ESOMAT 2018

Book Of Abstracts

11th European Symposium on Martensitic Transformations

Université de Lorraine · Georgia Tech Lorraine · LEM3 Laboratoire d'Étude des Microstructures et de Mécanique des Matériaux

CNRS · Institut Jean Lamour · Arts et Métiers ParisTech · GeorgiaTech-CNRS UMI 2958

August 27 — 31, 2018, Metz, FRANCE

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ESOMAT 2018

11^{th} European Symposium on Martensitic Transformations

August 27-31, 2018

Welcome on behalf of the Local Organizing Committee

On behalf of the Local Organizing Committee, we would like to welcome all the participants at Metz, at the Campus of Ile du Saulcy of the University of Lorraine. This 11^{th} European Symposium on Martensitic Transformations is jointly hosted by the University of Lorraine, Georgia Tech Lorraine, the European Campus of the Georgia Institute of Technology and Arts et Métiers Paris Tech.
We would like to acknowledge financial support by our sponsor, especially the Region Grand Est and Metz Metropole. This support has helped us to organize an attractive program in keeping the costs for participants low and offering reduced fees for PhD students.
We also want to acknowledge the participants, because the real success of a conference comes from interactions between the participants and we have did our best to offer you an attractive scientific program and a pleasant social program to promote these interactions and strengthen the relationships between martensite researchers in Europe and worldwide.
Participants come from 24 countries, with notable participation of young researchers and PhD students who will have a great opportunity to meet and exchange with renowned researchers.
The Symposium will cover different aspects related to martensitic transformation including Mechanical and Micromechanical Modelling, Computational Material Design, Advanced Characterization of Martensite, Martensite in Carbon Steel, NiTi Shape Memory Alloys, Magnetic Shape Memory and Ferroic Transition, Ti-based Alloys, Alloy Development and Application, Advances Processing Techniques and Elasto-caloric Cooling. Oral and poster presentations dealing with these topics are included in the conference program. In addition to the contributed presentations, the International Advisory Committee has invited eminent internationally known researchers from various countries to deliver Plenary and Invited lectures at ESOMAT 2018.
We hope you will enjoy ESOMAT 2018 and we wish you a very fruitful conference and enjoyable days in Metz.

Tarak Ben Zineb & Etienne Patoor
Chairs ESOMAT 2018
27 31 August 2018
Metz, France

http://www.lem3.univ-lorraine.fr/ESOMAT2018/
tarak.ben-zineb@univ-lorraine.fr
etienne.patoor@georgiatech-metz.fr

Conference Committee

Conference chairs	
	
Prof. Etienne PATOOR Georgia Tech Lorraine, Metz, France *etienne.patoor@georgiatech-metz.fr* +33 (0)3 87 20 39 27	Prof. Tarak BEN ZINEB Université de Lorraine, Nancy, France *tarak.ben-zineb@univ-lorraine.fr* +33 (0)3 72 74 68 70

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General Conference Information

Registration Fees
Registration fees for regular participants or students include:

- conference registration
- conference materiall including: program, book of abstracts, USB key with extended abstracts
- coffee breaks
- welcome reception held by the major of Metz, at City Hall on Monday 27^{th}
- lunches at walking distance from conference location
- gala evening at Le Royal on Thursday 30^{th}
- conference excursion on Thursday 30^{th} afternoon
- Accompanying persons program: on Monday 27^{th}, Tuesday 28^{th}, Wednesday 29^{th}, and Thursday 30^{th}

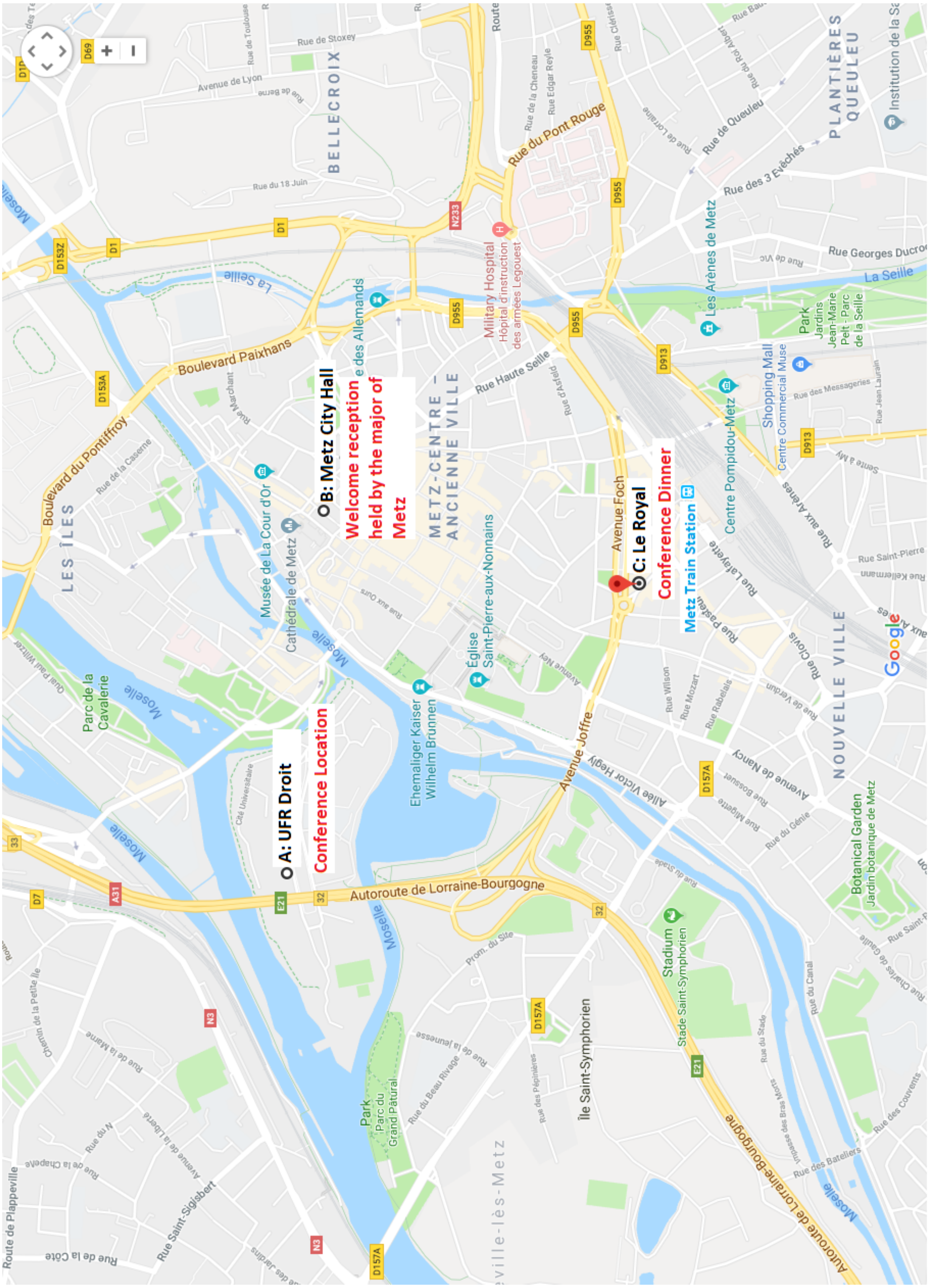
Name Badge
The conference name badge is required for admission to all conference events

Conference Location

Faculté de Droit, Economie et Administration, Ile du Saulcy, METZ
Amphi 1 & Amphi 4

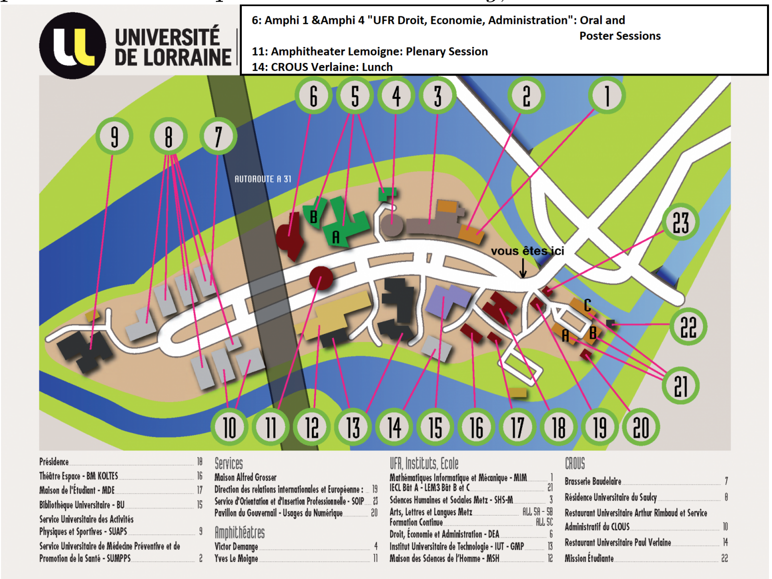
The Esomat 2018 conference will be held at "UFR Droit, Economie, Administration" of Lorraine University.

Amphitheater Lemoigne, Ile du Saulcy, METZ
Amphitheater


A: UFR Droit
Conference Location
B: Metz City Hall
Welcome reception held by the major of Metz
C: Le Royal
Conference Dinner
Metz Train Station
Map data ©2018 GeoBasis-DE/BKG (©2009), Google
200 m

A: UFR Droit, Economie, administration de l'Université de Lorraine: Ile du Saulcy, 57000 Metz

Tram Stops: *Mettis A: Square du Luxembourg; Mettis B: Université Saulcy*

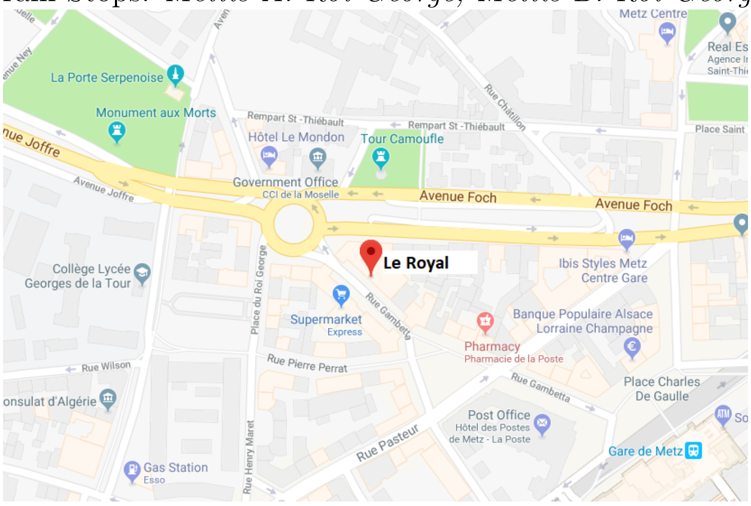


B: Metz City Hall: 1 Place d'Armes, 57000 Metz

Bus Stops: *L3: Place d'Armes; N83: Place d'Armes*

C: Le Royal: 2 Rue Gambetta, 57000 Metz

Tram Stops: *Mettis A: Roi George; Mettis B: Roi George*



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ESOMAT 2018 is financially supported by:

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Conference Program

- **Plenary lectures:** B. Appolaire, P.J. Jacques, M. Kohl, D.C. Lagoudas, Z. Moumni, E.V. Pereloma
- **Session 1:** Mechanical and Micromechanical Modelling
- **Session 2:** Advanced Characterization of Martensite
- **Session 3:** Computational Material Design
- **Session 4:** Martensite in Carbon Steel
- **Session 5:** Magnetic Shape Memory and Ferroic Transitions
- **Session 6:** Ti-based Alloys
- **Session 7:** Alloy Development and Applications
- **Session 8:** Advanced Processing Techniques
- **Session 9:** NiTi Shape Memory Alloys
- **Session 10:** Elasto-caloric cooling
- **Poster Sessions**

  Poster session A: Posters from session 1, 3, 4 & 6

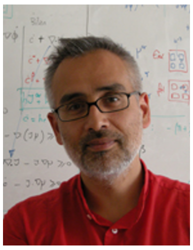
  Poster session B: Posters from session 1 & 2

  Poster session C: Posters from session 5 & 10

  Poster session D: Posters from session 7 & 8

Time	Mon. 27 Amphitheater	Tue. 28 Amphi 1	Tue. 28 Amphi 4	Wed. 29 Amphi 1	Wed. 29 Amphi 4	Thu. 30 Amphi 1	Thu. 30 Amphi 4	Fri. 31 Amphi 1	Fri. 31 Amphi 4
8:30		Session 1 Chair *D.C Lagoudas* Invited Lecture *H. Sehitoglu*	Session 2 Chair *D. Schyvers* Invited Lecture *G. Laplanche*	Session 3 Chair *H. Sehitoglu* Invited Lecture *R. Umetsu*	Session 6 Chair *E. Aeby-Gautier* Invited Lecture *F. Prima*	Session 5 Chair *V. A. Chernenko* Invited Lecture *D. Wang*	Session 8 Chair *P. Laheurte* Invited Lecture *B. Brailovski*	Session 9 Chair *A. Tuissi* Invited Lecture *C. Maletta*	Session 10 Chair *M. Kohl* Invited Lecture *S. Seelecke*
9:00		Oral Session	Oral Session	Oral Session	Oral Session	Oral Session	Oral Session	Oral Session	Oral Session
10:20	**Registration**	Coffee Break							
10:50		Session 1 Chair *Y. Chemisky* Oral Session	Session 2 Chair *T. Waitz* Oral Session	Session 5 Chair *I. Karaman* Oral Session	Session 2 Chair *S. Prokoshkin* Oral Session	Session 9 Chair *P. Šittner* Oral Session Invited Lecture *P. Sedlak*	Session 7 Chair *G. Eggeler* Oral Session	Amphitheater Plenary Lectures Chair *T. Ben Zineb* *Z. Moumni* *M. Kohl*	
12:30	**Lunch**								
12:40								**Closing ceremony**	
13:00									
13:30	**Opening ceremony**								
14:00	Lecture in remembrance of M. Landa *H. Seiner*	**Poster Session A**	**Poster Session B**	**Poster Session C**	**Poster Session D**	**Conference Excursion**			
15:30	Plenary Lectures Chair *E. Patoor* *D. C. Lagoudas* *P. Jacques*	Session 1 Chair *A.E. Volkov* Invited Lecture *Q. Sun*	Session 4 Chair *S. Denis* Invited Lecture *S. Berveiller*	Session 5 Chair *C. Esling* Invited Lecture *V. Chernenko*	Session 7 Chair *J. Pons* Invited Lecture *G.Y. Firstov*				
16:00	Oral Session	Oral Session	Oral Session	Oral Session	Oral Session				
16:10	Coffee Break								
16:40	Plenary Lectures Chair *T. Ben Zineb* *E. Pereloma* *B. Appolaire*	Coffee Break							
17:10		Session 1 Oral Session	Session 4 Oral Session	Session 5 Oral Session	Session 7 Oral Session				
18:10									
18:30				**ESOMAT ICA Board Meeting**	**ESOMAT ICA Board Meeting**				
19:00	**Welcome drink reception Metz Town Hall**					**Gala evening**			

Plenary Lectures



Prof. Benoît APPOLAIRE
Institut Jean Lamour - Université de Lorraine, Nancy FRANCE
Presentation title: *Achievements and perspectives on Phase Field modeling of Martensitic Transformation*

Prof. Pascal J. JACQUES
Université catholique de Louvain (UCL), BELGIUM
Presentation title: *Deformation-induced phase transitions, a playground for tailored mechanical properties of Fe-based engineering alloys*

Prof. Manfred KOHL
Karlsruhe Institute of Technology (KIT),Institute of Microstructure Technology (IMT), GERMANY
Presentation title: *State-of-the Art of Shape Memory Micro actuators*

Prof. Dimitris LAGOUDAS
John and Bea Slattery Chair of Aerospace Engineering, Director,
Texas Institute for Intelligent Materials and Structures (TiiMS),
Texas A& M University College Station,USA
Presentation title: *State of art on SMA thermomechanical behavior modeling*

Prof. Ziad MOUMNI
UME - ENSTA - ParisTech, Palaiseau, FRANCE
Presentation title: *Fatigue Analysis of Shape Memory alloys*

Prof. Elena V. PERELOMA
UOW - Electron Microscopy Centre, University of Wollongong, AUSTRALIA
Presentation title: *Deformation-induced phase transformations in metastable β-Ti alloy*

Plenary Lectures: Program

Lecture in remembrance of M. Landa: Monday August 27	
14:00 Amphitheater	**H. Seiner** "20 years of ultrasonic characterization of martensites: a lecture in remembrance of M. Landa (1965-2018)"
Plenary lecture 1: Monday August 27 Chairperson: E. Patoor	
14:30 Amphitheater	**D. C. Lagoudas** "State of art on SMA thermomechanical behavior modeling"
Plenary lecture 2: Monday August 27 Chairperson: E. Patoor	
15:20 Amphitheater	**P. J. Jacques** "Deformation-induced phase transitions, a playground for tailored mechanical properties of Fe-based engineering alloys "
Plenary lecture 3: Monday August 27 Chairperson: T. Ben Zineb	
16:40 Amphitheater	**E. V. Pereloma** "Deformation-induced phase transformations in metastable β-Ti alloy "
Plenary lecture 4: Monday August 27 Chairperson: T. Ben Zineb	
17:30 Amphitheater	**B. Appolaire**,B., Y. Le Bouar, A. Finel "Achievements and perspectives on Phase Field modeling of Martensitic Transformation"
Plenary lecture 5: Friday August 31 Chairperson: T. Ben Zineb	
10:50 Amphitheater	**Z. Moumni** " Fatigue Analysis of Shape Memory alloys"
Plenary lecture 6: Friday August 31 Chairperson: T. Ben Zineb	
11:40 Amphitheater	**M. Kohl** "State-of-the Art of Shape Memory Micro actuators"

Contents

Deformation-induced phase transitions, a playground for tailored mechanical properties of Fe-based engineering alloys

Plenary Lecture 2 Monday 27 03:20pm Amphi- -theater

Jacques P.J.

UCLouvain, Institute of Mechanics, Materials and Civil Engineering (iMMC), Place Sainte Barbe 2, B-1348 Louvain-la-Neuve, BELGIUM

Since decades, the future of structural engineering alloys has been mostly dictated by the quest for continuously higher levels of strength. While stronger steels, titanium or aluminium alloys were required, for example in transport applications for structural lightweight or crashworthiness improvement, (de)formability was also required to be kept at an increasing (or not too shortened) level. Different "classical" strengthening mechanisms (solid solution, precipitation, grain refinement, in situ composite, ...), all showing globally the same reverse trend between strength and ductility, that means the same work hardening rate, were successively tested, optimised and successfully used in numerous applications.
The concept of "dynamic" microstructure with deformation-induced mechanisms, i.e. martensitic transformation or mechanical twinning that bring larger levels of work hardening when it is needed was then considered. It is worth emphasising that improvement of the work hardening results from (positive) interactions between these deformation-induced transitions and the basic mechanisms related to dislocation glide. Indeed, large dislocation activity remains a mandatory condition for the improvement of the work hardening without substitution or compensation (like in hard-to-deform hcp alloys).
Metallurgy of Fe-based alloys is a good example of this story of successive mechanisms to continuously improve the combination of strength and ductility. Starting from solid solution strengthening, mostly by carbon and manganese, precipitation hardening and grain refinement were developed in the sixties as a response to the development of welding, while Dual Phase steels constituted the answer to the oil crisis in the 1970s. Since the 1990s, more continuous developments based on phase transitions appeared in order to answer to the constant urgency of greenhouse gas decrease and improvement of crashworthiness.
It is well established now that deformation-induced phase transitions is an effective strategy to avoid the decrease with strain of the work hardening rate, bringing about unprecedented levels of work hardening in steels, and more recently in titanium alloys or high entropy alloys. Specific grades, designed owing to more or less established theories and criteria, exhibit levels of work hardening close to the theoretical limit.
While much efforts are still devoted to characterise the microstructure properties relationship in these alloys, better understanding and potential future improvements would rely on the full and complete consideration of the role of the work hardening rate. It is definitely of primary importance to highlight the role of the microstructure modifications brought by these deformation-induced phase transitions on the net balance between the sources and sinks of dislocations and to identify the storage mechanisms. A huge number of specific interfaces are created either by the martensitic transformation or the mechanical twinning. These interfaces strongly interact with dislocations in a way that sometimes still needs to be understood. On the other hand, both martensite and mechanical twins exhibit different mechanical behaviour from the matrix, so that mechanical contrast and intrinsic composite effect are activated during straining. Finally, future prospects for these ultra-high strength engineering alloys could be based on the analysis of potential interactions with the damage and fracture mechanisms. While on the one hand, the levels of strength that are reached are so high that damage is prone to dramatically appear, such a large work hardening rate could on the other hand heal early occurring damage.

Deformation-induced phase transformations in metastable β-Ti alloys

Plenary Lecture 3 Monday 27 04:40pm Amphi- -theater

Pereloma E.[1],Naseri R.[1,2],Mitchell D. R.G.[2],Casillas G.[2],Ahmed M.[1],Gazder A. A.[2],Saleh A. A.[1]

[1]*School of Mechanical, Materials, Mechatronic and Biomedical Engineering, University of Wollongong, NSW 2522, AUSTRALIA;*
[2]*UOW Electron Microscopy Centre, University of Wollongong, NSW 2500, AUSTRALIA*

Metastable β-Ti (body centred cubic, bcc) alloys exhibit a unique combination of strength and ductility as they accommodate deformation via stress-induced phase transformation, twinning and slip. The matrix bcc β phase typically transforms during loading to α'' martensite (orthorhombic) and ω (hexagonal) phases. The relative activities of the operative deformation mechanisms depend on several factors including the stability of the β phase, loading path and deformation conditions (temperature and strain rate).

In the present ongoing research project, various in-situ and ex-situ experimental techniques are used to evaluate the effects of the β matrix characteristics, strain, loading path (tension, compression and cyclic tension-compression) and strain rate on the microstructure evolution and the associated deformation mechanisms in metastable β Ti-V-Fe-Al alloys. Detailed microstructural characterisation was undertaken using high resolution scanning transmission electron microscopy and electron back scattering diffraction. The electron microscopy studies revealed the formation of a relatively small fraction of ω phase within the β matrix, at β/α'' martensite interfaces and within deformation twins. At the same time, the formation of primary and secondary α'' martensite was much more prolific but its extent was affected by both the loading path and deformation conditions. It was found that with increasing β phase stability (by introducing increasing fractions of α (hexagonal close packed, hcp) phase in the initial microstructure), the deformation mechanisms during tension change from the dominant formation of α'' martensite + twinning in addition to slip to α'' martensite + slip. Contrarily, the formation of α'' martensite becomes more restricted during compression as the β phase stability increases. The effect of higher strain rates on the stress-induced phase transformations is similar to increasing the β phase stability with enhanced deformation twinning activity. The underlying reasons for the observed mechanisms will be discussed in detail. Lastly, in-situ neutron diffraction during cyclic tension-compression loading ($\pm 2\%$ strain) of fully β microstructure revealed a significant tension-compression asymmetry (with the maximum stress in compression always higher than tension) along with a pronounced strain recovery during unloading. The former is associated with easier α'' martensite formation under tension than compression, whereas the latter suggests that reverse transformation of α'' martensite back to the β matrix could be occurring during load reversal.

Plenary Lecture 4
Monday 27
05:30pm
Amphi- -theater

Deformation-induced phase transformations in metastable β-Ti alloys

Appolaire B.[1,2], Le Bouar Y.[2], Finel A.[2]

[1] *Institut Jean Lamour, Université de Lorraine, FRANCE;*
[2] *Laboratoire d'Étude des Microstructures, CNRS-ONERA, FRANCE*

The phase field approach has progressively invaded the field of materials science these last 25 years to become one of the most popular approach to compute microstructure formation and evolution. In this talk, I will give a small overview of the historical development of the approach to explain why two kinds of phase field models are used to describe martensitic transformations: (i) models with an order parameter indirectly coupled with displacements through some eigenstrain;
(ii) and models with strain components as the order parameters themselves.
I will explain their very difference from a physical point of view before discussing their pros and cons, including their implementation with different methods. I will illustrate their respective achievements taking examples from the literature or from fresh calculations, so as to give the broadest possible overview of the different issues addressed with the phase field approach (besides the nice pictures that are usually shown). I will finish my talk by giving some perspectives with the emerging trends in the phase field community, together with some advices to potential newcomers.

Plenary Lecture 5
Friday 31
10:50am
Amphi- -theater

Fatigue Analysis of Shape Memory alloys

Moumni Z.

ENSTA-Paristech FRANCE

Shape memory alloys (SMAs) exhibit interesting properties when subjected to mechanical or thermal loadings. For instance, they can accommodate large recoverable strains, or recover their shape by simple heating after being inelastically strained. In many applications, shape memory alloys are subjected to cyclic loadings, which could induce failure of the SMA structure by fatigue. Hence, a better understanding of fatigue of SMAs thus seems important in order to further promote the use of these materials in high-tech applications.
In this talk, we present a comprehensive approach for fatigue of SMAs developed in our research group the last 10 years. It includes four steps: i) the development of an accurate constitutive model to predict the stabilized thermo-mechanical state of a SMA structure under cyclic loading; ii) an energy-based criterion to predict low-cycle fatigue of SMAs, iii) a shakedown-based fatigue model to predict high-cycle fatigue of SMAs and iv) a structural optimisation procedure to design SMAs components with respect of fatigue.

Our approach takes into account the main features related to the unusual SMAs behaviour such as the strong thermo-mechanical coupling resulting from the dependence of the fatigue lifetime on the loading frequency.

State of the Art of Shape Memory Microactuators

Plenary Lecture 6 Friday 31 11:40am Amphi-theater

Kohl M.

Karlsruhe Institute of Technology, IMT, Karlsruhe, GERMANY

The ongoing miniaturization and increase of functionality have enabled the development and widespread use of smart devices and systems. The field of microelectromechanical systems (MEMS) has undergone an exceptionally dynamic evolution from silicon micromechanics to a highly diverse field comprising a large variety of materials and corresponding technologies. However, actuation on small scales has been an issue since the early nineties, where MEMS technology was still in its infancy. Silicon is not a transducer material, therefore, additional materials have to be introduced for conversion of energy into mechanical work. Shape memory microactuators are potential candidates for MEMS applications as they exhibit highest work densities compared to other actuation principles in the order of 10^7 J.m^{-3} [1].
Two approaches for producing planar shape memory alloy (SMA) materials for microactuation have been developed: (1) magnetron sputtering of SMA thin films and (2) the integration of rolled SMA foils, which both turned out to be very successful creating a paradigm change in microactuation technology. An intriguing feature of SMA materials is their multifunctionality allowing for, e.g.,structural stability, electrical and thermal conductance, actuation and self-sensing at the same time. This feature of being a "smart material" becomes particularly advantageous on small scales, at which technology constraints pose severe limitations on the number, geometry and size of functional structures. As a consequence, monolithic SMA microparts have been designed with different subunits and multifunctional performance that can be fabricated in a single micromachining step. Following this philosophy of "the material is the machine" opened up new routes for creating fabricable smart SMA microactuators and corresponding devices [2].
This review covers important milestones of the research and development of both, SMA film- and foil-based microactuators. Major material properties will be summarized and key processing technologies for fabrication of functional SMA microactuators will be presented. Selected demonstrators will be discussed including their potential applications and transfer to commercial products.

[1] S. Miyazaki, Y. Q. Fu, and W. M. Huang, Eds., Thin Film Shape Memory Alloys: Fundamentals and Device Applications., Cambridge, 2009.

[2] M. Kohl, "Shape memory microactuators", Springer book series on Microtechnology and MEMS, Springer-Verlag Berlin Heidelberg, 2004.

Session 1: Mechanical and Micromechanical Modelling

Invited Lecture: Tuesday August 28	
8:30 Amphi 1	**H.Sehitoglu**,S. Alkan, Y. Wu "Recent Developments in Understanding Slip Resistance of Shape Memory Alloys"
15:30 Amphi 1	**Q. Sun**, M. Li "Effects of length and time scales on phase transition behavior of shape memory alloys"

Oral presentations: Tuesday August 28	
9:00 Amphi 1	**T. Baxevanis**, A. Hossain "A Thermomechanically-Coupled Constitutive Model for Transformation-Induced Plastic Deformation in NiTi Single Crystals"
9:20 Amphi 1	**Y. Congard**, L. Saint-Sulpice, S. Arbab Chirani, S. Calloch "Determination of the low cycle fatigue lifetime of a Ni-Ti shape memory alloy using simplified methods : application to endodontic instrument"
9:40 Amphi 1	**M. Frost**, P. Sedlák, P. Sittner "Numerical study on macroscopic localization patterns in NiTi samples under various loading modes"
10:00 Amphi 1	**M. Peigney** "On shakedown and high-cycle fatigue of shape memory alloys in the presence of permanent inelasticity and degradation effects"
10:50 Amphi 1	**N. Ulff**, C. Bouby, A. Lachiguer, T. Ben Zineb, T. Bouraoui "Modelling of hydrogen effects on the thermomechanical behaviour of NiTi-based shape memory alloys"
11:10 Amphi 1	**H. Seiner**, P. Sedlák, L. Heller, P. Sittner "Kinematic compatibility conditions in presence of plastic slip a case study of Ni-Ti B19' "
11:30 Amphi 1	**M. D. Fall**, E. Patoor, O. Hubert, K. Lavernhe "Comparative study of two multiscale thermomechanical models of polycrystalline shape memory alloys: Application to a representative volume element of titanium-niobium"
11:50 Amphi 1	**G. Chatzigeorgiou**, L. Cheng, Y. Chemisky , F. Meraghni "Fully coupled thermomechanical model for SMAs for phase transformation, martensitic reorientation, transformation - induced plasticity and functionally fatigue damage"
12:10 Amphi 1	**C. Cayron** "Beyond shears"
16:00 Amphi 1	**A. E. Volkov**, F. S. Belyaev, M. E. Evard, E. S. Ostropiko, A. I. Razov "Aging Effect on the One-way and Two-way Shape Memory in TiNi-based Alloys"
16:20 Amphi 1	**R. Lechuga-Taboada**, F. N. Garcia Castillo, J. Cortés Pérez, G. A. Lara Rodriguez, A. Reyes-Solis "Determination of the correlations between a quantitative criteria, experimental evidence and finite element model of a polycrystalline sample of Cu-Al-Be undergoing uniaxial tension"
17:10 Amphi 1	**C. Collard**, T. Ben Zineb "Micromechanical Analysis of Elastic-Plastic Precipitate Effect on Shape Memory Alloy Behaviour"
17:30 Amphi 1	**Y. Chemisky**, D. Hartl, T. Baxevanis, F. Meraghni "Three-dimensional numerical analysis of functional and structural fatigue in of shape memory alloy actuators"
17:50 Amphi 1	**Y. You**, Z. Moumni, W. Zhang "Effect of thermomechanical coupling on stress-induced martensitic transformation in the crack tip region of NiTi"

Poster Session A: Tuesday August 28
M. Evard, A. E. Volkov, F. S. Belyaev "The effect of loading rate on mechanical behavior of edge cracked NiTi"
P. Hannequart, M. Peigney, J.-F. Caron, E. Viglino "A micromechanical model for polycrystalline shape memory alloy wires embedded into smart structures"

G. Helbert, J. Simon, S. Arbab Chirani, X. Balandraud, L. Dieng ”Experimental and numerical investigations of the propagation of austenite-to-martensite transformation bands”
R. Xu, C. Bouby, H. Zahrouni, T. Ben Zineb, H. Hu ”A multiscale approach for the thermomechanical modeling of shape memory alloy fiber reinforced composites”
G. Anlas, **F. Mutlu** ”The effect of loading rate on mechanical behavior of edge cracked NiTi”
A. Volkov, M. Evard, **E. Iaparova** ”Elucidation of the role of the structure of porous TiNi for its mechanical and functional properties”

Contents

Recent Developments in Understanding Slip Resistance of Shape Memory Alloys

28 Aug
8:30pm
Amphi 1

Sehitoglu H. Alkan S., Wu Y.

University of Illinois at Urbana-Champaign, UNITED STATES

In this presentation, we overview the importance of plastic deformation via slip in shape memory alloys (SMAs) utilizing results from the literature and our own work. The plastic flow affects the irrecoverability and hysteresis in shape memory alloys and typically initiates from austenite-martensite interfaces. Its occurrence in all SMAs including NiTi, CuZnAl, NiTiCu and Fe based SMAs is well-known, but the stress levels to trigger dislocation-mediated flow are not well established. Because these alloys have a cubic structure in the austenitic phase, we analyzed the spreading of the dislocation core and developed a theory to predict the critical resolved shear stress (CRSS). The theory is compared with experiments on single crystals and it predicts the tension-compression asymmetry and crystal orientation dependence of CRSS. We outline future directions to predict the transformation stress utilizing similar concepts of shearing on conjugate planes. Ideally, the design of new shape memory alloys must include a rapid assessment of their CRSS for slip and CRSS for transformation. A wide gap between the two, ie. high slip stress relative to transformation stress, would result in a large superelastic window and the achievement of high transformation strains close to theoretical levels.

Effects of length and time scales on phase transition behavior of shape memory alloys

28 Aug
3:30pm
Amphi 1

Sun Q.[1], Li M.[2]

[1]*Hong Kong University of Sci. and Tech., CHINA;*
[2]*Wuhan University, Wuhan, CHINA*

We report recent advances in the experimental and theoretical study of effects of material internal length scales (grain size, grain boundary and phase boundary) and time scales (loading time and heat conduction time) on the instability, pattern formation/evolution and hysteresis dissipation of NiTi polycrystalline SMAs which have nano-grain microstructure. Particular emphasis is paid to the significant effects of length and time scales on the properties and behaviors of the material. For length scale effect, it is shown that, with grain size reduction, the energy of the elastic non-transformable grain boundary will gradually become dominant in the phase transition process, and eventually bring fundamental changes of the deformation behaviors: breakdown of two-phase coexistence and vanishing of superelastic hysteresis. Such effects of length scale reduction has huge potential to improve and control the performance of the existing NiTi SMA by grain size engineering. The predictions are supported by experimental data. For the effects of time scales, it is shown that competition of physical processes of different time scales brings the emergence of the length scale in the phase rtansition domain patterns and domain spacing. In summary, nucleation and growth of new phase or domains, two-phase coexistence and the hysteresis dissipation are the key signatures of the first-order phase transition in materials and are widely used as the basic paradigms in the continuum modelling of martensitic phase transformation of most shape memory alloys where grain size is much larger than the length scales of grain boundary and phase boundary. The current technology has been able to manufacture SMAs with grain size down to 510 nm. With the grain size reduction and strong thermomechanical coupling involved in the high strain rate loading conditions, many unusual phenomena and properties will emerge, which not only open up new possibilities in the application but also provide opportunity for new paradigm building and shift in modelling and understanding.

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28 Aug
9:00am
Amphi 1

A Thermomechanically-Coupled Constitutive Model for Transformation-Induced Plastic Deformation in NiTi Single Crystals

Baxevanis T., Hossain A.

University of Houston, UNITED STATES

Shape memory alloys (SMAs) are a class of materials that exhibit the ability to recover large inelastic deformations induced from forward transformation of a high-symmetry crystallographic phase to a low-symmetry one through reverse phase transformation. Taking advantage of this functional property of SMAs in engineering applications that involve passing back and forth through phase transformation many times is subject to accumulated irrecoverable strains. Phase transformation introduces significant distortion at the austenite–martensite interfaces and grain boundaries that drives dislocation activity resulting in an observable macroscopic TRasformation-Induced Plastic (TRIP) deformation, which occurs at effective stress levels much lower than the plastic yield limit of the material. To account for and analyze the inelastic deformations at play in SMA single crystals undergoing cyclic transformation, a 3-D thermomechanically-coupled phenomenological constitutive model considering finite strains and finite rotations is proposed in this work. A Gibbs free energy of a representative volume element of an SMA single crystal is utilized for this purpose that describes the interaction between the two phases and approximates the local plastic deformation using the Mori-Tanaka micromechanical method. In the developed numerical implementation, all the tensorial variables are cast in a corotational configuration for finite deformation analysis, based on the framework proposed by Xiao and Brunhs (1997, 1998), to achieve frame objectivity at each loading increment. The capability of the proposed model to describe the cyclic deformation behavior of NiTi single crystals is verified by comparing the simulated results with experimental data.

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28 Aug
12:10am
Amphi 1

Beyond shears

Cayron C.

EPFL IMX LMTM, SWITZERLAND

The link between crystallography and mechanics in martensitic transformations is generally made by the use of simple shears, or by their generalized forms called invariant plane strains. The shear paradigm is 150 years old; it was close to collapse in the 1940s but was saved in extremis thanks to the discovery of dislocations. However, the shear-based theories are phenomenological, they are unnecessarily complex, they focus on the lattices and forget the atoms, and their power of prediction is less effective than usually claimed. Our aim is to present a hard-sphere angular-distortive approach we have developed for the last decade, and how it can be associated with linear algebra and group theory to form a coherent and predictive theory. The concepts of orientational variants, distortional variants and correspondence variants are detailed. The habit planes are calculated according an "untilted plane" criterion, and variant selection can be predicted by calculating the energy of formation. Examples will be taken with martensitic transformations in steels and deformation twinning in magnesium alloys.

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28 Aug
11:50am
Amphi 1

Fully coupled thermomechanical model for SMAs for ph ase transformation, martensitic reorientation, transformation - induced plasticity and functionally fatigue damage

Chatzigeorgiou G.[1], Cheng L.[2], Chemisky Y.[1], Meraghni F.[1]

[1]*LEM3, UMR 7239, Arts et Métiers ParisTech FRANCE;*
[2]*University of Lorraine, FRANCE*

Shape memory alloys (SMAs) have found tremendous development and usage in several innovative applications, such as actuators in mechanical, aerospace and biomechanics industry. These actuators often experience large number of cyclic loads. The current state-of-the art models are still incomplete in properly describing the SMA cyclic response under complex loading onditions. The present work proposes a 3D phenomenological model, based on the thermodynamical coupling of different deformation mechanisms such as the forward and reverse phase transformation, the martensitic reorientation, the transformation-introduced plasticity, accounting for fatigue damage. To achieve this goal, all the above mentioned

mechanisms are described through the martensitic volume fraction as the coupling parameter. A recently developed, thermomechanically coupled, SMA constitutive law [1], including both the phase transformation and martensitic reorientation mechanisms, has been validated under non-proportional loading conditions through a series of comparisons between numerical and experimental results. This model is extended further in order to capture the accumulated TRIP residual strain induced by the martensitic transformation, accounting in addition for the accumulated fatigue damage, which evolves during the cyclic loading [2]. The fatigue damage is incorporated into the constitutive law through the concepts of continuum damage theory. Numerical investigation under strongly non-proportional thermomechanical loading conditions demonstrate the capabilities of the new framework.

[1] D. Chatziathanasiou, Y. Chemisky, G. Chatzigeorgiou, F. Meraghni (2016) International Journal of Plasticity 82:192-224.
[2] Y. Chemisky, D. Hartl, F. Meraghni (2018) International Journal of Fatigue, in press.

Three-dimensional numerical analysis of functional and structural fatigue in of shape memory alloy actuators

28 Aug
5:30pm
Amphi 1

Chemisky Y.[1], Hartl D. [2], Baxevanis T. [3], Meraghni F.[4]

[1]Arts et Métiers, Metz, TIMC-IMAG, UMR CNRS 5525, Grenoble, FRANCE
[2]Texas A&M University, Texas, USA
[3]University of Houston, Houston, USA
[4]Arts et Métiers, LEM3, UMR CNRS 7239, Metz, FRANCE

With the design of new complex devices in shape memory alloys (SMAs) and to take advantage of their large recoverable strains, SMA components are more and more subjected to loadings in severe conditions. The two major mechanisms responsible for the loss of functionality of SMA actuators, namely structural and functional fatigue, is here investigated. A three-dimensional constitutive model is presented that describes the behavior of shape memory alloy actuators undergoing a large number of cycles leading to the development of internal damage and eventual failure.
Physical mechanisms such as transformation strain generation and recovery, transformation - induced plasticity, and fatigue damage associated with martensitic phase transformation occurring during cyclic loading are all included within a thermodynamically consistent framework. The numerical implementation of such complex model, in the framework of Finite Element Analysis, is detailed in this contribution. The validated model is utilized to analyse the influence of various loading modes (uniaxial, multiaxial, non-proportional) on three-dimensional structures. Both lifetime and the evolution of irrecoverable strain are accurately analysed and tracked by the developed model to determine structural and functional fatigue limit.

Micromechanical Analysis of Elastic-Plastic Precipitate Effect on Shape Memory Alloy Behaviour

28 Aug
5:10pm
Amphi 1

Collard C.[1], Ben Zineb T.[2]

[1]CEMES, CNRS UPR 8011, Toulouse, FRANCE;
[2]Université de Lorraine, CNRS, Arts et Métiers ParisTech, LEM3, Nancy, FRANCE

Shape memory alloy (SMA) processing induces presence of defects and heterogeneities such as precipitates. On the other hand, precipitates may be introduced voluntarily during elaboration process in order to improve the material properties. So it is interesting to model the effect of elastic-plastic precipitates on SMA behavior in order to be able to predict their impact on the material properties (phase transformation, transformation hysteresis, shift of the start and finish forward and reverse transformation temperatures , material hardening ...). Modelings are based on the description of the local behavior of precipitates and grains, and a double scale transition technique in order to lead to the effective thermomechanical behavior. At the grain level, we consider an SMA matrix embedding precipitates. The accommodation of the phase transformation induced strain in the SMA matrix could generate plastic deformation in precipitates. To take into account this induced plasticity, precipitate behavior is described by an elastic-plastic constitutive law. The SMA matrix behavior is described with a crystalline plasticity based micromechanical constitutive law. A Mori Tanaka homogenization model allows to derive the effective behavior at the grain scale. For polycrystalline materials, made of several grains, a self-consistent approach is adopted to determine the effective macroscopic behavior of the polycrystalline aggregate. Numerical obtained results are discussed for the case of CuZnAl SMA embedding hard and soft elastic-plastic precipitates. The impact of precipitates on the local and global behavior is analyzed taking into account the sensitivity to the crystallographic orientations and the inclusion volume fraction. Moreover the simulation accounting with elastic-plastic precipitates are compared with the same model considering a pure SMA matrix (without precipitates), and an SMA matrix embedding purely elastic precipitates.

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28 Aug
9:20am
Amphi 1

Determination of the low cycle fatigue lifetime of a Ni-Ti shape memory alloy using simplified methods : application to endodontic instruments

Congard Y.[1], Saint-Sulpice L.[1], Arbab Chirani S.[1], Calloch S.[2]

[1]*ENIB, FRE CNRS 3744, IRDL - Brest, FRANCE;*
[2]*ENSTA Bretagne, FRE CNRS 3744, IRDL - Brest, FRANCE*

More and more industrial applications are using shape memory alloys for their specific properties. This is particularly true for endodontic instruments used to devitalize decayed teeth. However, their use sometimes results in fatigue failure at low number of cycles due to rotational bending loading. This phenomenon has not been much studied in the literature so far. The objectives of this work are to study and predict the low cycle fatigue of endodontic instruments made of NiTi shape memory alloys. First, an experimental study of the alloy fatigue behavior is carried out on different geometries of NiTi test specimens subjected to rotational bending. The analysis of the fatigue results and observations of the fracture surface allowed the choice of a fatigue criterion adapted to this materials. Finite element simulation are often used in fatigue lifetime prediction in order to calculate local response of the material. In low cycle fatigue non linear finite element simulation are needed, which can still be prohibitive for lifetime prediction. In order to reduce the complexity and to optimize the duration of the calculations, a simplified method of modeling the cyclic behavior of the alloy is used. These models are based on inclusion theory [1] [2]. Simplified laws are used to predict confined nonlinear behavior in a given region of interest, where a crack could be initiated. The simulation of the cyclic nonlinear multiaxial behavior at the critical point is thus performed from the local loading determined from an elastic finite element calculation of the structure on a single loading cycle. The results of the predictions are then compared with the results of rotational bending fatigue tests performed on endodontic instruments.

[1] A. Darlet, and R. Desmorat. Stress Triaxiality and Lode Angle along Surfaces of Elastoplastic Structures. International Journal of Solids and Structures 67, no. Supplement C (August 15, 2015): 71-83.
[2] T. Herbland. Une méthode de correction élastoplastique pour le calcul en fatigue des zones de concentration de contraintes sous chargement cyclique multiaxial non proportionnel. Thèse de doctorat, École Nationale Supérieure des Mines de Paris, 2009.

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28 Aug
11:30am
Amphi 1

Comparative study of two multiscale thermomechanical models of polycrystalline shape memory alloys: Application to a representative volume element of titanium-niobium

Fall M. D.[1], Patoor E.[1], Hubert O.[2], Lavernhe K.[2]

[1]*Georgia-Tech Lorraine (Georgia-Tech/CNRS UMI 2958), Metz Technopôle, Metz, FRANCE;*
[2]*LMT (ENS-Paris Saclay/CNRS /Université Paris-Saclay), Cachan, FRANCE*

The behavior of shape memory alloys (SMA) is governed by the martensitic transformation mechanisms occurring at the scale of the microstructure. The martensitic transformation can be induced by thermal and/ or mechanical stresses and in a coupled manner. The development of reliable design tools requires a better predictability of the actual constitutive behavior of shape memory alloys under complex thermomechanical loadings. The choice of multiaxial and multiscale modeling is relevant. The aim of this study is to compare two different micro-macro modeling approaches. In a first part, we introduce the model proposed by Siredey and al. [1], which uses crystallographic data to describe martensitic transformation in polycrystalline SMA. This 3D multivariant model relies on micromechanics to propose a simplified expression of the interaction energy within the material. An interaction matrix, which only depends on the known transformation strains, is used for the description of the interactions between martensitic variants during the domains' formation. In a second part, we present the model proposed by Maynadier and al. [2], which describes the behavior of a polycrystalline SMA volume from the physics of martensitic transformation at the crystal lattice scale. The model is based on the comparison of the Gibbs free energies of each component, without topological description. A probabilistic comparison is made (Boltzmann distribution function) to determine the volume fractions of variants as internal variables. Interactions at the interfaces are not taken into account. The modeling uses a unique adjusting numerical parameter, As, which drives interfacial effects (indeed As parameter introduces the inertial effects ignored by the modeling) and is identified using a simple differential calorimetry measurement (DSC). Both models are based on thermodynamics and involve transition rules from variant to grain then from grain to polycrystalline scale. The macroscopic behavior of the polycrystalline SMA is estimated by using a self-consistent scale transition scheme. Modeling results from both models are compared to each other and to experimental results carried out on a metastable β-titanium-niobium biocompatible alloy. The latter is a good candidate for biomedical applications thanks to its interesting properties such as superelasticity, shape memory behavior, high resistance to corrosion, and excellent cold workability.

[1] Siredey, N., Patoor, E., Berveiller, M., Eberhardt, A., Constitutive equations for polycrystalline thermoelastic shape memory alloys: partI. Intragranular interactions and behavior of the grain. Int. J. Solids Struct. 36 (28), 4289-4315, 1999.
[2] A. Maynadier, D. Depriester, K. Lavernhe-Taillard, O. Hubert, Thermo-mechanical description of phase transformation in Ni-Ti Shape Memory Alloy, Procedia Engineering, 10: 22082213, 2011.

Numerical study on macroscopic localization patterns in NiTi samples under various loading modes

28 Aug
9:40am
Amphi 1

Frost M.[1], Sedlák P.[1], Sittner P.[2]

[1] *Institute of Thermomechanics, CAS CZECH REPUBLIC;*
[2] *Institute of Physics, CAS CZECH REPUBLIC*

Having found many applications in medicine, civil engineering or aerospace industries, NiTi-based alloys are a prominent class within SMAs usually utilized in the form of thin structures, e.g. wires, strips or tubes. In such structures, the martensitic transformation often does not occur in a spatially homogeneous manner; instead, localized "martensitic bands" form within the austenitic sample and their fronts propagate leaving a material with modified dimensions and microstructure (phase) behind. The macroscopic picture is very similar to the localization of plastic deformation in certain steels and alloys well-known as Lüders bands: the onset is usually accompanied by a stress overpeak and followed by a stress plateau, material rehardens after exhausting the available portion of inelastic strain and the formation of bands depends on microstructure of the material. There are also some specific features of NiTi alloys: a) the attainable inelastic (transformation) strain is reversible and strongly loading mode-dependent, b) physical properties of the phases differ pronouncedly and transformation precursor effects are well-documented, c) microstructural aspects of martensitic transformation in polycrystalline SMA (e.g. compatibility and interactions of phases and/or martensitic variants in severely constrained polycrystalline environment) are rather complex and not fully understood yet.
Because localization gives rise to high gradients of strain concentrated in small volumes of material, it is supposed to be a critical factor for the fatigue lifetime. Hence, good understanding and effective modeling of the phenomenon would have direct practical impacts. Despite a considerable body of experimental data and more than two decades of modeling efforts, a reliable computational tool capable to reproduce (and even predict) occurrence localization is not available yet. In this contribution we introduce an extension of a well-established constitutive model for NiTi SMA aiming at this challenge. By particular tuning of parameters of both internal energy (with Eshelbys inclusion-motivated form) and dissipation function it is possible to impose strain-softening in tension and strain-hardening in compression simultaneously. Finite element implementation then allows to study localization patterns in common sample geometries (wires, ribbons, tubes) and loading modes (tension, bending, torsion) and compare them with available experimental data (DIC, XRD, optical methods).

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28 Aug
4:20pm
Amphi 1

Determination of the correlations between a quantitative criteria, experimental evidence and finite element model of a polycrystalline sample of Cu-Al-Be undergoing uniaxial tension

Lechuga-Taboada R.[1], García-Castillo F. N.[2], Cortés-Pérez J.[1], Lara Rodríguez G. Á.[3], Reyes-Solís A.[4]

[1] *Laboratorio de Mecánica Aplicada, Centro Tecnológico Aragón, FES Aragón, UNAM, MEXICO;*
[2] *Departamento de Materiales y Manufactura, DIMEI, UNAM, MEXICO;*
[3] *Instituto de Investigaciones en Materiales, UNAM, MEXICO;*
[4] *Centro de Investigación Multidisciplinaria Aragón, F.E.S. Aragón, UNAM, MEXICO*

The martensitic transformation in polycrystalline shape memory alloys (PSMA) occurs in a much more complex way than in the monocrystalline case, this is due many factors affect the mechanical conditions in each grain, even between regions of the same grain. Besides the crystal orientation of the grain, the number and orientation of the neighbor grains are factors to affect the mechanical behavior of each grain in the sample and in consequence, impacts in the formation of the variant or variants who can appear in each grain, however the correlations between the variant or variants formed in a grain and the local states of stress and strain that activates these variants are not stablished yet. In this work a simulation in finite element method (FEM) of the central section of a PSMA of Cu-11.5% wtAl-0.6% wt Be undergoing uniaxial tension is presented. The morphology of the grains was idealized like a cluster of hexagonal prisms. This model supposes only one layer of grains in his transverse section. The topology of the model was based in a sample used in a previous work in which the crystalline orientation of several grains was reported. Each hexagonal prism in the model poses a different crystal orientation but the same elastic constants. The FEM analysis considers only the elastic behavior, in other words, the state of stress and strain before martensitic transformations occur. This tensor can be obtained for some nodes ubicated in different regions of the boundaries and center zones of selected grains. That tensors are referred to each martensite variant (24 in the case of Cu-Al-Be) and the results was compared with experimental evidence and a quantitative criterion who selects the variants with most probability of appear, the variants who presents the highest values of maximum shear stress are compared with the variants predicted by the quantitative criteria and with the experimental evidence. The FEM analysis results shows how the grain boundaries act like stress concentrators in several cases, in some cases, the stress and strain tensor presents high variations in the value of his components trough the sections of grain boundary due to strong influence of the neighbor grains. This fact impacts in the activation of variant or variants of martensite in each region of the grain.

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28 Aug
5:50pm
Amphi 1

The effect of loading rate on mechanical behavior of edge cracked NiTi

Mutlu F., Anlaş G.

Department of Mechanical Engineering, Bogazici University, TURKEY

In this work, the effect of loading rate on mechanical behavior of NiTi shape memory alloys (SMAs) is investigated. For this purpose,
rst, tensile experiments on dog-bone specimens are conducted at different quasistatic-range loading rates, with simultaneous Digital Image Correlation (DIC) measurements; transformation stresses, strains, and strain maps of the specimen surfaces are obtained. An increase in the forward transformation stresses-strains, and a decrease in the reverse transformation stresses-strains are observed. The strain maps obtained from DIC show strain localizations in the specimen under tensile loading, that manifest themselves in the form of strain bands, the number of which increases with increasing loading rate. Next, fracture experiments under Mode I loading on thin Compact Tension (CT) specimens are performed. Stress Intensity Fac- tors (SIFs) are calculated using empirical equations of the ASTM E399. SIFs are also calculated by least squares
tting the displacement data obtained from DIC measurements to the asymptotic displacement equation of LEFM. In addition, transformation region sizes around the crack tip are evaluated both experimentally and analytically; they are found to be decreasing with increasing loading rate.

On shakedown and high-cycle fatigue of shape memory alloys in the presence of permanent inelasticity and degradation effects

28 Aug
10:00am
Amphi 1

Peigney M.

Laboratoire Navier, Université Paris-Est, FRANCE

For elastic perfectly plastic bodies under cyclic loadings, Melans and Koiters theorems give conditions for the energy dissipation to remain bounded with respect to time. That last situation is classically referred to as shakedown, and is associated with the intuitive idea that the body considered behaves elastically in the large time limit. Regarding fatigue design, shakedown corresponds to the most beneficial regime of high-cycle fatigue, as opposed to the regimes of low-cycle fatigue or ratcheting. Although the physical mechanisms in shape memory alloys (SMA) differ from plasticity, the hysteresis observed in the stress-strain response shows that some energy dissipation occurs, and it can be reasonably assumed that situations where the energy dissipation remains bounded is the most favorable regarding the fatigue of SMA bodies. This raises the issue of extending Melans and Koiters shakedown theorems to shape memory alloys. In particular, SMA models coupling phase-transformation with permanent inelasticity have been proposed lately to capture degradation effects which are frequently observed experimentally for cyclic loadings: although phase transformation in SMAs is the main inelastic mechanism, dislocation motions also exist and are (partly) responsible for such effects as training and degradation in cyclic loadings. To model such a behavior, two internal variables are generally introduced: in addition to the (constrained) variable describing the phase transformation, an additional variable is used to describe permanent inelasticity. A coupling term between those two variables is generally introduced in the free energy. In this communication, the classical shakedown theorems of Melan and Koiter are extended to a class of such SMA material models. Those theorems gives conditions for the energy dissipation to remain bounded, and are be relevant for the fatigue design of SMA structures. An attractive feature of those theorems is that their application requires elastic calculations only, thus bypassing incremental nonlinear analysis. Moreover, only a partial knowledge of the loading (namely the extreme values) is needed.

Kinematic compatibility conditions in presence of plastic slip a case study of Ni-Ti B19'

28 Aug
11:10am
Amphi 1

Seiner H.[1], Sedlák P.[1], Heller L.[2], Sittner P.[2]

[1]*Institute of Thermomechanics, the Czech Academy of Sciences, CZECH REPUBLIC;*
[2]*Institute of Physics, the Czech Academy of Sciences, Prague, CZECH REPUBLIC*

The coupling between martensitic transitions and the plastic slip in NiTi and NiTi-based alloys is a thighly multi-scale problem. At the homogenized continuum scale, i.e. the macro-scale, the plasticity evolves according to the macroscopic boundary conditions imposed onto the tested sample, such as imposed stresses or strains or controlled temperature fields. Nevertheless, the macroscopic rules governing the behavior at the homogenized continuum level result from finer-scale processes, including those taking place at single austenite-martensite interfaces inside the individual grains.

This presentation will discuss the possible mechanisms of coupling between motion of austenite-martensite interfaces and plastic deformation in single crystals of Ni-Ti SMAs within the framework of continuum mechanics, using the mathematical theory of martensitic microstructures in combination with crystal plasticity. It will be shown that these mechanisms are in principle different for the forward and reverse transitions, giving rise to more pronounced unrecoverable strain accumulation during the reverse run. While transition-induced plastic slip is admissible for both directions, synergistic coupling of the transition and plasticity in order to relax the external loading is possible mainly for the reverse transition. Activation of specific slip planes in austenite will be discussed for two basic austenite-martensite morphologies: a single habit plane and a wedge microstructure.

In the second half of the presentation, implications at the single-crystal theory for the meso- and macro-scale behaviors of the polycrystalline SMAs will be discussed, in particular with respect to the compatibility strains at the grain boundaries, including sub-grain boundaries created by irreversible (non-ferroelastic) twinning. It will be shown that the kinematic compatibility conditions can be formulated also at these larger spatial scales, giving, again, different predictions for the forward and reverse transitions. However, at the polycrystalline level, the theory predicts also significant plastic strains during the forward transition, which is in agreement with the experimental observations. The higher plastic strain observed macroscopically for the reverse transition is, hence, resulting from the underlying mechanisms at the single-crystal scale.

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28 Aug
10:50am
Amphi 1

Modelling of hydrogen effects on the thermomechanical behaviour of NiTi-based shape memory alloys

Ulff N.[1], Bouby C.[1], Lachiguer A. [2], Ben Zineb T.[1], Bouraoui T.[2]

[1] *Université de Lorraine, CNRS, Arts et Métiers ParisTech, LEM3, Nancy, FRANCE ;*
[2] *LGM, ENIM, Université de Monastir, Monastir, Av. Ibn ElJazzar, TUNISIA*

Nowadays, it is well known that NiTi Shape Memory Alloys (SMAs) exhibit a martensitic transformation. It induces a recovery deformation at constant force widely considered for orthodontic treatments despite some fractures observed after few months in buccal cavity. A degradation of the mechanical properties of NiTi arches appears due to the presence of hydrogen. Indeed, the maximum strain decrease and the area of the hysteresis become smaller. Accounting for effects of hydrogen diffusion on the NiTi SMA behaviour, a coupled chemo-thermo-mechanical constitutive model needs to be formulated. Based on the work of Lachiguer et al. [1], a first step consists in introducing material parameter dependencies (transformations temperatures, maximum transformation strain, hysteresis size) to the normalized concentration of hydrogen in the NiTi constitutive law developed by Chemisky et al. [2]. Based on experimental results, it allows to formulate a first hydrogen concentration dependant SMA behaviour model.
The main limitation of this model is that the hydrogen concentration can only be considered as homogeneous. As nano-indentation tests reveal a heterogeneous distribution of hardness (which is indirectly related to the hydrogen concentration), it becomes necessary to take into account the gradient of hydrogen distribution from the surface to the cross section center. The temperature is also considered due to its influence on the mechanical response and the hydrogen diffusion.
To this end, we have to write equilibrium equations for each field (thermal, mechanical and chemical fields). These equations are then discretized to be solved numerically by finite element method. A special finite element with coupled degrees of freedom (displacements, temperature and hydrogen concentration) is developed and implemented in the Abaqus finite element software through the UEL subroutine. We consider firstly a two dimensional plane stress element which has four nodes, with linear interpolation functions. The obtained global numerical results are compared to experimental ones. This numerical tool allows to access to the local information in a given material point with a given local hydrogen concentration (stress state, transformation state). Such informations are mandatory for the design of SMA archwires considering the effect hydrogen diffusion. As a prospect of this work, this formulation will be extended to the three dimensional case. The obtained numerical tool will allow to analyze the effect of hydrogen diffusion on the performance of SMA-based orthodontic arches.

[1] A. Lachiguer, C. Bouby, F. Gamaoun, T. Bouraoui and T. Ben Zineb. Modeling of hydrogen effect on the superelastic behavior of Ni-Ti shape memory alloy wires. Smart Materials and Structures, 25 115047, 2016.
[2] Y. Chemisky, A. Duval, E. Patoor and T. Ben Zineb. Constitutive model for shape memory alloys including phase transformation, martensitic reorientation and twins accommodation. Mechanics of Materials, 43(7) : 361-376, 2011.

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28 Aug
4:00pm
Amphi 1

Aging Effect on the One-way and Two-way Shape Memory in TiNi-based Alloys. Experimental Study and Microstructural Modeling

Volkov A. E., Belyaev F. S., Evard M. E., Ostropiko E. S., Razov A. I.

Saint Petersburg State University, RUSSIAN FEDERATION

The influence of aging of TiNi-based shape memory alloy (SMA) specimens on the one-way and two-way shape memory was studied experimentally and simulated with the use of a microstructural model. Experiments have shown that a long-time storage of preliminarily strained TiNi and TiNiFe specimens does not change the strain recovery on heating. However, strain variations due to the two-way shape memory are bigger than before aging. A study of assembled thermomechanical couplings shows that the contact stresses in them practically do not decrease during aging. Computer modeling of these phenomena was done within the frames of a microstructural model, in which the macroscopic strain of a representative volume is considered to be the average of the micro strains strains of micro volumes (grains, austenite phase and Bain's domains of martensite). The micro strains are assumed small enough to be described by the small-strain tensors, and they are assumed to be the sum of the elastic, thermal, phase, and irreversible micro-plastic strains. For calculation of the phase strains a martensitic transformation is formulated in terms of the generalized thermodynamic forces. The irreversible strains are described from

the standpoint of the plastic flow theory. Isotropic hardening and kinematic hardening are taken into account and are related to the densities of scattered and oriented deformation defects. To describe the effects of aging special terms are introduced into the evolution equations for the defects densities. Thus modified equations are able to account for the age softening with the decrease of the deformation defect densities. A procedure for finding the material constants was developed. The results of modeling show a good qualitative agreement with the experimental data, particularly an increase of the two-way shape memory deformation after aging.
The work was supported by the Russian Foundation for Basic Research, grant 18-01-00594.

Microstructural Simulation of Fatigue Fracture of TiNi and FeMn-based Shape Memory Alloy Samples

28 Aug
2:00pm
Poster
Session A

Evard M., Volkov A. E., Belyaev F. S.

Saint Petersburg State University, RUSSIAN FEDERATION

A microstructural model of functional and mechanical behavior shape memory alloys (SMAs) has been proposed. This model has been modified for simulation both TiNi and FeMn-based SMAs behavior. Bain's deformation matrixes are used in this model as the basic characteristics of the martensitic transformations. The internal variables of the model, describing the amounts of martensite obtained by different variants of Bains deformation, are introduced in a way to consider the existence of different orientation variants of the direct transformation and the single-variant (as in TiNi-based alloys) or multi-variant (as in FeMn-based alloys) character of the reverse martensitic transformation. Self-accommodation of martensite has been taken into account by grouping of the variants into correspondence variants pairs for TiNi and triplets reflecting the threefold symmetry of the hcp and fcc phases for FeMn. Another set of internal variables specifies the measures of deformation defects produced by the microplastic deformations providing the plastic accommodation of martensite. Conditions of the transformation and microplastic deformation have been proposed and used for deduction of the evolution equations for the variation of the internal variables. The model has been supplemented by a deformation-and-stress criterion of fracture allowing to predict accumulation of damage both for a one-side loading and thermal and mechanical cycling. This criterion takes into account the effect of hydrostatic pressure, deformation defects and material damage. It has been shown that the model can describe the fatigue fracture of TiNi and FeMn SMAs under various thermomechanical cycling regimes. Results of calculating the number of cycles to failure at thermocycling under a stress, at symmetric two-sided cyclic deformation, at straining-unloading cycles, at cycling in the regime of loading imitating that for endovascular stents, vibration protection devices and the thermodynamic cycles of a SMA working body in the hard (strain controlled) and soft (stress controlled) working cycles and are presented for different parameters of these cycles.
This research has been supported by the grant of Russian Foundation of Basic Research 16-01-00335.

A micromechanical model for polycrystalline shape memory alloy wires embedded into smart structures

28 Aug
2:00pm
Poster
Session A

Hannequart P.[1], Peigney M.[1], Caron J.-F.[1], Viglino E.[2]

[1] *Université Paris-Est, Laboratoire Navier (UMR 8205), CNRS, Ecole des Ponts ParisTech, IFSTTAR, Marne la Vallée, FRANCE;*
[2] *Arcora, Groupe Ingérop, Rueil-Malmaison, France*

Thermomechanical models for shape memory alloys (SMA) can be categorized into phenomenological models and micromechanical models. Most micromechanical SMA models are 3-dimensional, computationally intensive and can only hardly be implemented in engineering codes to model smart structures. Some attempts at developing a simplified one-dimensional model have been made but often lack robustness. The SMA behavior is modelled by using a free energy with constrained state variables, which makes the numerical implementation delicate. This research aims at providing a computationally efficient one-dimensional micromechanical model for polycrystalline SMA wires. A texture is usually described by a set of crystalline orientations along with their volume fraction. In the present work, each orientation relates to the martensitic transformation strain of its most favorably oriented martensitic variant (with respect to the loading direction), projected onto the wire axis. Two internal state variables characterize each crystalline orientation: the volume fraction of self-accommodated martensite, and the volume fraction of

the most favorably oriented martensitic variant. The one-dimensional mechanical model of the wire in traction thus considers the influence of the 3-dimensional texture. The texture of Nickel-Titanium wires has been chosen in this study. This model can describe the specific thermomechanical behavior of shape memory alloy wires, such as superelasticity, self-accommodation and reorientation of martensite, as well as the one-way shape memory effect. It has been implemented numerically in an efficient computational tool. A user-material subroutine (UMAT) resorting to numerical constrained optimization tools has been developed for the finite element software ABAQUS. Several thermomechanical experiments on Nickel-Titanium wires have been carried out in order to identify the model parameters and the wire texture. The model has been used to simulate the response of morphing structures including SMA wires, actuated by shape memory effect. Such structures have also been manufactured with fiber reinforced polymers, and their displacements as well as their actuation temperatures could be compared to the model predictions.

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28 Aug
2:00pm
Poster
Session A

Experimental and numerical investigations of the propagation of austenite-to-martensite transformation bands

Helbert G.[1], Simon J.[2], Arbab Chirani S.[1], Balandraud X.[3], Dieng L.[4]

[1]*ENI Brest, FRE CNRS 3744, IRDL,Brest, FRANCE;*
[2]*ENSTA Bretagne, FRE CNRS 3744, IRDL, Brest, FRANCE;*
[3]*Clermont Université, Institut Français de Mécanique Avancée, UMR CNRS 6602, Institut Pascal, Aubière, FRANCE ;*
[4]*IFSTTAR, MAST, SMC, Bouguenais, FRANCE*

Thanks to the martensitic transformation, NiTi-based Shape Memory Alloys perform a significant dissipated energy. It is the reason why their use is attractive under the form of wires into damper devices, such as in anti-seismic applications. The dissipated energy and the fatigue life strongly depend on the strain field heterogeneity, due to the nucleation-propagation process of phase transformation fronts. This heterogeneity can be removed by an appropriate training process. Dissipation is maximal over the first loading cycle, and then decreases cycle after cycle. In this case, the training process is unwanted. In order to optimize the effectiveness of these devices by controlling the heterogeneity of the strain fields, this one must be investigated.
In this study, we propose (i) to investigate numerically the effect of fastening in the jaws and concentration stress at the interface between austenitic and martensitic areas to characterize the nucleation and propagation of transformation bands, respectively. Then (ii), a 0D macroscopic thermomechanically coupled three-phase super-elastic model is extended to a one-dimensional (1D) model, using a Cellular Automata Finite Element approach. It enables naturally create transformation bands in NiTi wires, under tensile loadings at room temperature. Indeed, three-dimensional (3D) phenomena associated with different behaviours on both sides of the transformation front, while the model remains 1D, and heat exchanges between the sample and the environment are taken into account. Eventually (iii), experimental observations, provided from simple image processing, and simulation results are compared.
This study enables to highlight and discuss some interesting points: (a) The superelastic hysteresis loop area size and shape arise from a competition between thermal and mechanical effects at the transformation front. (b) By introducing artificial defects in the material, one can link the imposed loading rate with the number of transformation bands. (c) Due to R-phase transformation intrisic properties, this intermediate transformation is homogenous, unlike the martensitic transformation.

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28 Aug
2:00pm
Poster
Session A

A multiscale approach for the thermomechanical modeling of shape memory alloy fiber reinforced composites

Xu R.[1,2], Bouby C. [2], Zahrouni H.[2], Ben Zineb T.[2], Hu H. [1]

[1]*School of Civil Engineering, Wuhan University, PR CHINA;*
[2]*Université de Lorraine, CNRS, Arts et Métiers ParisTech, LEM3, Nancy, FRANCE*

Hybrid materials with multi-physical behavior could contribute to the emergence of innovative applications taking advantage of the interesting properties of shape memory alloys (SMAs) and polymer components. The fiber-matrix or multilayer composites SMA-Polymers could result in applications in energy recovery and conversion or in sensor-actuators. It is therefore important to have numerical tools for predicting the multi-physical, multi-scale and non-linear behavior of these composite materials. Toward this end, we propose a numerical tool for modeling the effective behavior of SMA / Polymers composites based on the multilevel finite element (FE) method. It is an iterative numerical approach where the deformations calculated at any integration point of the structure are applied as boundary conditions at the level of the associated RVE. The SMA phase behavior is described by a constitutive law based on a thermodynamic approach where the driving forces associated with the internal martensite volume fraction and mean transformation deformation are derived from the postulate of Gibbs free energy expression. The behavior of the polymer is assumed to be linear and isotropic elastic. The FE method is adopted for multi-scale modeling. It can realize the transition of numerical scale between a complex heterogeneities microstructure discretized by finite elements and the macrostructure, where the responses in two scales are calculated simultaneously and coupled. The procedure is implemented in the ABAQUS finite element code via the UMAT routine. The state of constraints, volume fraction of transformation, and the corresponding tangent operators are thus calculated and considered as input at each point of integration of the mesh of the structure for the calculation of the global equilibrium. This multi-scale approach is validated on thermomechanical test cases in the literature. It will subsequently be used for the designing of a composite SMA / polymer application.

Effect of thermomechanical coupling on stress-induced martensitic transformation in the crack tip region of NiTi

28 Aug
2:00pm
Poster
Session A

You Y.[1], Moumni Z.[2], Anlaş G.[3], Zhang W.[4]

[1]*Northwestern Polytechnical University, CHINA;*
[2]*UME - ENSTA - ParisTech - Palaiseau, FRANCE;*
[3]*Department of Mechanical Engineering, Bogazici University ;*
[4] *State IJR Center of Aerospace Design and Additive Manufacturing, Northwestern Polytechnical University, CHINA*

In the present study, the effect of the theromomechanical coupling on stress-induced martensite phase transformation in the crack tip region of compact tension (CT) specimens of pseudoelastic NiTi SMAs is analyzed both experimentally and numerically. To this end, six CT specimens were submitted to tensile loads at different loading rates and the temperature distributions around the crack tip were captured by an infrared camera. Furthermore, the extended thermo-mechanically-coupled ZM (Zaki-Moumni) model was derived and implemented into the finite element code ABAQUS, so the temperature and phase transformation distributions are evaluated. The comparison between experiments and simulations results show a very good agreement. In addition, the results show that phase transformation region size decreases when the loading frequency increase s and the change of phase transformation area is evaluated (12% decreases in this case). To sum it up , numerically evaluation presented in this paper explains that the effect of thermomechanical coupling should be considered for analyzing SMAs fracture and crack propagation problems.

Elucidation of the role of the structure of porous tini for its mechanical and functional properties

28 Aug
2:00pm
Poster
Session A

Volkov A., Evard M., Iaparova E.

Saint Petersburg State University, RUSSIAN FEDERATION

Porous NiTi is a promising material for using as a bone implant in medicine. There is a large number of works devoted to experimental investigation of such material. Deformation of porous shape memory alloy (SMA) samples is determined by features of its heterogeneous structure and martensitic phase transformation in it. The problem of differentiation between contributions of one or other unelastic deformation mechanisms into mechanical and functional properties of porous SMAs is an actual question. Based only on an analysis of experimental results it hardly can be solved. An attempt to separate different mechanisms of deformation by modeling is presented in this work.
There are different approaches to describing behavior of porous SMAs. However, most of them do not allow taking into consideration the peculiar properties of porous structure, such as shape and orientation of porous channels. The model proposed in this work permits to consider the most common types of pore channels' direction and presents appropriate methods of calculation for each of them. The microstructure of porous samples is simulated by the earlier developed models based on the bending theory of beams.

Representative volume of each type of porous samples undergoes martensitic transformation, and its description requires the using of special equations. The approach under consideration is based on the microstructural model (Volkov et al., 2014). Its main constitutive relations regard hierarchy of structural levels in shape memory alloys and interaction between deformation mechanisms at the adjacent levels.
In this work TiNi porous samples with two types of pore channels direction have been considered. Simulation of isothermal compression at different temperatures and shape memory effect has been performed for three types of modeling samples: bulk SMA, porous SMA and porous sample without martensitic transformations. Comparison of these results allows to establish linkage between peculiarities of deformation behavior and structure of the porous SMA sample.

Session 2: Advanced Characterization of Martensite

Invited Lecture: Tuesday August 28	
8:30 Amphi 4	**G. Laplanche**, J. Frenzel, T. Birk, S. Schneider, G. Eggeler "On the effect of temperature and texture on the reorientation of martensite variants in NiTi shape memory alloys"
Oral presentations: Tuesday August 28	
9:00 Amphi 4	**T. Waitz**, M. Kerber, E. Schafler, F. Spieckermann, J. Bednarcik, T. Fischer "Studying phase transformations of NiTi shape memory alloys subjected to severe plastic deformation using in-situ synchrotron experiments"
9:20 Amphi 4	**G. Geandier**, L. Vautrot, B. Denand, S.Denis "Analysis of internal stresses during cooling by in situ high energy X-ray diffraction in a metal matrix composite with martensitic transformation of the matrix"
09:40 Amphi 4	**Y. El Hachi**, B. Malard, S. Berveiller, J. Wright, W. Ludwig, X. Morel, D. Bouscaud, B. Piotrowski "Influence of grain neighbours on the stress-induced martensitic transformation in individual grains studied by synchrotron 3DXRD and Diffraction Contrast Tomography (DCT)"
10:00 Amphi 4	**D. Langenkämper**, A. Paulsen, C. Somsen, J. Frenzel, E. Karsten, H. Jürgen Maier, G Eggeler "Synchrotron radiation and transmission electron microscopy investigations on the formation and dissolution temperatures of the ω -phase in Ti-Ta high temperature shape memory alloys"
10:50 Amphi 4	**G. Eggeler**, J. Burow, J. Frenzel, C. Somsen, E. Prokofiev, R. Valiev "An In-situ TEM Study of the Nucleation and Growth of New Grains in Strongly Deformed NiTi"
11:10 Amphi 4	**P. Thome**, M. Schneider, M. Ersanli, E. J. Payton, V. A. Yardley "In-depth EBSD investigation of spatially coupled crystallographic properties in binary FeNi alloys"
11:30 Amphi 4	**Y. Feng** "Microstructure and deformation behavior of Fe nanoparticle reinforced CuZnAl composite"
11:50 Amphi 4	**S. Pourbabak**, B. Verlinden, J. Van Humbeeck, D. Schryvers "Effect of DSC cycling on room temperature and low temperature aged nano-grain Ni-Ti microwires"
12:10 Amphi 4	**E. Nobre Dantas Grassi**, D. Favier, G. Chagnon "Influence of anisotropy on strain localization phenomenon in tension of superelastic NiTi thin walled tubes"
Oral presentations: Wednesday August 29	
10:50 Amphi 4	**E. Alarcon**, L. Heller, P. Sittner, S. Arbab Chirani , L. Saint-Sulpice "Mechanical dissipation of superelastic NiTi upon low-strain loading resolved by infrared thermography"
11:10 Amphi 4	**M. Vronka**, J. Vesely , J.Manak , M. Karlik , O. Heczko "Twinning in Micro and Nanoscale Pillars-Size effect in Cu-Ni-Al shape memory alloy"
11:30 Amphi 4	**M. Nishida**, Y. Soejima, H. Akamine "Dynamic Visualization of Thermoelastic Martensitic Transformation with In-situ SEM Observation"
11:50 Amphi 4	**X. Chang**, K. Lavernhe Taillard, O. Hubert "Observation of martensite phase transformation in shape memory alloys using a combined XRD-DIC technique"
12:10 Amphi 4	**L. Straka**, J. Drahokoupil, P. Veřtát, M. Zelený, J. Kopeček, A. Sozinov, O. Heczkon "Low temperature {110} nanotwins in Ni-Mn-Ga and Ni-Mn-Ga-Fe 10M martensites"

Poster Session B: Tuesday August 28
S. Belyaev,N. Resnina, R. Konopleva, V. Chekanov, A. Nakin, A. Shelykov "Influence of the neutron irradiation on NiTi-based alloys with different crystalline structure"
L.-G. Bujoreanu, M. Popa, C. Gurau, G. Gurau, B. Pricop, R. I. Comaneci, M. Vollmer, P. Krooss, T. Niendorf "On the structure and properties of $Fe_{43.5}Mn_{34}Al_{15-x}Ni_{7.5+x}$ shape memory alloys
A. Churakova, D. Gunderov "Change in enthalpy and energy of martensitic transformation in ultrafine-grained and nanocrystalline states of $Ti_{49.1}Ni_{50.9}$ alloy"
W.-N. Hsu, E. Polatidis, M. Smid, S. Van Petegem, **H. Van Swygenhoven** "Phase transformation and degradation of superelastic NiTi during uniaxial and multiaxial loading"
F. Meraghni, N. Bourgeois, R. Echchorfi, L. Peltier, Y. Chemisky, E. Patoor "Strain paths and superelastic anisotropy in a textured NiTi under isothermal uniaxial and biaxial loading using DIC analysis"
S. Pourbabak, A. Orekhov, D. Schryvers "Ni-Ti in-situ stress induced martensitic transformation and strain mapping in a TEM"
J. Racek, J. Duchoň, M. Vronka "TEM observation of austenite twins in biomedical NiTi wire after superelastic cycling"
E. Ryklina, K. Polyakova, N. Tabachkova, N. Resnina, S. Prokoshkin "Effect of B2-austenite Grain Size and Isothermal Aging on Microstructure of Ti_3Ni_4 Precipitates and Transformation Sequence in Titanium Nickelide"

Contents

On the effect of temperature and texture on the reorientation of martensite variants in NiTi shape memory alloys

28 Aug
8:30am
Amphi 4

Laplanche G., Frenzel J., Birk T., Schneider S., Eggeler G.

Ruhr-Universitaet Bochum, GERMANY

In the present study, martensitic NiTi wires and sheets with similar transformation temperatures, similar grain sizes but different textures were tensile strained at temperatures ranging between -100°C and 60°C to study the effect of texture and temperature on reorientation of martensite variants [1]. The stress-strain curves of the NiTi alloys are characterized by a stress-plateau which occurs concomitantly with the propagation of a Lüders band. The tensile tests were interrupted after the end of the stress-plateau. After unloading, the tensile specimens were heated to trigger the one way shape memory effect and the associated recoverable strains were measured. A comparison between the mechanical data obtained for tensile specimens taken from wires and sheets revealed a strong effect of texture. The plateau stresses of wires were found to be 25-33% lower and their recoverable strains were 30% higher than for sheets. However, the product of plateau stress and recoverable strain, which represents the external work per unit volume required for martensite variants reorientation is found to be independent of texture. The tensile tests performed at different temperatures revealed that the recoverable strain is independent of temperature. In contrast, the plateau stress as well as the external work required to reorient martensite variants increase with decreasing temperature. These findings could be rationalized in the light of a thermodynamic approach involving the elastic strain energy associated with the growth of reoriented martensite variants.

[1] G. Laplanche, T. Birk, S. Schneider, J. Frenzel, G. Eggeler, Effect of temperature and texture on the reorientation of martensite variants in NiTi shape memory alloys, Acta Materialia 127 (2017) 143-152

Mechanical dissipation of superelastic NiTi upon low-strain loading resolved by infrared thermography

29 Aug
10:50am
Amphi 4

Alarcon E.[1], Heller L.[1], Sittner P.[1], Arbab Chirani S.[2], Saint-Sulpice L.[2]

[1]*Institute of Physics of the CAS, Nuclear Physics Institute of the CAS. CZECH REPUBLIC;*
[2]*Ecole Nationale d'Ingénieurs de Brest, Technopole Brest-Iroise, Brest, FRANCE*

As in traditional metals, the fatigue damage of superelastic NiTi is characterized by the activation and evolution of dissipative mechanisms. Therefore, the amount of dissipated energy in a mechanical loading cycle may be considered as a measure of the fatigue performance of superelastic NiTi. In the high cycle fatigue regime, where the material may behave linear-elastically or display stress-induced R-phase transformation, the amount of dissipated energy is difficult to quantify as the stress-strain curves show imperceptible hysteresis loops. To correlate the mechanical dissipation with the high cycle fatigue behavior of NiTi, we propose an experimental method based on temperature field measurements during small strain fatigue loading. For this purpose, we used an infrared detector with a high acquisition rate frequency and great temperature and spatial resolutions. The temperature field measurements and the applied loading of the testing machine were synchronized, which allowed us to characterize precisely the temperature evolution of our samples with the applied stresses. From monotonic loading tensile tests in the stress range of the high cycle fatigue regime, we identified that the global temperature evolution of superelastic NiTi is caused by an interaction of three heat effects: i) the thermoelastic coupling of the austenite phase, ii) the latent heat exchange upon the austenite to R-phase transformation, and iii) mechanical dissipation. To deconvolve these three heat components, we carried out a series of temperature field measurements upon constant-frequency-amplitude cyclic tests. Then, we evaluated the constituent frequencies of the recorded temperature fields. When deformation mechanisms other than elasticity are activated, we found that the temperature evolution of the sample contains two higher harmonics besides the fundamental one, and a DC component related to the stabilized mean temperature of the sample (self-heating). By simulating the thermomechanical response of NiTi upon cyclic loading, we resolved the contributions of each heat effect to the DC component and the first three temperature harmonics. The simulations combined a phenomenological SMA model with the resolution of a 1D-heat diffusion problem reproducing the experimental boundary conditions. As a result, we show that the thermoelasticity and latent heat exchange influence the evolution of the first harmonics with respect to the applied stress. On the other hand, the latent heat exchange and the dissipation affect the evolutions of the second and third harmonics. We propose a procedure for extracting the dissipation from the second harmonics evolution based on the identification of the latent heat in the evolution of the first harmonics. Finally, we correlate the dissipation evolution with the high cycle fatigue behavior of superelastic NiTi.

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29 Aug
11:50am
Amphi 4

Observation of martensite phase transformation in shape memory alloys using a combined XRD-DIC technique

Chang X., Lavernhe K., Hubert O.

LMT (ENS Paris-Saclay / CNRS / Université Paris-Saclay, FRANCE

Shape memory alloys (SMA), given to their pseudo-elastic behaviors and/or shape memory properties, have increasing industrial applications in domains such as robotic, medicine and aerospace. NiTi is probably the most widely used material for these alloys. However the potential coexistence of three principal phases (Austenite, Martensite, and R phase) depending on the thermomechanical loading conditions and exhibiting very different transformation deformation, makes the proposition of a robust multiaxial thermo-mechanical modeling a difficult task.
The combined DIC-XRD (Digital Images Correlation / X-Ray Diffraction) observation thus becomes extremely relevant for a precise evaluation, in terms of the characteristics and conditions of appearance for the phases, and for the validation of a model if it needs to be. Indeed tensile strengthening of such type of material is well known to develop multiple localized transformation bands. On one hand, DIC grants a continuous global observation of strain field and strain gradient at the interface between bands and matrix over a large region of interest (ROI) as function of time. On the other hand, a series of XRD scanning permit us to construct a spatial distribution of volume fraction for each phase.
Such experiment has been carried out on a quasi-equiatomic Ni-Ti 1D strip. By using proper optimization procedure, the experimental distribution of Austenite, Martensite and R phase has been identified at various strain levels, showing that the R-phase is always produced upstream of the production of the Martensitic phase. This distribution allows on the other hand to evaluate the associated strain field by considering the free transformation deformation of each phase as a given data. This evaluation is shown to be in accordance with the strain field obtained by DIC. Experimental results are finally compared to the results of a finite difference thermomechanical modeling using a multiaxial and multiscale stochastic model as constitutive behavior.

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28 Aug
10:50am
Amphi 4

An In-situ TEM Stuy of the Nucleation and Growth of New Grains in Strongly Deformed NiTi

Eggeler G.[1], Burow J.[1], Frenzel J.[1], Somsen C.[1], Prokofiev E[2], Valiev R.[3]

[1] *Ruhr-Universität Bochum, GERMANY;*
[2] *Saint Petersburg State University, RUSSIA;*
[3] *Ufa State Aviation Technical University, RUSSIA.*

We investigates the nucleation and growth of nano and micrograins in severely plastically deformed NiTi. Two deformed NiTi alloys were subjected to in situ annealing in a transmission electron microscope (TEM) at 400 and 550 ˚C: An amorphous material state produced by high pressure torsion (HPT) and a mostly martensitic partly amorphous alloy produced by wire drawing. In situ annealing experiments allowed to directly observe the microstructural evolution from the initial states towards energetically more favorable microstructures. In general, the formation and evolution of nanocrystalline microstructures are governed by the nucleation of new grains and their subsequent growth. Austenite nuclei which form in HPT and wire-drawn microstructures have sizes close to 10 nm. Grain coarsening occurs in a sporadic, non-uniform manner and depends on the physical, chemical and microstructural features of the local environment. The mobility of grain boundaries in NiTi is governed by the local interaction of each grain with its environment. Nanograin growth in thin TEM foils seems to follow similar kinetic laws as observed in bulk microstructures. A short movie will be shown which documents the formation and growth of new grains in a severely deformed amorphous NiTi material

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Influence of grain neighbours on the stress-induced martensitic transformation in individual grains studied by synchrotron 3DXRD and Diffraction Contrast Tomography (DCT)

28 Aug
09:40am
Amphi 4

El Hachi Y.[1], Malard B.[2], Berveiller S. [1], Wright J.[3], Ludwig W.[4], Morel X.[1], Bouscaud D.[1], Piotrowski B.[1]

[1]*Arts et Métiers ParisTech, LEM3, Metz, FRANCE;*
[2]*CIRIMAT, Université de Toulouse, CNRS, Toulouse INP, UPS, Toulouse, FRANCE;*
[3]*ESRF, Grenoble, FRANCE;*
[4]*MATEIS, INSA Lyon, UMR5510 CNRS, Villeurbanne, FRANCE*

In shape memory alloys, single crystal analysis has delivered a great deal of useful information furthering the understanding of interfacial motions between parent and product phases, for both uniaxial loading and during temperature changes. Unfortunately, these results cannot be easily extended to martensitic transformation in polycrystalline materials. Strain incompatibilities occurring at grain boundaries and stress transfer between transforming grains strongly influence the transformation kinetics in polycrystals. As a consequence, the macroscopic behaviour differs strongly between polycrystals and single crystals. Therefore, synchrotron techniques have been used to study both morphology and strain-stress state of individual grains embedded in a polycrystal. Diffraction Contrast Tomography allows the three-dimensional morphology of individual grains to be determined with a spatial resolution of $\sim 1.5\ \mu$m as well as their positions in the specimen. The 3D-XRD technique gives access to the crystallographic orientation, average position and the stress state of each grain; due to various constraints, internal stresses were determined only in the austenite phase. Both techniques were coupled to follow one hundred grains during an in-situ tensile test on a Cu-Al-Be superelastic alloy. The microstructure of the polycrystal was reconstructed from DCT data. Correlations with 3DXRD results show that individual grains exhibit large stress heterogeneities, as much as a factor of 3. The stress state depends on the crystallographic orientation but also strongly on the local neighbour environment; we observed differences when comparing pairs of grains with the same orientation. In some cases, intergranular interactions inhibited the martensitic transformation. Finally, grains located at the surface carry lower stress values than grains embedded in the volume. However, when calculating the critical resolved shear stress from the local stress tensor values, we found that all the grains have similar experimental values.

Microstructure and deformation behavior of Fe nanoparticle reinforced CuZnAl composite

28 Aug
11:30am
Amphi 4

Feng Y.

China University of Petroleum-Beijing CHINA

A Fe particle reinforced CuZnAl composite was prepared by means of friction stir processing and wire drawing. The microstructure of composite was studied by using of SEM, TEM and XRD. Reinforcing effect of the Fe particle and deformation behavior in the composite were investigated by means of in-situ synchrotron X-ray diffraction. The maximum elastic strain of the Fe particle achieved was 0.7%, implying a component stress of 140 MPa on the particles. The Fe particles, with a volume fraction of 10%, carried 30% of stress fraction during tensile deformation.

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28 Aug
9:20am
Amphi 4

Analysis of internal stresses during cooling by in situ high energy X-ray diffraction in a metal matrix composite with martensitic transformation of the matrix

Geandier G.[1,2], Vautrot L.[1,2], Denand B.[1,2], Denis S.[1,2]

[1] *Université de Lorraine, CNRS, IJL, Nancy, FRANCE;*
[2] *LabEx DAMAS, Nancy, FRANCE*

In metal matrix composites (MMC), internal stresses are generated during cooling due to the differences in the coefficient of thermal expansion between the matrix and the reinforcements. When the matrix presents a phase transformation, the associated deformations induce also internal stresses. The evolutions of these stresses are a key factor in understanding the residual stress states which play an important role on the final mechanical properties of the MMC. In situ high energy X-ray diffraction using a synchrotron source performed on a steel metal matrix composite reinforced by TiC allows following the evolutions during cooling of the phase fractions and the mean cell parameters (Rietveld analysis). Thanks to the development of a new experimental device (a transportable radiation furnace with a controlled rotation of the specimen) the evolutions during cooling of the stress tensor components in all phases of the composite have been obtained too. It has been shown that the matrix and the reinforcements are under a hydrostatic stress state all along cooling even during the martensitic transformation. Using the stress free cell parameters and their evolutions versus temperature, it comes out that from 900°C to the beginning of the martensitic transformation, high compressive stresses are generated in the reinforcements due to the difference of the thermal expansion coefficients of the phases. As the martensitic transformation occurs, the stresses relax largely in the reinforcements, martensite undergoes tensile stresses and relatively small stress variations occur in the austenite. Micromechanical simulations by finite elements have been used to understand better these stress evolutions.

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28 Aug
10:00am
Amphi 4

Synchrotron radiation and transmission electron microscopy investigations on the formation and dissolution temperatures of the ω -phase in Ti-Ta high temperature shape memory alloys

Langenkämper D.[1], Paulsen A.[1], Somsen C.[1], Frenzel J.[1], Karsten E.[2], Maier H. J.[2], Eggeler G.[1]

[1] *Institut für Werkstoffe, Ruhr-Universität Bochum, Bochum, GERMANY;*
[2] *Institut für Werkstoffkunde (Materials Science), Leibniz Universität Hannover, Garbsen, GERMANY*

Shape memory alloys (SMAs) are attractive for technical and medical applications. The shape memory effect of SMAs is based on a reversible martensitic transformation between an austenitic high-temperature phase and a martensitic low-temperature phase. The transformation temperatures limit the field of application of SMAs. Several technical applications, e.g. in the sectors of aerospace and automotive sectors, require phase transformation temperatures higher than 120°C and a demand exists for a new class of SMAs with high martensitic transformation temperatures. Alloys based on the Titanium-tantalum (Ti-Ta) system are promising candidates as high-temperature shape memory alloys (HT-SMA) due to their martensite start temperature (Ms) which is close to 250°C. Ti-Ta alloys with a Ta content of 20 to 40 at.-% exhibit on cooling a martensitic transformation from the austenitic β-phase (bcc) to the martensitic α"-phase (orthorhombic). In addition, the metastable hexagonal ω-phase can be observed after tempering in the austenitic phase at medium temperatures. The ω-phase suppresses the martensitic α'-phase resulting in a loss of functional properties. In the present study synchrotron radiation and in situ transmission electron microscopy experiments were performed to investigate the temperature dependent formation and dissolution behavior of the ω-phase in a $Ti_{75}Ta_{25}$ alloy. For synchrotron investigations an initially martensitic sample has been heated up to 650°C and then cooled down to ambient temperature with a temperature rate of 20 °C/min. The data reveals a transformation from the martensitic α"-phase to the austenitic β-phase at about 300°C on heating. Faint reflections associated with the ω-phase appear additionally to the reflections of the β-phase. The intensity of the ω-phase reflections increases with time and temperature up to about 500°C. On further heating the intensity of the ω-phase reflections decreases and disappears at about 550°C, while only reflections of the austenitic β-phase are present up to 650°C. In contrast, on cooling the reflections of the ω-phase appear at lower temperature of about 400°C next to the reflections of the β-phase. On cooling down to ambient temperature the ω-phase suppresses the martensitic transformation and only β-phase and ω-phase can be detected, also confirmed by post mortem transmission electron microscopy.

Financial support by Deutsche Forschungsgemeinschaft within the Research Unit Program "Hochtemperatur-Formgedächtnislegierungen" (Contract nos. EG 101/22-2, FR 2675/4-2, MA 1175/34-2 and SO 505/2-2) is gratefully acknowledged.

Dynamic Visualization of Thermoelastic Martensitic Transformation with In-situ SEM Observation

29 Aug
11:30am
Amphi 4

Nishida M., Soejima Y., Akamine H.

Kyushu University, JAPAN

The nucleation and growth of the martensitic phase, the interaction between habit plane variant clusters (HPVC), and the forward and reverse transformation sequences were examined by in-situ scanning electron microscopy. In the Ti-Ni shape memory alloy, nucleation of the V-shaped 2HPVC triggers the formation of the hexangular 6HPVC in the grain interior. The autocatalytic transformation spreads from the 6HPVCs to the grain boundary and the reverse transformation proceeds like a rewind of the forward transformation. On the other hand, the martensitic phase nucleates from grain boundary in Cu-Al-Mn, Cu-Al-Ni and Ni-Mn-Ga alloys. The relation between the induced dislocations with thermal cycling and the self-accommodation morphologies in Ti-Ni alloy has been investigated by electron channeling contrast imaging. The induced dislocations are confined within the self-accommodated HPVC and there are no dislocations in the untransformed parent phase area. Many of dislocation loops are observed along $< 011 >$ type II twin trace near the habit plane. This fact shows that the plastic deformation hardly occurs in the parent phase upon the transformation. The forward and reverse movements of stress induced martensite were characterized in thermomechanically treated Ti-Ni alloys by in-situ tensile observations. Isothermal and multistage martensitic transformations in aged Ni-rich Ti-Ni with Ti_3Ni_4 precipitates were also visualized. We demonstrate that advanced SEM with in-situ stages has a great potential for dynamic visualization of thermoelastic martensitic transformation.

Effect of DSC cycling on room temperature and low temperature aged nano-grain Ni-Ti microwires

28 Aug
11:50am
Amphi 4

Pourbabak S.[1], Verlinden B.[2], Van Humbeeck J.[2], Schryvers D.[1]

[1] *University of Antwerp, EMAT group, BELGIUM;*
[2] *University of Leuven, Department of Materials Engineering, MTM, BELGIUM*

In this work the effect of Differential Scanning Calorimetry (DSC) measurements on room temperature and low temperature aged NiTi microwires is investigated. A $Ni_{50.8}Ti_{49.2}$ wire of diameter 150μm was recrystallized by annealing at various temperatures and times followed by water quenching and yielding samples with different grain size and low amounts of dislocations. The sample annealed at 600°C for 20 minutes which results in a grain size of about 1500nm diameter was selected for investigation. The obtained samples are divided into two sets, the first to be aged during all operations at room temperature (RT), the second at 100°C. Each set is divided into several sub-sets, treated as follows:
A. 1 sample undergoing 10 consecutive DSC cycles at the day of annealing (not for the 100°C aged sample)
B. 1 sample undergoing 10 DSC cycles, one at each of the following days 0, 1, 2, 4, 8, 15, 30, 60, 90, 120 after annealing, with in between the respective aging at RT or 100°C
C. 6 samples each undergoing 1 DSC cycle at respectively day 0, 2, 8, 30, 90, 120 after annealing, with in between the respective aging at RT or 100°C
The martensitic transformation start temperature (Ms) of different tests are compared with each other. The following results are obtained:
The set aged at RT shows lowering of Ms for part A, which means the martensitic transformation is suppressed during cycling. Part B with RT aging between DSC cycles shows an even faster lowering of Ms. No difference between Ms of the 6 samples in C measured at different days is observed.
The set aged at 100°C shows lowering of Ms for part B, plus the appearance of a small peak before Ms belonging to the R-phase. The suppression of Ms is also observed for samples C, which means that aging at low temperature of 100°C without cycling has a detectable effect on the martensitic transformation. In both B and C tests the first peak belonging to the test before putting the sample in the furnace has a different shape than the peaks in the following runs, indicating an immediate effect of aging at 100°C

on the structure. In order to understand the microscopic origin of such behavior, Transmission Electron Microscopy (TEM) and Automated Crystal Orientation Mapping (ACOM-TEM) will be performed on the samples after the treatment procedure is completed.

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28 Aug
12:10pm
Amphi 4

Influence of anisotropy on strain localization phenomenon in tension of superelastic NiTi thin walled tubes

Nobre Dantas Grassi E., Favier D., Chagnon G.

TIMC-IMAG Laboratory / Université Grenoble Alpes FRANCE

Tubes of Nickel-Titanium Shape Memory Alloy (NiTi SMA) are employed to produce 60% of self-expanding cardiovascular stents. Such small-scale devices require tubes with small thickness, which in turn implies a material with highly textured microstructure. Previous works demonstrate that this texture causes significant anisotropy, affecting various thermomechanical key properties. This knowledge is of critical value for the modeling of thermomechanical behavior and design of superelastic devices. As well as thermomechanical properties, the localization phenomenon observed in tensile tests is affected by the anisotropy in a NiTi tube and also needs investigation. The present work aims to address the effect of anisotropy on localization phenomenon through a series of tensile tests performed at three temperatures above Af (austenite finish temperature). Tensile dogbone samples were cut in five orientations from a flattened NiTi thin walled tube (0.165 mm of thickness). The orientations were 0°(drawing direction), 22.5°, 45°, 67.5° and 90°(transverse direction). Using Digital Image Correlation (DIC) technique, strain rate fields were calculated over a zone of interest in the gauge area of the samples, measuring 10 x 2 mm^2. Localization characteristics (in particular the angles of the localization bands with respect to the tension direction) were determined from these fields. Localization phenomena were observed for the samples cut at 0°, 22.5°, 67.5°and 90° at the three test temperatures. The samples cut at 45°, however, did not present strain localization at any of the three temperatures; the strain rate fields were very uniform both during loading and unloading. The localization morphologies of the samples cut at 0°, 22.5°, 67.5°and 90° were similar. All showed clear bands inclined from the longitudinal direction of the sample. The paths taken by the bands were essentially the same during loading and unloading, but bands were much more stable during unloading. The influence of the anisotropy on the localization phenomenon was analyzed regarding the band inclination angles from the sample axial direction. The inclination angles were measured estimated for all stra in range using the measured strain rate fields. The inclination angles measured in the samples oriented at 0°and 90° were similar and around 60°. In comparison, the inclination angles measured for the samples 22.5°and 67.5° were smaller, between 50°and 55°. The anisotropic mechanical state behaviour of the material was analyzed using Hill's quadratic anisotropic yield function. With the obtained Hill's anisotropic parametersparameters, the inclination of the bands wasere calculated for each tensile sample orientation. When confronted with experimental measurements, good qualitative results were obtained from this analysis, allowing to evaluate the relation between the angles of localization bands with samples' orientation.

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28 Aug
11:10am
Amphi 4

In-depth EBSD investigation of spatially coupled crystallographic properties in binary FeNi alloys

Thome P., Schneider M., Ersanli M., Payton E. J., Yardley V. A.

Universitätsstraße 150 GERMANY

Binary Fe-Ni alloys are well known to show a martensitic phase transformation. The formation of martensitic microstructures is governed by the crystallography of the parent and product phase. Fe-Ni alloys with Ni contents > 28 at. % Ni are reported to show a plate-like martensite (OR close to N-W), whereas alloys with < 28 at. % nickel show a lath-type morphology (Orientation Relationship (OR) close to K-S). In order to investigate this microstructural transition, custom algorithms for the MATLAB toolbox MTEX were developed and applied to extract spatially coupled OR data from extensive EBSD data sets. Visualization procedures were developed which help to identify the relationships between morphological and crystallographic features. Based on a quantitative measurement method of the OR, the microstructure was investigated in terms of different accommodation processes. The EBSD scans were performed on 10 specimens in the composition range 20-32.5 at.% Ni after the martensitic transformation was induced via DSC measurements. After the cooling procedure, detailed EBSD scans with step sizes of

100-200 nm were performed to investigate both the combinations of variants and the spatial distribution of point orientations within these variants. Lattice constants were determined by XRD for selected material states. By performing in-situ cooling experiments, the lattice constants of both austenite and martensite phase were measured at the transformation temperature. The determined lattice constants were used to calculate expected ORs by PTMT. The predictions of the PTMT were compared with the average OR and spatial deviations determined by EBSD.

Twinning in Micro and Nanoscale Pillars Size effect in Cu-Ni-Al shape memory alloy

29 Aug
11:10am
Amphi 4

Vronka M.[1], Vesely J.[2], Manak J.[1], Karlik M.[3], Heczko O.[1]

[1] *Institute of Physics of the Czech Academy of Sciences, CZECH REPUBLIC;*
[2] *Charles University, Department of Physics of Materials, Prague, CZECH REPUBLIC;*
[3] *Czech Technical University in Prague, Department of Materials, Prague, CZECH REPUBLIC*

Low twinning stress, i.e. high mobility of twinning boundaries, is a crucial condition for magnetically induced reorientation (MIR).This intensively studied effect observed in NiMnGa magnetic shape memory single crystals is promising for applications in micro-electromechanical systems for wearable healthcare and flexible electronic technologies such as sensors or actuators. However, some of the fundamental aspects of low twinning stress remain unclear, including the question how the twinning and twin boundary mobility is influenced by micro and nanoscale dimensions. In NiMnGa the twinning stress measured on bulk samples can be as low as 0.1 MPa for Type II twin boundary in 10M structure [Heczko, O., Mater. Sci. Technol. 30, 15591578 (2014)]. Such a low stress is impossible to measure on micro and nanoscale samples due to insufficient resolution of force sensors. However, relatively high mobility of twin boundary was noted also in well-known prototype of non-magnetic CuNiAl shape memory alloy [Novak, V., Sittner, P., Ignacova, S., Cernoch, T., Mater. Sci. Eng. A 438440, 755762 (2006)]. In a recent work [Vronka, M., Seiner, H., Heczko, O., Philos. Mag. 97(18), 14791497 (2017)], we have shown that particular CuNiAl single crystal having mobile twin boundaries can be considered as non-magnetic analogue to NiMnGa magnetic shape memory alloy. In CuNiAl the twinning stress is approximately ten times higher than in NiMnGa, thus it is measurable even on nanoscale samples. Here we present an observation of a remarkable size effect on the twinning stress for CuNiAl shape memory single crystals during reorientation upon compressive loading. The nano- and micro-scale pillars were in-situ compressed inside transmission and scanning electron microscopes. The pillars with the pillar base dimensions less than 200 nm do not twin at all. Larger pillars start twinning but reveal very high twinning stresses ($\sim$ 1.2 GPa). With the increasing cross-section of the pillars the twinning stress decreases heading towards to the values measured on bulk samples ($\sim$ 20 MPa). Similar size effect was previously observed on the critical stress for stress-induced martensite transformation but here we report the size effect on twin boundary motion resulting in the reorientation of the martensite.

Studying phase transformations of NiTi shape memory alloys subjected to severe plastic deformation using in-situ synchrotron experiments

28 Aug
9:00am
Amphi 4

Waitz T.[1], Kerber M.[1], Schafler E.[1], Spieckermann F.[2], Bednarcik J.[3], Fischer T.[3]

[1] *University of Vienna, Faculty of Physics, Physics of Nanostructure Materials, AUSTRIA;*
[2] *Erich Schmid Institute of Materials Science, Austrian Academy of Sciences, AUSTRIA;*
[3] *Deutsches Elektronen-Synchrotron DESY, Hamburg, GERMANY*

Bulk ultrafine-grained, nanocrystalline, and even amorphous materials can be processed using methods of severe plastic deformation (SPD). The small grain size as well as the high density of defects can significantly impact the phase stability as well as the kinetics of martensitic transformations and thus the functional properties of shape memory materials. In the present work, SPD is applied in-situ in the synchrotron using a dedicated high-pressure torsion (HPT) device. For the first time, structural changes and amorphization of NiTi induced by HPT were observed in-situ, using synchrotron experiments at the DESY (Hamburg, Germany). The HPT device allows quasi-constrained deformation of discs (2.5 mm in diameter and about 0.5 mm thick) at various pressures, rotation speeds and temperatures. At room temperature, coarse grained NiTi samples containing B19' martensite were compressed in the HPT device to build up a hydrostatic pressure of 6 GPa. Concomitant broadening of the B19' reflections

indicates large lattice strains. During torsional deformation at 6 GPa, amorphization is visible by diffusely diffracted intensity superimposing the B19' reflections already at rather low values of strain about 2. At a strain of about 30, complete disappearance of the reflections indicates almost full amorphization. HPT processed NiTi alloys were further investigated using a heating/cooling stage in the synchrotron. Upon heating, crystallization of the amorphous phase occurs by the formation of B2 nanocrystals. Heating was continued until a fully nanocrystalline structure was obtained. Upon cooling, in the nanograins the B2 to B19' martensitic phase transformation was observed to occur via the R-phase. The phase fractions of B2, R-phase and B19 were analysed with Rietveld refinement methods as a function of temperature.

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29 Aug
12:10pm
Amphi 4

Low temperature {110} nanotwins in Ni-Mn-Ga and Ni-Mn-Ga-Fe 10M martensites

Straka L.[1], Drahokoupil J.[1], Veřtát P.[1], Zelený M.[2,3] , Kopeček J.[1], Sozinov A.[4], Heczko O.[1,2]

[1]*Institute of Physics, Czech Academy of Sciences, Prague, CZECH REPUBLIC;*
[2]*Faculty of Mathematics and Physics, Charles University in Prague, Prague, CZECH REPUBLIC;*
[3]*Institute of Materials Science and Engineering, NETME Centre, Faculty of Mechanical Engineering, Brno University of Technology, Brno, CZECH REPUBLIC;*
[4]*Lappeenranta University of Technology, Material Physics Laboratory, FINLAND*

The magnetic shape memory alloys exhibit interesting magneto-mechanical effects such as giant magnetic-field induced strain up to 12%. The effects originate from the coupling between ferromagnetic domain structure and ferroelastic martensite. The martensite structure and twin microstructure are consequently defining most of the the material functional properties. The structure of alloys close to Ni_2MnGa composition has been studied very intensively during recent decades but there are still some controversies, particularly about the nature of the lattice modulation.
Here we report on our finding of low temperature {110} nanotwins in a five-layered modulated martensite phase of Ni-Mn-Ga and Ni-Mn-Ga-Fe. Evolution of the structure with decreasing temperature was studied by X-ray diffraction using single crystals exhibiting magnetic shape memory effect. The coincidence of (400) and (040) lines upon cooling indicated {110} nanotwinning. Scanning electron microscopy confirmed gradual refinement of twins with decreasing temperature both in Ni-Mn-Ga as well as in Ni-Mn-Ga-Fe alloy. Using adaptive diffraction condition we determined that the nanotwin size was less than 17 nm. Detailed study of satellites related to lattice modulation and nanotwinning indicated that the nanotwin size was even smaller, around 3.5 nm. We identified the particular region in phase diagram where the nanotwinning occurs. The extrapolation of our results suggests that the 110 nanotwinning may also appear in stoichiometric alloy Ni_2MnGa. This possibility has not been considered before.
The finding can have a strong impact on any study attempting to determine or refine the structure of Ni-Mn-Ga compounds, since in the case of nanotwinning the diffracted pattern is distorted and false crystal symmetries can appear. For any structural study on Ni-Mn-Ga it is critically important to recognize, whether the nanotwinning occurs or not for the particular alloy under investigation.

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28 Aug
2:00pm
Poster
Session B

Influence of the neutron irradiation on NiTi-based alloys with different crystalline structure

Belyaev S.[1,2], Resnina N.[1], Konopleva R.[2], Chekanov V.[2], Nakin A.[2], Shelykov A.[3]

[1]*Saint Petersburg State University, RUSSIAN FEDERATION;*
[2]*Nuclear Physics Institute NRC "Kurchatov Institute", RUSSIAN FEDERATION;*
[3]*National Research Nuclear University MEPhI (Moscow Engineering Physics Institute), RUSSIAN FEDERATION*

The aim of the present work was to study the influence of the neutron irradiation on the structure and martensitic transformation in the NiTi-based alloys. The $Ti_{40.7}Hf_{9.5}Ni_{44.8}Cu_5$ and $Ti_{50}Ni_{25}Cu_{25}$ melt spun ribbons with different structure (amorphous, amorphous-crystalline and fully crystalline) were subjected to neutron irradiation by fast neutrons (E > 0, 5 eV) at a temperature of 60 °C for different fluence up to 4·1019 neutron/cm^2. It was found that the irradiation of the samples in fully amorphous state influenced the crystallization behaviour of the samples during further heating. It was shown that an increase in neutron fluence hardly changed the crystallization temperatures in $Ti_{40.7}Hf_{9.5}Ni_{44.8}Cu_5$ alloy and decreased these temperatures in $Ti_{50}Ni_{25}Cu_{25}$ alloy. It was found that an increase a neutron fluence up to 1·1019 neutron/cm^2 resulted in a decrease in energy that released during the crystallization despite the alloy composition. It was concluded that a decrease in crystallization energy was due to the structure relaxation that occurred in the amorphous alloy during irradiation. It was found that an irradiation of the $Ti_{50}Ni_{25}Cu_{25}$ alloy with a fluence that was more than 1·1019 neutron/cm^2 led to sharp increase in the crystallization temperature and energy and it was assumed that this phenomenon was due to a disordering the crystalline clusters that existed in amorphous alloy. It was shown that a neutron irradiation of the amorphous-crystalline as well as fully crystalline alloys led to a decrease in martensitic transformation temperature. However, a decrease in transformation temperatures on an increase in neutron fluence in the alloys with ultra-fine grain structure was less than in coarse-grained materials. It was assumed that it was caused by a large density of the grain boundaries and existence of the amorphous-crystalline interface that acted as point defects sink that prevented from the irradiation damage of the crystalline grains. It was found that an irradiation of the $Ti_{50}Ni_{25}Cu_{25}$ alloy with a fluence that was more than 1·1019 neutron/cm^2 led to a suppression of the martensitic transformation.

On the structure and properties of $Fe_{43.5}Mn_{34}Al_{15-x}Ni_{7.5+x}$ shape memory alloys

28 Aug
2:00pm
Poster
Session B

Bujoreanu L.-G.[1], Popa M.[1], Gurau C.[2], Gurau G.[2], Pricop B.[1], Comaneci R. I.[1], Vollmer M.[3], Krooss P.[3], Niendorf T.[3]

[1] *"Gheorghe Asachi" Technical University of Iasi, ROMANIA;*
[2] *"Dunarea de Jos" University of Galati, ROMANIA;*
[3] *Universität Kassel, GERMANY*

The $Fe_{43.5}Mn_{34}Al_{15}Ni_{7.5}$ alloy became an eligible candidate for high temperature applications, characterized by a very wide thermal range (∼40 °C) of superelastic behaviour with a small temperature dependence of the stress plateau (0.53 MPa/ 0 °C), following 24 hours ageing at 200°C. The reversible stress induced martensitic transformation that governs superelastic behaviour in this alloy system is an isothermal α - body centred cubic (bcc) ↔ γ' - face centred cubic (fcc) transition. It is considered that the thermoelastic character is induced by β-NiAl (B2) coherent ordered precipitates, while superelasticity is enhanced by the increase of relative grain size expressed by the ratio between mean grain size, d and plate thickness or wire diameter, D. In spite of their isomorphic unit cells, Ni and Al atoms are prone to chemically combine in such a way that intermetallic compounds rather than a solid solution are formed. Thus, besides NiAl other ordered intermetallic compounds could form, such as Ni_3Al, Ni_2Al_3, $NiAl_3$ and Ni_2Al_9, which have the potential to contribute to the reinforcement of α-bcc parent phase lattice. Based on this assumption, the effects of Al substitution with Ni were investigated at five $Fe_{43.5}Mn_{34}Al_{15-x}Ni_{7.5+x}$ chemical compositions, where x = 0; ± 1.5 and ± 3 at. %. After hot rolling, annealing and cold rolling, the specimens were heat treated such as to cause the phase transitions necessary to enhance the superelastic behaviour: (i) abnormal grain growth and (ii) the precipitation of coherent Ni-Al intermetallic compounds. Abnormal grain growth was induced by heating-cooling cycles performed with constant temperature variation rate, around solvus temperature (determined by differential scanning calorimetry). The precipitation of coherent Ni-Al intermetallic compounds was achieved by solution treatment followed by low-temperature ageing. Microstructural analysis was performed by optical and scanning electron microscopy coupled with energy dispersive spectroscopy. Micro-hardness measurements were performed across specimen thickness, aiming to observe parent phase strengthening caused by low temperature ageing. Tensile and rectangular specimens were cut by electro-discharge machinig for tensile loading-unloading tests and for isothermal dynamic mechanical analysis, respectively. The effects of Al substitution by Ni were finally discussed by corroborating the results of microstructural analysis with those of mechanical tests.

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28 Aug
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Poster
Session B

Change in enthalpy and energy of martensitic transformation in ultrafine-grained and nanocrystalline states of $Ti_{49.1}Ni_{50.9}$ alloy

Churakova A., Gunderov D.

Ufa State Aviation Technical University, Ufa, RUSSIAN FEDERATION
Institute of Molecule and Crystal Physics, Ufa Federal Research Centre RAS, Ufa, RUSSIAN FEDERATION
Saint Petersburg State University, Saint Petersburg, RUSSIAN FEDERATION

The TiNi alloys belong to the class of functional materials with shape memory effects caused by thermoelastic martensitic transformations. The object of investigation was chosen of $Ti_{49.15}Ni_{50.85}$ (in at.%) alloy of the stoichiometric composition. Calorimetric studies were carried out on samples in various structural states in the temperature range from -120 ˚C to 100 ˚C. On the DSC curves in the CG state, peaks corresponding to direct and inverse martensitic transformation are observed. As a result of the TC, the temperature of the martensitic transformations shifts to higher temperatures with an increase in the number of thermocycles from 0 to 100. The most significant change is observed for the temperatures of the reverse martensitic transformation, for n = 100 cycles the temperatures become positive. At the same time, the transformation temperatures R → B2 remain relatively stable with increasing number of cycles. Such a change of transitions in alloys with a higher Ni concentration with respect to stoichiometry was also observed earlier. For the UFG state, only B19' → B2 and B2 → B19' transformations are recorded by the DSC method. The characteristic transition temperatures do not change monotonically depending on the number of cycles, but with the maximum number of thermal changes (n = 100), their values become close to the values before thermal cycling. In the state after the HPT, no peaks are observed on the calorimetric curves, which agrees with the structural data and the amorphous character of the material. The structure formed by annealing of the NC structure undergoes a martensitic transformation; on the DSC curves, an endothermic peak is recorded upon heating, corresponding, probably, either to the inverse martensitic transformation B19 → B2, or to the R transformation. After thermocycling with the maximum number of heat changes, all characteristic temperatures are shifted to lower temperatures, and peak temperatures are also lowered. On the basis of this, it can be concluded that in the NC state, like a coarse-grained material, some accumulation of defects occurs. An analysis of the presented results shows that as a result of thermocycling with a maximum number of cycles in comparison with the initial state, an increase in the direct conversion energy is observed both in short-circuit and in the UFG alloy with a simultaneous decrease in the energy of the inverse, which also affects the entropy change.

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28 Aug
2:00pm
Poster
Session B

Phase transformation and degradation of superelastic NiTi during uniaxial and multiaxial loading

Hsu W.-N.[1,2], Polatidis E.[1], Smid M. [1], Van Petegem S.[1], Van Swygenhoven H.[1,2]

[1]*Photons for Engineering and Manufacturing Group, Swiss light source, Paul Scherrer Institute, SWITZERLAND;*
[2]*Neutrons and X-rays for Mechanics of Materials, IMX, Ecole Polytechnique Federale de Lausanne, SWITZERLAND.*

Until now most of the knowledge of the martensitic transformation and the degradation mechanisms in NiTi is based on uniaxial loading experiments. The effect of multiaxial loading or changes in load path on the behavior of the material remains unclear. In this study, the deformation behavior and degradation of commercial superelastic NiTi is investigated with in situ synchrotron X-ray diffraction at the MS beamline of the Swiss Light Source and complemented by EBSD and HRDIC measurements. The initial microstructure consists of large austenitic grains subdivided in bands of nanoscaled subgrains [Acta Mat 144(2018)874]. Cruciform shaped samples are subjected to various loading conditions including uniaxial tension, equibiaxial tension and load paths that involve a sudden change in loading direction. The transformation behavior and martensitic variant selection is discussed in terms of the initial microstructure and the different loading paths. The degradation of the material after multiple load-unload cycles is found to depend strongly on the loading path.
This research is performed within the ERC Advanced Grant MULTIAX (339245).

Strain paths and superelastic anisotropy in a textured NiTi under isothermal uniaxial and biaxial loading using DIC analysis

28 Aug
2:00pm
Poster
Session B

Meraghni F.[1], Bourgeois N.[2], Echchorfi R.[1], Peltier L.[1], Chemisky Y.[1], Patoor E.[1]

[1]*LEM3, UMR 7239, Arts et Métiers ParisTech FRANCE;*
[2]*University of Lorraine, FRANCE;*
[3]*Georgia Tech Lorraine, FRANCE*

This work presents the experimental results obtained from the thermomechanical characterization of a textured superelastic NiTi SMA. Uniaxial tension, uniaxial compression and tension-tension, tension-compression biaxial tests have been performed at different temperatures between 30 and 70°C. Vital information about the strain fields for each test is measured using Digital Image Correlation. This method features a characterization technique which provides high fidelity data at local and global scales. It accurately characterizes the difference in the thermomechanical behavior between the rolling and the transverse direction under a wide range of load states. The carried-out loading configurations confirm the effect of temperature on martensitic transformation and the anisotropic superelastic behavior of the NiTi. Indeed, the martensitic transformation exhibits a non-uniform distribution in the gauge zone of the specimen and brings about heterogeneous strain fields. The performed tests demonstrate a significant anisotropy in the behavior due to material processing and specifically cold-working, resulting in a higher forward transformation stress. For the specimens tested uniaxially in the rolling direction, the stress-strain curve exhibits a pseudo-steady plateau as transformation progresses. However, the forward stress still continues to increase during the transformation for the specimen loaded inthe transverse direction. The experimental database on the behavior of the studied NiTi SMA was enriched with results on the compressive behavior, and also in tension-tension and tension-compression at 50°C temperature. Only the digital image correlation allows exploiting these tests configuration: it gives access to the evolution of the heterogeneity of the local and global fields, and thus to the strain paths at different points of the specimens, in connection with the prescribed applied loading. Measuring heterogeneity of the strain fields and the related variety of the strain path induced by the specificities of a textured NiTi SMA (anisotropy, tension-compression asymmetry, etc) would be of high interest to describe the actual thermomechanical behavior of such SMAs and for FE simulation of SMA structures.

[1] Meraghni, F., Chemisky, Y., Piotrowski, B., Echchorfi, R., Bourgeois, N., Patoor, E.: Parameter identification of a thermodynamic model for superelastic shape memory alloys using analytical calculation of the sensitivity matrix. Eur. J. Mech. - A/Solids. 45, 226237 (2014).
[2] Chemisky, Y., Meraghni, F., Bourgeois, N., Cornell, S., Echchorfi, R., Patoor, E.: Analysis of the deformation paths and thermomechanical parameter identification of a shape memory alloy using digital image correlation over heterogeneous tests. Int. J. Mech. Sci. 9697, 1324 (2015)

Ni-Ti in-situ stress induced martensitic transformation and strain mapping in a TEM

28 Aug
2:00pm
Poster
Session B

Pourbabak S., Orekhov A., Schryvers D.

University of Antwerp, EMAT group, BELGIUM

$Ni_{50.6}Ti_{49.4}$ bulk is annealed at 1273K for 2 hours followed by room temperature water quenching yielding a precipitate-free sample. An electropolished Transmission Electron Microscope (TEM) specimen is prepared from the obtained sample and studied by TEM. A region with the following properties is selected: a) dislocation-free, b) homogeneous thickness, c) from grain interior, and d) with a low-index zone perpendicular to the surface of the sample. A dog-bone shape specimen of the size of 0.7nmx1.7nm from the selected region is cut by Focused Ion Beam (FIB) and mounted on a Micro-Electro Mechanical System (MEMS) push-to-pull (PTP) device. The obtained specimen is then investigated in an FEI Tecnai G2 equipped with beam precession-enabled nanoscale strain mapping. The specimen is pulled with different strengths and nucleation and propagation of variants of martensite are investigated by Bright Field TEM (BF-TEM). Moreover, strain mapping of the specimen before, during and after pulling is acquired. The residual martensite after release is linked to the shift of the obtained stress-strain curve.

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2:00pm
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Session B

TEM observation of austenite twins in biomedical NiTi wire after superelastic cycling

Racek J., Duchoň J., Vronka M.

Institute of Physics of the Czech Academy of Science, CZECH REPUBLIC

Despite improving of fatigue life by surface finish or microstructure treatment the crystallographic defects induced by phase transformation in NiTi represent the crucial issue for service life and still lack understanding of their origin. TEM observation of biomedical NiTi wire after superelastic cycling allowed discovering of the unique defect band consisting of austenite twins in heavily deformed grains. Twins are accumulated by non-transforming pathway under conditions exceeding the critical stress during periodical propagation of diffused martensite band front at inclined angle of 55° to the wire axis. Austenite {1 1 4} twins analyzed by nano-electron beam diffraction forming the persistent defect band represent the key issue for functional fatigue of superelastic NiTi.

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28 Aug
2:00pm
Poster
Session B

Effect of B2-austenite Grain Size and Isothermal Aging on Microstructure of Ti_3Ni_4 Precipitates and Transformation Sequence in Titanium Nickelide

Ryklina E.[1], Polyakova K.[1], Tabachkova N.[1], Resnina N.[2], Prokoshkin S.[1]

[1]National University of Science and Technology "MISIS", RUSSIAN FEDERATION;
[2]Saint Petersburg State University, RUSSIAN FEDERATION

The particle size and morphology, transformation behavior under isothermal aging were studied in the Ti-50.7 t.%Ni shape memory alloy with various sizes of B2-austenite grain. The 0.3 mm thick band with the accumulated strain of $\varepsilon = 44$ % (e = 0.6) was obtained by the cold rolling. The samples of this band were then solution-treated at 600-800° for 0.3-1 hr and then quenched in water to obtain the structure with grain size (GS) from 5 to 15 μm. Then the samples were subjected to isothermal annealing at 430°C, for 1, 3 and 10 hr. The SEM observations were obtained using a JSM-6460LV; the microstructure was studied using a JEOL 2100 TEM. Energy dispersive spectroscopy (EDS) was carried out using the EDS stage of a JEOL 2100. The studies were carried out using samples with a homogenized structure obtained as a result of CR with accumulated strain of 0.6 and subsequent RA at 600° for 1 h and 800° for 1 h. Characteristic temperatures were measured using a "Mettler Toledo" DSC. The results of microstructure study prove that the GS strongly affects Ti_3Ni_4 particle size and morphology, as well as degree of micro-scale microstructure heterogeneity, transformation kinetics and transformation sequences. This influence is nonunique and depends on duration of isothermal annealing. After 1 hr aging, the particle thickness and length keep the nanometer range in the whole GS range of 5-15 μm. The increase of aging time up to 3 and 10 hr leads to localization of nano-sized precipitates in the grain-boundary region; the width of these regions decreases with the GS increase. In a fine-grained structure (GS = 5 m) after aging for 1 h, one B2→R peak with a tendency for bifurcation is determined, while after aging for 10 h, two distinct peaks, of abnormal two-stage B2→R transformation are recorded; B19' transformation is suppressed. The increase in the GS up to 15 m and aging duration up to 10 hr is accompanied by multiplication of martensitic transformations up to four stages. The study explains the influence of GS on the precipitated particle size and morphology, transformation kinetics and sequences.
The present work was carried out with the financial support of the Ministry of Education and Science of the Russian Federation in the frameworks of State Task No.11.1495.20174.6.

Session 3: Computational Material Design

Invited Lecture: Wednesday August 29	
8:30 Amphi 1	**R. Umetsu**, M. Tsujikawa, X. Xu , W. Ito , M. Shirai , R. Kainuma "Change of the electronic state for Ni-Mn-Sb alloys in martensitic phase transformation"
Oral presentations: Wednesday August 29	
9:00 Amphi 1	**S. E. Kulkova**, A. V. Bakulin "Oxygen diffusion and self-diffusion mechanisms in TiNi and TiAl alloys"
9:20 Amphi 1	**M. Zelený**, M. Heczko, L. Straka, A. Sozinov, O. Heczko "First-principle study of martensitic transformations in Ni-Mn-Ga alloy"
9:40 Amphi 1	I. Karaman, A. Solomou, G. Zhao, S. Boluki, J. K. Joy, X. Qian, R. Arróyave, **D. C. Lagoudas** "Multi-Objective Bayesian Materials Design: Application on the Design of Precipitation Hardened Ni-Ti SMAs"
10:00 Amphi 1	**F. Motazedian** "The possibility of utilizing NiTi phase transformation in transferring large elastic strain to the deposited thin film"

Poster Session A: Tuesday August 28
A. V. Bakulin, S. E. Kulkova , Q. M. Hu , R. Yang " Influence of impurities on oxygen sorption properties of TiNi and TiAl alloys and their oxidation resistance"
V. Buchelnikov, O. N. Miroshkina, A. T. Zayak, T. Roy "About Magnetic and Structural States of Ni_2CrZ (Z=Ga, Ge, Si, Sn) Heusler alloys"
O. Pavlukhina, V. Buchelnikov, V. Sokolovskiy, M. Zagrebin "Theoretical investigation of magnetic and structural properties of $FeRh_{1-x}Pd_x$ (x=0.5-1) alloys"
E. Smolyakova, **M. Zagrebin** "Effect of Cr-doping on Properties of Ni-Mn-(Ga, Sb) Heusler alloys"

Contents

Change of the electronic state for Ni-Mn-Sb alloys in martensitic phase transformation

29 Aug
8:30am
Amphi 1

Umetsu R.[1], Tsujikawa M.[2], Xu X.[3], Ito W.[4], Shirai M.[2], Kainuma R.[3]

[1]*Institute for Materials Research, Tohoku University, JAPAN;*
[2]*Research Institute of Electrical Communication, Tohoku University, JAPAN;*
[3]*Department of Materials Science, Graduate School of Engineering, Tohoku University, JAPAN;*
[4]*Sendai National College of Technology, JAPAN;*

Since a unique martensitic phase transformation in off-stoichiometric Heusler alloys, Ni-Mn-Z (Z = In, Sn, and Sb), were first reported by Sutou et al., they have attracted widespread interest as high-performance multiferroic materials. These alloys show many interesting physical properties, such as metamagnetic shape memory effect, inverse magnetocaloric effect and giant magnetoresistance effect. These interesting properties are related to drastic changes in magnetic properties between the martensite (M) phase with weak magnetism and the ferromagnetic parent (P) phase. Such large changes of the various physical properties will be associated with the change in the electronic state during the martensitic phase transformation. In the present study, low-temperature specific heat experiments were performed to investigate the electronic specific heat coefficient (γ), which will provide information on the density of states (DOS) around the Fermi level, and the Debye temperature (θD). Furthermore, firstprinciples density-functional calculations were carried out with using the VIENNA ab initio simulation package. The spin-polarized generalized gradient approximation was adopted for the exchange and correlation energies, and the atomic core potential was described by the projector augmented wave method. The specimens of polycrystalline $Ni_{50}Mn_{50-x}Sb_x$ ($0 \leq x \leq 25$) alloys were fabricated by induction melting in an Ar atmosphere. The obtained ingots were annealed at 1173 K for 1 day and quenched in water. The magnetic measurements were carried out by a superconducting quantum interference device magnetometer. Specific heat measurements were carried out with the relaxation method using a physical properties measurement system in temperature regions below 20 K. In the composition region of $x \leq 12$, where the ground state is M phase, θD decreases linearly and γ increases with increasing Sb. For $x \geq 18$, where the ground state is the ferromagnetic P phase, γ increases with decreasing Sb. Extrapolations of the composition dependence of γ in both the M and P phases suggest that the γ discontinuously changes during the martensitic phase transformation. The value of γ is slightly larger in the P phase, in good agreement with the calculated DOS. Here, DOS in the M phase was calculated with the distortion of c/a = 1.31 for x = 12.5. Such a coincidence has also been confirmed in $Ni_{50}Mn_{50-x}In_x$ ($0 \leq x \leq 25$) alloys.

Multi-Objective Bayesian Materials Design: Application on the Design of Precipitation Hardened Ni-Ti SMAs

29 Aug
9:40am
Amphi 1

Karaman I., Solomou A., Zhao G., Boluki S., Joy J. K., Qian X., Arróyave R., Lagoudas D. C.

Texas A&M University, College Station, UNITED STATES

Recent advances in manufacturing and computational resources have led to sophisticated designs of new devices suitable for aerospace, automotive and biomedical applications among others. The functionality of such devices is based on the exploitation of the capabilities of multifunctional materials, and therefore, their development has motivated the search for novel materials with optimal behavior. Materials discovery is significantly time- and resource-intensive and this calls for the acceleration of the materials discovery process through the integration of experiments and simulations within an informatics framework. In this study, a framework for the multi-objective discovery of materials based on Bayesian approaches is developed. The capabilities of the framework are demonstrated, on a test case related to the precipitation strengthened NiTi shape memory alloys (SMAs) in order to demonstrate how the required number of computational experiments can be minimized in order to identify SMA materials with a set of desired properties. To this end a Bayesian Optimal Experimental Design (BOED) process that operates in a closed loop is developed that uses a Gaussian Process Regression (GPR) model to emulate the response (and its uncertainty) of the experiment/simulation data. The Expected Hyper-Volume Improvement (EHVI) acquisition function is used to carry out the optimal sequential exploration of the design space. The EHVI scalar metric provides a measure of the utility of querying the design space at different locations, irrespective of the number of objectives in the optimization/design task. A validated and thermodynamically consistent SMA micromechanical model which predicts the materials response as a function of microstructure is used to perform the computational experiments. Finally the presented results demonstrate how a protocol for computational experiments, based on the EHVI metric, provides an optimal policy for the exploration of the NiTi SMA design space. Moreover, the present work shows that when the design task is subject to resource constraints, the most efficient strategy is to allocate as many resources as possible to the optimal sequential acquisition of information.

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29 Aug
9:00am
Amphi 1

Oxygen diffusion and self-diffusion mechanisms in TiNi and TiAl alloys

Kulkova S. E., Bakulin A. V.

Institute of Strength Physics and Materials Science, SBRAS; TSU, RUSSIAN FEDERATION

It is known that mechanical properties of Ti-based alloys are related directly to the atomic diffusion. Interrelation of point defect structures and atomic mobility with engineering alloy functional properties is very important for technological applications. Since the biocompatibility of TiNi is resulted from the formation of a thin passive layer of TiO_2 at the surface the understanding of the oxygen diffusion mechanism in the alloy is also important for explanation of oxidation behavior and formation of stable alloy-oxide interfaces. However, these phenomena are far from complete understanding even in binary alloys. In present work we discuss the peculiarities of self-diffusion and oxygen diffusion mechanisms in TiNi and TiAl alloys. The calculations of atomic and electronic structure of alloys were performed by the projector augmented wave method. It was revealed that among considered self-diffusion mechanisms such as the nearest-neighbor (NN) jumps, the next-nearest-neighbor (NNN) jumps, four-jump cycles (4JC) and six-jump cycles (6JC), the 6JC [001] bent mechanism of Ni-vacancy diffusion is found as preferential one in case of TiNi. The migration barrier is equaled to 0.82 eV that is in good agreement with experiment. We demonstrate that NNN jumps of Ni-vacancy become important with temperature increase. In case of TiAl both atoms self-diffusion take place by vacancy jumps in the nearest-neighbor sites along own sublattices. Our calculations demonstrate that Al is a slower diffuser than Ti in consistent with experimental data. At the $TiNi/TiO_2$ interface the jump of Ni atom from interface into subinterface layer needs of $\sim$ 1 eV less energy than opposite jump. This suggests that Ni diffuses toward bulk rather than into oxide layers. It was shown that in TiNi the migration barrier for O diffusion between the most preferential Ti-rich nearest octahedral O1-sites (1.14 eV) is by $\sim$ 0.6 eV lower than that between next nearest O1-sites. Both energy barriers are significantly lower than that between Ti-rich O1-sites in TiAl (3.02 eV) along (001) plane. In TiAl oxygen diffusion is energetically favorable from O1-site to tetrahedral T-site (1.15 eV) rather to Al-rich O2 (1.64 eV) along [001] direction. The preferential diffusion path of oxygen in this direction passes along tetrahedral interstices through the aluminum layer and through an octahedral interstice in the titanium layer. In general, the migration barriers along preferential paths are comparable in both alloys.

The work is partly supported by the Russian Foundation for Basic Research under grant No. 18-32-00264$_a$ and 18-03-00064$_a$.

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29 Aug
10:00am
Amphi 1

The possibility of utilizing NiTi phase transformation in transferring large elastic strain to the deposited thin film

Motazedian F.

School of Engineering, The University of Western Australia, AUSTRALIA

This research, investigates the possibility and mechanisms of transferring large elastic strain from a phase transforming substrate (NiTi) to a deposited thin film. Thin films, as a two-dimensional nano-scaled material, have an intrinsic ability to exhibit large elastic strains close to their theoretical strain limits. However, the attempt to create a practically useful thin film on substrate composite form that is able to sustain this large strain ability has limited success. This is due to the inability of conventional substrates to produce large and uniform elastic strains to match the inherent strain ability of the thin films. NiTi shape memory alloys are a unique class of materials capable of generating large recoverable strains of up to 7% via austenite to martensitic phase transformation and reorientation of self-accommodated martensite phase. Thus, as a substrate, NiTi is expected to be able to support large elastic strains of thin films. Moreover, a recent breakthrough was achieved in our research team, which induced large elastic strains (up to 6.5%) in a bulk composite material consisting of thousands of niobium (Nb) nano-wires embedded in a NiTi matrix. The innovative principle behind this breakthrough discovery is the lattice strain matching at the atomic level between martensitic transforming matrix and the ultra large elastic strain of the Nb nanowires embedded within. Therefore, taking advantage of the thermomechanical behaviour of NiTi, and the proven ability of niobium in nano scale, a NiTi-Nb thin film composite system designed to investigate the strain transfer ability and to quantify the induced elastic strains in the niobium thin film from the NiTi substrate. This study reveals that the XRD out of plane d-spacing of the deposited thin film of Nb (110) on NiTi substrate, contracted more than 1.8% strain after the applied 5.3% residual axial tensile load to the NiTi shape memory substrate through re-orientation of martensitic phase. This suggests that the actual induced strain in the loading direction to Nb thin film is well above previous reports on elastically strained thin films. This discovery shows "elastic strain engineering" potential of NiTi substrates coupled with functional thin film and further enhancement of the thin film properties by inducing large elastic strains from a phase transforming substrate.

First-principle study of martensitic transformations in Ni-Mn-Ga alloy

29 Aug
9:20am
Amphi 1

Zelený M.[1,2], Heczko M.[2], Straka L.[1,3], Sozinov A.[4], Heczko O.[1,3]

[1] *Charles University, Faculty of Mathematics and Physics, CZECH REPUBLIC;*
[2] *Faculty of Mechanical Engineering, University of Technology, Brno, CZECH REPUBLIC;*
[3] *Institute of Physics, Czech Academy of Sciences, Prague, CZECH REPUBLIC;*
[4] *Material Physics Laboratory, Lappeenranta University of Technology, Savonlinna, FINLAND*

The martensitic transformation to low-symmetry structures in Ni-Mn-Ga magnetic shape memory alloy is associated with characteristic features in the electronic DOS [1]. Using first-principles calculations combined with the generalized solid state nudged elastic band method [2] we determined the minimum energy path leading to the different modulated phases of martensite (4O, 10M, and 14M) and to non-modulated (NM) phase [3]. In stoichiometric Ni_2MnGa alloy there is no energy barrier on the path to the 10M phase. Geometry of the lattice in the initial part of the path confirms that this transformation is driven by a softening of the TA_2 $[\xi\xi0]$ phonon branch [1] corresponding to the shift of (110) planes. In later part of the path individual NM nanotwins are stabilized by band Jahn-Teller effect. Transformation paths to other structures including NM martensite exhibit more or less significant barriers in the beginning, hindering such a transformation from austenite although these structures exhibits lower total energy. This finding corresponds to experiment and demonstrates that the kinetics of the transformation is decisive for the selection of the particular low-symmetry structure of martensite. On the other hand, in off-stoichiometric alloys the barrier-less transformation was found between austenite and NM martensite corresponding to tetragonal distortion of the lattice due to Jahn-Teller effect.

[1] Zayak, A.T. et al. Phys. Rev. B 72, 054113 (2005).
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[3] Niemann, R. & Fhler, S.J. Alloy. Compd. 703, 280 (2017).

Influence of impurities on oxygen sorption properties of TiNi and TiAl alloys and their oxidation resistance

28Aug
2:00pm
Poster
Session A

Bakulin A. V.[1], Kulkova S. E.[1], Hu Q. M.[2],Yang R.[2]

[1] *Institute of Strength Physics and Materials Science, SBRAS; TSU, RUSSIAN FEDERATION;*
[2] *Shenyang National Laboratory for Materials Science, IMR, CAS, CHINA*

The biocompatibility of TiNi shape memory alloys for medical applications is resulted from the formation of a thin passive layer of TiO_2 at the surface. At the same time segregation of the impurities can influence on the adsorption of oxygen on the TiNi surface and the stability and strength of the $TiNi/TiO_2$ interface. The similar problem exists in case of TiAl, in which both components of the alloy have affinity to oxygen but the growth of TiO_2 and Al_2O_3 mixed layers leads to a low oxidation resistance at high temperatures. Thus, the improving of the oxidation resistance is the important problem for both alloys. In present work the comparative study of oxygen adsorption mechanisms on the doped TiNi and TiAl surfaces is performed. The electronic structure of the alloy surfaces with and without impurities are calculated by using the projector augmented wave method based on the density functional theory. Impurities of 3d-5d transition metals and Al, Si are considered on both Ti and Ni(Al) sublattices. We demonstrate that almost all 3d metal impurities on Ni sublattice lead to increasing O adsorption energy on TiNi (110) surface. For the 4d and 5d metals, only the impurities up to the middle rows of the periodic table increase the O adsorption energy. All considered metals on Ti-sublattice decrease the O adsorption energy on doped TiNi surface. The oxidation energies of the doped rutile TiO_2 and corundum Al_2O_3 with the alloying atoms on the Ti or Al sites were calculated as well. It was revealed that most alloying elements increase the oxidation energies of TiO_2 and Al_2O_3 except for several early transition metal elements (e.g., Zr and Hf for TiO_2 and Sc for Al_2O_3). We show that the oxidation energies increase with the number of d-electrons for the alloying elements in the same row of the periodic table. At the same time the oxidation energies of the Al_2O_3 increase for the isoelectronic elements from 3d to 5d period of the periodic table whereas they are almost the same in case of TiO_2. Furthermore, it is shown that all the 3d-elements lower the formation energy of the oxygen vacancy in TiO_2 but the elements with number of d electrons from 2 to 4 in the 4d and 5d periods, especially Hf and Ta, increase the formation energy of oxygen vacancy that decreases the oxygen diffusivity. Besides, almost the same elements decrease the relative stability of Al_2O_3 to TiO_2. This suggests that mentioned above elements are beneficial to the TiAl oxidation resistance as well.

The work is partly supported by the Russian Foundation for Basic Research under grant N 18-03-00064$_a$.

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28 Aug
2:00pm
Poster
Session A

About Magnetic and Structural States of Ni_2CrZ (Z=Ga, Ge, Si, Sn) Heusler alloys

Buchelnikov V.[1], Miroshkina O. N.[1], Zayak A. T.[2], Roy T.[1,3]

[1] *Chelyabinsk State University, RUSSIAN FEDERATION;*
[2] *Bowling Green State University, Bowling Green, USA;*
[3] *Raja Ramanna Centre for Advanced Technology, INDIA*

In this work, magnetic and structural states of Ni_2CrZ (Z = Ga, Ge, Si, Sn) Heusler alloys are studied. Both ferromagnetic and antiferromagnetic configurations of these alloys were considered. Cr atoms having the antiparallel direction of the magnetic moment set antiferromagnetic configuration. It was demonstrated that the total energy of the antiferromagnetic states of considered alloys is lower than the ferromagnetic ones in case of $L2_1$ cubic structure. However, both magnetic configurations of considered Ni_2Cr-based alloys are unstable in the cubic phase. In fact, $L1_0$ phase with antiferromagnetic configuration is realized with ratios $c/a \simeq 1.2$ for Ni_2CrGa, Ni_2CrGe, and Ni_2CrSi and $c/a \simeq 1.1$ for Ni_2CrSn [1, 2]. In addition, the analysis of the phonon dispersion curves of Ni_2CrZ (Z = Ga, Ge, Si, Sn) alloys for $L2_1$ cubic structure was carried out. It was found that the frequencies of the one or several acoustic modes are negative. Thus, the phonon calculations confirm the instability of cubic phases. Vienna ab initio simulation package (VASP) and PHONON software were used as tools for the pursuance of the described above investigation [3-6]. With the help of VASP, the minimizing the total energy of the crystals with respect to the electronic part, lattice constants, atomic positions, the possibility of the tetragonal distortions, and calculations of the Hellmann-Feynman forces were carried out. Then using obtained results the phonon dispersion curves were calculated with the help of PHONON. Calculations were conducted for 40-atoms supercells, which were created by merging 5 primitive cells along the [110]c direction of the cubic Heusler structure, which is the [010]t direction of the tetragonal cell.

This work is supported by Young Scientist Support Fund of Chelyabinsk State University and RSF-Russian Science Foundation No. 17-72-20022\17.

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[2] T. Roy, et al., J. Magn. Magn. Mater., 401, 929 (2016).
[3] G. Kresse, et al., Phys. Rev. B, 54, 11169 (1996).
[4] G. Kresse, et al., Phys. Rev. B, 59, 1758 (1999).
[5] K. Parlinski, et al., Phys. Rev. Lett., 78, 4063 (1997).
[6] K. Parlinski, Software Phonon, Cracow (2013).

Theoretical investigation of magnetic and structural properties of $FeRh_{1-x}Pd_x$ (x=0.5-1) alloys

28 Aug
2:00pm
Poster
Session A

Pavlukhina O., Buchelnikov V., Sokolovskiy V., Zagrebin M.

Chelyabinsk State University, RUSSIAN FEDERATION

Fe-Rh-based alloys have attracted a lot of attention because of their possible application in magnetic cooling, thermally assisted magnetic recording and spintronic devices. Fe-Rh alloys exhibit a metamagnetic phase transition (AFM-FM). The metamagnetic phase transition in Fe-Rh succeeds also the large change in magnetization, which is responsible for a giant magnetocaloric effect upon variation of a magnetic field. It well known, that the magnetic order in FeRh compounds depends strongly on the concentration. In this work, we present theoretical investigations of the structural and magnetic properties $FeRh_{1-x}Pd_x$ (x = 0.5, 0.625, 0.75, 0,875, 1) alloys. The structural and magnetic properties of Pd-doped Fe-Rh alloys are investigated by using the density functional theory calculations as implemented in the VASP package. The ab initio calculations have been carried out by using the 16-atom supercell approach with different initial spin configurations. The energy calculations were performed for the supercell ($FeRh_{1-x}Pd_x$). Calculations were carried out for ferromagnetic, paramagnetic and three kinds of antiferromagnetic states as functions of the lattice parameter. The equilibrium lattice parameters $a = 3.012$ for $FeRh_{1-x}Pd_x$ (x = 0.5) up to 3.05 for $FeRh_{1-x}Pd_x$ ($x = 1$). It can be concluded that the addition of Pd atoms leads to an increase in the lattice equilibrium parameter due to the larger atomic radius of Pd compared to the lower Rh value. The calculation of the total energy for the tetragonal distortion of the cubic structure along the z axis is performed also. To accomplish this, we fixed the volume of a supercell as $V_0 = a_0^3 \simeq a^2c$. In this case, the zero value of E corresponds to the austenitic phase for each compound. In the case of $FeRh_{1-x}Pd_x$ (x=0.5) the antiferromagnetic configuration solution is favorable for martensite states. For the parent $FeRh_{1-x}Pd_x$ (x=0.625, 0.75, 0,875) compound, the ferromagnetic spin configuration is energetically favorable compared to other configurations. Our calculations have shown that the substitution of Pd for Rh results in an appearance of stable body-centered tetragonal state. While the cubic phase becomes unstable. We also calculated the lattice constants, volume cell, partial and total magnetic moments.

This work was supported by RSF-Russian Science Foundation No. 17-72-20022\17.

Effect of Cr-doping on Properties of Ni-Mn-(Ga, Sb) Heusler alloys

28 Aug
2:00pm
Poster
Session A

Smolyakova E., Zagrebin M., Sokolovskiy V., Buchelnikov V.

Chelyabinsk State University, RUSSIAN FEDERATION

It is well known that the addition of the fourth element into Heysler alloys can strongly affect martensitic transformation and the Curie temperatures[1-4]. A series of Cr-doped Ni-Mn-(Sb, Ga) alloys have been theoretically investigated. Ground state calculations were performed within the framework of quantum-chemical modeling in the software package VASP (Vienna Ab-initio Simulation Package)[6,7] in the framework of the density functional theory (DFT), while calculations of structural parameters were performed using the density functional theory as part of the spin-polarized relativistic Korringa-Kohn-Rostoker (SPR-KKR)[8] package. Generally, the composition dependences of crystal lattice parameters, bulk modulus, magnetic moments, magnetic exchange parameters were found. The investigation was performed for 16-atom supercell possesses the fcc $L2_1$-type of structure (t he space group Fm3m, No. 225).This structure with the general formula X2YZ consists of four mutually penetrating fcc sublattices, in which two X atoms are located in 8c Wyckoff positions ((1/4, 1/4, 1/4) and (3/4, 3/4, 3/4)), while the Z and Y atoms occupy positions 4a and 4b ((0, 0, 0) and (1/2, 1/2, 1/2)), respectively. The most energetically favorable magnetic state for both austenite and martensite phases were found based on calculated results. Also, a complex analysis of the effect of magnetic stoichiometry in alloys on the favorable energy state has been done. Besides, the energy gap between martensite and austenite increases with increasing of Cr concentration. Within the Heisenberg model and Monte Carlo technique, the Curie temperatures are obtained. Theoretical results are compared with other theoretical end experimental results.

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Session 4: Martensite in Carbon Steel

Invited Lecture: Tuesday August 28	
15:30 Amphi 4	**S. Berveiller**, R. Kubler, R. Guiheux, C. Mauduit, D. Bouscaud, E. Patoor, L. Barrallier, P. Osmond, M. Monin, Q. Puydt, B. Weber "Martensitic transformation in a shot peened TRIP steel: influence of shot peening conditions on residual stresses and their relaxation"
Oral presentations: Tuesday August 28	
16:00 Amphi 4	**D. Brandl**, M. Stockinger, S. Ploberger, G. Ressel "In-situ observations of martensite reversion and austenite memory phenomenon in PH 15-5 corrosion resistant steel"
16:20 Amphi 4	E. Polatidis, M. Smid, **W.-N. Hsu**, T. Panzner, H. Van Swygenhoven "How austenitic TRIP steels accommodate strain under multiaxial loading: the effect of stacking fault energy and deformation state"
17:10 Amphi 4	**E. Nagy**, A. Talgotra, M. Sepsi, M. Benke, V. Mertinger "Thermomechanical treatment induced martensitic transformations in TWIP steels"
17:30 Amphi 4	**T. Sawaguchi**, I. Nikulin, Y. Inoue, A. Kushibe, Y. Chiba, T. Nakamura "Martensite Microstructure at Dissimilar weld between Ferrous-High Manganese Seismic Damping Alloy and Carbon Steel"
17:50 Amphi 4	**J. Teixeira**, S. D. Catteau, H. P. Van Landeghem, J. Dulcy, M. Dehmas, A. Redjaïmia, S. Denis, M. Courteaux "Bainite formation in carbon and nitrogen enriched low alloyed steels: kinetics and microstructures"

Poster Session A: Tuesday August 28
H. Abreu, G. Ribamar, H. Miranda "Study of the relationship between stacking fault energy and martensitic transformation in a weld fusion zone of a Fe-Mn electrode deposit"
S. Allain, G. Geandier, S. Gaudez, JC. Hell, M. Soler, M. Goune, F. Danoix, S. Aoued "Interest of High Energy X-Ray diffraction experiments for the investigation of Q& P steels"
B. Amini, G. Helbert, Y. Demmouche, S. Arbab Shirani, S. Calloch "Effect of martensitic transformation on the self-heating behavior of 304 metastable stainless steel"
A. Baur, C. Cayron, R. Logé "Variant selection in surface martensite"
S. Dieck, M. Ecke, S. Fritsch, M. F.-X. Wagner,T. Halle "SD effect in martensitic stainless steel under Q& P heat treatment condition"
A. Talgotra, E. Nagy, M. Sepsi, M. Benke, V. Mertinger "Martensitic transformation and PLC phenomena in austenitic FeMnCr TWIP steels"
W. Tasaki, T. Sawaguchi, I. Nikulin, K. Tsuchiya, I. Kireeva, Y. Chumlyakov "Structures at the intersection of ε-martensite variants in 316-type austenitic stainless steel single crystal compressively deformed at cryogenic temperature"

Contents

Martensitic transformation in a shot peened TRIP steel: influence of shot peening conditions on residual stresses and their relaxation

28 Aug
3:30pm
Amphi 4

Berveiller S.[1], Kubler R.[2], Guiheux R.[1,3], Mauduit C.[2,3], Bouscaud D.[1], Patoor E.[1,4], Barrallier L.[2], Osmond P.[5], Monin M.[3,6], Puydt Q., Weber B.[7]

[1]Arts et Métiers ParisTech, MSMP, 2, Cours des Arts et Métiers, 13617 Aix-en-Provence, FRANCE;
[2]Arts et Métiers ParisTech, MSMP, 2, Cours des Arts et Métiers, 13617 Aix-en-Provence, FRANCE;
[3] IRT M2P, 4, rue Augustin Fresnel, 57078 Metz Cedex 03, FRANCE;
[4]GeorgiaTech Lorraine, 2, rue Marconi, 57070 Metz, FRANCE;
[5] Now in Safran Tech, 1 Rue Geneviève Aube, 78114 Magny-les-Hameaux, FRANCE;
[6] Now in ArcelorMittal, Florange, FRANCE;
[7] ArcelorMittal Research SA, Voie Romaine, 57280 Maizières-lès-Metz, FRANCE

Shot peening process is commonly used in mechanical industries to increase life duration of mechanical and structural parts, as automotive gears for instance, by introducing residual stresses (RS). However, RS are likely to be relaxed during the material life [1]. In the case of TRIP-effect steels, the metastable austenite can transform into martensite during shot peening and fatigue life. The final stress state is complex as it results from mechanical strain due to the process and the martensitic transformation that leads to stress redistribution between austenite and martensite. The aim of this work is to study the behaviour of TRIP-effect steels submitted to shot peening by taking into account martensitic transformation, and its evolution during subsequent cycling bending tests. The studied material is TRIP780 steel which exhibits a multiphased microstructure with bainite, ferrite and retained austenite. First, samples were shot peened, using cut wire shots of two different diameters. Then plane bending cyclic test were performed at different numbers of cycles and loading ratios. At each processing step, retained austenite and RS were determined using X-ray diffraction in the depth of shot-peened specimens. Due to the complex microstructure, RS were analyzed in austenite and in ferritic phases (ferrite + bainite + martensite). Cyclic tests were also performed up to failure to determine the Wlher curves for the different shot peening conditions. In parallel, finite element simulations were performed taking into account residual stresses, plastic strains and hardening parameters for each phase due to shot peening. It is based on the shot peening model with stress and microstructure gradients developed previously; a semi-phenomenological transformation behaviour law for unstable austenite has been implemented to consider microstructure phase evolution. A similar approach was developed to predict also relaxation in each phase, as well as the evolution of the retained austenite fraction.

[1] Schulze, V., Modern mechanical surface treatment: states, stability, effects, John Wiley Sons, 2006

In-situ observations of martensite reversion and austenite memory phenomenon in PH 15-5 corrosion resistant steel

28 Aug
4:00pm
Amphi 4

Brandl D.[1], Stockinger M.[2], Ploberger S.[2], Ressel G.[1]

[1]Materials Center Leoben Forschung GmbH, AUSTRIA;
[2]voestalpine Böhler Aerospace GmbH & Co KG, AUSTRIA

PH 15-5 is a precipitation hardenable and corrosion resistant high alloyed steel and consists of lath-martensitic matrix with a carbon content below 0.04 wt.-% and nm-sized Cu precipitations. The alloy combines high strength with good toughness properties and therefore, is used e.g. for structural components in aerospace industry. In order to improve mechanical properties a special heat treatment, consisting of solution annealing and aging, is applied. During solution annealing the PH 15-5 undergoes a ferrite - austenite transformation and during aging the Cu-particles are formed. Several studies in literature found that alloys with low carbon and high nickel amounts deviate from classical diffusion controlled transformation during austenitization. Investigations of Shirazi et al. on binary iron-nickel alloys exhibit phenomena such as austenite memory or spontaneous recrystallization which are related to diffusionless transformation from martensite to austenite. The low austenite formation temperature of these alloys is assumed to impede long range diffusion. In alloys with significant lower Ni content and higher austenite formation temperatures such as 13Cr - 5Ni, Liu et al. observed austenitic spontaneous recrystallization by means of in-situ laser scanning confocal microscopy measurements. The presence of austenite memory is assumed due to equal size and shape of prior and reversed austenite grains. However, a diffusive transformation from martensite to austenite is postulated by the author for this alloy. This work shows that also the commercial PH 15-5 steel exhibits akin effects during heating up to austenite. Therefore, to clarify the formation of austenite memory and austenitic spontaneous recrystallization of the PH 15-5 steel, detailed investigations of these phenomena are proposed in the actual work. In the presented study in-situ electron backscatter diffraction and laser scanning confocal microscopy measurements are carried out in order to investigate martensite reversion in PH 15-5. Austenite memory is verified by comparing the crystal orientation of prior and reverted austenite and changes in crystal orientation during spontaneous recrystallization are analysed. Additionally, the heating rate dependence of austenite grain refinement due to spontaneous recrystallization is characterized by means of dilatometry measurements.

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28 Aug
5:10pm
Amphi 4

Thermomechanical treatment induced martensitic transformations in TWIP steels

Nagy E., Talgotra A., Sepsi M., Benke M., Mertinger V.

Institute of Physical Metallurgy, Metalforming and Nanotechnology, University of Miskolc, HUNGARY

Steels with high Mn content (15-30%) have high quality and extraordinary versatility inferable from either twinning-initiated versatility (TWIP) or the transformation induced plasticity (TRIP) that outcomes from various martensitic changes, for example, γ fcc (austenite) $\rightarrow \varepsilon$ hcp (hcp-martensite)$\rightarrow \alpha'$ bcc (bcc-martensite). The formation of these martensitic phases affects the strength, toughness, and formability and corrosion resistance of the steel. The mechanism of the austenite-martensite transformation depends on the composition, deformation rate and temperature. The ratio and quantity of the resulting phases determine the properties of the product. During the thermomechanical transformation of austenite to martensite, the texture evaluation and phase transformation both take place simultaneously. The martensitic transformation of three steels with varying Cr content at various temperatures and strain rates was studied in this work. The effects of loading parameters and temperature on the texture and volume fraction of the martensite were investigated. The developed texture of ε martensite, α' martensite and γ austenite was examined on different scales as by Electron Backscattered Diffraction (EBSD) orientation mapping and X-ray-diffraction pole figure measurements. Correlations between the developed textures and the formation of the martensitic phases are discussed.

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28 Aug
4:20pm
Amphi 4

How austenitic TRIP steels accommodate strain under multiaxial loading: the effect of stacking fault energy and deformation state

Polatidis E.[1], Smid M.[1], Hsu W.-N.[1,2], Panzner T.[3], Van Swygenhoven H.[1,2]

[1] *Photons for Engineering and Manufacturing Group, SLS, Paul Scherrer Institute, SWITZERLAND;*
[2] *Neutrons and X-rays for Mechanics of Materials, IMX, EPFL, SWITZERLAND;*
[3] *Laboratory for Neutron Scattering, NUM, Paul Scherrer Institute, SWITZERLAND.*

The effect of uniaxial/multiaxial loading on the martensitic transformation behavior of two metastable austenitic stainless steels (SS201 and SS304) with different stacking fault energy was studied by performing in-situ neutron diffraction at the Swiss spallation source SINQ. Both cruciform and dogbone-shaped samples were tested in-situ using the multiaxial deformation rig that the POLDI beamline is equipped with [1]. The neutron diffraction results are complemented by EBSD and digital image correlation in SEM to reveal the dominant strain accommodation mechanism for a given material under a given loading state and to relate them with the crystallographic texture of the microstructure. A discrepancy was observed between the two steels on whether biaxial loading favors the martensitic transformation In low SFE steel, i.e. 201 stainless steel, the martensitic transformation is facilitated during uniaxial loading [2], whereas biaxial loading produces more martensite in medium SFE steel, i.e. 304 stainless steel. The initial and evolving crystallographic texture strongly affects the preferred deformation mechanisms in these steels. In specific, the combination of texture and loading direction defines whether the partial dislocations will repulse or attract each other, i.e. whether ε martensite, which is a precursor for α' martensite, will form in low SFE 201 steel or whether twinning in the medium SFE 304 steel will suppress the formation of α martensite.
This research is performed within the ERC Advanced Grant MULTIAX (339245).
[1] Acta Materialia, 105 (2016) 404. [2] Scripta Materialia, 147 (2018) 27.

Martensite Microstructure at Dissimilar weld between Ferrous-High Manganese Seismic Damping Alloy and Carbon Steel

28 Aug
5:30pm
Amphi 4

Sawaguchi T.[1], Nikulin I.[1], Inoue Y.[2], Kushibe A.[2], Chiba Y.[3], Nakamura T.[1]

[1]*National Institute for Materials Science, JAPAN;*
[2]*Takenaka Corporation, JAPAN;*
[3]*Awaji-Materia Co Ltd, JAPAN*

A novel coaxial multi-layer weld wire was developed for a Fe-15Mn-10Cr-8Ni-4Si seismic damping alloy, which undergoes FCC (γ) to HCP (ε) martensitic transformation under cyclic loading and has almost 10-times longer low-cycle fatigue life than usual steels. The seismic damping alloy and the weld wire are practically used in manufacturing long-lived seismic dampers for architectural constructions. In the present publication, we report microstructural characteristic of the weld. The seismic damping alloy was welded with a SM490B-grade construction steel into fillet welded joints using the wire. Small dog-bone specimens were taken from the weld metal and subjected to tensile testing to failure. Microstructures of the weld metal before and after the tensile deformation were observed by means of scanning electron microscopy, and the crystallographic characteristics of the martensite variants were analyzed with electron backscattering diffraction. 24 crystallographic variants of α'-martensite were observed. They can be classified into four close-packed plane (CP) groups and three Bain groups consisted of two different CP directions with small angular difference, as have been reported for martensitic and bainitic steels. Characteristics of the present martensite microstructure developed in the weld metal are: 1) The martensite structure consists of packets with a plate-like morphology belonging to one or two CP group(s) (Plated packet) and packets without the plate-like morphology and high area fraction and larger lath size than the former (Matrix packet). 2) The plated packets are running on the traces of the corresponding CP planes. (3) A small amount of ε-martensite exists in between the plated packets along the CP plane. (4) The matrix packets exhibit the adjacency relationship of V1/V4 (different CP directions, same Bain) and V1/V2, 3, 5 (twinning relationship), likewise the usual mrtensitic steel, while the plated packets exhibit the adjacency relationship of V1/V6 (same CP direction, different Bain). The characteristics of the plated packets suggest the two-step $\gamma \rightarrow \varepsilon \rightarrow \alpha$' martensitic transformation. The tensile plastic deformation brings about the distortion and rotation of the martensite laths and the formation of shear bands, where fine laths with large misorientation angles appear, which implies deformation twinning of the original laths.

Bainite formation in carbon and nitrogen enriched low alloyed steels: kinetics and microstructures

28 Aug
5:50pm
Amphi 4

Teixeira J.[1,2], Catteau S. D.[1,2,3], Van Landeghem H. P.[1,2,4], Dulcy J.[1], Dehmas M.[1,2,5], Redjamia A.[1,2], Denis S.[1,2], Courteaux M.[3]

[1]*Université de Lorraine, CNRS, IJL, FRANCE;*
[2]*Labex DAMAS "Design of Alloy Metals for Low-mass Structures", Université de Lorraine, FRANCE;*
[3]*PSA Peugeot-Citroën, Centre Technique de Belchamp, FRANCE;*
[4]*SIMaP, UMR 5266, Grenoble INP, CNRS, UGA, FRANCE;*
[5]*CIRIMAT, Université de Toulouse, CNRS, INPT, UPS, FRANCE.*

The effect of an enrichment in carbon and nitrogen of the austenite of a low-alloyed steel on its decomposition during cooling was examined by in situ high-energy synchrotron X-ray diffraction (HEXRD) and the final microstructures were analyzed by transmission electron microscopy. The introduction of nitrogen induces new microstructures and drastic kinetics effects which were scarcely examined in literature, which is focused on model Fe-N and Fe-C-N systems. Samples of a 0.246C-1.21Mn-1.31Cr-0.237Si-0.184Ni (wt%) steel were enriched homogeneously in the austenite or austenite+CrN phase field at 900 °C in carbon and/or nitrogen by cracking respectively methane or ammonia by using an in-house thermobalance. Three enrichment treatments were considered: carburizing (0.6wt%C), nitriding (0.12wt%C-0.25wt %N) or carbonitriding (0.7wt%C-0.25wt%N). During the enrichments in nitrogen, CrN nitrides precipitated, as expected from thermodynamics, either at grain boundaries or intragranularly. AlN, VN and $MnSiN_2$ nitrides were observed as well, with much smaller number density. They formed frequently aggregates with the CrN nitrides. Classical slowing down effect on kinetics is observed by increasing the carbon content, in carburized steel compared to initial steel. Conversely, the enrichment in nitrogen induces a strong acceleration of the bainite transformation kinetics in carbonitrided steel compared to carburized steel, despite the -stabilizing character of nitrogen. This is attributed to the nucleation of bainitic ferrite

on the CrN nitrides which precipitated during the enrichment. The bainitic ferrite microstructure is much finer than in initial or carburized steel. It shares some common features with intragranularly nucleated bainite, i.e. acicular ferrite. The ferrite probably nucleated on CrN nitrides which are frequently observed at the boundaries of the ferritic domains. The high density of the nucleation sites may hinder the organization of the ferrite laths into packets by autocatalytic nucleation. This refinement of the microstructure leads to an improvement of the mechanical properties, according to hardness measurements. From HEXRD, the chronology of the phase formation (ferrite and precipitates) during bainite formation as well as cell parameter evolutions are analyzed. For initial and carburized steels, the results are in agreement with the assumption of a non-diffusive mechanism. In presence of nitrogen, analysis of austenite cell parameters and peaks profiles showed clearly the rejection of interstitials from ferrite into austenite, leading to composition heterogeneities, without making possible to propose firm hypotheses on the phase transformation mechanism.

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28 Aug
2:00pm
Poster
Session A

Study of the relationship between stacking fault energy and martensitic transformation in a weld fusion zone of a Fe-Mn electrode deposit

Abreu H., Ribamar G., Miranda H.

Universidade Federal do Ceará, BRAZIL

Steels with high manganese content have been gaining more prominence due to their high toughness in cryogenic environments and excellent allied resistance and high ductility. These materials have a structure composed of austenitic matrix, and may present ferrite, α'-martensite and ε-martensite, depending on the content of the alloying elements. The properties of these materials are often related to the mechanism of austenite deformation, either martensite formation or twins formation during deformation. This deformation mechanism is often related to the stacking fault energy (SFE) that the material possesses. Studies show that SFE values between 0 and 8 mJ/m^2 provide formation of ε martensite during deformation, whereas only twins are formed for energies above 18 mJ/m^2. Intermediate values provide the formation of both ε martensite and twins. In this way it is possible to predict the microstructure and, consequently, mechanical properties with the SFE knowledge of the material. In this work a weld consumable with high manganese content was deposited on a SAE 1012 carbon steel plate with the flux-cored arc welding process producing different chemical compositions and consequently different stacking fault energies. An algorithm was developed for the calculation of SFE by the thermodynamic method; the microstructures to verify the prediction made with the energy of stacking failure were obtained by optical microscopy and scanning electron microscopy. The deposition of the electrode on the SAE 1012 steel surface generated different chemical compositions in the weld metal that varied as a function of the dilution. Conditions with dilution values between 7 and 41% were produced. Low dilution conditions presented positive SFE, while its microstructure showed only austenite. For the medium and high dilution conditions, the chemical composition of the molten zone presented negative SFE, and its microstructure was composed of austenite and ε-martensite.

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28 Aug
2:00pm
Poster
Session A

Interest of High Energy X-Ray diffraction experiments for the investigation of Q&P steels

Allain A.[1], Geandier G.[1], Gaudez S.[1], Hell J. C.[2], Soler M.[2], Goune M.[3], Danoix F.[4], Aoued S.[3,4]

[1]*Institut Jean Lamour, UMR CNRS-UL 7198, Nancy, FRANCE;*
[2]*Maizières Automotive Products, Arcelormittal Maizières Research SA, Maizières les Metz, FRANCE;*
[3]*Institut de Chimie de la Matière Condensée de Bordeaux, UPR 9048, Pessac, FRANCE;*
[4]*Normandie Univ., UNIROUEN, INSA Rouen, CNRS, Rouen, FRANCE*

High Energy X-Ray diffraction (HEXRD) experiments conducted on synchrotron beamlines offer opportunities to go deeper in the understanding of Q&P steels. Such in situ experiments allow not only following in real-time the complex phase transformations processes and their interactions taking place during manufacturing but also investigating the phase behavior during mechanical tests on as-transformed microstructures. In the project CAPNANO funded by the French ANR, both kinds of experiments have been conducted on the same model alloys at ESRF, France (ID 15A et ID11) and at DESY, Germany (Petra-07). The experiments have been made possible by the use of brilliant and high energy sources and fast 2D detectors (powder diffraction set-up in transmission), which enable acquisition rate higher than

10 Hz. Our in situ experiments during thermal treatments have revealed for the first time that intense second-order internal stresses are generated all along the processing route of Q&P steels. The stresses affect in turn the way to measure carbon enrichment content in austenite during partitioning and the apparent stability of retained austenite at room temperature. In situ HEXRD experiments are thus the sole reliable method to measure carbon enrichment in austenite as they allow deconvoluting unambiguously the chemical and mechanical contribution in austenite lattice parameters' evolution. In parallel, the couplings between carbon diffusion from martensite to austenite and possible carbide-free bainitic transformation have been investigated during partitioning step. This work is based on consolidated carbon mass balances which take into account mean concentration and local distributions of carbon in austenite, tetragonality of martensite/bainite and eta carbide precipitation in martensite. Finally, our in situ tensile tests have allowed assessing the stability of the retained austenite in microstructures elaborated under different manufacturing conditions. For both kinds of experiments, the limits of the analysis and the possible opportunities for the future will be discussed.

[1] S.Y.P. Allain, G. Geandier, J.C. Hell, M. Soler, F. Danoix, M. Gouné, In-situ investigation of quenching and partitioning by High Energy X-Ray Diffraction experiments, Scr. Mat. 131 (2017) 1518.
[2] S.Y.P. Allain, G. Geandier, J.C. Hell, M. Soler, F. Danoix, M. xGouné, Effects of Q&P Processing Conditions on Austenite Carbon Enrichment Studied by In Situ High-Energy X-ray Diffraction Experiments, Metals 7 (2017) 232-245.
[3] S.Y.P. Allain, S. Gaudez, G. Geandier, J.C. Hell, M. Gouné, F. Danoix, M. Soler, S. Aoued, A. Poulon-Quintin, Internal stresses and carbon enrichment in austenite of Quenching and Partitioning steels from high energy X-ray diffraction experiments, MSEA 710 (2018) 245-250.

Effect of martensitic transformation on the self-heating behavior of 304 metastable stainless steel

28 Aug
2:00pm
Poster
Session A

Amini B.[1], Helbert G.[1], Demmouche Y.[2], Arbab Chirani S.[1], Calloch S.[2]

[1]*ENIB, IRDL, FRE CNRS 3744, Brest, FRANCE;*
[2]*ENSTA Bretagne, IRDL, FRE CNRS 3744, Brest, FRANCE*

Stainless steel materials have found a wide range of applications due to interesting properties such as high ductility, good corrosion resistance, low cost, easy for machining and plenty availability. However, their mechanical properties are significantly affected by the deformation-induced martensitic transformation. The martensitic transformation (MT) can occur upon monotonous mechanical loadings or cyclic loading (even below the yield stress value). Hence, in order to use these steels to their full potential, it is essential to have good knowledge regarding this martensitic phase transformation and specially its effect on fatigue properties and so on. The traditional methods of fatigue estimation are extremely time consuming. In the case of fatigue for classic metals, evolution of micro-plasticity (MP) causes heat generation due to a detectable temperature rising at specimen surface. This phenomenon is the principle to evaluate the fatigue limit by self-heating method. The self-heating method allows the rapid determination of the high cycle fatigue limit, but its relation with fatigue should be confirm for special materials like metastable stainless steels. Moreover, the results of self-heating measurements can be used to better understanding the deformation mechanism of simultaneous micro-plasticity and martensitic transformation during cyclic loading. In this study, we considered 304 metastable austenitic steel and we paid special attention to the comprehension of material's thermal response (i.e. self-heating) during cyclic tests. These tests were carried out in different levels of stress and temperatures to get a better comprehension and to be able to relate obtained results from self-heating with those obtained from classical fatigue tests. Thus, for some stress amplitudes, the fatigue tests were carried out up to 10^6 cycles and the mean temperature evolutions were measured during the tests. Hence, the aim of the present research is to study the self-heating behavior of a metastable austenitic stainless steel under fatigue loading for stress amplitudes lower than the yield stress value. Furthermore, the deformation-induced phase transformation from γ-austenite to α'-martensite is globally evaluated by means of magneto-inductive measurements. The obtained results show that the martensite volume fraction increases after cyclic loading tests and the appearance of martensite phase effects on the behavior of this material. This study confirms that fatigue criteria have to consider microstructure changes (both MP and MT) in order to improve fatigue life prediction of stainless steel structures. After good comprehension of self-heating for this material, in future, we can use this method for studying fatigue behavior.

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28 Aug
2:00pm
Poster
Session A

Variant selection in surface martensite

Baur A., Cayron C., Logé R.

Ecole polytechnique fédérale de Lausanne (EPFL), SWITZERLAND

Variant selection in martensitic steels is a widely studied phenomenon because of its implications on the industrial process of steels. Variant selection is generally reported when stress are applied during or before transformation, and the majority of the models developed for the prediction of variant selection considers external loading as inputs for the computation. Here, we report a significant variant selection in surface martensite formed isothermally on the electropolished surface of a as-cast Fe-30%Ni sample. The variant selection phenomenon is observed even though no external stress, nor any deformation are applied to the material before transformation. The crystallographic features of surface martensite as well as the variant selection have been characterized using EBSD measurements. In particular, the orientation relationship, the habit plane and accommodation process are studied. A variant selection rule that captures the phenomenon is proposed. Different crystallographic descriptions are compared in order to see the ones that better account for the crystallographic features of surface martensite and for the observed variant selection. Namely, the PTMC matrices, the Jaswon and Wheeler distortion and the continuous FCC-BCC model are compared. Additionally, the effect of applied stress before the formation of surface martensite is investigated, and its impact on variant selection in surface martensite characterized.

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28 Aug
2:00pm
Poster
Session A

SD effect in martensitic stainless steel under Q&P heat treatment condition

Dieck S.[1], Ecke M.[1], Fritsch S.[2], Wagner M. F.-X.[1], Halle T.[1]

[1]*Institute for Materials and Joining Technology, Otto-von-Guericke-University Magdeburg, GERMANY;*
[2]*Institute for Materials Science and Technology, Technical University Chemnitz, GERMANY*

The quenching and partitioning (Q&P) heat treatment enables a higher deformability of high strength martensitic steels. Therefore it is necessary to have some metastable austenite in the microstructure, which transforms in martensite during plastic deformation (TRIP-effect). This condition is guaranteed by the two-step heat treatment, consisting of quenching in a way, which retains a certain amount of austenite. A subsequent low temperature annealing, the so called partitioning, stabilises the retained austenite due to carbon diffusion. The Q&P heat treatment was investigated for the martensitic stainless steel 1.4034 ($X_{46}Cr_{13}$) concerning the influence of partitioning time. In line with these efforts extensive characterizations of the mechanical properties, focussing on the materials behaviour under different mechanical load scenarios, were performed. Therefore quasi static tension and compression tests were carried out. Additional micro-hardness mappings should clarify the homogeneity of mechanical properties over the sheet thickness. Moreover, a comprehensive analysis of the microstructural evolution was performed for different stages of heat treatment, as well as after mechanical loading. For this purpose light optical microscopy and different methods of electron microscopy were executed. The comparison of common quenching and tempering with the Q&P heat treatment verifies the extensively enhanced materials strength whereat the formability is still acceptable. The microstructural reason was detected to be an increasing austenite fraction due to austenite reversion at sub-grain boundaries of martensite besides the stabilising of retained austenite. Further a distinctive strength differential effect was observed.

Martensitic transformation and PLC phenomena in austenitic FeMnCr TWIP steels

28 Aug
2:00pm
Poster
Session A

Talgotra A.[1], Nagy E.[2], Sepsi M.[1], Benke M.[1], Mertinger V.[1]

[1]*IPMMN, University of Miskolc, HUNGARY;*
[2]*MTA-ME Material Science Research Group, University of Miskolc, HUNGARY*

Austenitic steels are best suited for automobile applications including press-formed parts because of their energy absorption or structural reinforcement. Austenitic FeMnCr steels have high strength, high toughness and formability because of the martensitic transformation. In these steels the temperature as well as strain-induced non thermoelastic martensitic phase transformation takes place. The austenitemartensite transformation depends on composition, deformation rate and temperature. During thermo-mechanical treatments, when a well-controlled combination of the temperature and the deformation rate is applied an extraordinary combination of microstructures can be achieved. Because of the ratio and quantity of resulted phases determines the properties of the product an extraordinary mechanical property can be predicted after or during the treatments. In our research work the PLC or effect was seen in the tensile curves investigations during the thermo-mechanical treatments of the high strength austenitic TWIP steels. Many serrations of different intensities and amplitudes were observed in the tensile curves of the steel test samples with different composition. The cause for jerky flow or serrated yielding (or load drops) could be because of many reasons. It could be due to phase transformation (martensite formation), due adiabatic heating, order disorder transformation. The 1st serration (or load drop) usually occurs at a given "critical plastic strain", which varies with strain rate and temperature. Aim of our study is to solve the pattern of the serrations to get the answers and results for the occurrence of the PLC effect in the pathway of our research work. Therefore the true stress- true strain were characterised in more detail and various iterations of the test samples were used on the basis of temperature, composition and the deformation rate.

Structures at the intersection of ε-martensite variants in 316-type austenitic stainless steel single crystal compressively deformed at cryogenic temperature

28 Aug
2:00pm
Poster
Session A

Tasaki W.[1], Sawaguchi T.[1], Nikulin I.[1], Tsuchiya K.[1], Kireeva I.[2], Chumlyakov Y.[2]

[1]*National Institute for Materials Science, JAPAN;*
[2]*National Research Tomsk State University, Siberian Physical-Technical Institute of V.D. Kuznetsova, RUSSIAN FEDERATION*

Martensitic transformation from FCC (γ) austenite to HCP (ε) martensite is responsible for functional and mechanical properties of Fe-Mn-Si-based shape memory alloys and high-Mn austenitic steels. It is also known that the ε-martensite serves as a precursor of the BCC (α') martensite in austenitic stainless steels, where the intersection of different ε-martensite brings about the rearrangement of atoms to nucleate the α'-martensite. Depending on the chemical composition and the crystallographic orientation, however, not only the second martensitic transformation from ε to α', but also twinning of the ε-martensite and even the reverse transformation into the γ-austenite have been reported at the intersection of the ε-martensite variants. In this study, we carried out a single crystal study to clarify the selection rule of the intersection structure. A single crystal of 316-type austenitic stainless steel was compressively deformed at 173 K with respect to the compressive axis parallel to <001> direction. The pre-polished side surface in {110} plane was observed by means of scanning electron microscopy and the structures at the ε-ε intersections and their crystallographic characteristics were analyzed with electron backscattering diffraction. Among the four different ε-martensite variants with comparable Schimid factors above 0.45, there are two types of combinations of crossing shears, depending on the shear angles either perpendicular to or 30 °-inclined from the intersection axis. The former stimulated the reverse transformation into the γ-crystal rotated from the parent γ phase by 90 °with respect to the intersection axis, while the latter produced the second martensitic transformation into the α'-martensite. The atomic rearrangement at the two types of the intersection reactions can be visualized by the geometric consideration on the double-shear deformation of the Thompson tetrahedron.

Session 5: Magnetic Shape Memory and Ferroic Transitions

Invited Lecture: Wednesday August 29	
15:30 Amphi 1	**V. A. Chernenko**, P. Sratong-on, J. Feuchtwanger, H. Hosoda "Magnetomechanical effects in NiMnGa/polymer composite"
Invited Lecture: Thursday August 30	
08:30 Amphi 1	**D. Wang** "Combined caloric effects in magnetic shape memory alloy with broad working region"
Oral presentations: Wednesday August 29	
10:50 Amphi 1	**A. Saren**, A. Sozinov, D. Musiienko, S. Kustov, K. Ullakko "Magnetostriction in 10M Ni-Mn-Ga martensite"
11:10 Amphi 1	**B. Yang**, Z. Li, H. Yan, Y. Zhang, X. Zhao, C. Esling "Martensitic transformation in <110>A oriented Ni-Mn-Ga thin films"
11:30 Amphi 1	**R. Chulist**, P. Czaja, M. Faryna, A. Wojcik, L. Straka, A.Sozinov "Adaptive phase or variant formation in modulated Ni-Mn-Ga martensite ?"
11:50 Amphi 1	**O. Heczko** "Direct observation of a/b twin laminate and its evolution under stress in 10M martensite exhibiting magnetic shape memory effect"
12:10 Amphi 1	**O. Sevestre**, A. Bouterf, B. Smaniotto, O. Hubert "X-Ray DVC monitoring of martensitic reorientation in a Ni_2MnGa single crystal"
16:00 Amphi 1	**A. Soroka**, A. Sozinov, N. Lanska, M. Rameš, L. Straka, K. Ullakko "Tailoring the mobility of compound-type twin-boundaries in magnetic shape memory alloys"
16:20 Amphi 1	**L. Demchenko**, L. zlv, A. Titenko, O. Shevchnk "Structural Features of Martensitic Transformations in Ferromagnetic Fe-Ni-Co-Ti Shape Memory Alloys"
17:10 Amphi 1	**V. Sánchez-Alarcos**, J. López-García, I. Unzueta, J. I. Pérez-Landazábal, V. Recarte, J. J. Beato-López, J. A. García "Influence of defects on the martensitic transformation and the magnetic properties of Co-doped Ni-Mn-Sn and Ni-Mn-In metamagnetic shape memory alloys"
17:30 Amphi 1	**T. Odaira**, X. Xu, A. Miyake, T. Omori, M. Tokunaga, R. Kainuma "Thermal-, magnetic-field- and stress-induced transformation in $Co_2Cr(Al, Si)$ Heusler-type shape memory alloys"
17:50 Amphi 1	**D.L. Beke**, M. K. Bolgár, L.Z. Tóth, L. Daróczi "On the asymmetry of the forward and reverse martensitic transformations in shape memory alloys"
Oral presentations: Thursday August 30	
9:00 Amphi 1	**D. Salas**, O. Eliseeva, Y. Wang, T. Duong, Y. Ren, R. Arróoyave, I. Karaman, Y. Chumlyakov "Microstructural dependences of the multi-ferroic transformation in Ni-Co-Mn-In metamagnetic shape memory alloys"
9:20 Amphi 1	**E. Villa**, A. Nespoli, F. Passaretti, I. Pallecchi "NiMnGaFe alloys polycrystalline samples: modulation of gamma-phase and related functional properties"
9:40 Amphi 1	**M. Leitner**, P. Neibecker, J. Schmidbauer, W. Petry "Premartensite in $Ni_2MnAl_{0.5}Ga_{0.5}$"
10:00 Amphi 1	**M.J. Szczerba**, W. Maziarz, A. Brzoza, A. Wierzbicka-Miernik, A. Wójcik, M. Kowalczyk, J. Wojewoda-Budka, M. Sikora "Structure and magneto-mechanical properties of Ni-Mn-Ga-Co-Cu melt-spun ribbons"

Poster Session C: Wednesday August 29
L. Bodnarova, P. Sedlák, H. Seiner, M. Zelený, L. Straka, O. Heczko, A. Sozinov, M. Landa "Elastic properties of pure and Co- Cu- doped non-modulated Ni-Mn-Ga martensite"

Poster Session C: Wednesday August 29
M.K. Bolgár, Daróczi, L. Z. Tóth, E. E. Timofeeva, E. Y. Panchenko, Y.I. Chumlyakov, D. L. Beke "Magnetic noise emitted during martensitic transformation in $Ni_{49}Fe_{18}Ga_{27}Co_6$ single crystals"
A. Brzoza, P. Czaja, T. Czeppe, A. Wierzbicka-Miernik, A. Wójcik, J. Wojewoda-Budka and M.J. Szczerba, E. Cesari "Influence of Cu addition on crystal structure and martensitic transformation in Heusler $Ni_{50}Mn_{25}Ga_{25-x}Cu_x$ alloys"
R. Chulist, T. Tokarski, Y. I. Chumlyakov, P. Czaja "Orthogonal shear process in Ni-Mn-Ga and Ni-Mn-Sn single crystals"
L. Daróczi, A. Hudk, L. Z. Tóth, Dezsö L., Beke D. L. "Investigation of magnetic and acoustic emission during stress induced superplastic deformation of martensitic Ni_2MnGa single crystal"
V. Kopecky, M. Rameš, O. Heczko "Martensitic transition in Fe-doped Ni_2MnGa"
P. Krystian, E. Matyja, M. Wąsik, M. Zubko "Martensitic transformation, structure and mechanical properties of the Mo doped NiCoMnIn magnetic shape memory alloys"
Y. V. Kudryavtsev, N. V. Uvarov, A. E. Perekos, J. Dubowik, L. E. Kozlova "Effect of temperature and magnetic field induced martensitic transformation on electronic structure and physical properties of bulk $Fe_{45}Mn_{26}Ga_{29}$ alloy"
A. Kyianytsia, C.-S. Chang, I. Cinar, R. Lima de Miranda, P. Boulet, L. Calmels, E. Gaudry, S. Migot, J. Ghanbaja, B. Kierren, T. Hauet "Magnetic characteristics of polycristalline NiTi film"
A. A. Likhachev, A. Sozinov, K. Ullakko "Effect of magnetostatic energy on the magnetization behavior and magnetic driving forces in NiMnGa FMSMA's"
J. López-García, J. A. Rodríguez-Velamazán, V. Recarte, V. Sánchez-Alarcos, J. I. Pérez-Landazábal, I. Unzueta, J. A. García, F. Plazaola "Correlation between defects and magneto-structural properties in Ni-Mn-Sn metamagnetic shape memory alloys"
D. Musiienko, A. Saren, L. Straka, L. Klimša, K. Ullakko "High-speed actuation of Ni-Mn-Ga micropillars by pulsed magnetic field"
E. Panchenko, E. E. Timofeeva, Y.I. Chumlyakov, K. S. Osipovich, N.G. Larchenkova, M. V. Pichkaleva, A. B. Tokhmetova, G. Gerstein, H. J. Maier "Stress-induced martensite ageing in single crystals of Ni-based Heusler alloy: processing, effect of orientation and functional properties"
N. Samy, M.K. Bolgár, N. Barta, L. Daróczi, Y.I. Chumlyakov, I. Karaman, D. L. Beke "Thermal and magnetic noises emitted during martensitic transformation in a metamagnetic $Ni_{45}Co_5Mn_{36.6}In_{13.4}$ single crystal"
C. Seguí, J. Torrens-Serra, E. Cesari, P. Lázpita "Characteristics and functional properties of Ni-Mn-Ga-Cu alloys"
A. Titenko, **L. Demchenko**, M. Babanli, L. Kozlova, A. Perekos, S. Huseynov, S. Sidorenko "Effect of thermomechanical treatment on deformational and magnetic behaviour of ferromagnetic Fe-Ni-Co-Ti alloy"
L. Z. Tóth, L. Daróczi, D. L. Beke "Effect of the partial heating/cooling cycles on the asymmetry of the forward and reverse martensitic transformation of single crystalline Ni_2MnGa alloy"
J. Xia, X. Xu, T. Omori, R. Kainuma "Temperature dependence of critical stress and entropy change for martensitic transformation in Fe-Mn-Al-Ni shape memory alloy"
X. Xu, Y. Yoshida, T. Omori, T. Kanomata, R. Kainuma "Martensitic transformation and magnetic phase diagram of $Ni_{50}Mn_{50-x}Ga_{x/2}In_{x/2}$ magnetic shape memory alloys

Contents

29 Aug
3:30pm
Amphi 1

Magnetomechanical effects in NiMnGa/polymer composite

Chernenko V. A.[1-4], Sratong-on P.[2], Feuchtwanger J.[4], Hosoda H.[2]

textsuperscript1BCMaterials, Basque Center for Materials, Applications and Nanostructures, SPAIN;
[2]Institute of Innovative Research, Tokyo Institute of Technology, JAPAN;
[3]Ikerbasque, Basque Foundation for Science, SPAIN;
[4]University of the Basque Country (UPV/EHU), SPAIN.

Ni-Mn-Ga FSMAs exhibit outstanding actuation properties in a bulk, such as, e.g., magnetically induced reorientation of martensitic variants resulting in magnetostrains up to 12%, alongside conventional shape memory and superelasticity effects. These effects are associated with a high work output per unit volume, in the order of 10^7 Jm^{-3}, making FSMAs interesting materials for magnetically activated micro-electromechanical systems (MEMS). While an effective downscaling was already proved for the thermomechanically controlled actuation properties of the low-dimensional FSMAs, such as NiMnGa thin films, ribbons, foams and small particles, this is still challenging task for the case of their magnetomechanical activation. We will overview the literature results on the dynamical mechanical response of NiMnGa-powder/polymer composites revealing the transformation behavior and stress-induced twin boundary motion effect in the NiMnGa particles. On the other hand, no clear evidences have been obtained about magnetic field induced twin boundary motion in single particles which may partially explain a tiny magnetostrain response in NiMnGa/polymer composites observed so far. We will discuss in detail the latter problem and formulate preconditions of getting large magnetostrains in these materials. In this regard, we will present experimental evidences about possible constraint conditions when particles are elastically interacting. Recently we have developed NiMnGa/silicone composites as prototype materials suitable for the magnetic actuation, mechanical damping controlled by magnetic field and stress sensing. The composites contain 20-30 vol. % of fillers representing oriented ferromagnetic martensitic particles (with spherical or irregular shape). For the first time we have observed the effect of magnetostrain in both individual particle (by the three-dimensional X-ray micro-computed tomography) and in composite as a whole (optical detection), produced by the magnetic-field-induced twin boundaries rearrangements. Strikingly, the magnetostrain response of composite (up to 4% in the field of about 0.5 T) was reversible along particle chains and in the perpendicular direction, which means an appearance of the local internal stresses driving twin boundaries back after field removal.
Grant-in-Aid for Scientific Research (Kiban S 26220907) from Japan Society for the Promotion Science is greatly acknowledged.

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30 Aug
8:30am
Amphi 1

Combined caloric effects in a magnetic shape memory alloy with broad working region

Wang D.

Nanjing University, CHINA

Solid-state refrigeration based on the caloric effects of ferroic materials has attracted more and more attention. However, the refrigeration temperature regions of most caloric effects are usually in a limited scale, which has been a key drawback for applications. The earlier reports showing limited ability of broadening the refrigeration temperature region indicate that the narrow working temperature region is still a challenge for the caloric refrigeration. We investigate the magnetocaloric and elastocaloric effects of a directionally solidified magnetic shape memory alloy, $Ni_{49.5}Mn_{28}Ga_{22.5}$ (at. %). Since their refrigeration temperature regions are adjacent, a broad operating temperature range (280-400 K) is realized in a single magnetic shape memory alloy by combining magnetocaloric and elastocaloric effects.

On the asymmetry of the forward and reverse martensitic transformations in shape memory alloys

29 Aug
5:50pm
Amphi 1

Beke D. L., Bolgár M. K., Tóth L. Z., Daróczi L.

Department of Solid Stet Physics, University of Debrecen, HUNGARY

Differential Scanning Calorimetric, DSC, runs taken during martensitic phase transformations in shape memory alloys, often look differently during cooling and heating. Similar asymmetry is observed e.g. for the numbers of hits or the critical exponents of energy and amplitude distributions in acoustic and magnetic emission measurements. It is illustrated that, in accordance with empirical correlations, the above asymmetry of acoustic noises can be classified into two groups: the relative changes of the exponents during cooling and heating are either positive or negative. For positive values the number of hits and the total energy of acoustic emission are larger for cooling, and the situation is just the reverse for negative asymmetry. It was illustrated in a set of our previous measurements [1,2,3,4,5] that surface roughening did not resulted in changes of the above asymmetry, i.e. the role of the usual frictional interactions of the moving interface (nucleation, pinning/depinning events) are active in both directions. Thus our interpretation is based on the different ways of relaxation of the elastic energy during cooling as well as heating. It is illustrated that if the relaxed fraction of the total elastic energy (which would be stored without relaxations) during cooling is larger than the corresponding relaxed fraction during heating, then the asymmetry is positive. Magnetic emission noises, accompanied with martensitic phase transformations in ferromagnetic alloys, show similar asymmetry than those observed for thermal (DSC) and acoustic noises and depends on the constant external magnetic field too. Experimental results obtained in different shape memory alloys on thermal, acoustic and magnetic noises are gathered and analysed in the light of the interpretation based on the different ways of elastic energy relaxations during heating and cooling.

[1] L.Z. Toth, S. Szabo, L. Daroczi, D.L. Beke, Phys. Rev. B 90 (2014), 224103.
[2] M. Bolgar, L. Daroczi, L. Toth, E. Timofeeva, E. Panchenko, Y. Chumlyakov, D.L. Beke, J. Alloy. Comp. 705 (2017) 840.
[3] M. Bolgar, L. Toth, S. Szabo, S. Gyongyosi, L. Daroczi, E. Panchenko, Y. Chumlyakov, D. Beke, J. Alloy. Comp. 658 (2016) 29.
[4] L.Z. Toth, L. Daroczi, S. Szabo, D.L. Beke, Phys. Rev. B 93 (2016), 144108. [5] D.L. Beke, M.K. Bolgar, L.Z. Toth, L. Daroczi J. Alloy. Comp. 741 (2018) 106.

Adaptive phase or variant formation in modulated Ni-Mn-Ga martensite?

29 Aug
11:30am
Amphi 1

Chulist R.[1], Czaja P.[1], Faryna M.[1], Wojcik A.[1], Straka L.[2,3], Sozinov A.[4]

[1]*Institute of Metallurgy and Materials Science, Polish Academy of Sciences POLAND;*
[2]*Faculty of Mathematics and Physics, Charles University in Prague, CZECH REPUBLIC;*
[3]*Institute of Physics ASCR, CZECH REPUBLIC;*
[4]*Material Physics Laboratory, Lappeenranta University of Technology, FINLAND*

The crystal structure and microstructure of the modulated martensite phases have a strongly adaptive character. Depending on twin configuration (one, two or multi-variant) different diffraction pattern indicating a change of lattice parameters are obtained. On the other hand, the concept of adaptive martensite proposed more generally by Khachaturyan et al. and particularly for the 14M Ni-Mn-Ga alloys by Kaufman et al. shows that the lattice mismatch formed at a habit plan is compensated by nanotwinned martensite. For this reason, the 14M martensite is understood as an intermediate martensite phase built out of nanotwinned non-modulated phase of tetragonal blocks twinned on the scale of five and two atomic layers yielding the 14M martensite structure. Nevertheless, this concept was never confirmed for the 10M martensite. Therefore, to determine the real structure of Ni-Mn-Ga modulated phases and confirm or exclude the adaptive concept the real single variant state of 14M and 10M martensite is studied. The results show the role of type I, type II, modulation and a/b boundaries in self-accommodation process as well as the sequence of twin boundaries formation. In-situ experiments combined with a high-resolution scanning electron microscope and synchrotron radiation provide a significant insight into the view of adaptive character of martensitic transformation.

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29 Aug
4:20pm
Amphi 1

Structural Features of Martensitic Transformations in Ferromagnetic Fe-Ni-Co-Ti Shape Memory Alloys

Demchenko L.[1], Kozlova L.[1], Titenko A.[1], Shevchenko O.[2]

[1]Institute of Magnetism, National Academy of Sciences, UKRAINE;
[2]Frantsevich Institute for Problems in Material Science of NAS of Ukraine, UKRAINE.

The Fe-Ni-Co-Ti alloy system belongs to the family of new smart ferromagnetic materials exhibiting the shape memory effect and superelasticity. The formation of spherical nanoparticles of γ'- phase (Ni_3Ti) homogeneously distributed in austenite matrix and coherent with it occurs under the ageing of this alloys. The nanoparticles retain their coherency with the matrix under martensite transformation, that results in tetragonality of the martensite crystal lattice. It makes easier the coherent connection of the austenite and martensite lattices and decreases the level of elastic strains between them that determines the thermoelastic martensite transformation behaviour. By the proper choice of the alloy composition (taking into account the invar anomaly advantages) and ageing regimes, it has been managed to obtain the thermoelastic transformation with hysteresis from 250 to 20 in the above alloys. The aim of this research was to study thermoelastic martensite structure of the Fe-Ni-Co-Ti alloys and its correlation with the hysteresis of transformation. Three different compositions of Fe-Ni-Co-Ti alloys have been studied by metallography, X-ray, magnetic measurements at low temperatures. The characteristic temperatures of martensitic transformation were determined by the temperature dependences of low field magnetic permeability. It has been shown that temperature hysteresis of $\gamma \leftrightarrow \alpha$ transformation in the investigated alloys depends on the ageing time. So, the hysteresis value was 65, 100, 185 and 92 K for the alloys 1, 2, 3 and 4, and the tetragonality of martensite for this alloys was equal to 1,15; 1,13 and 1,05, respectively. The martensite in these alloys is the thin plates with twins and straight borders that evidenced of high mobility of the latter and that there was no relaxation of the strains arising during the plates growth owing to the coherent connection of the austenite and martensite lattices. In austenite grains, the relaxation occurs because of the energy-advantageous directions of martensite plates growth and the formation of parallel plates. Smaller hysteresis of direct and inverse martensitic transformation corresponds to thinner plate martensite. Also, the martensite relief on the preliminary polished specimens becomes finer, that evidenced about the smaller form deformation under the transformation while the volume changes are approximately equal. Thus, the increasing the degree of tetragonality (/) of the martensite crystal lattice resulted in the narrowing hysteresis of direct and inverse martensitic transformation in Fe-Ni-Co-Ti alloys.

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29 Aug
11:50am
Amphi 1

Direct observation of a/b twin laminate and its evolution under stress in 10M martensite exhibiting magnetic shape memory effect

Heczko O.

Institute of Physics, Department of Magnetic Measurements and Materials, CZECH REPUBLIC

High mobility of twin boundary aka extremely low twinning stress is, together with anisotropic (ferro-)magnetic properties, basic condition for magnetically induced reorientation, one of the magnetic shape memory effects. In 10M modulated martensite the mobile twin boundaries providing the reorientation are of monoclinic Type I and II. These boundaries differ not only in magnitude of twinning stress but also in temperature dependence of it. The most mobile twin boundary is of a-c Type II boundary in 10M martensite, which can exhibit the twinning stress less than 0.1 MPa.
Although the origin for extreme mobility is not known, one of the reasons is expected to be the complex hierarchy of twinning systems. Based on experimental investigation of structure and microstructure by X-ray diffraction the complex twin hierarchy was suggested from theoretical model based on elastic continuum theory. Suggested hierarchy was partially identified by direct observation but observation of whole hierarchy is still missing. Optical microscopy using Nomarski contrast and polarization contrast was instrumental in the identification of the mobile a/c twin boundary and monoclinic twinning and the evolution of a-c and monoclinic twinning under stress was also studied. The limited resolution of optical microscopy however, does not allow finding expected a/b twin laminate. Also the usual EBSD fails in identification of a/b laminate due to small difference between lattice constants and minuscule orientation tilt in this type of twinning.
However, recently we demonstrate that submicron a/b laminate can be directly visualized using backscattered electrons in SEM due to channelling contrast. This observation opens new venue for studying full hierarchy of twinning and comparison with a model. It makes possible the investigation of the evolution

of the twinned structure under stress and magnetic field. We demonstrate that in the vicinity of mobile a-c twin boundary, the a-b laminate is of submicron size and its width varies with temperature. In agreement with the model the a-b laminate terminated straight on Type I and by branching on Type II twin boundary.
In extension to static observation in our search to reveal the reason behind high mobility of twin boundaries we present the study of the evolution of twin microstructure and mutual interaction between various twin types under mechanical stress using combined optical and electron scanning microscopy. Moreover, the previously published results are critically discussed and reinterpreted in the view of current knowledge.

Premartensite in $Ni_2MnAl_{0.5}Ga_0.5$

30 Aug
9:40am
Amphi 1

Leitner M.[1], Neibecker P.[1], Schmidbauer J.[2], Petry W.[1,2]

[1] *Heinz Maier-Leibnitz Zentrum (MLZ) / FRM II, Technische Universität München, GERMANY;*
[2] *Physik-Department, Technische Universität München, GERMANY*

In many systems having martensitic transitions, which includes the prototypical examples NiTi, AuCd and NiAl, a range of materials properties display anomalies above the transition temperature, which are known as premartensitic effects. Specifically for the ferromagnetic shape-memory system Ni_2MnGa, a well-defined premartensitic phase exists, located between the minimum of the soft phonon frequency at 260 K and the transition to the proper martensite state at 220 K in the stoichiometric case [1]. The corresponding structure was identified in terms of a transversal shifting of (110)-planes of the underlying bcc lattice with a six-fold repetition, giving an orthorhombic symmetry, while the primary lattice reflections retain cubic symmetry [2]. Later, it has been reported that, just as with the martensitic structures, also the premartensitic structure should rather be considered as incommensurate [3]. Still, the mechanism of stabilization of the premartensitic phase is not settled.
In the present study, we investigate the low-temperature structural behaviour of $Ni_2MnAl_0.5Ga_0.5$ by neutron powder diffraction. In contrast to Ni_2MnGa, which always shows $L2_1$-order, and Ni_2MnAl, where the formation of $L2_1$ from the disordered B2 is very sluggish, intermediate compositions allow to adjust the state of order by the annealing treatment [4]. While in the as-quenched state the austenite is stable down to 10 K, annealing at 623 K leads to the emergence of a modulated structure that is equivalent to the premartensitic structure of Ni_2MnGa, with a transition temperature to the austenite at around 150 K after 3 h annealing, and at 210 K after 10 d annealing. Most importantly, in neither case a further transition to a proper martensite is found. Our results underline the role of ordering in determining the stability of the martensite, which previously had been discussed primarily in terms of composition, while the large region of stability has implications also for the technologically relevant Ni_2MnGa case.
[1] A Zheludev et al., Phys. Rev. B 51, 11310 (1995)
[2] PJ Brown et al., J Phys Condens Matter 14, 10159 (2002)
[3] S Singh et al., J Phys Condens Matter 25, 212203 (2013)
[4] H Ishikawa et al., Acta Mat 56, 4789 (2008)

Thermal-, magnetic-field- and stress-induced transformation in Co_2Cr(Al, Si) Heusler-type shape memory alloys

29 Aug
5:30pm
Amphi 1

Odaira T.[1], Xu X.[1], Miyake A.[2], Omori T.[1], Tokunaga M.[2], Kainuma R.[1]

[1] *Department of Material Science, Graduate School of Engineering, Tohoku University, JAPAN;*
[2] *The Institute for Solid State Physics, The University of Tokyo, JAPAN;*

Recently, our research group has reported new Co-Cr-Ga-Si Heusler alloys showing martensitic transformations [1]. One of the characteristic feature is the re-entrant martensitic transformation, in which the martensite phase induced from the paramagnetic parent phase by cooling transforms to the ferromagnetic parent phase by further cooling. Moreover, the Co-Cr-Ga-Si alloys exhibit a cooling-induced shape memory effect, as well as a heating-induced shape memory effect, and superelasticity with inverse temperature dependence of critical stress [1]. In addition, due to the high Cr content, Co-Cr-based shape memory alloys may be attractive as high corrosion-resistant shape memory alloy for biomaterial applications. However, the cost of this alloy system is relatively high because of a large amount of Ga. Very recently, inexpensive Co-Cr-Al-Si alloys have been found to show the $L2_1/D0_{22}$ martensitic transformation and superelasticity [2]. However, the composition dependence of the martensitic transformation temperatures and Curie temperature have not been determined. In this study, these transformation temperatures were measured in Co-Cr-Al-Si alloys. Moreover, temperature dependence of superelastic behavior and the magnetic-field-induced transformation were investigated. The phase diagram, including the martensitic and magnetic transformations, was determined by thermoanalysis and thermomagnetization measurements in the $Co_xCr_{79-x}Al_{10.5}Si_{10.5}$ section. It was found that the martensitic transformation is strongly suppressed when the parent phase is ferromagnetic due to the magnetic effect [3] and that the martensitic transformation is obtained only from paramagnetic parent phase. By magnetization measurements under pulsed magnetic fields, magnetic field-induced reverse martensitic transformation was conformed at room temperature. Superelastic behavior was detected in the temperature range of 198 to 423 K for a pseudo-single crystal obtained by the cyclic heat treatment [4]. The critical stress of the martensitic transformation was found to show a negative temperature dependence at temperatures lower than approximately 310 K due to the magnetic effect, in contrast to the normal temperature dependence at higher temperatures.

[1] X. Xu et.al: Appl. Phys. Lett. 103 (2013) 164104
[2] K. Hirata et.al: J. Alloys Comp. 642 (2015) 200-203
[3] X. Xu et.al: Phys. Rev. B 91 (2015) 104434
[4] T. Omori et.al: Science 341 6153 (2013) 1500-1502

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30 Aug
9:00am
Amphi 1

Microstructural dependences of the multi-ferroic transformation in Ni-Co-Mn-In metamagnetic shape memory alloys

Salas D.[1], Eliseeva O.[1], Wang Y.[1], Duong T[1], Ren Y.[2], Arróyave R.[1], Karaman I.[1], Chumlyakov Y.[3]

[1]Department of Materials Science and Engineering, Texas A& M University, USA;
[2] X-ray Science Division, Advance Photon Source, Argonne National Laboratory, USA;
[3]Siberian Physical-Technical Institute, Tomsk State University, RUSSIAN FEDERATION.

Ni-Co-Mn-In Meta-Magnetic Shape Memory Alloys (MMSMAs) undergo a martensitic transformation (ferroelastic transition) which is coupled with a strong decrease in the magnetic ordering from a ferromagnetic parent phase to a weak-magnetic product phase (ferromagnetic transition). The properties of this multi-ferroic transformation (MFT) have been shown to depend on composition, thermal history (including atomic ordering) and external applied stress and magnetic fields lending many tuning options for advance applications. It is well-known that many of these MFT properties, like transformation hysteresis and magnetization change which are crucial for the magneto-caloric effect, depend on the magnetic contributions to the thermodynamic energy balance between austenite and martensite. As consequence, these properties will be function of the difference between the characteristic temperatures of the ferromagnetic transition in the austenite, Tc, and of the multi-ferroic transformation, like Ms. However, the dependence of the Tc-Ms difference on thermal history is complex and not well understood. In particular, MFT Ms temperature presents unexpected non-monotonic dependence on the duration of thermal treatments when annealing below the order-disorder transition temperature. We propose that this behavior is linked to the evolution of microstructural features modifying the energy barriers to the transformation. The goal of the present work is to investigate the evolution of the atomic ordering domains in order to unravel the dependence of the MFT on the structural and microstructural changes occurring during the B2-$L2_1$ ordering transition. With this objective, different Ni-Co-Mn-In samples have been investigated by transmission x-ray diffraction using high energy synchrotron radiation. The evolution of materials structure have been obtained during in-situ experiments simulating thermal treatments conditions and for pre-heat treated samples for revealing the final state after quenching. These experiments permitted us to understand the evolution of overall degree of $L2_1$ ordering, average size of the ordering domains and lattice strains. Results show fast kinetics for the change in overall degree of $L2_1$ ordering but slow logarithmic kinetics for ordering domain coarsening which can be related to previous observations on the time-evolution of MFT properties.

Influence of defects on the martensitic transformation and the magnetic properties of Co-doped Ni-Mn-Sn and Ni-Mn-In metamagnetic shape memory alloys

29 Aug
5:10pm
Amphi 1

Sánchez-Alarcos V.[1], López-García J.[1,2], Unzueta I.[3,4], Pérez-Landazábal J.I.[1], Recarte V.[1], Beato-López J. J.[1], García J. A.[4]

[1] *Departamento de Física. Universidad Pública de Navarra, Institute for Advanced Materials (INAMAT), SPAIN;* [2] *Institut Laue Langevin, FRANCE;*
[3] *Elektrizitate eta Elektronika Saila, Euskal Herriko Unibersitatea UPV/EHU, SPAIN;*
[4] *BC Materials (Basque Centre for Materials, Applications and Nanostructures), SPAIN;*

The effect of microstructural defects on the magnetostructural properties has been analyzed in Ni-Co-Mn-Sn and Ni-Co-Mn-In alloys subjected to soft milling and subsequent annealing treatments. A huge stabilization of the martensite (shifts of the reverse martensitic transformation temperature larger than 100 K) is observed in the milled Ni-Mn-In-Co alloy whereas no stabilization at all is detected in the Ni-Mn-Sn-Co alloy, thus pointing out a higher amount of mechanically-induced defects interacting with the moving interphases in the former. The presence of defects degrades the magnetic properties of both austenite and martensite in the In-containing alloy but, surprisingly, a very different behavior is observed depending on whether the alloy was milled in austenitic or in martensitic phase. In the Ni-Mn-Sn-Co alloy, in turn, the presence of microstructural defects, far for worsening, it is found to make the magnetocaloric effect to be higher in the as-milled state than after subsequent annealing. This unusual beneficial presence of defects can be explained as a result of the combination of the effect Co on the magnetic exchange coupling between Mn atoms, the effect of defects on the Mn-Mn distance and the effect of defects on the vibrational entropy change at the transformation. The presented results show that the induction of microstructural defects can be an effective way to enhance the multifunctional properties of these alloys.

Magnetostriction in 10M Ni-Mn-Ga martensite

29 Aug
10:50am
Amphi 1

Saren A.[1], Sozinov A.[1], Musiienko D.[1], Kustov S.[2], Ullakko K.[1]

[1] *Lappeenranta University of Technology, FINLAND;* [2] *University of the Balearic Islands, SPAIN*

Investigation of magnetostriction in magnetic shape memory (MSM) alloy Ni Mn Ga has been attracting big attention due to so called "magnetoelastic model" [1], according to which the magnetostrictive deformation triggers the twinning detwinning process, and so, magnetoelastic interaction is the physical origin of the magnetically induced reorientation (MIR) of twinned martensitic variants observed in Ni-Mn-Ga alloys. On the other hand, in the literature there are only few experimental studies related to the magnetostriction measurements in Ni Mn-Ga 10M modulated martensites, which contradict each other [2-5]. The obvious reason for this is hindering of magnetostriction by much stronger pronounced (MIR). In addition, no direct observation of the lattice deformation was reported so far. In our study, we measured magnetostriction in 10M Ni-Mn-Ga martensite, in single variant unconstrained samples, at room temperature. A special setup was utilized to measure the sample's strain under a pulsed magnetic field application, with a non contact optical measurement technique. The magnetostriction was found to be negligible (less than 5 ppm) when magnetic field was applied along the easy c-axis. For the hard a(or b)-axis direction, we obtained more than 500 ppm contraction at saturation field which is comparable with such magnetostrictive materials like galfenol. To prevent the easy variant nucleation/growing in the latter case we used a special sample preparation and mounting procedure also avoiding any constrain related effects Thus, we conclude that the observed large magnetostriction is not related to the a-c twin boundary motion, i.e. no MIR occurs. Due to the large observed magnetostriction values for the hard a axis magnetization, we conducted X-Ray diffraction (XRD) measurements on single crystal samples under saturation field applied along the hard axis and without field. The XRD measurements were performed using PANalytical Empyrean diffractometer with Co tube (λ=0.17890 nm) and high resolution optics included hybrid monochromator and PIXcel3D-Medipix3 detector. Using high resolution reciprocal lattice mapping we studied set of peaks {620} most sensitive to lattice parameter change due to high two theta diffraction angles and also recorded intensity change of other peaks including satellites which are related to lattice modulation. Based on the XRD data, the possible origins for the observed large magnetostriction are considered, in terms of field induced redistribution of the a-b domains, modulation domains, and change of lattice modulation amplitude.

[1] V.A. Chernenko V.A. L'vov (2008) Mat Sci Forum 583:1

[2] R. Tickle and R.D. James (1999) J.Magn.Magn.Mat. 195:627
[3] O. Heczko (2005) J.Magn.Magn.Mat. 290-291:846
[4] J. M. Barandiarn et al (2012) Appl.Phys.Lett. 100:262410
[5] T. Sakon et al. (2017) Metals 7:410

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29 Aug
12:10am
Amphi 1

X-Ray DVC monitoring of martensitic reorientation in a Ni_2MnGa single crystal

Sevestre O., Bouterf A., Smaniotto B., Hubert O.

Laboratoire de Mécanique et et Technologie, Ecole Normale Supérieure Paris-Saclay, FRANCE

Our time is highly characterized by the use of increasingly widespread electromagnetic actuators, from largest to smallest scales. The development of efficient micro-actuators through the development of new materials is relevant, using for example the pseudo-elasticity of Shape Memory Alloys (SMA). SMA belong to a family of materials whose behavior is characterized by an austenite-martensite phase transformation. This transformation can be activated by heating or cooling and/or mechanical stress. Among the SMA, Magnetic Shape Memory Alloys (MSMA) are furthermore ferromagnetic. Under the action of a magnetic field, through the existence of a magnetostatic coupling as well as a magneto-mechanical coupling, a phase transformation and/or a reorientation of martensite variants can occur. The behavior of these alloys, dependent of the amount of stress, temperature and/or magnetic field applied, is difficult to observe and model.A mechanical test on this type of material is often difficult to achieve given the low volume generally available, and the brittle behavior of these materials generally exhibiting an ordered metallic structure. These tests are however interesting because they make it possible to develop a martensitic reorientation more easily and in a more controlled way than under the action of a magnetic field. The difficulty lies in the measurement of kinematic quantities. In this work, a three points bending test has been realized on a Ni2MnGa single crystal inside an X-ray tomograph. As the structure of the alloy is perfect, it shows no X-ray contrast. Therefore tungsten particles have been sprayed on the surfaces. Using this new contrast, and after reconstruction of the volume at each loading level, the displacement field between levels has been calculated by Digital Volume Correlation (DVC). The measurement highlights a clear variant reorientation in relation with the macroscopic force/displacement curve. The martensitic reorientation threshold has been consequently precisely estimated.

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29 Aug
4:00pm
Amphi 1

Tailoring the mobility of compound-type twin-boundaries in magnetic shape memory alloys

Soroka A.[1], Sozinov A.[1], Lanska N.[1], Rameš M.[2], Straka L.[2,3], Ullakko K.[1]

[1]*Lappeenranta University of Technology, Material Physics Laboratory, FINLAND;*
[2]*Institute of Physics, Academy of Sciences of the Czech Republic, CZECH REPUBLIC;*
[3]*Faculty of Mathematics and Physics, Charles University in Prague, CZECH REPUBLIC.*

Magnetic shape memory (MSM) alloys with low twinning stress (σTW) and high maximum magnetic equivalent stress (σMAG) are of high scientific as well as practical interest for actuators, sensors, energy harvesters, micro-pumps and other applications. Currently important task is development of high-temperature MSM materials, which satisfy the MSM-requirement σTW< σMAG with good marginal, enabling the giant magnetic field-induced strain (MFIS) with high efficiency in broad operating temperature range. In Ni-Mn-Ga-based systems the alloys possessing non-modulated (NM) martensite structure with lattice tetragonality ratio c/a¿1 exhibit the higher martensite-austenite transformation temperatures (TA) than the modulated 10M and 14M martensites. We studied a possibility to decrease the σTW of mobile compound-type twin boundaries (CTB) in single-crystals of NM $Ni_{50-x}Mn_{25-y}Ga_{25-z}Co_xCu_{y+z}$ MSM-alloys, by varying measurement temperature and composition (9 alloys). We observed that in each alloy the σTW decreases with increasing temperature and reaches lowest value near the alloy-specific TA. We found that the rate with which the σTW decreases is significantly composition dependent and varies between -0.008 and -0.018 MPa/K. For comparison, in ternary NM Ni-Mn-Ga alloys the CTBs have considerably more severe temperature dependence of -0.04 MPa/K, which is same value as for the TBs type I in 10M Ni-Mn-Ga alloys. Thus, by Co and Cu alloying of the Ni-Mn-Ga base alloy we have succeeded to decrease the temperature dependence of σTW in NM martensite by up to 5 times, which enables MSM-actuation in considerably broader temperature range. This new highly interesting finding opens up new paths for tailoring the thermo-mechanical properties of MSM-alloys. By more detailed analysing of the alloys with different compositions we discovered that the magnitude of σTW is clearly proportional to the c/a ratio, when σTW and c/a are measured just below TA of each alloy. Lowering the c/a ratio by change of composition results in decrease of the σTW with slope 14 MPa/1. Important conclusion following from this finding is, that we can expect a very low twinning stress, σTW¡0.1MPa, and high actuation efficiency by MSM-effect, in the NM Ni-Mn-Ga-Co-Cu-based alloys possessing c/a$\simeq$1.085-1.104. To our knowledge, this is the first reported prediction that also the non-modulated MSM-alloys could in future achieve high actuation efficiencies and become competitive or perhaps even superior to the existing electrostrictive and magnetostrictive actuation materials.

Structure and magneto-mechanical properties of Ni-Mn-Ga-Co-Cu melt-spun ribbons

30 Aug
10:00am
Amphi 1

Szczerba M. J.[1], Maziarz W.[1], Brzoza A.[1], Wierzbicka-Miernik A.[1], Wójcik A.[1], Kowalczyk M.[2], Wojewoda-Budka J.[1], Sikora M. [3]

[1]*Institute of Metallurgy and Materials Science PAS POLAND;*
[2]*Faculty of Materials Science and Engineering, Warsaw University of Technology, POLAND;*
[3]*Academic Centre for Materials and Nanotechnology, AGH UST, POLAND.*

The Ni-Mn-Ga Heusler alloys are a subject of intense research due to promising magneto-mechanical properties which may find practical use in actuation and sensor devices. Recently, chemical modification of this alloy, by minor addition of Co and Cu, further expands these unique functional properties to non-modulated martensite structures of Ni-Mn-based single crystalline materials. Thus, in view of this recent progress, we study the possibility of triggering magnetic field-induced strain in $Ni_{45}Mn_{25}Ga_{20}Co_5Cu_5$ alloys fabricated by conventional melt-spinning technique. The microstructure of these ribbons turned out to be highly anisotropic evolving from small equal-axial grains at the wheel side, through columnar grains in the middle and finally to dendritic one at the free side. Such strong microstructural anisotropy, has been also reflected in the phase composition. At the wheel side, a dual-phase microstructure composed of non-modulated martensite and a high temperature $L2_1$ austenite phase occurs. Further from the wheel side, the austenite phase gradually disappears and a single non-modulated martensite is present at the free side where the grain size is substantially larger. As the chemical composition along the cross-section, of ribbons is constant, the observed stabilization of austenite should be ascribed in this case to grain size reduction observed when approaching the wheel side of ribbons. Transmission electron microscopy observations, apart from the twinned microstructure of the non-modulated martensite phase, have revealed large amount of dislocations within the grain interior. The grain boundaries, on the other hand, are free of precipitates. Calorimetric measurements have revealed two fundamentally different stages of heat treatment. The first stage of heat treatment consisting of heating up to 560°C triggers thermally activated annihilation of dislocations and other atomic ordering effects which significantly increases magnetic properties, e.g. magnetic susceptibility. Interestingly, up to 560°C no grain growth occurs. Substantial grain refinement is observed during the second stage of heat treatment performed up to 900°C. The microstructure evolution, however, takes place at the very beginning stage of heating, i.e. after 10 minutes at 900°C. Further heating at this temperature up to 180 minutes does not affect the microstructure. Interestingly, the observed grain growth also does not influence magnetic susceptibility which seem to be affect by atomic ordering and defects annihilation only. The applied mechanical training, consisting of a series of bending experiments, significantly refined the microstructure which changed the magneto-mechanical response of both the as-grown and heat-treated NiMnGaCoCu melt-spun ribbons.

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30 Aug
9:20am
Amphi 1

NiMnGaFe alloys polycrystalline samples: modulation of gamma-phase and related functional properties

Villa E.[1], Nespoli A.[1], Passaretti F.[1], Pallecchi I.[2]

[1] *CNR ICMATE Unit of Lecco, ITALY;*
[2] *Università degli Studi di Genova, Dipartimento di Fisica, ITALY.*

The NiMnGaFe alloys, with Fe addition in substitution of Ni or Mn in the NiMnGa system, were considered in the last years to obtain improvement of the functional properties, particularly in the magnetic response and in the ductility of the material. Generally when the Fe content in the alloy overcomes the 10 at% , there is a formation of secondary phase denominated gamma-phase, particularly rich in Fe content. In literature there are different works about magnetic performances and cycling stability of these alloys, but only a preliminary investigation of mechanical properties which are very important in the use of these materials like magnetic actuators. Moreover the presence of the gamma-phase could be one way to solve the high brittleness of the polycrystalline samples. In our study two series of polycrystalline NiMnGaFe alloys were prepared and thermal treated to obtain different distribution of gamma-phase in the material. The microstructural conditions were investigated by calorimetric and microscopic analysis. The influence of gamma-phase in the mechanical performance was characterized by dynamic and mechanical measurements in flexion and compression configuration at different temperatures. Furthermore some preliminary investigation of magnetoresistance has been carried out. The gamma-phase generally reduces the brittleness of the material and modifies the pseudoelastic and shape recovery properties of the alloys. The best condition has been reached when the gamma-phase is localized at grain boundaries.

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29 Aug
11:10am
Amphi 1

Martensitic transformation in <110>A oriented Ni-Mn-Ga thin films

Yang B.[1], Li Z.[1], Yan H.[1], Zhang Y.[2,3], Esling C.[2,3], Zhao X.[1], Liang Z.[1]

[1] *Key Laboratory for Anisotropy and Texture of Materials, Northeastern University, CHINA;*
[2] *LEM3, CNRS UMR 7239, Université de Lorraine, FRANCE;*
[3] *Labex DAMAS "Design of Alloy Metals for low-mAss Structures", Université de Lorraine, FRANCE*

Ni-Mn-Ga ferromagnetic shape memory alloys exhibit multifunctional behavior such as giant magnetic field induced strain, magentocaloric effect and so on, owing to the twin variant reorientation under external magnetic field. To date, giant magnetic field induced strain as much as 6-12% has been achieved in the Ni-Mn-Ga bulk single crystals. In recent years, epitaxial Ni-Mn-Ga thin films are conceived as promising sensor and actuator materials in the magnetic micro-electro-mechanical systems and have been extensively investigated. Although the magnetic field induced variant reorientation has been observed in the Ni-Mn-Ga thin films epitaxial on MgO (100) substrate, large magnetic field induced strain is still not observed, owing to the martensite microstructure of <001>A oriented Ni-Mn-Ga thin films are complicated [1, 2]. In 2007, G. Jakob et.al proposed that the <110>A oriented Ni_2MnGa thin films grown on (110) sapphire substrate (Al_2O_3) [3]. For <110>A oriented films on Al_2O_3, the martensitic phase is significantly more stable than for the <100>A oriented films on MgO. However, the martensitic transformation temperature is 260-280 K, which indicates that the Ni2MnGa thin film are of Austenite at ambient temperature. The magnetic properties, microstructure and crystal structure of martensite in <110>A oriented Ni-Mn-Ga thin films have not been revealed. In the present work, the <110>A oriented Ni-Mn-Ga films with different thickness was prepared on MgO (111) and Al_2O_3 (110) substrates by magnetron sputtering. X-ray diffraction results showed that all the films are of 7M martensite in the ambient temperature. Transmission electron microscopy characterization revealed that the twin interfaces of 7M martenstie are parallel to the substrate surface. Finally the magnetic-field-induced martensitic variant reorientation were observed in the Ni-Mn-Ga films with a thickness of 200 nm.

[1] P. Ranzieri, M. Campanini, S. Fabbrici, et al. Adv Mater 2015;27:4760-4766.
[2] B. Yang, Y. D. Zhang, Z. B. Li, et al. Acta Mater 2015;93:205-17.
[3] G. Jakob, T. Eichhorn, M. Kallmayer, et al. Phys Rev B 2007;76: 174407.

Elastic properties of pure and Co- Cu- doped non-modulated Ni-Mn-Ga martensite

29 Aug
2:00pm
Poster
Session C

Bodnarova L.[1], Sedlák P.[1], Seiner H.[1], Zelený M.[2], Straka L.[3], Heczko O.[3], Sozinov A.[4], Landa M.[1]

[1] *Institute of Thermomechanics, CAS, CZECH REPUBLIC;*
[2] *Institute of Materials Science and Engineering, NETME Centre, Brno university of Technology, CZECH REPUBLIC;*
[3] *Institute of Physics, CAS, CZECH REPUBLIC;*
[4] *Lappeenranta University of Technology, Material Physic Laboratory, FINLAND.*

The Ni-Mn-Ga magnetic shape memory alloy belongs among the most intensively studied material systems nowadays. A particular attention is dedicated the modulated structures of Ni-Mn-Ga, as these structures exhibit extremely high mobility of twin boundaries. According to the adaptive concept of martensite, the modulated phase can be represented by a laminate of basic units of non-modulated martensite (NM), and so the elastic properties of modulated structures are supposed to be predictable from NM coefficients. A remarkable influence of Co- and Cu- doping on magnetic and mechanical properties of NM martensite has been reported. With these additions, NM martensite exhibits highly mobile twin boundaries comparable with those in 10 M martensite, and the Co- and Cu- doped alloys are supposed to be the first step to designing new high-temperature magnetic SMAs with wider operating temperature interval. This work will present the room temperature elastic coefficients of tetragonal NM martensites of pure and Co and Cu doped Ni-Mn-Ga alloys determined using ultrasonic methods. The complete tensor of elastic coefficients was obtained from resonant ultrasound spectroscopy (RUS) measurement complemented by pulse-echo longitudinal waves velocities (measured in the directions perpendicular to the sample faces). Two NiMnGaCoCu alloys with slightly different compositions were examined, both having a tetragonal crystal structure with the c/a ratio equal to 1.14 (i.e. significantly lower than for pure Ni-Mn-Ga NM, where c/a=1.21). The design of the used specimens allowed switching between two different tetragonal c-axis orientations, which significantly improved the accuracy and reliability of the resulting elastic constants. It was observed that the Co and Cu additives have a significant influence on material elasticity. The material remains non-modulated and tetragonal, but the doping effectively decreases the elastic anisotropy. Even more importantly, the orientation of soft shearing planes changes with the doping. This evolution of elasticity with Co and Cu additions is in a perfect agreement with predictions of ab-initio calculations, that also reproduce well the related decrease in the c/a ratio.

Magnetic noise emitted during martensitic transformation in $Ni_{49}Fe_{18}Ga_{27}Co_6$ single crystals

29 Aug
2:00pm
Poster
Session C

Bolgár M. K.[1], Daróczi L.[1], Tóth L. Z.[1], Timofeeva E. E.[2], Panchenko E. Y.[2], Chumlyakov Y. I.[2], Beke D. L.[1]

[1] *Department of Solid State Physics, University of Debrecen HUNGARY;*
[2] *Tomsk State University, Siberian Physical Technical Institute, RUSSIAN FEDERATION*

Emitted magnetic noises were measured in single crystalline $Ni_{49}Fe_{18}Ga_{27}Co_6$ (at%) ferromagnetic shape memory alloys during austenite-martensite first order phase transformation driven by heating and cooling. In this alloy the magnetization of the martensite phase is lower than that of the austenite phase, thus during the transformation the magnetization undergoes an abrupt change, similar to metamagnetic transformation. In my presentation I will first show that the statistical characteristics of the emitted magnetic noise are different from those measured in ferromagnetic Ni_2MnGa single crystals (magnetic emission was measured only in this alloy so far): i) the critical exponents of the noise distributions and the scaling law are different, ii) in contrast to Ni_2MnGa, the corresponding critical exponents of the acoustic [1,2] and magnetic noises are different. These differences, besides the difference in the mode of phase transformation (single interface transformation in homogeneous $Ni_{49}Fe_{18}Ga_{27}Co_6$ sample and formation of many needles in Ni_2MnGa) are most probably also related to the fact that the change in the magnetic properties of the austenite and martensite is different in the two alloys compared. Secondly an interesting effect of the presence of the γ-precipitates will be demonstrated. The presence of nano-sized γ-particles increased the noise activity and resulted in bipolar magnetic noise in contrast to mostly unipolar signals observed in homogeneous sample. Application of moderate external magnetic field (up to 34 mT) decreased the bipolar nature of the noise. The presence of nano-sized precipitates resulted in more fine steps of the jerky motion of the interface and caused barriers against of easy development of the magnetic structure of the ferromagnetic austenite. For understanding the details of the effect of the external magnetic field further investigations are necessary.

[1] M. K. Bolgár, L. Z. Tóth, S. Szabó, Sz. Gyöngyösi, L. Daróczi, E. Y. Panchenko , Y. I. Chumlyakov, and D. L. Beke, Journal of Alloys and Compounds 658, (2016) 29.
[2] M. K. Bolgár , L. Daróczi, L. Z. Tóth, E. E. Timofeeva, E. Y. Panchenko, Y. I. Chumlyakov, D. L. Beke, Journal of Alloys and Compounds, 705 (2017) 840.

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29 Aug
2:00pm
Poster
Session C

Influence of Cu addition on crystal structure and martensitic transformation in Heusler $Ni_{50}Mn_{25}Ga_{25-x}Cu_x$ alloys

Brzoza A.[1], Czaja P.[1], Czeppe T.[1], Wierzbicka-Miernik A.[1], Wójcik A.[1], Wojewoda-Budka J.[1], Szczerba M. J.[1], Cesari E.[2]

[1]*Institute of Metallurgy and Materials Science, Polish Academy of Science POLAND;*
[2]*Department de Fisica, Universitat de les Illes Balears, SPAIN*

Ni-Mn-Ga Heusler alloys have attracted large interest over past few decades due to their functional properties including magnetic field-induced strain effect. It is well-know that the low operating temperature and brittleness remain the major drawbacks for industrial applications of these alloys. The addition of fourth element such as copper to the ternary Ni-Mn-Ga system could be used for improving ductility and increasing operating temperature. The series of polycrystalline alloys of nominal composition $Ni_{50}Mn_{25}Ga_{25-x}Cu_x$ (x=1-10 at.%) were fabricated from high purity elements using conventional arc-melting method and thereafter re-melted four times under argon protective atmosphere. Subsequently, the samples were encapsulated at vacuum condition, homogenized at 1173 K for 48h and then slowly cooled with furnace to ambient temperature. Then the samples were cut into two piece. One part was again encapsulated in quartz ampoules heated at 1173 K for 30 minutes and then water quenched. The final chemical composition of alloys were checked by an energy-dispersive spectrometer attached to a scanning electron microscope. Generally, a strong composition dependence of the type of martensite structure has been observed. At ambient temperature, four types of crystal structure were detected: $L2_1$ austenite and 5-layered modulated, 7-layered modulated and non-modulated martensite. The substitution of Ga for Cu in the $Ni_{50}Mn_{25}Ga_{25-x}Cu_x$ system results in an increase of martensitic transformation temperature (TM) which in turn is proportional to the e/a ratio. Moreover, the quenching process has also influence on the martensitic transformation temperature and crystal structure shifting TM to lower temperatures and stabilizing the $L2_1$ austenite and 7-layered modulated martensite phases. Electron microscopy observations revealed that the Cu addition significantly affects the martensite microstructure. At higher Cu concentration, i.e. 9 and 10 at.%, a so-called γ phase precipitates of face-centred cubic crystal structure form being substantially enriched with Cu. This effect in turn decreases the e/a ratio of the martensite phase (as a matrix of the dual-phase microstructure) and consequently decreases the TM.

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29 Aug
2:00pm
Poster
Session C

Orthogonal shear process in Ni-Mn-Ga and Ni-Mn-Sn single crystals

Chulist R.[1], Tokarski T.[2], Chumlyakov Y. I.[3], Czaja P.[1]

[1]*Institute of Metallurgy and Materials Science, Polish Academy of Sciences, POLAND;*
[2]*AGH University of Science and Technology, Faculty of Physics and Applied Computer Science, Department of Solid State Physics, POLAND;*
[3]*Siberian Physical-Technical Institute, RUSSIAN FEDERATION*

Martensitic transformation results in several martensitic variants the number of which heavily depends on the symmetry change. Adjusting the structure of single crystalline Ni-Mn-Ga or Ni-Mn-Sn alloys by magneto-mechanical treatments allow obtaining a well-defined microstructure i.e. to reduce the number of variants and to decrease the twinning stress. The training process, as the last step in the preparation procedure of Ni-Mn-Ga alloys consists of multi-axis compression and is necessary to obtain magnetic field-induced strain in Ni-Mn-Ga single crystals. In the first stage of deformation, the variant reorganization is strongly dominated by the so-called Muellner-King mechanism (or orthogonal shear process). This process plays an essential role in progress towards the single variant state. The coordinate change of conjugate twin plane affects the other configuration of variants leading to the removal of conjugation boundary by coordinated secondary twinning and reorganization of neighboring areas. The given mechanism appears to be fully controlled by martensite crystallography being fully consistent with the distribution of martensitic variants. The orthogonal shear process or the opposite process which may operate during martensitic transformation is also shown for NiMnSn single crystals. It strongly indicates that this process is general and cannot be limited only to Ni-Mn-Ga alloys.

Investigation of magnetic and acoustic emission during stress induced superplastic deformation of martensitic Ni_2MnGa single crystal

29 Aug
2:00pm
Poster
Session C

Daróczi L., Hudák A., Tóth L. Z., Beke D. L.

Department of Solid State Physics, University of Debrecen, HUNGARY

Since it's discovery the magnetic shape memory effect (MSM) is one of the most widely investigated phenomena because of it's outstanding practical and theoretical importance. The prototype of the ferromagnetic shape memory materials is the Ni_2MnGa alloy. In these materials martensitic transformation can be observed and the magnetic field induced variant rearrangement in the martensitic phase is the origin of the magnetic shape memory effect. In martensitic phase a duplex domain structure can be observed: the ferromagnetic domains and the martensite variants. The high magnetic anisotropy energy results in simultaneous changes of both structures during the magnetization process and the evoked rearrangement of the martensite variants leads to a large shape change of the sample. The movement of the domain walls as well as of the variant (twin) boundaries is a discontinuous process resulting magnetic and acoustic noises. In this work the noisy character of the superplastic deformation, induced by application of uniaxial compressive stress, in martensitic Ni_2MnGa single crystalline sample was investigated by detecting the emitted acoustic and magnetic noise signals (AE and ME, respectively). Note, that in [1] the AE and ME was investigated in the bar-shaped sample, magnetized and elongated perpendicularly to the magnetic field, due to the well known magnetic shape memory effect. In the present study the initially elongated sample was compressed producing superplastic deformation. The compression was performed by stress controlled (soft) driving using an electromechanical system. During this process acoustic and magnetic emission signals were generated by the jerky twin boundary motions. The magnetic and acoustic noises were detected simultaneously and statistical analysis was performed on the recorded signals. The dependence of the shape and the statistical characteristics (exponents of noise distributions) of the noise signal on the external magnetic field was also investigated. During the deformation process two noise packets was observed in both types of signals. The separation of these packets shows increasing tendency with increasing external magnetic field is explained by the effect of the external field on the possible variant rearrangements.

[1] L. Daróczi, E. Piros, L. Z. Tóth, D.L. Beke, Phys. Rev. B96 (2017) 014416

Martensitic transition in Fe-doped Ni_2MnGa

29 Aug
2:00pm
Poster
Session C

Kopecky V., Rameš M., Heczko O.

Institute of Physics of the Czech Academy of Sciences, CZECH REPUBLIC

Magnetic shape memory (MSM) effect includes several effects and the most promising one is called magnetically induced reorientation (MIR) which provides a large strain (up to 12 %) in medium magnetic field below 1 T. The strain is product of microstructure reorientation which takes place by twin boundary motion. The MIR effect is possible only in low-symmetry martensite, therefore, an existence of diffusionless martensitic transition from cubic austenite to martensite is for the effect crucial.

It is anticipated that the highest utilization of the MIR effect would be in automotive industry. Their applications, however, need quite high working temperatures that are out of range of the current MSM alloys. The best potential alloys for applications are slightly off-stoichiometric with Mn excess Ni_2MnGa alloys. Nowadays, it seems to be proven that changing the ratio of Ni/Mn/Ga is not sufficient to shift martensitic transition high enough to obtain ferromagnetic martensite with highly mobile twin boundaries at the desired working temperatures. Adding other elements into this alloy can be the way to reach higher temperature of martensitic and also ferromagnetic transformation.

Here we study the effect of Fe alloying. We prepared three Ni_2MnGa alloys doped with Fe using arc-melting furnace. For the sake of simplicity we used same doping amount 5 % of Fe. The alloys differ by the element which is substituted for, i.e. $(Ni_{45}Fe_5)Mn_{25}Ga_{25}$, $Ni_{50}(Mn_{20}Fe_5)Ga_{25}$ and $Ni_{50}Mn_{25}(Ga_{20}Fe_5)$. In order to determine transformation temperatures we used AC magnetic susceptibility and saturation

magnetization measurement that are usual methods to analyse the martensitic transitions in magnetic or even non-magnetic materials as the transition is driven by changes in electron structure. The transformation is detected as a step in magnetization and that can be measures in wide temperature range from 10 K to 1000 K.
In our presentation, we will discuss how the "position" of doped element or the substitution of given element affect the Curie and martensitic transformation temperatures. We show that highest transition temperature was achieved by substituting Ga. In addition, we will show effect of annealing and ageing on the transition temperatures.

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29 Aug
2:00pm
Poster
Session C

Martensitic transformation, structure and mechanical properties of the Mo doped NiCoMnIn magnetic shape memory alloys

Krystian P., Matyja E., Wąsik M., Zubko M.

University of Silesia, Institute of Materials Science, POLAND

NiCoMnIn alloys as magnetic shape memory alloys (MSMA) exhibit reversible martensitic transformation which may be driven either by temperature, external strain or magnetic field resulting in macroscopic shape change. Large recoverable strain and high frequency response make this materials promising magnetic actuators. Despite of many advantages NiCoMnIn alloys are very brittle. There are many strategies to improve ductility of ordered intermetallic crystal structures including: controlling of phase stability, eliminating environmental embrittlement, promoting transformation induced plasticity, structure and chemistry grain-boundary engineering. One of the most useful method is microstructural optimization which may be realized by thermal treatment and/or chemical composition selection (e.g. by appropriate elements additions). In the present work the influence of the Mo additions on the martensitic transformation, structure and mechanical properties of the NiCoMnIn MSMA alloys have been studied. Series of polycrystalline NiCoMnIn alloys containing from 0 to 5 mas.% of Mo were produced by the arc melting technique. To observe the microstructure and texture of alloys SEM and EBSD were applied. For the alloys containing Mo two phase microstructure were observed. Mo-rich precipitates were distributed randomly in the matrix. Relative volume fraction of the precipitates depends on the Mo content. The DSC measurements reviled that the molybdenum additions decreases (of about 60 K) the martensitic transformation temperatures. The structures of the phases were determined by the X-Ray and TEM. The mechanical properties of the matrix and precipitates were studied by tensile tests by micro and nanohardness.

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29 Aug
2:00pm
Poster
Session C

Effect of temperature and magnetic field induced martensitic transformation on electronic structure and physical properties of bulk $Fe_{45}Mn_{26}Ga_{29}$ alloy

Kudryavtsev Y. V.[1], Uvarov N. V.[1], Perekos A. E.[1], Dubowik J.[2], Kozlova L.E.[3]

[1]*Institute of Metal Physics, NAS of Ukraine, UKRAINE;*
[2] *Institute of Molecular Physics, PAS, POLAND;*
[3]*Institute of Magnetism, NAS of Ukraine, UKRAINE*

Effect of temperature and magnetic field induced martensitic transformation (MT) on the electronic structure and some physical properties of bulk $Fe_{45}Mn_{26}Ga_{29}$ Heusler alloy has been investigated. According to the results of DSC, magnetic and transport measurements direct and reverse martensitic transformation without external magnetic field takes place within $194 \leq T \leq 328$ K temperature range with a hysteresis of about Δ T $\simeq$ 90 - 100 K. External magnetic field of μ 0H = 5 T causes high temperature shift of MT temperatures and reduces temperature hysteresis width down to ΔT 60 K. MT from parent austenite $L2_1$ phase to martensitic tetragonally distorted $L2_1$ one causes lattice distortion (c - a)/a of the tetragonal phase by 34.5 %, volume increase by $\Delta V = 0.36$ %, significant changes in the electronic structure of alloy, drastic increase in alloy magnetization, decrease in alloy resistivity, changes the sign of the temperature coefficient of resistivity from negative to positive. At the same time optical properties of $Fe_{45}Mn_{26}Ga_{29}$ Heusler alloy in austenite and martensite states look visually rather similar being noticeable different in microscopic nature. Experimentally observed changes in physical properties of alloy are discussed in terms of the electronic structures of austenite and martensite phases.

Magnetic characteristics of polycristalline NiTi film

29 Aug
2:00pm
Poster
Session C

Kyianytsia A.[1,2], Chang C. S.[2], Cinar I.[3], Lima de Miranda R.[4], Boulet P.[2], Calmels L.[5], Gaudry E.[2], Migot S.[2], Ghanbaja J.[2], Kierren B.[2], Hauet T.[2]

[1] *Cryoscan, FRANCE;*
[2]*Institut Jean-Lamour, UMR 7198, CNRS, Université Lorraine, FRANCE;*
[3]*Physics Department, Bogazici University, Istanbul, TURKEY;*
[4]*Acquandas GmbH, Kiel, GERMANY;*
[5]*CEMES, CNRS-Université de Toulouse, FRANCE*

Although Nitinol physical properties have been heavily studied along with the development of a wide range of applications in industry, military technologies and health care, its magnetic characteristics have been only rarely discussed in the litterature. For instance, some works have been motivated by magnetic resonance imaging artifacts due to the paramagnetic signal from NiTi devices [1]. Here we measured, with commercial SQUID and VSM, the paramagnetic features of a freestanding 20μm thick NiTi polycristalline samples grown by DC magnetron sputtering [2], as a function of magnetic field, temperature and strain. We correlated them with NiTi lattice structure features characterized by TEM and XRD techniques. We showed that structural transformation from B2 to B19 (and vis-versa) can be easily revealed from non-invasive magnetometry measurements. Finally, the origin of the paramagnetic signal observed in our NiTi film, as well as its behavior under temperature and strain variation, was investigated in comparing our experimental data to ab-initio calculations and data from the literature.

Effect of magnetostatic energy on the magnetization behavior and magnetic driving forces in NiMnGa FMSMA's

29 Aug
2:00pm
Poster
Session C

Likhachev A. A.[1], Sozinov A.[2], Ullakko K.[2]

[1]*Institute for Metal Physics, Nat.Acad.Sci. of Ukraine, UKRAINE;*
[2]*Savonlinna Lappeenranta University of Technology, School of Engineering Science, Material Physics Laboratory, FINLAND.*

Ni-Mn-Ga based Ferromagnetic Shape Memory Alloys (FMSMA's) have have a unical ability to show extremely large magnetic field induced deformation effects which is about 30-50 times larger compared to the best known ordinary magnetostrictive materials. First these effects were discovered in two different nonstochiometric ferromagnetic martensitic phases (6% in 5M [1] and then 10% in 7M) [2]. It has been found that the strain mechanism in FMSMA's is based on the twin boundary motion and resulting redistribution between two twin related variants of the martensitic phase which easy magnetization axes are perpendicular each to other. Twinning processes are driven by the macroscopic magnetostrictive forces developed during the magnetization of FMSMA's. Our present results obtained on the basis of micromagnetic theory [3] show that along with the magnetic anisotropy and Zeeman's energies, magnetostatic energy plays an important role and produces a special coupling effect between the twin related martensite variants caused by the demagnetizing effects both on the surface of the MSM material and also on the internal twin boundaries. As a result, the magnetostatic energy produces the nonlinear dependence of the magnetic free energy and the magnetic driving force both on the magnetic field and on the volume fractions of twin variants.

[1] Sozinov A., Likhachev A. A., Lanska N., and Ullakko K, Appl. Phys. Lett, 2002, Vol. 80, pp 1746-1748.
[2] Likhachev A.A. and Ullakko K, Journal de Physique IV France, 2001, Vol. 11, Pr8-293-298, pp 435-440.
[3] A.A. Likhachev, Materials Science Forum, 2013, Vols. 738-739, pp 405-410.

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29 Aug
2:00pm
Poster
Session C

Correlation between defects and magneto-structural properties in Ni-Mn-Sn metamagnetic shape memory alloys

López-García J.[1], Rodríguez-Velamazán J. A.[1], Recarte V.[2], Sánchez-Alarcos V.[2], Pérez-Landazábal J. I.[2], Unzueta I.[3], García J. A.[3], Plazaola F.[3]

[1] *Institut Laue-Langevin, SPAIN;*
[2] *Universidad Pública de Navarra, SPAIN;*
[3] *Universidad Pública del País Vasco, SPAIN*

The effect of combined mechanical and thermal treatments in the magnetostructural properties was studied in Ni-Mn-Sn metamagnetic shape memory alloys, in which the extraordinary high stability of the $L2_1$ structure precludes the variation of atomic order by means of conventional thermal treatments. A $Ni_{50}Mn_{35}Sn_{15}$ alloy has been mechanically milled and then annealed at different temperatures in order to produce different microstructural states. The evolution of both the internal stresses and the crystallite size upon annealing has been quantified and correlated to the evolution of the martensitic transformation features and the magnetic properties. It is found that the relaxation processes brought by annealing leads to recovery of the martensitic transformation and the enhancement of the magnetism at both macroscopic and local level. In particular, the density of non-magnetic inclusions (defects) and their stress field decrease upon annealing, thus leading to an increase of the saturation magnetization and a decrease of the martensitic transformation temperature range, respectively, which results in a higher magnetocaloric effect. The obtained results confirm that, once the transition temperature has been fixed by the composition, the modification of the microstructure through thermomechanical treatments appears as the best way to tune the functional properties of these alloys.

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29 Aug
2:00pm
Poster
Session C

High-speed actuation of Ni-Mn-Ga micropillars by pulsed magnetic field

Musiienko D.[1], Saren A.[1], Straka L.[2], Klimša L.[2], Ullakko K.[1]

[1] *Lappeenranta University of Technology, FINLAND;*
[2] *Institute of Physics of the Czech Academy of Sciences, CZECH REPUBLIC.*

Magnetic shape memory (MSM) alloys exhibit large reversible magnetic-field-induced strain (MFIS) in martensitic phase. Those materials combine the large strain capability with the fast and remote actuation or sensing by magnetic fields. MSM alloys offer prospects for novel applications and embody a new mechanism for magnetic-to-mechanical energy conversion. The scaling down to micro-scale became one of the most advantageous development directions in the field. Similarly to microelectromechanical systems, here the key idea is to replace existing complex machinery by magnetomechanically active materials. Recently we have shown that if one dimension of the bulk Ni-Mn-Ga single crystal is decreased down to 1 μm there is no hindering of the MSM effect [D. Musiienko et al., Scr. Mater. 139 (2017) 152 - 154]. In the present study, we continue our research on the microstructural engineering of magneto-mechanically active devices. We report on pulsed magnetic field actuation of $50 \times 50 \times 100$ μm^3 cuboid pillars, created by decreasing two dimensions of a bulk single crystal. Focused ion beam (FIB) milling was used as one of the most advanced microstructure prototyping technologies. Surface stresses caused by ion beam milling play one of the main roles in prohibition of the MSM effect in microns-sized pillars. Thus, custom electro-polishing technique was used to remove the damaged surface layer of a pillar with a thickness of ~ 2 μm. After the treatment, pillars exhibited fully reversible magnetically induced reorientation (MIR) of crystal lattice of 6 ± 0.5 The actuation of the pillar was observed by a high-speed camera with a frame rate up to 775000 frames per second. Video footages were synchronized with a magnetic field pulse. Laser Doppler vibrometry technique was used for direct observation of the actuation speed of the pillar. Applied magnetic field pulse had a square front with rise time of less than 3 μs, the length of 60 μs and the magnitude about 1 T. Two different actuation speed patterns were observed which are in a good agreement with previously reported results for type 1 and type 2 twin boundaries (TB) [A. Saren et al., Scr. Mater. 113 (2016) 154 - 157]. Video clips clearly show full MIR of the pillar within ~ 23 μs for one set of experiments and $\sim$7 μs for the other, which are assumed to represent TB type 1 and TB type 2 motion, correspondingly. These important results indicate that MSM materials can provide fast actuation at microscale.

Stress-induced martensite ageing in single crystals of Ni-based Heusler alloy: processing, effect of orientation and functional properties

29 Aug
2:00pm
Poster
Session C

Panchenko E.[1], Timofeeva E. E.[1], Chumlyakov Y. I.[1], Osipovich K. S.[1], Larchenkova N. G.[1], Pichkaleva M. V.[1], Tokhmetova A. B.[1], Gerstein G.[2], Maier H. J.[2]

[1] *Tomsk State University, RUSSIAN FEDERATION;*
[2] *Institut für Werkstoffkunde (Materials Science), Leibniz Universität Hannover, GERMANY*

The objective of the present study was to develop an effective thermal-mechanical treatment to significantly enhance the functional properties due to creating internal stress fields in ferromagnetic NiFeGaCo and NiMnGa single crystals. Internal long-range stress fields are the key parameter to grow the oriented martensite upon cooling without any external applied stresses, control the martensitic transformations temperatures, and induce the two-way shape memory effect (TWSME) and the martensite rubber-like behavior (RLB). Effective regimes of the low-temperature stress-induced martensite ageing (SIM-ageing) along various crystallographic directions in high-temperature superelasticity range were worked out for the NiFeGaCo and NiMnGa single crystals. The temperature and stress level of the effective SIM-ageing regime are determined by the crystal chemical composition, the initial microstructure of single crystals, and the crystallographic orientation of the applied stress axis during ageing. It was shown experimentally, that the SIM-ageing leads to the stabilization of stress-induced martensite in both investigated NiFeGaCo and NiMnGa single crystals independently on their orientation and the initial microstructure ($(L2_1 + \gamma)$- and B2-austenite). This is accompanied by increase of martensitic transformation temperatures, creation of internal stress fields and decrease in 1.5-3.0 times both the thermal and stress hysteresis as compared with the quenched crystals. The high-temperature shape memory effect and superelasticity at T>373 with both minimum thermal hysteresis of 7-10 K and stress hysteresis of 12-15 MPa are observed in [001]A-oriented $Ni_{51}Fe_{18}Ga_{27}Co_4$ (at.%) single crystals after SIM-ageing at 423-498 K along the [001]A||[110]$L1_0$-direction. The best TWSME with a giant reversible tensile strain up to + 8.5% was obtained in [001]A-oriented $Ni_{51}Fe_{18}Ga_{27}Co_4$ single crystals after SIM-ageing along [110]A||[100]$L1_0$-direction at T = 423 K for 1.0 h. In addition, in these SIM-aged crystals a RLB with a reversible compressive strain of up to -7.0% in loading/unloading cycles due to martensite variant reorientation was observed. The obtained results indicate that the SIM-aged NiFeGaCo crystals can be used to develop not only thermal-mechanically but also magnetically controlled actuators that exploit the TWSME and RLB. Physical reasons of effect of applied stress orientation during SIM-ageing on microstructure and functional properties in ferromagnetic NiFeGaCo and NiMnGa single crystals are under discussion.
This work was supported by the Russian Science Foundation (grant No. 16-19-10250).

Thermal and magnetic noises emitted during martensitic transformation in a metamagnetic $Ni_{45}Co_5Mn_{36.6}In_{13.4}$ single crystal

29 Aug
2:00pm
Poster
Session C

Samy N.[1], Bolgár M. K.[1], Barta N.[2], Daróczi L.[1], Chumlyakov Y. I.[3], Karaman I.[2], Beke D. L.[1]

[1] *Department of Solid State Physics, University of Debrecen, HUNGARY;*
[2] *Department of Mechanical Engineering, Texas A& M University, USA;*
[3] *Siberian Physical Technical Institute, Tomsk State University, RUSSIAN FEDERATION.*

Characteristics of thermal, acoustic and magnetic noises, detected during martensitic transformation in the $Ni_{45}Co_5Mn_{36.6}In_{13.4}$ single crystal, were investigated. From the statistical analysis of the thermal spikes, appeared on the DSC curves at low cooling/heating rates (typically below 1 K/min) the energy exponent of the power law behavior of this thermal noise was calculated as epsilon=1.6 plus or minus 0.2. This is in acceptable agreement with exponent of the energy distribution of acoustic emission: epsilon=1.9 plus or minus 0.1. These results agree very well with the values epsilon=2.0 plus or minus 0.1 and epsilon=1.7 plus or minus 0.2, obtained from similar measurements in $Ni_{49}Fe_{18}Ga_{27}Co_6$ [1]as well as Ni_2MnGa[2]single crystals.
Our sample shows an abrupt change in the magnetization across the martensitic transition (metamagnetic behaviour) and thus detection and statistical analysis of the emitted magnetic noise is very important. This type of measurements were not carried out before for this alloy system. We obtained that i) at zero external field the-signal-to-noise ratio was poor (not allowing the determination of power exponents), but interestingly the noise packages contained voltage peaks in the detector coils in both direction, ii) by the application of small external magnetic field (10 and 30 mT) the polarity of the peaks decreased (both in heating and cooling), but surprisingly with increasing tail at the end of the noise package for martensitic transformation. Furthermore the noise activity was higher for the austenite to martensite transformation

than that for the reverse transformation. The energy, area, width (duration), and height (amplitude) distributions of the signals exhibit a power-law behavior, with an exponential cutoff. The values of the exponent were independent of the external field, and are characteristic for the magnetic noise originating from the transition itself.

[1] M.K. Bolgar, L. Toth, S. Szabo, S. Gyongyosi, L. Daroczi, E. Panchenko,Y. Chumlyakov, D. Beke, J. Alloy. Comp. 658 (2016) 29.
[2] L.Z. Toth, S. Szabo, L. Daroczi, D.L. Beke, Phys. Rev. B 90 (2014), 224103.

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29 Aug
2:00pm
Poster
Session C

Characteristics and functional properties of Ni-Mn-Ga-Cu alloys

Seguí C.[1], Torrens-Serra J.[1], Cesari E.[1], Lázpita P.[2]

[1]*Departament de Fsica. Universitat Illes Balears, SPAIN;*
[2]*Dept. Electricidad y Electrnica. Fac. de Ciencia y Tecnologa. UPV/EHU, SPAIN.*

When considering the possibility of magnetic field induced martensitic transformation (MFI MT) in ferromagnetic shape memory alloys, alloy systems such as the so-called metamagnetic alloys Ni-Mn-X (X=In, Sn, Sb) are usually considered. In these alloys, ferromagnetic austenite transforms into a low magnetization martensite giving rise to a magnetization drop (ΔM). According to Claussius-Clapeyron, dT/dH=-ΔM/ΔS, application of magnetic field decreases the MT temperatures, thus helping to induce the reverse MT. The magnetic field levels necessary to induce magnetic actuation can be significantly reduced with the help of applied stress; however, for the case of reverse transformation induced by field, the magnetic field and external stress play an antagonistic role. Instead, for alloys undergoing paramagnetic martensite to ferromagnetic austenite MT, such as Ni-Mn-Ga-Cu, the magnetic field promotes forward transformation, in the same way as applied stress. A certain composition range of Ni-Mn-Ga-Cu alloys with paramagnetic austenite and ferromagnetic martensite phase has already been defined, which is promising, not only for MFIMT at low applied fields, but also as magnetocaloric materials. Their characteristics and functional properties, significantly field-induced strain (MFIS) with and without external stress, will be presented and analyzed in the present work.

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29 Aug
2:00pm
Poster
Session C

Effect of thermomechanical treatment on deformational and magnetic behaviour of ferromagnetic Fe-Ni-Co-Ti alloy

Titenko A.[1], Demchenko L.[2], Babanli M.[3], Kozlova L.[1], Perekos A.[4], Huseynov S.[3], Sidorenko S.[2]

[1]*Institute of Magnetism, NAS of Ukraine, UKRAINE;*
[2]*National Technical University of Ukraine "Igor Sikorsky Kyiv Polytechnic Insitute", UKRAINE;*
[3]*Azerbaijan State University of Oil and Industry, AZERBAIJAN;*
[4]*G. V. Kurdyumov Institute for Metal Physics of the NAS. of Ukraine, UKRAINE*

Ferromagnetic shape memory alloys (FSMAs) have unique functional properties that are manifested in the unusual mechanical behaviour under thermal, mechanical, magnetic effects. Traditional alloys such as Ni-Ti, Cu-Al-Zn, Ni-Mn-Ga are replaced by disordered iron-based alloys that have good thermoelasticity, shape memory, high temperature and low-temperature superelasticity, etc. Thus, in the polycrystalline FeNiCoAlTaB alloy, an anomalously large superelastic strain εSE of 13% was found at the tension, exceeding the lattice deformation for a given orientation $\varepsilon 0 = 8.7\%$. These indicators are almost 2 times higher than those for commercial Ni-Ti, both in the value of εSE and the stress of martensite deformation induction σc. According to the magnetic characteristics, the Ni-Mn-Ga Heusler alloys are inferior to the Fe based FSMA on the saturation magnetization and the Curie temperature (Tc). In the process of deformation by stretching due to the induction of the martensitic γ - α' transformation (MT) in FeNiCoAlTaB, the saturation magnetization changes up to 4 times and the anisotropy field when the structure is transformed. To improve the functional properties of a ferromagnetic alloy with shape memory, a thermomechanical treatment consisting of drawing, quenching with high-temperature annealing is proposed, as a result of which a nanostructured state is formed. Systemic studies of structural features of both the structural phase transformation of the martensitic type, as well as the magnetic transition, have been carried out. The mechanical characteristics of the phases of the alloy were studied under uniaxial tension conditions. Thermomechanical treatment (TMT) makes the alloy more thermoelastic, although this is accompanied by a large temperature hysteresis of the martensitic transformation, but leads to a significant hardening of the matrix, which in turn improves the effects of shape memory and pseudoelasticity. It has been established that TMT in Fe-Ni-Co-Ti alloy with shape memory contributes to an increase in reversible pseudoelastic deformation to $\sim 3\%$. With a superelastic cycle with large mechanical hysteresis, a large dissipative scattering energy is obtained, which can promote the use of this alloy as a damper for mechanical oscillations. The change in the magnetic characteristics as a function of the preliminary thermomechanical treatment is associated with a decrease in the volume fraction of the ferromagnetic phase upon aging and the formation of a system of micro-inhomogeneities in the material, which leads to the appearance of additional induced magnetic anisotropy both during the processing itself, and especially during deformation.

Effect of the partial heating/cooling cycles on the asymmetry of the forward and reverse martensitic transformation of single crystalline Ni2MnGa alloy

29 Aug
2:00pm
Poster
Session C

Tóth L. Z., Daróczi L., Beke D. L.

Department of Solid State Physics, University of Debrecen HUNGARY

The austenite/martensite transformation is a stop-and go type discontinuous process, which is associated with the emission of thermal, acoustic and magnetic signals (if the sample is ferromagnetic). The differential scanning calorimetry (DSC) curves at small heating/cooling rates often look different for heating and cooling runs, as well as the acoustic and magnetic exponents are also not the same in most cases. This asymmetry of the noise parameters suggest, that the dynamics of the forward and reverse transition are different. Using the notations of our recent publication in this topic [D. L. Beke, et al. J. Alloys Compd. 741 (2018): 106-115], the asymmetry is positive, if the critical exponents for heating is higher than the cooling exponents, and the asymmetry is negative for the reverse case. In this paper the asymmetry is attributed to the different ways of relaxation of the elastic strain energy during cooling as well as heating. For the fully understanding of the asymmetry of the forward and reverse martensitic transformations, special measurements are needed. Since the nucleation of the martensite phase can not interpret both the positive and negative asymmetry, it is worth to try what happens if the martensite phase does not need nucleation. In a poster presentation, we will show the effect of partial heating and cooling cycles on the critical exponents obtained from acoustic emission measurements using a Ni_2MnGa shape memory alloy. Partial cycle means, that the heating was stopped at different temperatures, before the austenite finish temperature, in order to leave some martensite nuclei in the sample. For a full cycle, there is a positive asymmetry with amplitude exponents of $\alpha h \simeq 2.70$ for heating and $\alpha c \simeq 2.15$ for cooling resulting in a high asymmetry parameter ($\gamma\alpha = (\alpha h - \alpha c)/\alpha c \simeq 0.26$). In case of the partial cycles, the heating exponents remained the same, but the cooling exponents increased, resulting in a lower ($\gamma\alpha = 0.16$), but still present asymmetry.

Temperature dependence of critical stress and entropy change for martensitic transformation in Fe-Mn-Al-Ni shape memory alloy

29 Aug
2:00pm
Poster
Session C

Xia J., Xu X., Omori T., Kainuma R.

Department of Materials Science, Graduate School of Engineering, Tohoku University, JAPAN

Recently, it has been reported that a new Fe-based Fe-Mn-Al-Ni shape memory alloy shows superelasticity at temperatures between 77 K to 513 K associated with the α(bcc, ferromagnetic) / γ(fcc, weak magnetic) martensitic transformation [1]. The extremely small temperature dependence of critical stress for superelasticity in this alloy system, arising from small entropy change during martensitic transformation, allows it to be used in a wide temperature range and thus makes it a promising alternative for industry applications compared with Ti-based alloys. Moreover, the magnetic field induced transformation is considered possible in this alloy due to the large magnetization difference between parent and martensite phases. Till now, there are no report related to superelasticity under cryogenic temperatures and few reports related to magnetic field induced reverse transformation in this alloy.
In this study, compression tests from 10 K to room temperature were investigated on Fe-Mn-Al-Ni single crystals and superelasticity was found even at 10 K. The magnetic field induced reverse transformation was directly observed by in-situ observation of the optical microstructure. By an application of magnetic field up to 50 T, full reverse martensitic transformation was induced by magnetic field at low temperatures. The temperature dependence of both the critical stress and critical magnetic field was revealed to be

small and transformation hysteresis was less sensitive to temperature change. By using the Clausius-Clapeyron relation with critical stress and magnetic field, the temperature dependence of entropy change was obtained indirectly. In order to obtain further understanding of the extremely small temperature dependence of critical stress for superelasticity, specific heat measurements (2-473 K) for the parent and deformation-induced martensite phases were carried out, from which the temperature dependence of entropy change was obtained. The results of direct and indirect measurements of entropy change showed good consistency to each other. The Néel temperature of martensite phase as well as the Curie temperature of the parent phase was determined by thermomagnetization measurements. The values of entropy change during martensitic transformation were found affected by the magnetic transitions.
[1] T. Omori, K. Ando, M. Okano, X. Xu, Y. Tanaka et al. Science 333 (2011) 68-71.

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29 Aug
2:00pm
Poster
Session C

Martensitic transformation and magnetic phase diagram of $Ni_{50}Mn_{50-x}Ga_{x/2}In_{x/2}$ magnetic shape memory alloys

Xu X., Yoshida Y., Omori T., Kanomata T., Kainuma R.

Department of Materials Science, Graduate School of Engineering, Tohoku University, JAPAN

NiMn-based Heusler alloys generally encompass two families of alloy systems. Represented by the Ni-Mn-Ga alloys, the ferromagnetic shape memory alloys show magnetic field-induced variant rearrangement in the ferromagnetic martensite phase, where huge magnetostriction can be obtained. On the other hand, the metamagnetic shape memory alloys, represented by the Ni-Mn-In alloys, show magnetic field-induced reverse martensitic transformation, during which metamagnetic shape memory effect can be realized. The martensitic transformation and magnetic phase diagrams have been determined for both systems for the stoichiometric $Ni_{50}Mn_{50-x}Z_x$ (Z=In, Ga) sections. It has been a well-known fact that, as the concentration of Z atom increases, the martensitic transformation temperature approximately linearly decreases, however, it shows a bending behavior when it intercepts against the magnetic transition temperatures. The martensitic transformation temperature bends upwards for $Ni_{50}Mn_{50-x}Ga_x$, whereas it bends downwards for $Ni_{50}Mn_{50-x}In_x$. On the other hand, there is a large magnetization difference during martensitic transformation for $Ni_{50}Mn_{50-x}In_x$, whereas it is negligibly small for $Ni_{50}Mn_{50-x}Ga_x$. In this study, samples in a pseudo-binary section of $Ni_{50}Mn_{50-x}Ga_{x/2}In_{x/2}$ were prepared, where an equiatomic Ga:In=1:1 ratio was kept. The martensitic transformation and magnetic transition temperatures, B2-$L2_1$ atomic order-disorder transformation temperatures and spontaneous magnetization were systematically investigated. The B2-$L2_1$ atomic order-disorder transformation temperatures were determined for selected sample. They were found to show a parabolic-like behavior with a vertex composition at around 23.6% Ga+In composition. A magnetic phase diagram including the martensitic transformation and magnetic transition temperatures was determined. The martensitic transformation temperatures monotonically decreases with increasing Ga+In composition, and slightly bends downwards below the magnetic transition temperatures. The composition dependence of spontaneous magnetization was investigated for both parent and martensite alloys. The spontaneous magnetization was found to be higher in parent phase than that in the martensite phase. The above characteristics are also compared against those of the ternary Ni-Mn-Ga and Ni-Mn-In systems in this presentation.

Session 6: Ti-based Alloys

Invited Lecture: Wednesday August 29	
8:30 Amphi 4	**F. Prima**, F. Sun, L. Lilensten, Y. Danard, C. Brozek, P. Vermaut, I. Freiherr Von Thungen "New "strain - transformable" beta titanium alloys for improved resistance / ductility / strain hardening compromise "
Oral presentations: Wednesday August 29	
9:00 Amphi 4	W. Elmay, X.Gabrion, L. Peltier, **S. Berveiller**, B. Piotrowski, P. Laheurte "Influence of microstructure energy dissipation capacity of β metastable titanium alloys "
9:20 Amphi 4	**P. Krooß**, C. Lauhoff, A. Paulsen, J. Frenzel, C. Somsen, D. Langenk¨amper A. Reul, W. Schmahl, E. Karsten, H.J. Maier, T. Niendorf "Effect of the heating-cooling rate on the functional properties of Ti-Ta-Al HT-SMAs "
9:40 Amphi 4	**J. Nejezchlebová**, H. Seiner, P. Sedlák, M. Janovská, M. Landa, J. Šmilauerová, J. Stráský, M. Janeček "In-situ characterization of $\beta \rightarrow \omega$ phase transformation in metastable β-Ti alloys by ultrasonic methods "
10:00 Amphi 4	**M. Goetz**, B. Appolaire, M. Dehmas, E. Aeby-Gautier S. Andrieu, D. Mangelinck, M. Descoins "Decomposition of the Beta phase at low temperatures in Beta-metastable Ti-5553 alloy "

Poster Session A: Tuesday August 28
S. Dubinskiy, G. Markova, E. Yudina, S. Prokoshkin, V. Brailovski "Elinvar effect in thermomechanically treated Ti-Nb-Zr alloy "
K. Golasinski, E. Pieczyska, M. Maj, M. Staszczak, N. Takesue "Superelastic-like behavior of Gum Metal under compression inspected by infrared thermography "
Y. Zhang, K. Hua, H. Kou, J. Li, W. Gan, C. Esling "Deformation-induced α variant selection during β to α phase transformation in a metastable β Ti alloy"

Contents

New "strain - transformable" beta titanium alloys for improved resistance / ductility / strain hardening compromise

29 Aug
8:30am
Amphi 4

Prima F.[1],Sun F.[1],Lilensten L.[1], Danard Y.[1], Brozek C.[1], Vermaut P.[1], Freiherr Von Thungen I.[2]

[1]*PSL Research University, Chimie ParisTech - CNRS, Institut de Recherche de Chimie Paris,7 5005, Paris, FRANCE;*
[2]*SAFRAN TECH 1 Rue Geneviève Aube, 78114 Magny - les - Hameaux,FRANCE*

Owing to their high specific properties, titanium alloys have been , for a long time , highly competitive materials in fields such as aerospace industry. Among these alloys, research efforts were recently dedicated to design approaches for improved strength/ductility trade off. Guided by electronic parameters calculations, new "strain - transformable" Ti alloys have been developed and both single phase and dual phase materials have been optimized. Thanks to the synergy between stress induced martensitic transformation (TRIP effect) , intense mechanical twinning (TWIP effect) and dislocations glide, these new materials display a combination of high fracture strength (up to 1400MPa) , extra - large work - hardening and superior ductility (up to 45% at fracture).
In this talk, design strategy and microstructural optimization approach es of this new family of alloys will be discussed regarding the occurrence , chronology and synergy of the different deformation mechanisms. From this work, future directions towards both compositional and microstructural optimization pathways will be drawn and discussed

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Influence of microstructure energy dissipation capacity of β metastable titanium alloys

29 Aug
9:00am
Amphi 4

Elmay W.[1], Gabrion X.[2],Peltier L.[1],Berveiller S.[1],Piotrowski B.[1],Laheurte P.[1]

[1]*LEM3 Metz, UMR/CNRS 7239, F-57012 Metz, FRANCE;*
[2]*FEMTO-ST, UMR 6174, 25000 Besançon, FRANCE*

During the last decades, much attention has been paid to Ti-based alloys for several areas such as the medical domain (surgical implements and implants), aerospace (aircraft, armor plating,...) and automotive applications (connecting rods, valves...) due to their good mechanical properties, superior corrosion resistance, low density and excellent cold workability.
New β metastable type titanium alloys have been developed more recently [1]. The specificity which makes interesting this type of alloy is the reversible martensitic transformation from the body centered cubic (bcc) β phase to the orthorhombic α'' martensite phase, induced either by applying stress or by temperature variation. This reversible transformation is at the origin of superelasticity and/or shape memory effect. The advantage of β type titanium alloys is that they present a large spectrum of properties which can be modulated by controlling the thermomechanical treatment conditions.
Several studies have been conducted to characterize mechanical properties of these alloys as a function of their microstructure, however only few studies have dealt with the energy dissipation capacity of these alloys [2-3]. The characterization of this capacity of a binary titanium based alloy is the subject of our work. Dynamic mechanical analysis (DMA) was performed at different temperatures in order to activate different deformation mechanisms. A correlation between the microstructure induced by a thermomechanical treatment, the activated deformation mechanisms and the mechanical properties was established. A comparison of the role of the martensitic transformation and the reorientation mechanism on the energy dissipation capacity was investigated.

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[2] E. Bertrand, P. Castany, T. Gloriant, Acta Materialia 61, 511 518, 2013 .

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29 Aug
9:20am
Amphi 4

Effect of the heating-cooling rate on the functional properties of Ti-Ta-Al HT-SMAs

Krooß P.[1],Lauhoff C.[1],Paulsen A.[2], Frenzel J.[2], Somsen C.[2], Langenkämper D.[2], Reul A.[3], Schmahl W.[3], Karsten E.[4], Maier H.J.[4], Niendorf T.[1]

[1] *Institut für Werkstofftechnik,Universität Kassel, GERMANY;*
[2]*Institut für Werkstoffe, Ruhr-Universität Bochum, GERMANY ;*
[3]*Applied Crystallography and Materials Science, Ludwig-Maximilians-Universität, GERMANY;*
[4] *Institut für Werkstoffkunde, Leibniz Universität Hannover, GERMANY*

Shape memory alloys attract a lot of attention since decades due to their unique properties. In biomedical applications shape memory alloys based on binary NiTi are already used intensively. Besides these niche applications, numerous issues prevail hindering a more wide-spread use in other fields. Most conventional shape memory alloys, e.g. binary NiTi, lose their shape recovery ability above temperatures of about 100 °C. Therefore, shape memory alloys exhibiting transformation temperatures above 100 °C, referred to as high-temperature shape memory alloys (HTSMA), have been introduced some years ago. HTSMAs such as NiTiHf and NiTiPd show transformation temperatures up to 400 °C, teraining high recoverable transformation strains during thermal cycling. However, expensive alloying elements, poor workability and limited ductility of most actual HTSMAs hinder wide-spread industrial applications in mass products, e.g. in the automotive and the aerospace sector. At least in terms of cost, processability and formability, binary and ternary TiTa alloys seem to fulfill the given requirements as they show excellent workability and high ductility with deformation degrees up to 90 %.
As HTSMAs are also supposed to be used in applications, where a high number of thermal cycles is required, characterizing functional fatigue and underlying elementary mechanisms is crucial. It is known that thermal cycling at elevated temperatures may lead to degradation of shape memory properties as a result of a shift in transformation temperatures and a decrease in transformation strains due to the formation of additional phases, i.e. the ω-phase, generated during thermal cycling and dwell times in critical temperature regimes, most pronounced in the binary TiTa alloy.
Thus, this study follows the idea of suppressing the ω-phase formation during thermal cycling by adding Al as a α-stabilizing element. In order to evaluate the cyclic stability of ternary TiTaAl, functional fatigue, i.e. isostress thermal cycling experiments under tensile loading conditions tests were conducted. Heating-cooling rates were varied in order to study the impact of cycle number and dwell times separately. In order to highlight the microstructural evolution following thermal cycling in situ synchrotron and TEM investigations were made.

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29 Aug
9:40am
Amphi 4

In-situ characterization of $\beta \to \omega$ phase transformation in metastable β-Ti alloys by ultrasonic methods

Nejezchlebová J.[1],Seiner H.[1],Sedlák, P.[1],Janovská M.[1], Landa M.[1], Šmilauerová J.[2], Stráský J.[2],Janeček M.[2]

[1]*Institute of Thermomechanics, Czech Academy of Sciences CZECH REPUBLIC;*
[2]*Faculty of Mathematics and Physics, Charles University CZECH REPUBLIC*

Metastable β-Ti alloys exhibit various solid-solid phase transitions. Our study is focused on the characterization of $\beta \to \omega$ phase transition. The particles of ω phase play an important part in thermomechanical treatment since they serve as a heterogeneous nucleation sites for precipitation of finely dispersed particles of hexagonal α phase. This fine microstructure leads to a significant strengthening and general improvement of mechanical properties (strength and toughness are enhanced while ductility is maintained to a reasonable level).
Nano-sized particles of athermal ω phase (ωath) form by diffusion-less shuffle transformation during quenching from the temperatures above β-transus temperature. Upon subsequent heating, the particles of isothermal ω phase (ωiso) further evolve by coupled diffusional-displacive transformation mechanism. The alloying elements are rejected from the ωiso particles to the surrounding β matrix and collapse of $(111)\beta$ planes takes place simultaneously during this process. Detailed knowledge of the transformation processes is the foundation for the tailoring of thermomechanical treatment routes in order to obtain material with required properties.
The in-situ observation of the growth of particles of ω phase could be difficult by conventional techniques. Recently, it was shown that the ω phase significantly influence the elastic constants of the material and the different forms of ω phase have different effects on the elastic anisotropy, as well as on the internal

friction coefficients [1]. Therefore the $\beta \rightarrow \omega$ phase transformation could be in-situ observed by the precise measurement of the tensor of elastic constants. In this contribution, we present the study of the kinetics of the $\beta \rightarrow \omega$iso phase transformation by resonant ultrasound spectroscopy. Three metastable β-Ti alloys Ti5553, TIMETAL LCB and Ti15Mo were in-situ examined by this technique during isothermal and non-isothermal ageing. Furthermore, the influence of stress on the growth of ω phase during isothermal ageing will be discussed.

[1] J. Nejezchlebová, M. Janovská, H. Seiner, P. Sedlák, M. Landa, J. Šmilauerová, J. Stráský, P. Harcuba, M. Janeček, The effect of athermal and isothermal ω phase particles on elasticity of β-Ti single crystal, Acta Mater. 110, 185 - 191, 2016.
doi:10.1016/j.actamat.2016.03.033.

Decomposition of the Beta phase at low temperatures in Beta-metastable Ti-5553 alloy

29 Aug
10:00am
Amphi 4

Goetz M.[1], Appolaire B.[1], Dehmas M.[2], Aeby-Gautier E.[1],Andrieu S.[3],Mangelinck D.[4],Descoins M.[4]

[1]Institut Jean Lamour - SI2M, Université de Lorraine, 2 allée André Guinier, 5400 Nancy Cedex, FRANCE.;
[2]CIRIMAT - ENSIACET, 4 allée Emile Monso, BP 44363, 31030 Toulouse Cedex, FRANCE.;
[3]Safran Landing Systems, Etablissement de Bidos, BP 39, 64401 Oloron Ste Marie Cedex, FRANCE;
[4]IM2NP, Faculté des Sciences et Techniques, avenue Escadrille Normandie Niemen, 13397 Marseille Cedex, FRANCE

The Ti-5553 (Ti-5Al-5Mo-5V-3Cr, in wt%) alloy is a beta titanium alloy designed for the manufacturing of large section components such as landing gears. Its good quenchability results in a deep hardenability, provided that the decomposition of the beta phase (bcc) is controlled, and so well understood. Among the different issues remaining on this decomposition, we have addressed the nature of the transformations undergone by beta depending on temperature. For that purpose, we have analyzed quantitatively the composition of the different phases by means of different characterization techniques (APT, TEM) in specimens annealed at various temperatures for different holding times.
Whereas at high temperatures (>500°C) beta transforms into alpha (hcp) involving a partitioning compliant with the equilibrium phase diagram, we show that beta transforms into alpha$''$ (orthorhombic), without any significant partioning at the beginning of the transformation, suggesting that the transformation features a mixed mode character in agreement with phase field calculations.

Elinvar effect in thermomechanically treated Ti-Nb-Zr alloy

28 Aug
2:00pm
Poster
Session A

Dubinskiy S.[1], Markova G.[2], Yudina E.[2],Prokoshkin S.[1],Brailovski, V.[3]

[1]National University of Science and Technology "MISIS", Moscow, RUSSIA;
[2]Tula State University, Tula, RUSSIA,[3]Ecole de technologie superieure, Montreal, CANADA

Ti-Nb-based shape memory alloys (SMA) are mainly known as highly biocompatible low-stiffness metallic materials for load-bearing implant applications. Moreover, these alloys demonstrate some particular features which may also be of practical interest. For example, it was reported that Ti-Nb SMA doped with oxygen and plastically deformed to generate in the material a high density of crystal lattice defects, mainly dislocations, show elinvar behavior upon heating in the 77 to 500 K temperature range [Saito, 2003; Miyazaki, 2015]. In this work, the elinvar type behavior was observed in Ti-Nb-Zr alloy subjected to cold rolling with a true strain of e=0.3 and post-deformation annealing to form either polygonized (600°C, 30min) or recrystallized (750°C, 30min) structure. Contrarily to the oxygen-doped alloys in which the elinvar effect was observed on heating, the studied ternary alloy exhibits this effect on cooling. It was shown that in the studied 823 to 293 K temperature range, the temperature coefficient of the elastic modulus of the studied alloy ($\sim$ 1x10E-6 to 10E-5 1/K) is much lower than that of the conventional titanium alloys, such as Ti-6Al-4V ($\sim$ 1x10E-3 1/K in the 523 to 873 K temperature range) and comparable to that of the classic elinvar-type alloys, such as Ni-Span-C Alloy 902 ($\sim$ 1x10E-5 1/K, but in a lower and shorter temperature range of 240 ... 410 K). Based on the structure and phase transformation studies, hypotheses are made about the mechanisms of such an elinvar type behavior.
Acknowledgements.
This work has been carried out with the financial support of the Russian Foundation for Basic Research (grants 16 - 43 - 710688 and 18 - 08 - 01193) and of the Natural Science and Engineering Research Council of Canada (NSERC) (Discovery grant program).

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28 Aug
2:00pm
Poster
Session A

Superelastic-like behavior of Gum Metal under compression inspected by infrared thermography

Golasinski K.[1],Pieczyska E.[1],Maj M.[1],Staszczak M.[1],Takesue N.[2]

[1] *Institute of Fundamental Technological Research, Polish Academy of Sciences POLAND;*
[2] *Fukuoka University, Department of Applied Physics, Fukuoka, JAPAN*

Gum Metal (Ti36Nb2Ta3Zr0.3O in mass%) is a beta titanium alloy developed at Toyota Central R & D Labs., Inc. The alloy has attracted remarkable attention due to its exceptional properties, i.e. low elastic modulus, high strength, nonlinear superelastic-like deformation, excellent cold workability, as well as Invar- and Elinvar-like performance. This set of outstanding properties has been extensively discussed in the literature in the context of structural, compositional and thermal analyses. Specifically, unconventional deformation mechanisms occurring in Gum Metal under load have been a focal point in several publications. Recent studies confirmed that the nonlinear superelastic-like deformation of Gum Metal is mainly caused by two microstructural features martensite-like α nanodomains and ω phase precipitates.
In this work, Gum Metal cube samples were machined out of a Gum Metal rod, texturized along <110> direction. The samples were subjected to monotonic and cyclic compression with an increasing strain step on a testing machine. The deformation was simultaneously monitored by an infrared camera and a CCD camera for determining a thermal response of the alloy and strain changes, respectively. Stress-strain curves confirmed the superelastic-like deformation and high plastic performance of Gum Metal. The thermal response of the alloy under compression, determined within the large recoverable strain, gave an insight into the nature of the deformation mechanisms of Gum Metal. In this regime, significant changes in the growth rate of the thermal response were observed. As in conventional materials, the first stage of compression is a linear, fully elastic deformation accompanied by a temperature growth. The second stage is a nonlinear deformation which is reflected by change of the temperature growth rate. The change can be associated with martensite-like α nanodomains and ω phase precipitates activated during the loading of Gum Metal. Next stage of deformation is yielding, which was represented by a clearly pronounced yield point. Further high plastic deformation without hardening is accompanied by a rapid growth in temperature.
The technique of infrared thermography was successfully applied to register thermomechanical nature of the unconventional deformation mechanisms namely α'' martensite-like nanodomains and ω phase precipitates activated in Gum Metal under compression. The changes of the temperature rate can precisely indicate subsequent stages of Gum Metal deformation especially during the large superelastic-like deformation.
Acknowledgments
The research has been supported the Polish National Science Centre (NCN) under Grants nos. 2014 / 13 / B / ST8 / 04280 and 2016 / 23 / N / ST8 / 0368.

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28 Aug
2:00pm
Poster
Session A

Deformation-induced α variant selection during β to α phase transformation in a metastable β Ti alloy

Hua K.[1,2],Zhang Y.[1],Kou H.[2], Li J.[2],Gan W.[3], Esling C.[1]

[1] *Université de Lorraine, CNRS, Arts et Métiers ParisTech, LEM3, F-57000, Metz, FRANCE;*
[2] *State Key Laboratory of Solidification Processing, Northwestern Polytechnical University, 710072 Xi´an, PR CHINA;*
[3] *Helmholtz-Zentrum Geesthacht, Max-Planck-Straße 1, 21502 Geesthacht, GERMANY*

The mechanical properties of titanium alloys are thought to be related to α phase produced via the β to α phase transformation during thermomechanical process. Previous works have evidenced that there happens α variant selection during thermomechanical process. The formation of α variant could form the microtextures, leading to the modification of the mechanical properties of the alloy. The rules and mechanisms of this variant selection are still need to be illustrated. Thus, based on this, a study was conducted, in the present work, on the α variant selection in a metastable β Ti alloy under isothermal compression. It is found that two Burgers orientation variants with a disorientation of 90°around <1 1.38 0> α appeared concomitantly and often intersect each other, forming ¨cross - shaped¨ clusters. The selection of such variants is governed by the compatibility of the lattice deformation of the phase transformation to the imposed compressive strain. The selected variants are able to effectively accommodate the external compressive strain by their compatible normal lattice strain.

Session 7: Alloy Development and Applications

Invited Lecture: Wednesday August 29	
15:30 Amphi 4	**G. Y. Firstov**,T. Kosorukova ,A. Timoshevskii , Yu. Koval ,V. Odnosum ,Yu. Matviychuk ,G. Gerstein , H.J. Maier "Strategies for high entropy shape memory alloy design - from motivation to structure and properties "
Oral Presentations: Wednesday August 29	
16:00 Amphi 4	**D. Piorunek**, J. Frenzel, G. Eggeler " High Entropy Shape Memory Alloys Effects of Chemical Complexity on Martensitic Transformation "
16:20 Amphi 4	**B. Franco**, E. Dogan, S. Wang, I. Karaman "Engineering the transformation hysteresis by precipitation in NiTi and NiTiHf "
17:10 Amphi 4	**P. Xolin**, C. Collard, M. Engles-Deutsch, T. Ben Zineb " Experimental and numerical study of monocrystalline CuAlBe endodontic file response subjected to combined torsion-bending loading "
17:30 Amphi 4	**Y. Chumlyakov** , I. Kireeva , I. Kuksgausen, Z. Pobedennay , M. Panchenko, K. Reunova , D. Kuksgausen ,V. Kirillov "Shape Memory Effect and Superelasticity in high-strength Fe-based single crystals "
17:50 Amphi 4	**J. Pons**, S. Xu, J. Pons, R. Santamarta, I. Karaman "Behaviour of Ni-rich Ni-Ti-Zr shape memory alloys upon low temperature aging "
Oral Presentations: Thursday August 30	
10:50 Amphi 4	**F. Fouché**, A. Hautcoeur, D. Chassoulier, A. Blanc, J. Sicre " Development and Testing of a High - Temperature Shape Memory Actuator using CuAlNi for Hold Down and Release Mechanisms (HRM)"
11:10 Amphi 4	**A.I. Tagiltsev** , E.Y. Panchenko ,Y.I. Chumlyakov , H.E. Karaca , I. Karaman " The comparison of effect of different treatments on the high temperature superelasticity in Ni50.3Ti32.2Hf17.5 polycrystals "
11:30 Amphi 4	**H. O. Tugrul**, H. H. Saygili, B. Kockar " Comparison of the Transformation Behavior of Cold Rolling with Aging and Hot Extrusion with Aging Processed Ni50.3Ti29.7Hf20 High Temperature Shape Memory Alloy "
11:50 Amphi 4	**C. Lauhoff**, M. Vollmer, P. Krooß, A. Paulsen, J. Frenzel, Y.I. Chumlyakov, T. Niendorf " In situ characterization of functional properties in polycrystalline Co-Ni-Ga high-temperature shape memory alloys From bicrystals to hot-rolled material "
12:10 Amphi 4	**Y. Zhukova**,R. Drevet ,P. Kadirov ,Yu. Pustov ,S. Prokoshkin "Synthesis and characterization of FeMnSi alloys as potential biomedical materials "

Poster Session D: Wednesday August 29
Zh. M. Blednova ,P.O. Rusinov " Synthesis of functionally oriented surface compositions of high-entropy alloys with thermoelastic phase transformation "
F. Niccoli,C. Garion ,C. Maletta ,P. Chiggiato " Shape memory alloy technologies for ultra-high vacuum coupling in particle accelerators "
D. Wagner , P. Didier ,**B. Piotrowski**,P. Laheurte ,D. George "Quantification of the mechanical forces induced by NiTi SMA orthodontic archwires : experiments and numerical modeling "
A. Kushibe,Y. Inoue, K. Umemura, T. Sawaguchi, I. Nikulin, S. Takamori, T. Maruyama, S. Sugimura, K. Tsuzaki " Dynamic Loading Test of Fatigue-Resistant Fe-Mn-Si-based Alloy Seismic Damper "

Poster Session D: Wednesday August 29
T. Iwamoto ,B. Cao " Strain rate sensitivity during loading and unloading processes on shape recovery behavior in Fe-28Mn-6Si-5Cr shape memory alloy "
K. K. Jee, J. H. Han "Some effects of coil orientation change for shape memory alloys "
M. Czerny , T. Tokarski ,W. Maziarz , Y.I. Chumlyakov ,N. Schell , R. Chulist " Superelastic strain in FeNiCoAlTaB single crystals "
N. Resnina,S. Belyaev ,A. Savelieva ,A. Sibirev ,A. Gazizullina ,V. Nikolaev ,R. Timashov ,A. Soldatov " Peculiarities of the shape memory effects in Ni55Fe18Ga27 single crystals "
P. Velvaluri, M. Pravdivtseva , O. Jansen ,E. Quandt " Characterization of thin film flow diverter stents based on shape memory alloys: FEM, CFD and MRI study "
M. Vollmer,T. Arold , C. Lauhoff ,P. Krooß ,T. Niendorf " On the effect of Chromium and Titanium on abnormal grain growth of Fe Mn-Al-Ni-X shape memory alloys induced by cyclic heat treatment"
Y. Inoue,A. Kushibe ,T. Sawaguchi ,I. Nikulin ,S. Takamori ,T. Maruyama ,Y. Chiba , K. Tsuzaki " Mechanical Properties of Fatigue-Resistant Fe-Mn-Si-based Alloy for Seismic Dampers "
W. Trehern,H. Ozcan ,B. Franco , N. Malone ,N. Hite , B. Loveall ,I. Karaman " Ultra-low temperature shape memory effect in CuMnAl alloys "
O. Karakoc,C. Hayrettin ,D. Canadinc ,D. C. Lagoudas ,I. Karaman " Comparison of Actuation Fatigue Performance for Ni-rich NiTiHf and NiTiZr High Temperature Shape Memory Alloys"
U. Tejas,I. Karaman ,A. Talapatra ,R. Arroyave ,R. Santamarta " Effect of composition and thermal processing on transformation characteristics and equilibrium phase stability in NiTiHf high temperature shape memory alloys "
H. H. Saygili, H. O. TUGRUL , B. Kockar " Strain Limited Functional Fatigue Tests of 50,3(at%)Ni-29,7(at%)Ti-20(at%)Hf High Temperature Shape Memory Alloy "
T. Kosorukova, A. Timoshevskii ,Yu. Koval ,Yu. Matviychuk ,G. Firstov "Binary B2 vs. multicomponent high entropy shape memory intermetallics: crystal and electronic structure peculiarities "
E. Timofeeva,E.Yu. Panchenko ,N.G. Larchenkova ,Yu.I. Chumlyakov ,H.J. Maier Two-way shape memory effect in [001]-oriented Ni49Fe18Ga27Co6 single crystals

Contents

Strategies for high entropy shape memory alloy design - from motivation to structure and properties

29 Aug
03:30pm
Amphi 4

Firstov G.[1], Kosorukova T.[1],Timoshevskii A.[1], Koval Yu.[1],Odnosum V.[1],Matviychuk Yu.[1],Gerstein G.[2], Maier H.J.[2]

[1]G.V. Kurdyumov Institute for Metal Physics of the National Academy of Sciences of Ukraine, UKRAINE;
[2]Institut für Werkstoffkunde (Materials Science) Leibniz Universität Hannover GERMANY

The development of conventional shape memory alloys almost came to a halt and the main market is still nitinol for medical applications. The reason why industrial leaders became disenchanted with shape memory alloys is related to functional degradation upon thermal cycling and structural fatigue phenomena. These effects appear due to plastic deformation and/or diffusion processes. To cope with these, the so-called high entropy approach was employed with some success on as-cast multicomponent TiZrHfCoNiCu alloys using binary TiNi as a prototype [1]. Exceptional stability of the shape memory behavior has given some hope to renew industrial interest in shape memory alloy applications. Specifically, these novel high entropy shape memory alloys performed in a much wider temperature range than nitinol and even demonstrated a two-fold increase in yield strength and elastic modulus [2].
The present study is dedicated to the discussion of the key elements of high entropy shape memory alloys design including not just the common parameters like valence electron concentration, atomic size difference, enthalpy and entropy of mixing but also considers structural stability through the analysis of crystal and electronic structure, and interatomic interaction. The formation of different types of high entropy shape memory alloys will be analyzed, namely, multicomponent intermetallic compounds and solid solutions, and directions required for the successful development of high entropy shape memory alloys will be discussed.

[1] G.S. Firstov, T.A. Kosorukova, Yu.N. Koval, V.V. Odnosum, High entropy shape memory alloys, Materials Today: Proceedings 2S, S499 - S504, 2015.

[2] G.S. Firstov, T.A. Kosorukova, Yu.N. Koval, P. A. Verhovlyuk, Directions for High-Temperature Shape Memory Alloys Improvement: Straight Way to High-Entropy Materials? Shape memory and Superelasticity, 1, 400 - 407, 2015.

High Entropy Shape Memory Alloys Effects of Chemical Complexity on Martensitic Transformation

29 Aug
04:00pm
Amphi 4

Piorunek D.,Frenzel J.,Eggeler G.

[1] Institute for Materials (IFM), Ruhr University Bochum, 44801 Bochum, GERMANY

High entropy alloys have recently received significant scientific attention as they allow to study how chemical complexity affects elementary aspects such as phase stability, diffusion and deformation behavior. A few years ago, a new compositionally complex shape memory alloy (SMA) of type Ni-Cu-Co-Ti-Zr-Hf was introduced by G.S. Firstov and co-workers. This material, which is originally based on binary Ni-Ti, shows a reversible martensitic transformation at elevated temperatures. In the present study we investigate how the chemical complexity affects the martensitic transformation in NiTi-based shape memory alloys (SMAs) in terms of phase transformation temperatures, latent heats, cyclic stability and microstructures. We consider Firstov-type high entropy shape memory alloys (HESMAs) and SMAs with compositions from HESMA subsystems with lower configurational entropy. The different SMAs were prepared by arc melting and subsequent heat treatments. The phase transformation behavior was characterized by differential scanning calorimetry (DSC). It was observed that additions of ternary and quaternary elements to binary Ni-Ti significantly affect the dependence of transformation temperatures and latent heats on the Ni-concentration. The results also suggest that an increased chemical complexity provides a solution-strengthening effect which improves functional fatigue resistance. In the present work, we present first results on the formation of martensite in chemically complex SMAs during in-situ cooling in a scanning electron microscope.

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29 Aug
04:20pm
Amphi 4

Engineering the transformation hysteresis by precipitation in NiTi and NiTiHf

Franco B., Dogan E.,Wang S.,Karaman I.

Texas A&M University, USA

The ability to control the transformation temperatures and thermal hysteresis of NiTi and NiTiHf shape memory alloys is of great importance for engineering applications. For example, pipe coupling applications require a wide hysteresis in order to maintain coupling forces in a wide temperature range, while actuator applications require a narrow hysteresis in order to improve response time and efficiency. Aeronautical, automotive, and oil-and-gas environments require higher transformation temperatures, while biomedical and space applications require lower transformation temperatures. In the current work, we performed a systematic study in order to explore how aging heat treatments in a wide range of binary NiTi compositions (50.7-52.4 at% Ni) can be used to control the transformation temperatures and the thermal hysteresis with and without the presence of external stress. Aging treatments between 200°C-550°C for durations between 30 minutes 100 hours were performed, and differential scanning calorimetry was performed to determine the transformation behavior. The resulting data was used to demonstrate general trends in how aging conditions affect the transformation behavior. Additionally, 3-dimensional response surfaces and model fitting were used to generate tools to predict the aging time, temperature, and initial composition required to meet a set of transformation temperature requirements. Several selected conditions were subjected to isobaric-heating cooling in order to characterize the effect of these heat treatments on the transformation behavior under stress. Finally, we demonstrate some results from NiTiHf alloys to show that the same concepts can be extended to ternary NiTi alloys, which greatly extends the application space of heat treated NiTi based SMAs.

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29 Aug
05:10pm
Amphi 4

Experimental and numerical study of monocrystalline CuAlBe endodontic file response subjected to combined torsion-bending loading

Xolin P., Collard C.,Engles-Deutsch M.,Ben Zineb T.

Université de Lorraine, CNRS, Arts et Métiers Paris Tech, LEM3 FRANCE

In endodontics, root canal preparation is mechanized by using Nickel-Titanium (NiTi) files. The superelastic properties of this polycrystalline Shape Memory Alloy (SMA) provide to the dentists a safer, faster and reproducible work. Although, when files evolve inside the root canal, especially when the tooth presents curved roots, they undergo, under cyclic loading combining torsion and bending. These combined loadings induce a local complex stress state with shear and tension-compression components. Therefore, in order prevent any induced corresponding failure and uncomfortable aspiration sense, manufacturers regularly propose new enhanced instruments by optimizing their geometrical parameters and their material properties. To avoid this sensation and improve the ratio softening/strength, we suggest the possible use new family of endodontic files made of a monocrystalline SMA: copper-aluminium-beryllium alloy. In order to show their reliability, their response under bending-torsion complex loading state is numerically and experimentally studied. The experimental part allowed to predict the global response and calibrate the numerical one. This latter leads to the prediction of local stress state in each material point of the instrument. The experimental part considers one device specifically designed in order to apply a torsion and bending rotation and measures the induced corresponding moments. The numerical part allowed to predict the global and local response of the studied instrument by the finite element method. To this end the file-geometry is described and meshed with solid continuum elements. Boundary conditions and loading are applied in consistence with the experimental loading. The monocrystalline SMA behavior is described by two kinds of constitutive law taking into-account the effect of phase transformation. The first law is based on phenomenological approach and developed by Chemisky et al. (2011)[1] whereas the second law is based on micromechanical approach and proposed by Patoor et al. (1989)[2] and improved by Collard et al. (2008)[3]. Obtained results reveals that the deformation angle before failure can be highly increased when bending is combined with torsion compared to pure torsion loading. We show equally difference between NiTi and single crystal CuAlBe SMA files, and how bending and torsion can influence each other to modify the instrument response. The results showed equally an original response of the single crystal Cu-base SMA file.

[1] Chemisky and al., Constitutive model for shape memory alloys incrluding phase transformation martensitic reorientation and twins accomodation, Mechanics of Materials, 2011.

[2] Patoor and al., Thermomechanical behavior of shape memory alloys, ESOMAT 1989.

[3] Collardand al., Micromechanical analysis of precipitate effects on shape memory alloys behaviour, Materials Science & Engineering, 2008.

Shape Memory Effect and Superelasticity in high-strength Fe-based single crystals

29 Aug
05:30pm
Amphi 4

Chumlyakov Yu.,Kireeva I.,Kuksgausen I.,Pobedennay Z.,Panchenko M.,Reunova K., Kuksgausen D.,Kirillov V.

Tomsk State University RUSSIAN FEDERATION

On single crystals of high-strength FeNiCoAlX alloys (X = Ta, Nb, Ti), investigation of the fcc-bcc thermoelastic martensitic transformations, shape memory effect and superelasticity depending on the size of dispersed particles, the test temperature, the orientation of the crystals, the stress state tension/compression were carried out.
It is shown that in tension superelasticity experiments revealed that [001]-oriented crystals with dispersed particles d $<$ 3 5 nm in size showed large reversible strain of 10 15 % to be higher than theoretical strain level of 8.7 %. In compression the reversible strain of 14.3 % is close to theoretical strain level of 15.5 % in [001]-oriented crystals. In tension increasing the particle size d $>$ 5 15 nm leads to decrease in reversible strain and stress hysteresis $\Delta\sigma$. On the contrary, in compression $\Delta\sigma$ increases, this leads to a degeneracy of the superelasticity. For the first time in [001]-oriented crystals with particles d $<$ 3 nm in size, it is shown that in compression stress-induced martensite formed during in a forward fcc-bcc transformation at T = 203 K at $\epsilon = 14\%$ stabilizes. The heating of the stabilized stress-induced martensite leads to reverse transformation at a very high rate, which is accompanied by jump of sample of 3 5 m. With increasing the particle size d $>$ 5 10 nm such effect of burst-like behavior of fcc-bct martensitic transformation is not observed.
Physical causes of abnormally large reversible strain in tension are attributed to fcc-bcc thermoelastic martensitic transformations, and then $< 110 >$ 110 elastic twinning are discussed. The effect of $< 112 >$ 111 detwinning of the bct martensite on the transformation strain, the stress hysteresis $\Delta\sigma$ and the stabilization of stress-induced martensite are analyzed. In the same way, the asymmetry of the transformation strain, stress hysteresis $\Delta\sigma$, stabilization of stress-induced martensite in tension/compression is considered.

Behaviour of Ni-rich Ni-Ti-Zr shape memory alloys upon low temperature aging

29 Aug
05:50pm
Amphi 4

Xu S.[1], Pons J.[1], Santamarta R.[1],Karaman I. [2]

[1]*Departament de Fsica, Universitat de les Illes Balears, E07122 Palma de Mallorca, SPAIN;*
[2]*Department of Materials Science and Engineering, Texas A&M University, College Station, TX 77843, USA*

Slightly Ni-rich NiTiHf and NiTiZr alloys are considered as serious candidates for high temperature shape memory alloy applications, since they can exhibit good mechanical and functional properties above 200 ˚C when they are reinforced with nanoprecipitation of the so-called H-phase. However, the microstructures and functionalities of any HTSMA might be modified by long exposure of the material to high working temperatures.
In a previous work [1], the B2 phase of Ni50.3Ti29.7Zr20 was shown to be unstable upon prolonged ageing at 250 ˚C and undergo short range atomic reordering processes producing diffuse streaks in the diffraction patterns, which were considered as a precursor state to the H-phase precipitation that takes place upon ageing at higher temperatures (above $\sim$ 450 ˚C).
In the present work, new results of the instability of the B2 phase in Ni50.3Ti29.7Zr20 and Ni50.3Ti24.7Zr25 will be presented. For solution heat treated Ni50.3Ti29.7Zr20 samples, short ageing times (a few hours) at 250˚C produce a progressive decrease of the direct martensitic transformation temperatures until its complete suppression. The alloy with 25 at% Zr is more prone to decomposition and presents a completely suppressed martensitic transformation just after solution heat treatment, without any further ageing. Both alloys with suppressed martensitic transformation exhibit the typical features of the strain glass state.

[1] A.M. Perez-Sierra, J. Pons , R. Santamarta, I. Karaman, R.D. Noebe, Scripta Materialia 124, 47 - 50 ,2016.

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30 Aug
10:50am
Amphi 4

Development and Testing of a High - Temperature Shape Memory Actuator using CuAlNi for Hold Down and Release Mechanisms (HRM)

Fouché F.[1], Hautcoeur A.[1], Chassoulier D.[2], Blanc A.[2],Sicre J.[3]

[1] *Nimesis Technology FRANCE;*
[2] *Thales AleniaSpace FRANCE;*
[3] *CNES FRANCE*

Hold down and Release Mechanisms (HRM) are used in space applications, during launch, to hold rigidly a mobile part (antenna, solar array, mirror, etc.) on the satellite structure. Usually a tie rod tightened makes the link between the mobile and the fixed parts. This tie rod must be released to deploy the appendices. This operation is done by a pyrotechnic device or a low-shock unit.
In both cases, a shock (high or low) is exported on the satellite, due to the quick release of the tie rod tension.
The idea behind this project is to slowly release the tension before releasing the tie rod itself.
Copper based Shape Memory actuators manufactured by Nimesis are a good candidate to address this problematic:

- They generate a slow action (graduated in minutes),
- They are reliable,
- They can lengthen or shrink way more than classic metals while heating them,
- They can withstand the HRM high thermal environment (temperatures from -120°C to +120°C) before triggering (above 120°C).

The goal of this project is to develop a CuAlNi Shape Memory actuator for a specific HRM with triggering temperature above 120°C and able to reach 160 or 200°C. Benefits of this actuator make it interesting not only for HRM but also for any devices that must be actuated at high temperature (antenna or panels deployment, spacecraft dismantlement,...).
This paper presents the development of an improved version of THALES ALENIA SPACE HRM NG (Hold Down & Release Mechanism New Generation) with high triggering temperature. This mechanism will hold, store, untighten, then release the appendices. The CuAlNi Shape Memory actuator developed for this HRM allows a high temperature stress release of the holding tie-rod without any induced shock on the satellite. This development aims at demonstrating the feasibility of the concept through :

- Functional tests on a breadboard (in the air and under vacuum, in ambient and cold cases),
- Vibration tests,
- Exported shock measurement.

Development will cover qualification tests required to qualify CuAlNi as space approved material.

The comparison of effect of different treatments on the high temperature superelasticity in Ni50.3Ti32.2Hf17.5 polycrystals

30 Aug
11:10am
Amphi 4

Tagiltsev A.I.[1],Panchenko E.Y.[1],Chumlyakov Y.I.[1], Karaca H.E.[2],Karaman I. [3]

[1]*National Research Tomsk State University RUSSIAN FEDERATION;*
[2]*Kotelnikov Institute of Radio Engineering and Electronics, RAS, Moscow, RUSSIADepartment of Mechanical Engineering, University of Kentucky, Lexington, USA;*
[3]*Department of Materials Science and Engineering, Texas A&M University, College Station, USA*

The effect of different thermal treatment on the high-temperature superelasticity (SE) was investigated in the Ni50.3Ti32.2Hf17.5 (at. %) polycrystals, which undergo B2-B19' thermoelastic martensitic transformations (MTs). Polycrystals of NiTiHf alloy are famous for their high transformation temperatures (TTs), but the absence of SE limits their application. Now the common method to induce SE in polycrystals is the decrease of grains size due to extrusion followed by ageing [1]. In present study polycrystals in initial state with grains size of 27 μm were used, because of their cheapness.
In order to induce SE in as-cast polycrystals there were chosen two treatments: stress-induced martensite ageing (SIM-ageing) (regime is: 550 MPa of applied stresses at 150°C, 1h. in stress-induced martensite) and high-temperature heat treatment including homogenization at 1050°C, 8h. with air cooling, followed by heat treatment at 900°C, 3h. continued with quenching into water. It is shown [2],that heat treatment at 900°C, 3h. doesnt result in precipitation of dispersed particles.
The both of treatments induce a high-temperature SE in polycrystals. High temperature treatment results in significant increase of critical stresses on 300 450 MPa at the same temperature compared with initial state. The SE is observed from 175°C to 250°C and the following increase of stresses up to 1400 MPa leads to the destruction of samples. It should be noted, that the α coefficient characterizing the increase of stresses with the temperature growth decreases from 8.0 MPa/°C to 5.9 MPa/°C. Low α coefficient and higher strength properties provide larger temperature interval of SE.
The SIM-ageing provides next results: the SE interval is about 40°C and begins at 170°C. The increase of critical stresses at the same temperature compared with the initial state is 100 MPa, and the α coefficient doesnt change (8.0 MPa/°C). However, the SIM ageing provides not only high-temperature SE, but also the high-temperature two way shape memory effect (TWSME) with the maximum strain of 0.9($\pm$ 0.2) %. It should be noted, that the maximum reversible strain of shape memory effect (SME) in initial state doesnt exceed 1.1($\pm$0.2) %, so the value of TWSME is almost the same as SME. The TTs after the both SIM-ageing and high-temperature heat treatment dont shift.
Physical reasons of effect of heat treatments on microstructure and functional properties in high-strength NiTiHf polycrystals are under discussion.

[1] H.E. Karaca et. al., Acta Mater., 61,7422- 7431, 2013.

[2] S.M. Saghaian et. al., Acta Mater., 134 ,211 - 220, 2017.

Comparison of the Transformation Behavior of Cold Rolling with Aging and Hot Extrusion with Aging Processed Ni50.3Ti29.7Hf20 High Temperature Shape Memory Alloy

30 Aug
11:30am
Amphi 4

Tugrul H. O., Saygili H. H., Kockar B.

Hacettepe University, Mechanical Engineering Department, 06800, Beytepe, Ankara, TURKEY

Among NiTiHf high temperature shape memory alloys, nickel rich ternary Ni50.3Ti29.7Hf20 (at%) alloy has been studied extensively and found to be promising for high temperature applications especially in aerospace industry. NiTiHf alloys have very high strength and transformation temperatures proportional to their hafnium percentage. Therefore, these alloys are accepted as a hard to deform material. Hot extrusion at 900°C and solutionizing at 1050°C - 1100°C treatments have been generally used in literature for the homogenization of the cast microstructure and chemistry. In this study, one set of the as cast Ni50.3Ti29.7Hf20 alloy was hot extruded at 900C and then aged at 550°C for 3 hrs and the other set was solutionized at 1050°C for 2 hrs, cold rolled for 10% at room temperature and then aged at 550°C for 3 hrs. The transformation temperatures of the samples were measured using differential scanning calorimetry (DSC) and it was found that cold rolling led to a decrease in transformation temperatures despite the increasing transformation temperature effect of aging treatment due to the increase in the dislocation density. Heating-cooling experiments under increasing stress magnitudes starting from 50 MPa and increasing up to 600 MPa were conducted on cold worked with aging and hot extruded with aging processed alloy in order to compare the shape memory characteristics such as actuation strain, irrecoverable strain, transformation temperatures and overheating-undercooling magnitudes. Both of the samples showed very high dimensional stability with no irrecoverable strain values up to 500MPa. However, the actuation strain magnitudes of the cold rolled with aging processed sample was half of the sample which was hot extruded and aging treated. Additionally, higher undercooling and overheating were necessary to achieve full transformation in the cold worked samples. These results might be due to the very high dislocation density which was obtained during cold rolling process. Increasing the dislocation density leads to a decrease in the austenite-martensite mobility during transformation.

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30 Aug
11:50am
Amphi 4

In situ characterization of functional properties in polycrystalline Co-Ni-Ga high-temperature shape memory alloys From bicrystals to hot-rolled material

Lauhoff C.[1], Vollmer M.[1],Krooß P.[1],Paulsen A.[2],Frenzel J.[2], Chumlyakov Y.I.[3], Niendorf T. [1]

[1]*Institut für Werkstofftechnik (Materials Engineering), University of Kassel GERMANY;*
[2]*Institut für Werkstoffe, Ruhr-Universität Bochum GERMANY;*
[3]*Siberian Physical Technical Institute, Tomsk State University ,RUSSIA*

High-temperature shape memory alloys (HT-SMAs) have gained increasing attention in recent years. Due to their attractive functional properties at elevated temperatures, which are based on a fully reversible phase transformation from a high-temperature austenitic phase to a low-temperature martensitic phase, these alloy systems are of great interest for applications in the automotive, aerospace and energy sectors, where the operating temperatures often exceed 100°C. Both, actuation and damping applications are feasible. Over the last few decades, ternary Ni-Ti-X (X = Pt, Pd, Hf, Zr), Cu-based, Ni-Al-based and Ti-Ta-based alloys have been identified as attractive HT-SMA candidates. However, most HT-SMAs consist of expensive alloying elements and/or suffer from a brittle material behavior, finally hindering their wide spread application. In addition to this, many of these HT-SMAs show poor cyclic stability in both, thermal cycling and mechanical cycling at elevated temperatures, caused by distinct microstructural instabilities due to precipitation of secondary phases and plasticity. In this regard, Co-based Heusler-type Co-Ni-Ga alloys received considerable attention. Relatively inexpensive constituent elements, an improved workability due to the potential precipitation of a ductile second phase and a fully reversible pseudoelastic response up to 500 °C make them promising HT-SMA alternatives to Ni-Ti-based HT-SMAs. This study reveals the mechanisms responsible for degradation of functional and structural properties in polycrystalline Co-Ni-Ga HT-SMAs. Detailed microstructure analysis focusing on the meso- to micro-scale using in situ techniques employing optical and scanning electron microscopy was conducted. Results obtained allowed for correlation between the phase transformation, local microstructural features and evolution of structural as well as functional damage. Due to highly anisotropic material behavior Co-Ni-Ga HT-SMA polycrystals suffer from intergranular constraints. Grain boundary (GB) characteristics, i.e. GB misorientation, the orientation of the GB with respect to the loading direction and precipitation of secondary phases, are critical parameters to be considered in order to obtain a deep understanding of the damage evolution and, finally, to allow for adequate microstructure design.

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30 Aug
12:10am
Amphi 4

Synthesis and characterization of FeMnSi alloys as potential biomedical materials

Zhukova Y.,Drevet R.,Kadirov P.,Pustov Yu.,Prokoshkin S.

National University of Science and Technology "MISIS" RUSSIAN FEDERATION

The Fe-Mn-Si alloys are promising materials as biodegradable metallic implants for temporary healing processes inside thProkoshkine human body. Their appropriate degradation properties in a physiological environment are used to avoid re ProkoshkinpeatedProkoshkin surgical intervention for the implant removal after the Drevetcompletion of the healing process. Due to their shape memory and superelastic properties, such alloys can be used for temporary coronary stents applications. Indeed, the Ms temperature of the γ-austenite ϵ-martensite transformation decreases as the manganese content increases in the alloy and approaches the body temperature at 30 wt.% Mn. Moreover, this alloy is also expected to be used as a bone implant due to its low Youngs modulus (E = 118 GPa) that makes it more biomechanically compatible with bone tissues than pure iron (E = 220 GPa).

This work presents the synthesis and characterization of three Fe-(23-30)Mn-5Si (wt.%) alloys which physicochemical and mechanical characteristics make them promising for biodegradable materials. The phase composition and martensitic transformation features are modified as a function of the manganese content in the alloy. The Ms temperature of the γ-austenite $\rightarrow$ ϵ-martensite transformation decreases as the manganese content increases and approaches the body temperature at 30 wt.% Mn. That provides conditions for the premartensitic lattice softening and related lowering of the Youngs modulus, and indicates the way for the shape memory realization at sufficiently low temperature if required.
The Youngs modulus of the studied alloys is twice lower than that of pure iron, particularly for the highest manganese amount. This mechanical property together with sufficiently high yield stress makes these materials promising candidates for implant applications from the viewpoint of biomechanical compatibility and mechanical reliability.
The study of the corrosion properties of the Fe-Mn-Si alloys, pure iron and iron-manganese binary alloy in Hanks solution at 310 K (37°C) shows that the corrosion rate of the Fe-Mn alloy is much higher than that of iron. The silicon addition to the binary alloy increases the corrosion rate.
The Fe30Mn5Si alloy shows the most promising properties for biomedical applications as a biodegradable implant material which combines high biodegradation rate with biomechanical compatibility and reliability during lifetime.

Dynamic Loading Test of Fatigue-Resistant Fe-Mn-Si-based Alloy Seismic Damper

29 Aug
02:00pm
Poster
Session D

Kushibe A.[1],Inoue Y.[1],Umemura K.[1],Sawaguchi T.[2],Nikulin I.[2], Takamori S.[2],Maruyama T.[3], Sugimura S.[3], Tsuzaki K.[4]

[1]*Research and Development Institute, Takenaka Corporation JAPAN;*
[2]*National Institute for Materials Science JAPAN;*
[3]*Awaji Materia Co.,Ltd. JAPAN;*
[4] *Kyushu University JAPAN*

In recent years, large amplitude cyclic vibrations of high-rise buildings due to the long-duration and long-period ground motion have become a new problem. Therefore, the performance requirements for the seismic dampers that are installed in vibration-controlled buildings have increased dramatically and durability to cyclic deformation has become essential. In this context, we have developed fatigue-resistant seismic dampers that are made of a Fe-15Mn-4Si-10Cr-8Ni damping alloy are designed to counteract long-period ground motion. This shear panel-type seismic damper exhibits a hysteretic damping effect owing to its elastoplastic deformation. Under a maximum rated deformation angle of 1/25 rad, the damper can bear loads up to approximately 4,000 kN, which places it in the highest class of steel dampers. This study aims to verify the deformation performance and fatigue resistance of the developed seismic damper. Because of the load limit of the force-application actuator, the test samples were made with a shape similar to that of a seismic damper but with a height, width, and thickness for the movable portion of the alloy panel scaled to half of the actual size. To confirm stable deformation performance, the strain was progressively increased until the sample exhibited a deformation angle of 1/25 rad. Three cycles of sinusoidal wave with a period of 5 s (used on the basis of the natural period of the vibration of a typical high-rise steel building) were applied with various amplitudes. No discontinuous load decreases due to buckling or failure were observed during this test and relation between the load and deformation angle was stable over a range of deformation angles, resulting in a stable hysteresis loop. To examine the fatigue endurance of the seismic dampers, strain-controlled low-cycle fatigue tests were performed by applying a sinusoidal wave with a period of 5 s and were executed at representative share deformation angles of 1/100, 1/50, and 1/25 rad. The test results revealed that the developed damper has a significantly superior fatigue life in comparison with the conventional steel dampers and offers outstanding deformation performance and durability under low-cycle fatigue. In 2014, sixteen seismic dampers made of Fe-Mn-Si-based alloy were installed on floors one through four of a 196-meter-tall high-rise building. Structural analysis of this building revealed that it is possible to achieve an extra-high-grade vibration control with performance margins that allow the building to withstand long-period, long-duration ground motion, and repeated after-quakes.

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29 Aug
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Poster
Session D

Synthesis of functionally oriented surface compositions of high-entropy alloys with thermoelastic phase transformations

Blednova ZH. M.,Rusinov P.O.

Kuban State Technological University, Krasnodar RUSSIAN FEDERATION

To expand the field of practical use of high-entropy alloys with thermoelastic phase transformations, a technology has been developed for the formation of functionally oriented surface layers of five and six-component powder compositions CoCuTiZrHf, NiCoTiZrHf, NiCuTiZrHf, ZrCuNiCoTi, TiNiZrHfCoCu. The coatings were formed under high-energy conditions, including mechanical activation of the deposited material, high-speed gas-flame spraying in a chamber in argon medium with subsequent thermal and thermomechanical processing in a single technological cycle on patented equipment. The electron microscopic and X-ray diffraction studies showed that the resulting coatings in structure and phase state correspond to materials with shape memory effect (SME) with a grain size of 80-400 nm. The forecasting of the cyclic durability of steel samples with a surface layer of multicomponent highly entropy materials with an SME based on the energy criterion is performed, taking into account the energy content of the coating material and the structural factor. The energy intensity of the alloy was determined from the thermodynamic characteristics and the structural-phase state, and the structural factor of the alloy based on the estimation of the system's adaptability, determined by the multifractal parameterization of the structure. Using the example of the complex-rich highly entropic material cBN-Co-NiAlY, all stages of the formation of the surface composition from surface preparation and mechanoactivation of the sprayed material to the formation of a composition and thermomechanical processing providing the functional properties of a material with an SME.

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29 Aug
02:00pm
Poster
Session D

Ultra-low temperature shape memory effect in CuMnAl alloys

Trehern W.,Ozcan H.,Franco B., Malone N.,Hite N., Loveall B.,Karaman I.

Department of Mechanical Engineering, Texas A&M University, College Station TX USA UNITED STATES

Ultra-low temperature shape memory alloys (SMAs) a newly emerging class of active materials with applications in the aerospace industry and cryogenic environments. CuMnAl alloys are advantageous because of their low cost and good formability. However there is a lack of data on the shape memory response at ultra-low (¡-100˚C) temperatures. In this work, we have shown for the first time that CuAlMn SMAs can show shape memory effect below -100˚C. Additionally, the compositional dependence of transformation temperatures are investigated using physical property measurement system (PPMS) and it is found that CuMnAl alloys can transform as low as -181˚C. Shape memory properties are characterized using isobaric heating cooling experiments at different stress levels and superelastic response is investigated at different temperatures.

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29 Aug
02:00pm
Poster
Session D

Strain rate sensitivity during loading and unloading processes on shape recovery behavior in Fe-28Mn-6Si-5Cr shape memory alloy

Iwamoto T.[1],Cao B. [2]

[1]*Academy of Science and Technology, Hiroshima University JAPAN;*
[2]*Graduate School of Engineering, Hiroshima University JAPAN*

Fe-based shape memory alloy (Fe-SMA) can be separately employed with NiTi shape memory alloy in different applications. However, in order to utilize the Fe-SMA to further applications with great advantages such as cost-reduction, the improvement of the shape memory effect (SME) is required. To improve the SME of Fe-SMA, the thermomechanical treatments has have been reported.

However, the investigations of the thermomechanical treatments have been focused on levels of pre-straining as well as temperature for the straining and number cycles. However, few studies about the SME after the treatments at different deformation rate can be found about the SME after thermomechanical treatment at different deformation rate. Therefore, in order to obtain the optimum thermomechanical treatments, it is necessary to study the SME as well as the mechanical properties at different deformation rate.

Recently, some related research works indicated that the SME of Fe-SMA is sensitive negatively on loading strain rate under tensile tests. That is, the SME decreases with increasing loading strain rate. Nevertheless, the unloading strain rate is constant or unconsidered in these studies. Up to now, the strain rate sensitivity for unloading on the shape recovery behavior in Fe-SMA is still unclear. In the case of the Cu-based shape memory alloy, some research works reported that the temperature of the sample differs significantly in the tensile tests at different martensitic transformation. In other words, it is possible to enhance the SME of Fe-SMA by thermomechanical treatment based on different loading and unloading strain rate.

In the present study, to investigate the strain rate sensitivity for loading and unloading processes on the SME of Fe-SMA individually, tensile tests of rounded bar specimens made of Fe-28Mn-6Si-5Cr alloy, which is a kind of commercially supplied Fe-SMA, at maximum strain of 2% for different loading and unloading strain rate are conducted. After that, the specimen unloaded after tensile test is heated over austenite start temperature. Furthermore, the shape recovery strain and residual strain are measured by using high temperature strain gauge. Finally, the microstructural observation in the alloy is carried out to confirm the martensitic transformation.

Binary B2 vs. multicomponent high entropy shape memory intermetallics: crystal and electronic structure peculiarities

29 Aug
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Poster
Session D

Kosorukova T., Timoshevskii A.,Koval Yu.,Matviychuk Yu.,Firstov G.

G.V. Kurdyumov Institute for Metal Physics of the National Academy of Sciences of Ukraine, UKRAINE

It is known that some of the B2 intermetallic compounds have the tendency to undergo martensitic transformation and, therefore, exhibit shape memory behavior. In addition to the most famous TiNi, there is quite a number of AB compounds (A - Ti, Zr, Hf, Nb, Ta; B - Co, Ni, Cu, Ru, Rh, Pd, Ir, Pt, Au) that belong to this kind. It was shown for ZrCu and confirmed for TiNi that the reason for structural instability upon B2 phase symmetry decrease at martensitic transformation is the appearance of the B-B interatomic interaction in addition to the existing interaction of the A-B type in the high temperature B2 phase [1].

Recently, it was found that novel multicomponent high entropy shape memory alloys [2] are in fact intermetallics with distorted B2 structure [3]. Present report intends to underline similarities and differences in crystal and electronic structure while moving from binary to multicomponent shape memory intermetallics.

[1] G. Firstov, Yu. Koval, J. Van Humbeeck, A. Timoshevskii, T. Kosorukova, P. Verhovlyuk, Some physical principles of high temperature shape memory alloys design. In: N. Resnina and V. Rubanik (eds.) Shape Memory Alloys: Properties, Technologies, Opportunities, pp. 207 - 232, Trans Tech Publications Inc., Zurich, 2015.

[2] G.S. Firstov, T.A. Kosorukova, Yu.N. Koval, V.V. Odnosum, High entropy shape memory alloys, Materials Today: Proceedings 2S, S499 - S504, 2015.

[3] G. Firstov, A. Timoshevskii, T. Kosorukova, Yu. Koval, Yu. Matviychuk, P.Verhovlyuk, Electronic and crystal structure of the high entropy TiZrHfCoNiCu intermetallics undergoing martensitic transformation, MATEC Web of Conferences, 33, 06006, 2015.

Some effects of coil orientation change for shape memory alloys

29 Aug
02:00pm
Poster
Session D

Jee K. K.[1], Han J. H. [2]

[1]*Center for Opto-Electronic Materials and Devices, Korea Institute of Science and Technology REPUBLIC OF KOREA ;*
[2]*Div. of Nano Materials Engineering , Chungnam National Univ., Gung-dong, Yuseong-gu, Daejeon 305- 764, KOREA*

The present authors have invented a new method of changing coil orientation of a coil spring. The major merits of coil orientation change for SMA are reduction of space and control of two way shape memory effect. For instance, when a SMA coil is designed to work by lifting a load on heating above Af, the coil does not completely contracts. The leftover strain, attributed to elastic deformation of high temperature phase by the load, wastes a space. The elastic strain can be eliminated by changing coil orientation, reducing the necessary space. When a SMA actuator is designed using an extension SMA spring and a bias spring, more space reduction is expected. In case of SMA actuator with coil bias spring, miniaturization is expected due to space saving of both SMA and bias spring. Besides generation and control of two way shape memory effect is presented using the reverse of coil orientation.

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29 Aug
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Poster
Session D

Superelastic strain in FeNiCoAlTaB single crystals

Czerny M.[1], Tokarski T.[2],Maziarz W. [1], Chumlyakov Y.I.[3],Schell N.[4], Chulist R.[1]

[1]*CInstitute of Metallurgy and Materials Science, Polish Acade my of Sciences, Krakow, POLAND ;*
[2] *AGH University of Science and Technology, Academic Centre for Materials and Nanotechnology, Al. Mickiewicza 30, Krakow, POLAND;*
[3] *Siberian Physical-Technical Institute, Tomsk, RUSSIA;*
[4] *Institute of Materials Research, Helmholtz-Zentrum Geesthac ht, Max-Planck-Strasse 1, D-21502 Geesthacht, GERMANY*

Fe-based alloys (Fe-28Ni-17Co-11.5Al-2.5Ta-0.05B abbreviated NCATB) belong to a new family of shape memory alloys exhibiting a large superelastic strain. The superelastic effect is strongly related with precipitation hardening which is required to suppressed the plastic deformation of the parent phase. The main strengthening component of these alloys is the fcc γ' phase. In this paper the effect of heat treatment on precipitation hardening in multicomponent single crystalline NCATB materials to obtain optimal mechanical properties is studied. Heat treatment was performed for single crystal with $< 100 >$ and $< 111 >$ orientations at temperatures 973 K and for variable times (0.5h, 1h, 5h, 10h, 24h). Three different intermetallic phases of NiAl, Ni3Al and NiAl3 are analyzed with synchrotron diffraction to reveal the evolution of strengthening phases. Subsequently, to provide an insight into the mechanism of superelasticity observed in the NCATB alloys, single crystals with$< 100 >$, $< 110 >$, $< 111 >$ and $< 112 >$ orientations were compressed at different temperatures (77, 123, and 295 K). Depending on the orientation and deformation temperature elastic, plastic or elasto-plastic response are observed. The global and local orientation measurements were determined by diffraction of high-energy synchrotron radiation and electron backscatter diffraction, respectively. The results are discussed with respect to crystallographic orientation, deformation mode, precipitations and phase transformations.

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Shape memory alloy technologies for ultra-high vacuum coupling in particle accelerators

Niccoli F.[1],Garion C.[1],Maletta C.[1,2],Chiggiato P. [1]

[1]*CERN, European Organization for Nuclear Research, Geneva 23, SWITZERLAND;*
[2]*Department of Mechanical, Energy and Management Engineering (DIMEG), University of Calabria, Rende (CS), ITALY*

A new shape memory alloys (SMAs)-based system for ultra-high vacuum (UHV) coupling applications in particle in accelerators, is currently under investigation at the European organization for nuclear research (CERN). The use of SMAs as beam-pipe connectors in some restricted-access radioactive areas at CERN could result in noticeable advantages, especially during maintenance operations. In fact, bolt-free SMA couplers, can be activated remotely by temperature changes, resulting in significant reduction of the radiation doses collected by the technical personnel. The alloy composition of the SMA element has been selected and special thermo-mechanical training processes have been designed, to satisfy strict functional constraints. Both ternary (NiTiNb) and binary (NiTi) alloy systems have been analyzed and the main application limits/advantages for the two material classes have been outlined. Results have shown that leak rate constraints for UHV applications could be easily satisfied (leak rate <10-10 mbar l s-1). In addition, the thermal mounting/dismounting has been always obtained even after multiple actuation cycles. Furthermore, the effects of ionizing radiation on both mechanical and functional response of SMA couplers are under investigation by using special facilities within the CERN accelerator complex. Preliminary results have shown that leak tightness and thermal dismounting are mainly unaffected by irradiation (up to $\sim$ 140 kGy of absorbed dose).
Thanks to these results, possible applications in the field of particle accelerators and, in particular, in restricted access areas, are considered.

Comparison of Actuation Fatigue Performance for Ni-rich NiTiHf and NiTiZr High Temperature Shape Memory Alloys

29 Aug
02:00pm
Poster
Session D

Karakoc O.[1],Hayrettin C.[1],Canadinc D.[1,3], Lagoudas D. C.[2,1],Karaman I. [1]

[1] *Department of Materials Science and Engineering, Texas A&M University, College Station, Texas 77843 USA;*
[2] *Department of Aerospace Engineering, Texas A&M University, College Station, Texas 77843 USA;*
[3] *Department of Materials Science and Engineering, Texas AM University, College Station, USADepartment of Mechanical Engineering, Koç University, 34450 Istanbul, TURKEY*

The focus of this study is centered on investigation of actuation fatigue performance of Ni-rich NiTiHf and NiTiZr alloys. After proper heat treatment, Ni-rich NiTiHf and NiTiZr alloys exhibit decent shape memory properties due to formation of nano-scale H-phase precipitates. To implement these promising alloys in the high temperature applications, characterization of actuation fatigue performance of these alloys is needed. Fatigue test of specimens under different load levels from 200 to 500MPa is performed until failure of samples through-thermally induced phase transformations. Microstructural characterization displays thermal and mechanical evolution of microstructure prior to test and after failure of specimens using transmission and scanning electron microscopy, and differential scanning calorimetry. The fatigue test results indicate that Ni-rich Ni50.3Ti29.7Hf20 attains more fatigue life than its counterpart Ni-rich Ni50.3Ti29.7Zr20 at all applied stress levels. The fatigue life difference amongst them becomes more distinguishable as stress level increases. For instance, Ni-rich Ni50.3Ti29.7Hf20 achieves average 4,400 cycles at 400MPa while fatigue life is reduced to 180 cycles for Ni-rich Ni50.3Ti29.7Zr20. At 200MPa, Ni-rich Ni50.3Ti29.7Hf20 alloy has two times more number of cycles to failure than its counterpart. Comparison of actuation strain reveals that Ni-rich Ni50.3Ti29.7Hf20 provides advantages over its counterpart since Hf-based alloy exhibits 2.50% on the average at 300MPa, whereas actuation strain drops to 1.40% for Zr-based alloy. Evolution of actuation strain for both alloys follow similar trend, start from maximum value and decrease continuously until failure of specimen. Conversely, irrecoverable strain at failure is lower for Zr-based alloy than Hf-based alloy at all stress levels. However, it may stem from earlier failure of Zr-based alloys since more repeated transformations causes more plastic deformation. After extensive investigations by many researchers on microstructure of Hf and Zr-based alloys, current work becomes first fundamental research to compare actuation fatigue behaviours of Ni-rich Ni50.3Ti29.7Hf20 and Ni-rich Ni50.3Ti29.7Zr20 alloys.

Quantification of the mechanical forces induced by NiTi SMA orthodontic archwires : experiments and numerical modeling

29 Aug
02:00pm
Poster
Session D

Wagner D.[1], Didier P.[2],Piotrowski B.[2],Laheurte P.[2],George D. [3]

[1] *Dental Faculty, 8, rue Sainte Elisabeth, 67000 Strasbourg, FRANCE;*
[2] *Université de Lorraine, CNRS, Arts et Métiers ParisTech, LEM3, F-57000 Metz, FRANCE;*
[3] *ICUBE, CNRS, Université de Strasbourg, 67000 Strasbourg, FRANCE*

Orthodontic tooth movement (OTM) is based on a prolonged application of a mechanical force into a tooth bracket, leading to the tooth displacement through the remodeling of the surrounding bone. The application of both forces and moments leads to tissue remodeling at the level of the dental pulp, the periodontal ligament (PDL), the alveolar bone and the gingiva. This bone remodeling depends on the biological reactions occurring within the PDL, related to vascular changes through its mechanical deformation. One of the major challenges for orthodontists is to apply the "ideal force" to obtain the desired movement (tipping, translation, intrusion, extrusion, rotation or root displacement). Light forces are preferred to heavy ones, cause tissue necrosis resulting in a delay in the dental displacement, and undesirable side effects such as pain, root and bone resorption.
The orthodontic treatments are patient dependent and related to the efficiency of the used archwires. Adopted methodologies to reduce the misalignment of teeth are constituted by the consecutive use of various orthodontic wires, differing in their section or material, and chosen on the basis of experimental in - vitro and animal knowledge, and clinical experience. Orthodontists rely primarily on their clinical sense. Available predictive models are limited and based on a number of simplifying assumptions (2D).
In this study, we propose to develop a 3D Finite Element Model (FEM) to predict the reaction forces applied on the teeth on a real geometrical case, and depending on the material parameters of the orthodontic wire. This work requires to (i) conduct an experimental characterization study of the friction developed between the wire and brackets, (ii) determine through an in-house developed experimental set-up the wire reaction forces on the teeth, (iii) develop a FEM accounting for the phase transformation

of NiTi wire, and (iv), validate the numerical model by experimental/numerical comparisons. The developed numerical model can then be used as a predictive numerical tool for orthodontists to choose the optimum wire configurations for the patient treatment.

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29 Aug
02:00pm
Poster
Session D

Peculiarities of the shape memory effects in Ni55Fe18Ga27 single crystals

Resnina N.[1],Belyaev S.[1],Savelieva A.[1],Sibirev A.[1],Gazizullina A.[1],Nikolaev V. [2],Timashov R. [2],Soldatov A. [2]

[1]*Saint Petersburg State University RUSSIAN FEDERATION;*
[2]*Ioffe Institute RUSSIA*

The aim of the work was to study the one way and two-way shape memory effects in Ni55Fe18Ga27 single crystals with different orientations. The [001] and [011] single crystals were grown by the Czochralski method using [001] and [011] seed crystals. The sample was compressed at room temperature at which the alloy was in martensite 10 M state, unloaded, heated up to 150 ˚C at which the alloy was in the austenite state to study the one-way shape memory effect and subjected to cooling and heating within a temperature range of 25 to 150 ˚C to observe the two-way shape memory effect.
It was shown that despite the single crystal orientation the maximum value of the one-way shape memory effect was 5%. It was found that the recovery coefficient was equal to 90 % and did not change on an increase in the preliminary strain up to 7.5% (for [001] crystal) and 10 % (for [011] crystal). Further preliminary compression to 20 % led to a decrease in the recovery coefficient to 60 % in [001] crystal and to 45 % in [011] crystal. Thus, it was concluded that the [001] single crystal demonstrated the better resistance to the irreversible strain appearance during preliminary compression than [011] crystals. The martensite stabilization effect was found on heating of the preliminary compressed single crystals. It led to a decrease in a temperature range of the one-way shape memory effect and it was observed that 80 % of strain sharply recovered at constant temperature As while the 20 % of strain recovery took place on further heating from As to Af. During the two-way shape memory effect the strain varied smoothly in temperature ranges of the forward and reverse transformations. It was found that the value of the two-way shape memory effect depended on the preliminary strain in non-monotonic way. The maximum recoverable strain of 2.6 % was found in [011] crystal that was preliminary compressed to 7.5 % and it was four times larger than the maximum value of the two-way shape memory effect found in [001] crystal after compression to 10 %.

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29 Aug
02:00pm
Poster
Session D

Strain Limited Functional Fatigue Tests of 50,3(at%)Ni-29,7(at%)Ti-20(at%)Hf High Temperature Shape Memory Alloy

Saygili H. S., TUGRUL H. O., Kockar B.

Hacettepe University, Mechanical Engineering Department TURKEY

Strain limited functional fatigue tests were performed on 50,3(at%)Ni-29,7(at%)Ti-20(athigh temperature shape memory alloy (HTSMAs) in this study. High purity elemental Nickel, Titanium and Hafnium materials were used in order to fabricate 50,3(at%)Ni-29,7(at%)Ti-20(at%)Hf HTSMA. Material was hot extruded at 900C with an area reduction of 12:1 after it was fabricated via vacuum induction melting under high purity argon atmosphere. Tensile specimens were cut via electrical discharge machining in flat dog bone shape from bulk material. Specimens were aged at 550˚C for 3 hours in a furnace under high purity argon atmosphere and then, water quenched.
Functional fatigue experiments were conducted in a manner that the complete austenitic transformation was prohibited. The strain at martensite phase was selected as a reference and the specimens were heated until they reached to a specified recoverable strain level which was less than the recoverable strain value of the specimens for full austenitic transformation. Specimens were then cooled below the martensite finish temperature and the heating-cooling cycles were repeated. Reference martensite strain value was updated for each cycle in order to eliminate the effect of accumulated irrecoverable strain. Specimens were heated via electrical current passing through the specimens, while forced air convection was used for cooling. Measurements and control of the test system were performed on a program scripted in LabView. Functional fatigue tests were performed under 200 MPa constant stress level and 2% strain was set as the limited value for the experiment. Specimen showed the limited strain value successfully

until it fractured at the end of the 15500 cycles. However, upper cycle temperature (UCT), which was the temperature value in order to achieve the preset limited strain value, increased with the increase in the number of cycles. UCT was about 20°C for first cycle and increased up to 37°C at the last cycle. The strain limited functional fatigue experiment was compared to the functional fatigue test with full austenitic transformations and it was found that fracture occurred earlier at strain limited functional fatigue experiments. However, the specimen was able to present 2% actuation strain in all cycles of the strain limited tests while actuation strain was decreased gradually to the values less than 2% at full austenitic transformation fatigue tests.

Effect of composition and thermal processing on transformation characteristics and equilibrium phase stability in NiTiHf high temperature shape memory alloys

29 Aug
02:00pm
Poster
Session D

Tejas U.[1] ,Karaman I.[1],Talapatra A.[1],Arróyave R.[1],Santamarta R. [2]

[1]*Department of Materials Science and Engineering, Texas A&M University, College Station, TX 77843, USA ;*
[2]*Departament de Fisica, University de les Illes Balears, E07122, Palma de Mallorca, SPAIN*

Ni-Ti-Hf High Temperature Shape Memory Alloys (HTSMAs) are potential candidates for aerospace and space applications such as in space rovers and supersonic flights due to their high strength, superelasticity, tailorable transformation temperatures, and high actuation energies. As a result of martensitic transformation from B2→B19´, high transformation strains can be achieved along with excellent thermal and dimensional stability. Transformation temperatures of these alloys can be precisely tailored by varying composition and thermal processing. In this work, we aim at understanding the compositional dependence of transformation temperatures in Ni-Ti-Hf system, by varying nickel from 49.8% to 51.2% and hafnium from 0% to 30% (at.%). We found that the transformation characteristics are highly sensitive to nickel content for all respective hafnium contents. The nature of martensitic transformation is further studied in the light of crystallographic compatibility, strain-glass behavior, transformation arrest, and H-phase precipitation. We also studied, the phase equilibrium in Ni-Ti-Hf system at different temperatures using diffusion multiple experiments. Knowing the chemical equilibrium of the system aids us in understanding the H-phase precipitate chemistry more clearly and help in developing predictive precipitation models.

Two-way shape memory effect in [001]-oriented Ni49Fe18Ga27Co6 single crystals

29 Aug
02:00pm
Poster
Session D

Timofeeva E.[1],Panchenko E.Yu.[1],Larchenkova N.G.[1],Chumlyakov Yu.I.[1],Maier H.J. [2]

[1]*Tomsk State University RUSSIAN FEDERATION;*
[2]*nstitut für Werkstoffkunde (Materials Science), Leibniz Universität Hannover, 30823 Garbsen, GERMANY*

The effects of the thermo-mechanical treatment and mechanical training (loading/unloading cycles) on the two-way shape memory effect (TWSME) and its stability under thermal cycles in [001]-oriented Ni49Fe18Ga27Co6 single crystals were investigated.
In this study, TWSME in quenched, stress-free and stress-assisted aged crystals was induced by isothermal loading/unloading cycles (ϵ = 6 %, σmax = 140 MPa). The reversible TWSME strain increase with the number of loading/unloading cycles. Maximum TWSME strain up to 5.5 % in the stress-assisted aged single crystals and up to 2.2 % in the stress-free aged single crystals was observed after 100 loading/unloading cycles due to oriented internal stress fields. These fields are formed by the oriented particles variants during stress-assisted ageing and oriented antiphase boundaries and dislocations during mechanical training. Then the TWSME strain is decreased to 3.5 % after 1000 loading/unloading cycles and to 2.5 % after 20000 cycles. In stress-free aged crystals TWSME strains after 100 cycles are not more than 2.2 % and 1.2 % after 103 and 20000 cycles.
The cyclic stability of TWSME under thermal cycles (cooling/heating) at maximum TWSME strain after 100 loading/unloading cycles was investigated. After 100 thermal cycles the TWSME strain is decreased from 5.5 % to 2.5 % in the stress-assisted aged crystals and from 2.2 % to 1.2-1.5 % in the quenched and stress-free aged crystals. The mechanisms of functional degradation for quenched and aged Ni49Fe18Ga27Co6 single crystals oriented along the [001]-direction were developed. The mechanisms of degradation of the TWSME determine the aspects of the dislocation structure, which appeared during training in crystals with the B2- and L21-structure of austenite. It has been experimentally shown that

in quenched crystals, the degradation of the TWSME is completely irreversible and is associated with the increase in the volume fraction of residual martensite during thermal cycling and an irreversible change in the dislocation structure that promotes TWSME. In aged single crystals, in contrast to quenched crystals, the relaxation of internal long-range stress fields and the decrease in volume fraction of the oriented martensite with increasing thermocycles number are observed.
This work was supported by the Russian Science Foundation (grant No. 16 - 19 - 10250).

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29 Aug
02:00pm
Poster
Session D

Characterization of thin film flow diverter stents based on shape memory alloys: FEM, CFD and MRI study

Velvaluri P., Pravdivtseva M., Jansen O.,Quandt E.

Institute for Materials Science, Christian-Albrechts - Universität zu Kiel, GERMANY

Currently flow diverter stents are one of the most used devices in the treatment of intracranial aneurysms, the function of such a stent is to steer the blood flow away from the aneurysm and to promote endothelialization at the neck of the aneurysm. Majority of such commercially available stents are made from braided shape memory alloy wires and this can have certain limitations to the design and functionality. On the other hand, sputtered shape memory thin film devices have a lot of potential because of their possible complicated design structures, miniaturization [1] and an added ability to tailor the mechanical properties including fatigue [2] based on the requirements. However, to address a device as a flow diverter it needs to satisfy certain criteria, including the ability to crimp into catheters for intracranial use, stent deployment, nominal force exertion on the vessel and efficient blood flow diversion. It is however not feasible to manufacture all possible stent designs and then characterize them, as design can be very influential in its functionality. Following this, a better way of evaluating is analyzing them by finite element method (FEM).
In the present study, we have used Abaqus (Dassault Systèmes) for understanding the crimping behavior and the radial force exertion by the stents on the vessel walls. The stent deployment and the efficacy of flow diversion were evaluated using structural and computational fluid dynamics (CFD) modules from Ansys, Inc. Patient specific aneurysm design and input parameters were obtained from MRI (3T, Philips, TOF MRI geometry; 2D PC MRI- velocity profile) was used for the CFD study . Furthermore, 4D flow MRI data for in vivo flow pattern characterization of blood vessel was acquired and then it was compared with the CFD simulation to understand the flow behavior. Aneurysm rupture risk with and without stent was evaluated by using mathematical models from the literature with the parameters obtained from the CFD simulations. One can see that parameters like pressure, wall shear stress and oscillatory shear index have significant role in growth and rupture of aneurysms. We would like to discuss the influence of various design parameters on a stent functionality.

[1] C. Bechtold, R. Lima de Miranda and E. Quandt, Shap. Mem. Superelasticity 1, 286-293, 2015.

[2] C. Chluba, W. Ge, R. Lima de Miranda, J. Strobel, L. Kienle, E. Quandt and M. Wuttig, Science 348, 1004-100, 2015.

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29 Aug
02:00pm
Poster
Session D

On the effect of Chromium and Titanium on abnormal grain growth of Fe Mn-Al-Ni-X shape memory alloys induced by cyclic heat treatment

Vollmer M.,Arold T., Lauhoff C.,Krooß P.,Niendorf T.

University of Kassel, Institute of Materials Engineering, Metallic Materials, 34125 Kassel, GERMANY

Iron-based shape memory alloys, i.e. Fe-Ni-Co-Al-X and Fe-Mn-Al-Ni-X, are promising candidates for mass applications exploiting functional materials, e.g. damping elements for bridges and skyscrapers. Some of the biggest advantages of Fe-based alloy systems are the comparatively simple processing routes originating from steel industry and the relatively low material costs. However, one of the challenges for any industrial application is the adjustment of suitable microstructures with respect to phase fractions, grain size, grain morphology and grain orientations. Especially the control of grain size is essential since the pseudoelastic performance drastically improves, when the grain size exceeds the cross section of the samples and component, respectively.
Recently, a suitable process to control the grain size in Cu-Al-Mn by abnormal grain growth (AGG) was introduced. Omori, Kainuma et al. showed that AGG is stimulated by a cyclic heat treatment

between a single phase region and a two phase region and can be related to the formation of subgrain structures. Similar mechanisms prevail in one of the Fe-based SMAs, however, the grain growth kinetics of Fe-Mn-Al-Ni and Cu-Al-Mn differ by one order of magnitude. The current study focuses on the impact of small amounts of Chromium and Titanium on the abnormal grain growth behavior of Fe-Mn-Al-Ni-X. Thereby, it is possible to change the kinetic of the abnormal grain growth significantly. Microstructure evolution was characterized by means of scanning electron microscopy, electron backscatter diffraction, energy dispersive X-ray spectroscopy, optical microscopy and dilatometry. Furthermore, the role of a fifth element on the aging kinetics and pseudoelastic response was studied by analysis of the Fe-Mn-Al-Ni-Ti alloy. The results obtained in this study clearly reveal that the addition of a fifth element is vital for further development of the Fe-Mn-Al-Ni-base shape memory alloy system. From the results obtained so far potential directions for further alloy design will be deduced.

Mechanical Properties of Fatigue-Resistant Fe-Mn-Si-based Alloy for Seismic Dampers

29 Aug
02:00pm
Poster
Session D

Inoue Y.[1],Kushibe A.[1],Sawaguchi T.[2],Nikulin I.[2],Takamori S.[2],Maruyama T.[3],Chiba Y.[3],Tsuzaki K. [4]

[1]*Research and Development Institute, Takenaka Corporation JAPAN;*
[2]*National Institute for Materials Science JAPAN;*
[3]*Awaji Materia Co.,Ltd. JAPAN;*
[4]*Kyushu University JAPAN*

Seismic isolation and vibration control structures that protect buildings from damage caused by earthquake have become wide spread in earthquake-prone countries. Steel seismic dampers that absorb seismic vibrations through elastoplastic deformation have a large load capacity, high stiffness, and outstanding cost-performance ratio. These dampers are most widely used and indispensable for economical vibration control structures. In recent years, the long-period ground motion has been highlighted as a new problem. Because long-period earthquake waves resonate with the natural period of the high-rise buildings, these buildings vibrate for a long time and with large amplitude. In this situation, large-amplitude cyclic deformation occurs in the seismic dampers that are installed in high-rise buildings. To overcome this problem, the fatigue-resistance properties of the conventional steel dampers are insufficient. Thus, to withstand large-amplitude cyclic deformations, it is required to increase the durability of seismic dampers significantly. It has been reported that deformation-induced ϵ martensite in the FeMnSi-based alloys can cause the reverse transformation to γ austenite by counter-directional deformation, and the reversible two-way martensitic transformation under cyclic tensile-compressive loading improves the low-cycle fatigue lives. In this study, we focus on a Fe-Mn-Si-based alloy to develop a new steel seismic damper with superior fatigue resistance. This study aims to confirm the tensile and low-cycle fatigue properties of this alloy under relevant temperature and frequency conditions for use in seismic dampers. Toward this end, we present the results of static tensile strength tests of a Fe-15Mn-4Si-10Cr-8Ni alloy. These results indicate that this alloy has a small yield-tensile ratio, a large elongation, and undergoes stable deformation. In addition, the tensile properties are isotopic characteristics regardless of the rolling direction. Therefore, this alloy has mechanical properties that make it suitable for use as shear and bending type dampers as well as tensile-compressive type dampers. We also investigate the low-cycle fatigue of this alloy by applying symmetric tensile-compressive loading. Under broad amplitude conditions, this alloy has an outstanding fatigue life in comparison with the low-yield point steel that is used for the conventional steel dampers. It also has stable hysteresis loops from small to large strain region. In addition, the properties of this alloy are hardly affected by temperature and frequency in the relevant range for seismic dampers. Based on these results, we confirm that the tensile properties and low-cycle fatigue characteristics of this Fe-15Mn-4Si-10Cr-8Ni alloy make it suitable for use in seismic dampers.

Session 8: Advanced Processing Techniques

Invited Lecture: Thursday August 30	
8:30 Amphi 4	**V. Brailovski** "Additive manufacturing: influence of processing and post-processing parameters on structure, texture, and functional properties of biomedical shape memory alloys "

Oral Presentations: Thursday August 30	
9:00 Amphi 4	**A. Tuissi**, C. A. Biffi, A. G. Demir, B. Previtali "Microstructure and thermal analysis of NiTi shape memory alloy produced by additive manufacturing: comparison among laser metal wire deposition and laser powder bed fusion "
9:20 Amphi 4	**K. Ullakko**, V. Laitinen, A. Saren, A. Sozinov, D. Musiienko, M. Chmielus, A. Salminen "Ni-Mn-Ga actuating elements manufactured using 3D printing "
9:40 Amphi 4	**B. Kockar**, H. H. Saygili, H. O. Tugrul, I. Karaman "Work output response with functional fatigue behavior of severe plastically deformed Ni50Ti30Hf20 high temperature shape memory alloy "
10:00 Amphi 4	Y. Chen, P. Šittner, O. Tyc, X. Shen, **L. Heller** "Functional and mechanical property tuning of superelastic NiTi filaments by electric pulse heat treatment "

Poster Session D: Wednesday August 29
A. Eftifeeva, E. Panchenko, Y. Chumlyakov, G. Gerstein, H. J. Maier "The effects of stress-induced martensite ageing on shape memory behavior in Co35Ni35Al30 single crystals "
L. Demchenko, A. Titenko, L. Demchenko, A. Perekos, O. Gerasimov, Ya. Titenko " The effect of magnetic field and hydrostatic pressure on aging processes and properties of Cu-Al-Mn alloy "
N. Horandghadim, J. Khalil-Allafi "Effect of Voltage on Electrophoretic Deposition of HA and HA- Ta2O5 Nanocomposite Coatings for Biomedical Applications "
T. Omori, T. Kusama, Y. Araki, R. Kainuma "Large Single Crystals by Abnormal Grain Growth and Superelasticity in Cu- and Fe-Based Alloys "
P. Vondrous " NiTi welding by various methods "
O. Tyc, L. Heller, P. Šittner, M. Vronka " Effect of heat treatment and transformation stresses on fatigue of NiTi filaments "
S. Xu, X. Xu, T. Omori, R. Kainuma " Large $< 001 >$ single crystals via abnormal grain growth from columnar polycrystal in Cu-Al-Mn shape memory alloy "
C. Schneider-Maunoury, L. Weiss, P. Laheurte " Functionally graded binary alloys manufactured with DED-CLAD® process "

Contents

30 Aug
8:30am
Amphi 4

Additive manufacturing: influence of processing and post-processing parameters on structure, texture, and functional properties of biomedical shape memory alloys

Brailovski V.

École de technologie supérieure, CANADA

Additive manufacturing (AM) technologies are gaining a lot of interest in the biomedical field thanks to the possibility of manufacturing complex engineered components, which would otherwise be unfeasible or prohibitively expensive. In the laser powder bed fusion AM technology (LPBF), a thin layer of powder is spread on a platform and a laser beam selectively fuses powder particles to form one layer of the component at a time, in conformity with a 3D model of the component. Following the general overview of the AM technologies, the effect of LPBF parameters on the density, phase composition, structure, and texture of a selected biomedical Ti-Zr-Nb alloy will be presented. Based on the results obtained, the LPBF parameters leading to the fabrication of fully dense Ti-Zr-Nb components will be exposed. Next, influence of different post-processing treatments on the alloy structure, texture, static and fatigue mechanical properties will be revealed. Finally, examples will be given to illustrate how the LPBF technology can be employed to produce personalized and functionally-graded biomedical implants with enhanced biomechanical compatibility.

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30 Aug
9:00am
Amphi 4

Microstructure and thermal analysis of NiTi shape memory alloy produced by additive manufacturing: comparison among laser metal wire deposition and laser powder bed fusion

Tussi A.[1], Biffi C. A.[1],Demir A. G.[2],Previtali B. [2]

[1]*National Research Council, CNR ICMATE, Institute of Condensed Matter Chemistry and Technologies for Energy; Via Previati 1, 23900 Lecco, ITALY;*
[2]*epartment of Mechanical Engineering, Politecnico di Milano, Via La Masa 1, 20156 Milan, ITALY*

Additive Manufacturing (AM) of functional materials, like shape memory alloys, has been a challenging topic for developing advanced smart components. Several experimental studies on 3D printing of NiTi employed a powder bed fusion method, selective laser melting (SLM) with a poor control of the composition and a certain degree of porosity in the built components. Among the available AM technologies, thanks its relatively low cost and versatile material capability, the laser metal wire deposition can be applied for manufacturing of NiTi alloy too.

In this work Ni rich NiTi was printed starting from both, a 0.4 mm in diameter wire and a NiTi powder by using laser metal wire deposition and selective laser melting, respectively. The microstructure and functional properties of thin built elements were analyzed with SEM, differential scanning calorimetry (DSC) and Xray diffraction analyses. It was found that both the processes require post heat treatments for optimizing the martensitic transformation and a reduced porosity degree can be reached by wire feedstock. A comparison of macrostructures and thermal analysis characteristics obtained with the two experimented AM methods will be reported.

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30 Aug
9:20am
Amphi 4

Ni-Mn-Ga actuating elements manufactured using 3D printing

Ullakko K., Laitinen V.,Saren A., Sozinov A.,Musiienko D.,Chmielus M.,Salminem A.

Lappeenranta University of Technology FINLAND

We have recently demonstrated that it is possible to make polycrystalline foamy structures by 3D printing (Binder Jetting) [A. Mostafaei et al., Acta Materialia 131 (2017) 482-490], and demonstrated by modeling that it is possible to make single crystalline MSM elements by 3D printing [K. Ullakko, et al., Proc. of Int. Conf. FSMA 2016, Sendai, Japan, Sept 2016]. Our ultimate goal is to print MSM devices (e.g. valves and grippers) that include actuating parts whose shapes can be changed by an external magnetic field and other parts that act as the body of the device.

Laser Powder Bed Fusion (LPBF) is a commonly used 3D-printing process that utilizes a focused laser beam to manufacture near net shape components by selectively melting and fusing power material in layer by layer. Manufacturing of NiMnGa alloy by LPBF has previously not been reported. In this

study, an LPBF method was used for printing cubic NiMnGa components with dimensions of 4.5 x 4.8 x 5 mm3 on stainless steel substrates. The LPBF system equips an Ytterbium Fiber Laser that has a laser beam of 82 μm in diameter. Substrate pieces were made from 5 mm thick stainless steel. Manufactured components had a porous substructure that consisted of approximately 500 μm long and 200 μm wide columns extending from the substrate to the top of the component. The columns were manufactured by selectively melting numerous single tracks on each other in order to form thin columnar structures. These columns were positioned symmetrically on substrate surface, while leaving approximately 500 μm of empty space between each column. Two layers of transverse support structures were manufactured between columns every six layers in order to stiffen the structure. Hatch spacing used for the parallel support structures was 150 μm. The component exhibits polycrystalline structure with 5M martensite. The component revealed a magnetic-field-induced strain of 100 ppm.

Work output response with functional fatigue behavior of severe plastically deformed Ni50Ti30Hf20 high temperature shape memory alloy

30 Aug
9:40am
Amphi 4

Kockar B.[1],Saygili H. H.[1], Tugrul H. O.[1],Karaman I. [2]

[1]*Hacettepe University, Mechanical Engineering Department TURKEY;*
[2]*Department of Materials Science and Engineering, Texas A&M University, College Station, TX 77843, USA*

High purity Ni, Ti and Hf elements were used to produce Ni50Ti30Hf20 (at%) high temperature shape memory alloy via vacuum induction melting under high purity argon atmosphere. The material was placed in a mild steel can and hot extruded at 900 ˚C for the homogenization of the structure. Then, the extruded alloy was inserted in a stainless steel can to conduct severe plastic deformation via Equal Channel Angular Extrusion (ECAE) following route C for 2 passes and 4 passes at 700 ˚C. Transformation temperatures of the hot extruded and ECAE processed materials were measured using Differential Scanning Calorimetry (DSC) to determine the upper and lower cycle temperatures which were used in the functional fatigue experiments. Dog bone shape tensile specimens were cut using wire electrical discharge machining from extruded and ECAEd materials to perform functional fatigue experiments under constant stress magnitude.
Functional fatigue experiments were conducted via utilizing a custom-built test setup. Joule heating and forced air convection methods were used to heat and cool the material and temperature, displacement and number of cycle values were measured and recorded by a LabView Program. The constant stress was set to 200MPa using dead weights which were hung to the samples. Work output was calculated by multiplying the applied stress with the actuation strain values which were measured in functional fatigue experiments. Actuation strain values for extruded and ECAE processed samples were very stable throughout the cycles, however, ECAE led to an increase in the magnitude of the actuation strain as well as the work output. The calculated work output values were approximately 3 J/cm3, 3.8 J/cm3 and 5.4 J/cm3 for the extruded, 2 pass and 4 pass ECAE processed samples, respectively. Additionally, 2 pass ECAE processed sample showed the highest number of life cycles which was determined as 2500 and the work output values were stable enough for considering this alloy as an actuator in many applications.

Functional and mechanical property tuning of superelastic NiTi filaments by electric pulse heat treatment

30 Aug
10:00am
Amphi 4

Chen Y.[1,2], Šittner P.[2], Tyc O.[2], Shen X.[1], Heller L. [2]

[1] *State Key Laboratory of Mechanics and Control of Mechanical Structures, Nanjing University of Aeronautics and Astronautics, 210016, CHINA;*
[2]*Institute of Physics of the Czech Academy of Sciences, Na Slovance 1999/2, 18221 Prague 8, CZECH REPUBLIC*

Intermetallic shape memory alloy NiTi, due to its superelasticity and shape memory effect, has been a prominent material in scientific and engineering field. Microstructure and functional thermomechanical properties of the superelastic NiTi alloy can be deliberately modified by cold working and annealing.
In this work, cold-worked NiTi filaments were subjected to non-conventional electric pulse heat treatment with varied annealing time (5-25 ms) and fixed power density 160 W/mm3. Results of tensile tests up to the failure on annealed wires reveal that varied annealing time results in different functional properties, such as decrease in transformation stresses, increase in transformation strain and cyclic stability

of superelastic stress-strain response. However, the annealing affects also the yield stress for plasticity, strength and ductility of the annealed wire: longer annealing time (>15 ms) makes the NiTi filaments to attain large ductility ~55 % after a strain hardening, while in case of shorter annealing time (< 14 ms), the wire fractures at strains of ~13% in the absence of strain hardening. The yield stress for plasticity decreases from 1800 MPa to 400 MPa with increasing annealing time from 4 up to 25ms. It is concluded that the plastic deformability, hardening, strength and ductility plays essential role in functional property setting of NiTi filaments.

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29 Aug
2:00pm
Poster
Session D

The effects of stress-induced martensite ageing on shape memory behavior in Co35Ni35Al30 single crystals

Eftifeeva A.[1],Panchenko E.[1],Chumlyakov Y.[1],Gerstein G.[2],Maier H. J. [2]

[1]*National Research Tomsk State University RUSSIAN FEDERATION;*
[2]*Institut für Werkstoffkunde (Materials Science), Leibniz Universität Hannover, 30823 Garbsen, GERMANY*

In the present work, the improvement of the functional properties by stress-induced martensite ageing (SIM-ageing) is presented for single crystals of shape memory Co35Ni35Al30 alloy undergoing thermoelastic B2-L10 martensitic transformation. The ageing was performed at 423 K for 1.0 h under a constant compressive stress of 500 MPa along the [110]B2||[100]L10-direction in the stress-induced martensite. The internal tensile stress fields of 65 MPa along the [001]B2-direction are generated by SIM-ageing of single crystals. As a result, SIM-aged crystals demonstrate two-way shape memory effect (TWSME) and rubber-like behavior (RLB).
A RLB (a reversible strain during loading/unloading cycles at T TWSME (a reversible strain during stress-free cooling/heating cycles) is observed along the [001]B2-direction with a giant reversible tensile strain of up to ϵTWSME=+7.3 (±0.3) % due to the growth of one martensite variant at cooling. The forward and the reverse B2-L10 martensitic transformations during TWSME proceed in two stages. In the first stage, the transformation exhibits a burst-like nature, where the forward and reverse transformations occur in a very narrow temperature range of 5 . However, the second stage requires significant undercooling and spans a temperature range of 141 K. In the first stage, the forward martensitic transformation begins and ends at the same temperature, i.e. Ms=Mf=280 , where the transformation strain is +4.9 %. Similar to the forward martensitic transformation, the temperatures of reverse transformation are As=328 and Af=333 . As a result, an un-gripped SIM-aged sample was observed to jump actually high during both cooling and heating and the thermal energy was converted into mechanical work. The SIM-aged crystals possess a great work output of WTWSME=0.19 J.g^{-1}. Moreover, TWSME demonstrates a high cyclic stability during 100th cycles in stress-free thermal condition.
This work was supported by RFBR (grant No. 16 - 08 - 00179).

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29 Aug
2:00pm
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Session D

Additive manufacturing: influence of processing and post-processing parameters on structure, texture, and functional properties of biomedical shape memory alloys

Brailovski, V.[1],Kreitcberg A.[1], Sheremetyev V.[2], Prokoshkin S. [2]

[1]*École de technologie supérieure CANADA;*
[2]*National University of Science and Technology "MISIS" RUSSIA*

Additive manufacturing (AM) technologies are gaining a lot of interest in the biomedical fSheremetyev,ield thanks to the possibility of manufacturing complex engineered components, which would otherwise be unfeasible or prohibitively expensive. In the laser powder bed fusion AM technology (LPBF), a thin layer of powder is spread on a platform and a laser beam selectively fuses powder particles to form one layer of the component at a time, in conformity with a 3D model of the component. Following the general overview of the AM technologies, the effect of LPBF parameters on the density, phase composition, structure, and texture of a selected biomedical Ti-Zr-Nb alloy will be presented. Based on the results obtained, the LPBF parameters leading to the fabrication of fully dense Ti-Zr-Nb components will be exposed. Next, influence of different post-processing treatments on the alloy structure, texture, static and fatigue mechanical properties will be revealed. Finally, examples will be given to illustrate how the LPBF technology can be employed to produce personalized and functionally-graded biomedical implants with enhanced biomechanical compatibility.

The effect of magnetic field and hydrostatic pressure on aging processes and properties of Cu-Al-Mn alloys

29 Aug
2:00pm
Poster
Session D

Demchenkou L.[2],Titenko A.[1],Perekos A.[3],Gerasimov O.[3],Titenko Ya. [2]

[1] *Institute of Magnetism, National Academy of Sciences and Ministry of Education and Science of Ukraine, 36-b, Vernadskoho Blvd., Kyiv 03142, UKRAINE;*
[2]*National Technical University of Ukraine "Kyiv Polytechnic Institute", 37, Peremohy Ave., Kyiv 03056, UKRAINE;*
[3]*G. V. Kurdyumov Institute for Metal Physics of the N.A.S. UKRAINE*

To improve physical and mechanical properties of alloys, thermal, thermomeDemchenkochanical or thermomagnetic treatments are widely used. The creation of nanostructured state plays an important role in improving the properties. Magnetic nanomaterials exhibit some features in magnetic behavior: superparamagnetism, giant magnetoresistance, and mechanical one: shape memory effect, thermoelasticity, superelasticity, plasticity of transformation, etc.
Cu-Mn-Al alloys are promising materials for controlling mechanical and magnetic properties. The aging of Cu-Mn-Al alloys leads to the formation of a system of nanoscale ferromagnetic particles of Cu2MnAl phase in paramagnetic Cu3Al-matrix. Heat treatment allows controlling the amount and size of particles in the alloy, as well as the temperatures and hysteresis of martensitic transformation, depending on the particles size and quantity. To search for optimal parameters of physical and mechanical properties of Cu-Mn-Al alloys, the thermal treatment with additional influences of an external magnetic field of different strength and orientation and hydrostatic pressure, was proposed. This, in combination, makes it possible to significantly improve both mechanical and magnetic properties.
Moreover, to study the decomposition of a solid solution, the situations related to a change in the intensity and orientation of the magnetic field relative to the main axis of the sample and the hydrostatic pressure during annealing with a fixed temperature and time were considered. Significant differences in the mechanical and magnetic behavior of the material are obtained, depending on the chosen regime of their thermal-force treatment. Structural features that are associated with the decay of the solid solution and the coexistence of ordered and disordered phases are established. The main results of the work are explained from the position of the phenomenological theory of diffusional decay of solid solution with precipitation of new phase nanoparticles. The tendency of increase in microhardness of alloys, accompanied by a decrease in magnetic characteristics due to the reduction of the ferromagnetic nanoparticles size, was noted.

Effect of Voltage on Electrophoretic Deposition of HA and HA- Ta2O5 Nanocomposite Coatings for Biomedical Applications

29 Aug
2:00pm
Poster
Session D

Horandghadim N., Khalil-Allafi J.

Research Center for Advanced Materials and Mineral Processing, Faculty of Materials Engineering, Sahand University of Technology, Tabriz,IRAN

Hydroxyapatite (HA powders extracted from natural bovine bone by the calcination combined ball milling methods with mean particle sizes less than 100 nm) and HA-20%wt Ta2O5 (Ta2O5 with mean particle sizes of 100 nm) nanocomposite coatings were deposited via electrophoretic deposition (EPD). Stable suspensions of HA and HA-Ta2O5 nanoparticles in n-butanol were prepared in optimum concentration of TEA (16 mL/L) as surfactant. NiTi shape memory alloy (SMA, 50.9%atom Ni) was used as substrate. Before coating, chemical etching and passivation were applied as primitive surface modification methods for NiTi SMAs. Low angle-XRD results indicated that chemical etching and boiling of samples in 20% HNO3 solution causing formation of TiO and TiO2 thin films on the NiTi SMA surfaces. Coatings surface and cross section morphologies and thicknesses were studied by SEM. Among various conditions of EPD (50, 60 and 70 V for 60 s) uniform, crack-free and continuous coatings were obtained in 60 V for 60 s for both of HA and HA-Ta2O5 coatings. SEM micrographs revealed that compaction of HA coatings has enhanced after introducing Ta2O5 nanoparticles as secondary phase. Thickness measurements indicated that the thicknesses increased by increments of EPD voltages for HA (from 21 to 42 μm) and HA-Ta2O5 (from 18 to 37 μm) coatings. Furthermore, HA-Ta2O5 coatings had lower thicknesses than HA coatings in the same deposition conditions. To investigate the effects of deposition parameters on coating efficiency of prepared suspensions, in situ weights of deposits were measured using density kit of a 0.1 mg accuracy balance for 360 s. In contrast with thickness achievements, HA-Ta2O5 coatings had more in situ weights than HA coatings in the same deposition conditions. In order to increase the density and reduce the porosity of coatings, samples were sintered in a vacuum annealing furnace under 1.4 10-6 mbar at 850 °C for 1h. The heating and cooling rates were 5 °C min-1. The crystalline phases and microstructures of coatings were examined after sintering.

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Session D

Large Single Crystals by Abnormal Grain Growth and Superelasticity in Cu- and Fe-Based Alloys

Omori T.[1], Kusama T.[1],Kise S.[2],Araki Y.[3],Kainuma R. [1]

[1] *Tohoku University JAPAN;*
[2] *Furukawa Techno Material JAPAN;*
[31] *Kyoto University JAPAN;*

Shape memory properties are often influenced by the grain size because of grain constraint during deformation and many kinds of shape memory alloys show superior properties in single crystals. One of the drawbacks is the high cost and special techniques/equipment for production of single crystals. We have demonstrated that large grains can be simply obtained using abnormal grain growth (AGG) caused by the cyclic heat treatments (CHTs) in Cu-Al-Mn [1] and Fe-Mn-Al-Ni [2] shape memory alloys, which consist of cooling and heating through the phase transition temperature between the single-phase and two-phase states. In this presentation, the microstructural evolution in the CHT and AGG to a large single crystal in Cu-Al-Mn and Fe-Mn-Al-Ni alloys, as well as superelasticity, will be shown.
It was found that subgrains form during cooling in the matrix surrounding the precipitates and that the subgrain structure remains in the single phase at high temperatures. The microstructure in AGG preliminarily subjected to the CHT indicates that the abnormally growing grains encroach on the subgrains. These microstructural features are observed both in Cu-Al-Mn and Fe-Mn-Al-Ni alloys. While the misorientation angle analyzed by EBSD is 0.46°after one CHT, it increases up to 1.12°by repeating the low-temperature CHT between 500 °C and 740 °C five times in Cu-Al-Mn alloy, during which no AGG occurs. The growth rate is significantly increased after five CHTs, meaning that the sub-boundary energy is the dominant driving pressure in the present AGG phenomenon. The AGG changes to normal grain growth when abnormal grains come in contact with one another, which is a common characteristic in AGG phenomena, however, the subgrain structure is restored by further CHT in the present technique. Thus AGG can repeatedly be induced and a large crystal can consequently be obtained. Single crystal bars 700 mm in length and 15 mm in diameter were successfully obtained using the CHT in Cu-Al-Mn alloy and they showed good superelasticity [3]. This technique is advantageous for mass production of single crystals because of its simplicity of the process and is useful for applications of shape memory alloys as large-scale components, such as seismic devices.

[1] T. Omori and al. Science 341,1500, 2013.

[2] T. Omori and al. Mater. Design 101,263, 2016.

[3] T. Kusama, T. Omori and al., Nature Communications 8, 354, 2017.

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29 Aug
2:00pm
Poster
Session D

NiTi welding by various methods

Vondrous P.

Czech Technical University, Prague CZECH REPUBLIC

Difficulty of welding NiTi is well known and is troublesome for some prospective applications as stents, catheters etc. The main problem is ease of oxidation and moreover precipitation of brittle phases in WM and HAZ. Several fusion welding methods and pressure welding methods were used. Microplasma welding, electron beam, laser beam welding are evaluated by mechanical properties together with their ease of use. The comparison of results of NiTi base material condition, e.i. hard, SA, etched is done, and is found important.It was found out that fusion welding methods will even under perfect welding conditions (e.g. shielding, limited heat input) tend to suffer decrease of mechanical properties. Tensile strength drops to about 700 MPa for the best welds. There is tendency to brittle fracture in HAZ, which can not be fully eliminated by fusion welding methods.

Functionally graded binary alloys manufactured with DED-CLAD ® process

29 Aug
2:00pm
Poster
Session D

Schneider-Maunoury C.[1,2],Weiss L.[2],Laheurte P. [2]

[1]*REPA LASER, Parc dInnovation, Pôle API, 67400 Illkirch, FRANCE;*
[2]*LEM3, Université de Lorraine, 7 rue Félix Savart, 57073 Metz, FRANCE*

The objective of the current work is the manufacturing of a defect-free functionally graded material thin wall structure by the CLAD® process (Construction Laser Additive Direct). Functionally Graded Materials are materials whose chemical composition, and thus mechanical and microstructure characteristics are gradually varied along one or more spatial directions. The interest of these materials is thus to associate and concentrate the benefits of two or more materials into one part. Along with the widely used titanium alloy Ti64, refractory materials such as Mo and Nb were chosen in this study for their beta-gene alloying effect on titanium structures. Ti64/Mo graded materials could be used for applications requiring lightweight intermediate resistance temperature on one side, and high melting point on the other side. Ti/Nb graded materials could be used in biomedical applications for its biocompatibility and its mechanical properties which help to reduce the stress-shielding effect. Walls have been manufactured by starting with 100% Ti64 at the bottom of the wall to finish with 100% Mo or Nb at the top. Between the first and last layers, both Ti64 and Mo or Nb content have been adapted to achieve a composition graded structure.
The study examined the transition between the two different grades to understand microstructural evolution (shape and size of grain, phases, etc.) and evaluate mechanical properties. EDX analysis was performed to control the chemical composition into the deposits and thus validated the response between the expected and the actual chemical composition. Micro-hardness is used to evaluate the mechanical properties and homogeneity along the wall and thus the functionally graded Ti64 to Mo or Nb samples. Microstructure evolution along the wall has been studied under an optical microscope along the construction and has been analysed through the means of EBSD tool which allows to get information about crystallographic texture into the deposition. Tomography allows to validate CLAD® process thanks to an analyse of the unmelted particles distribution, and thus the homogeneity of the mixing into the sample.

Effect of heat treatment and transformation stresses on fatigue of NiTi filaments

29 Aug
2:00pm
Poster
Session D

Tyc O.,Heller L., Šittner P.,Vronka M.

Institute of Physics of the Czech Academy of Sciences CZECH REPUBLIC

Microstructure and thermomechanical functional response of Ni-rich NiTi alloys can be in a significant extent adjusted by heat treatments. Different types of processes (precipitation, recovery and recrystallization) are activated during a heat treatment depending on its parameters. On the other hand, it is not clear whether, how and/or in which extent fatigue of NiTi can be controlled by heat treatments. This is related to the fact that, despite extensive research, it is still not clear what is the dominating factor affecting the superelastic fatigue performance of NiTi (transformation strain, transformation stress, inclusions, strain localization or surface finishing). In this study, cold-worked NiTi filaments (100 μm in diameter) subjected to two types of the final heat treatment were used to investigate an influence of microstructure (precipitates) on its structural and functional fatigue performance in a wide temperature range (-20°C 100 °C). The first employed heat treatment was pulse heating by electric current (25Wmm-3/100ms) triggering recovery and recrystallization processes that yield approx. 100 nm grain size without Ni-rich precipitates. The second employed heat treatment is the same electro-pulse heating followed by aging 400°C/1h to introduce nanoprecipitates of Ni4Ti3 phase into the microstructure.
The results of fatigue tests proved that precipitation hardened filaments exhibit almost twice as long fatigue life compared to the electro-pulse treated filaments. The experiments also show that an accumulation of permanent strain recorded in a closed loop of thermomechanical loading cycles is generated only when the martensitic transformation proceeds under applied stress. Thus, precipitation is beneficial as transformation stresses are reduced and microstructure is strengthened. It is proposed that the accumulation of permanent strain and related microstructure changes accompanying martensitic transformation under stress upon cyclic superelastic loading represent evolution of material damage, which significantly affects fatigue performance of NiTi alloy.

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Poster
Session D

Large $< 001 >$ single crystals via abnormal grain growth from columnar polycrystal in Cu-Al-Mn shape memory alloy

Xu S., Omori T., Kainuma R.

Tohoku University JAPAN

Producing single-crystalline alloys with large size and specific crystal orientation is of great importance for practical applications. Typically, $< 001 >$-oriented single crystals in many cubic metals show superior properties, such as the enhanced superelasticity in Cu-based shape memory alloys. Recently, our group has developed a novel method for growing ultra-large single crystals in a Cu-Al-Mn superelastic alloy via abnormal grain growth (AGG), namely, cyclic heat treatment (CHT) that involves thermal cycling through β(bcc)$\rightarrow$ $\beta + \alpha$(fcc)$\rightarrow$ β phase transformation [1,2]. However, the crystal orientation is not controllable. Here, we conducted CHT on a directionally solidified Cu-Al-Mn alloy, yielding large $< 001 >$-orientated single crystals. A Cu71.5Al17.5Mn11 alloy was firstly prepared by the directional solidification method [3], showing a $< 001 >$ texture along solidification direction with columnar grains. CHT was then carried out on the columnar polycrystal, i.e. cooling from 900°C in the β single-phase region to 500°C in the $(\beta+\alpha)$ two-phase region and then reheating to 900°C in the β single-phase region. The microstructure was examined by the EBSD technique. The superelasticity was characterized by the cyclic loading-unloading tensile tests. Even though the columnar polycrystal contained a high fraction of low-mobility low-angle grain boundaries, the AGG could also be effectively induced by using CHTs, during which the formed subgrains associated to the phase transformation served as a very strong and dominant driving force to promote the fast migration of preexisting grain boundaries. The abnormally large grains or single crystals maintained a $< 001 >$ orientation ascribed to the initial as-cast $< 001 >$ texture. Finally, with applying multi-CHTs, Cu71.5Al17.5Mn11 single crystals orientated to $< 001 >$ with 50mm10mm1mm in dimension were successfully obtained, in which an excellent superelastic strain of more than 10% was observed.

[1] Omori T. and al. Science 341,1500 - 1502, 2013 .

[2] Kusama T. and al. Nature Communications 8, 354, 2017.

[3] Liu J. and al. Materials & Design 64,427-433 ,2014.

Session 9: NiTi Shape Memory Alloys

Invited Lecture: Thursday August 30	
12:10 Amphi 1	**P. Sedlák** "Coupled transformation and plasticity in NiTi "
Invited Lecture: Friday August 31	
8:30 Amphi 1	**C. Maletta** "NiTi fracture and fatigue aspects"
Oral Presentations: Thursday August 30	
10:50 Amphi 1	**S. Prokoshkin**, S. Dubinskiy, A. Korotitskiy, V. Sheremetyev, A. Glezer, V. Brailovski " TEM and in situ XRD study of the stress-induced transformation mechanism in Ti-50.61 at. %Ni alloy with extremely small grain sizes "
11:10 Amphi 1	**F. M. Braz Fernandes**, E. Camacho, P. Inácio, T. Santos, N. Schell " In situ Structural Characterization of Functionally Graded Ni-Ti Shape Memory Alloy During Tensile Loading "
11:30 Amphi 1	**P. Sittner**,L. Heller, P. Sedlák, H. Seiner, O. Tyc, L. Kadevrávek, M. Vronka, P. Sedmák " Beyond the strain recoverability limits of martensitic transformations in NiTi "
11:50 Amphi 1	**M. Barati**, S. Arbab Chirani, M. Kadkhodaei " Residual stress-induced martensite process in NiTi shape memory alloy during and after different training methods studied by electric resistivity measurement "
Oral Presentations: Friday August 31	
09:00 Amphi 1	**H. Martinni Ramos De Oliveira**, H. Louche, D. Favier " Influence of the heat treatment of nanostructured NiTi wires on the specific latent heat and martensite fraction during superelastic tensile tests "
09:20 Amphi 1	**Y. Kimura**, K. Han, K. Niitsu, X. Xu, T. Omori, R. Kainuma " R-phase and Intermediate-phase Transitions in Ti50.0-xNi47.0 +xFe3.0 Shape Memory Alloys"
09:40 Amphi 1	**D. Glazova**, N. Resnina, S. Belyaev, D. Glazova, A. Saveleva, V. Zeldovich, V. Pilyugin, N. Frolova, M.N. Mikheev " Influence of grain size on shape memory effects in NiTi alloy "
10:00 Amphi 1	**S. Hahn**, V. Klemm, D. Rafaja, S. Kaufmann-Weiß, S. Fähler, M.F.-X. Wagner " On the growth mechanism of hetero epitaxial {100} Nickel Titanium shape memory alloy thin films "

Poster Session B: Tuesday August 28
T. Castro da Silva, L. Contaifer de Moraes, A. Pinheiro Barcelos, E. Paulo da Silva "Cryogenic Treatment Effect on NiTi Wire Under Thermomechanical Cycling "
L. Heller, P. Šittner, O. Tyc, L. Kadeřávek, H. Seiner, P. Sedlák " Unrecovered strains generated during thermomechanical loading of NiTi wires - why, when and how ? "
L. Kaderavek, L. Heller, L. Klimša, P. Šittner " How localized superelastic deformation of NiTi wires in tension affects its fatigue performance "
S. Pejman, L. Heller, E. Alarcon, P. Sittner , P. Sandera, J. Hornikova, J. Pokluda " The Effect of Stress Risers on Superelasticity of NiTi Components "
E. Demidova, A. Shelyakov " Strain variation during isothermal martensitic transformation in the Ti40.7 Hf9.5 Ni44.8 Cu5 shape memory alloy"

Poster Session B: Tuesday August 28
V. Grishkov, A.I. Lotkov, A.A. Baturin, D.Yu. Zhapova, V.N. Timkin " The effect of the isothermal mechanical cycling on superelasticity and shape memory of Ti49.3Ni50.7 (at.%) alloy "
A. Jury, L. Heller, E. Alarcon, P. Sittner, P. Sedlak, M. Frost, R. Stalmans " Thermomechanical characterisation and modelling of NiTiFe and NiTiNb for design of couplings "
M. Sergey " Nanohardness and modulus of elasticity of single crystals of the TiNi-TiFe system "
E. Ostropiko " The influence of high-strain rate compression on the functional properties of the TiNi alloy "
S. Aleksei, N. Resnina, S. Belyaev " Influence of holding temperature on recovery in martensitic transformation temperatures in TiNi alloy after preliminary thermal cycling "
S. Belyaev, N. Resnina, A. Ivanov, E. Demidova, V. Andreev " Pre-martensitic phenomena, martensitic transformation and strain variation in quenched Ni-rich NiTi alloy "
V. Torra " Thinner and thick wires in NiTi SMA alloy and their applications "
A. Baturin, A. Lotkov, V. Grishkov, I. Rodionov "Effect of aging at room temperature after hydrogenation on the martensitic transformation and superelasticity in the TiNi alloys with coarse-grained and ultrafine-grained structure "
B. Ben Fraj, S. Zghal, A. Gahbiche, Z. Tourki " On the annealing temperature effect on the thermomechanical behavior of the Ni-rich NiTi SMA "
F. M. Braz Fernandes, P.F. Rodrigues, E. Camacho, A.S. Paula, N. Schell " In situ study of thermomechanical processing of NiTi shape memory alloy "
C.-H. Chen, S.-K. Wu, Y.-C. Wang " Martensitic Transformations and Shape Memory Characteristic of Ti-rich Ti51.9Ni48.0Si0.1 Shape Memory Ribbons"
A. Shelyakov, N. Sitnikov ,K. Borodako , A. Menushenkov "Effect of annealing conditions on transformation and shape memory characteristics of melt-spun TiNiCu alloys with high Cu content "
S. Samal, L. Heller, J. Brajer, J. Manak, P. Sittner " Gradient of hardness and Youngs modulus over crosssection of laser annealed NiTi wires "
E. E. Timofeeva, E.Yu. Panchenko, A.I. Tagiltsev, Yu.I. Chumlyakov " The features of the orientation dependence of reversible strain in aged TiNi single crystals at the variation of Ni concentration "

Contents

Coupled transformation and plasticity in NiTi

30 Aug
12:10am
Amphi 1

Sedlák P.[1],Seiner H.[2],Frost M.[2],Heller L.[3], Šittner P. [3]

[1] *Institute of Thermomechanics, CAS CZECH REPUBLIC;*
[2] *Institute of Thermomechanics, CAS CZECH REPUBLIC;*
[3] *Institute of Physics, CAS CZECH REPUBLIC*

Activation and intensity of various inelastic deformation mechanisms occurring in NiTi shape memory alloys strongly depend on temperature. Dedicated experiments on high-quality NiTi superelastic wires showed that at low temperatures (<50 °C), classical "functional" behaviors stemming from reversible martensitic transformation is dominant; within the intermediate temperature range (100-250°C), large plastic deformation is generated concurrently with the martensitic transformation and results in surprisingly high ductility of the wires. Finally, conventional plastic deformation of austenite was observed at even higher temperature (>300 °C) and high stress. On the crystallographic level, it was recently recognized that the dominant mechanism generating plastic deformation induced by martensitic transformation is the reverse transformation of martensite into twinned austenite. This twinning originates in {20-1} plastic twinning in martensite, and, after the transformation, it is inherited in the form of {411} twins in austenite [1]. However, complete quantitative description of these processes and their mutual interactions is still missing.
In this contribution, we present a possible phenomenological description on the macroscopic level extending our previous modeling efforts. Conventional approaches trying to capture the observed plastic deformation mechanism with models for metal plasticity considering phase-dependent yield surface was found insufficient to cover the complex evolution of plastic strain in experiments. It was also impossible to simply adapt equations usually used for description of transformation-induced plasticity (Greenwood-Johnson mechanism) since the assumption of proportionality between the rate of plastic strain and rate of martensitic volume fraction is generally not satisfied. Instead, it was necessary to develop a new mechanism which includes and relates the deformation twinning in martensite and the reverse phase transformation to plastically deformed (twinned) austenite. It is assumed that this mechanism accompanies the conventional reversible austenite-to-martensite phase transformation. Capability of the proposed approach will be demonstrated on an extensive set of simulations fitting well with experiments.

[1] P. Šittner and al., Esomat 2018

Fracture mechanics of NiTi SMAs: modeling and experimental approaches

31 Aug
8:30am
Amphi 1

Maletta C.

Universita della Calabria ITALY

Fatigue and fracture properties of nickel-titanium (NiTi) based Shape Memory Alloys (SMAs) were analyzed by both fatigue crack propagation and monotonic fracture tests, carried out at different testing temperatures within the pseudoelastic regime of the alloy. Digital Image Correlation (DIC) was adopted to capture the near crack-tip displacement and strain fields during crack propagation.
Displacement data were used to estimate the effective Stress Intensity Factor (SIF), by fitting the Williams expansion series. It was found that temperature plays an important role on SIF and on both stable and critical fast fracture conditions. As a consequence, Linear Elastic Fracture Mechanics (LEFM) approaches are not suitable to predict fatigue and fracture properties of SMAs, as they do not consider the effects of temperature. On the contrary, good agreements between DIC results and the predictions of an ad-hoc analytical model of the author were observed. In fact, the model takes into account the whole thermo-mechanical loading condition, including both stress and temperature. Results revealed that crack tip stress-induced transformations do not represent a toughening effect. Furthermore, it was demonstrated that the analytical model can be actually used to define a temperature independent fracture toughness parameter. Therefore, a new approach is proposed, where both mechanical load and temperature are considered as loading parameters in SIF computation.

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30 Aug
10:50am
Amphi 1

TEM and in situ XRD study of the stress-induced transformation mechanism in Ti-50.61 at. %Ni alloy with extremely small grain sizes

Prokoshkin S.[1],Dubinskiy S.[1],Korotitskiy A.[1],Sheremetyev V.[1],Glezer A.[1],Brailovski V. [2]

[1]*National University of Science and Technology MISIS RUSSIAN FEDERATION;*
[2]*Ecole de Technologie Superieure, Montreal, CANADA*

It has already been shown that stress-induced transformation of B2-austenite in a nickel-rich Ti-Ni shape memory alloy keeps a "discrete" (martensitic) nature down to austenite grain size as small as 12 nm. This work is aimed to extend the previous study to grain sizes smaller than 10 nm. For this purpose, Ti-50.61at.%Ni alloy wire was subjected to a thermomechanical treatment comprising severe cold rolling with a true (logarithmic) strain of e=1.7 (82% reduction) and post-deformation annealing at 350 °C for 5 and 10 min followed by water-cooling. A TEM study showed that after these treatments, a predominantly nanograined structure with an average grain size of 8 nm (5 min) and 10 nm (10 min annealing) forms in B2-austenite. Next, combined TEM and XRD analyses showed that the parent phase state of the thermomechanically treated alloy is not a single-phase (B2) but a two-phase (B2+R) state. The quantitative analysis of the in situ XRD profiles recorded during tensile deformation showed that a reliable separation of strong multi-component X-ray diffraction profiles consisted of {110}B2, (330)R, (3-30)R, (11-1)B19$'$, (002)B19$'$ and (111)B19$'$ lines is indeed impossible. In this situation, to be capable of drawing a conclusion about the nature of phase transformation mechanisms, some relatively weak, but well-separated lines, such as 110B19$'$, (212)19$'$, and, less reliable, (032)B19$'$, were used as key diffractogram features. Tracing the evolution of these key lines across the entire path of stress-induced transformation unambiguously evidenced its discrete (not continuous) nature. Although continuous stress-induced transformation was not detected in such an extremely fine-grained Ti-Ni alloy, its realization in the case of austenite grains smaller than 8 nm cannot be excluded.

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30 Aug
11:10am
Amphi 1

In situ Structural Characterization of Functionally Graded Ni-Ti Shape Memory Alloy During Tensile Loading

Braz Fernandes F. M.[1],Camacho E.[1],Rodrigues P.[1],Incio P.[2],Santos T.[2],Schell N. [3]

[1]*CENIMAT/I3N, Department of Materials Science, Faculdade de Ciências e Tecnologia, Universidade Nova de Lisboa, Monte da Caparica, PORTUGAL;*
[2] *UNIDEMI, Department of Mechanical and Industrial Engineering, NOVA School of Science and Technology, NOVA University Lisbon, 2829-516 Caparica, PORTUGAL;*
[3] *Institute of Materials Research, Helmholtz-Zentrum Geesthacht, Max-Planck-Str. 1, D-21502 Geesthacht, GERMANY*

A functionally graded NiTi shape memory alloy wire was investigated by in-situ synchrotron radiation-based x-ray diffraction during cyclic tensile deformation. The transformation temperatures were determined by DSC and the thermomechanical behavior was analyzed by three-point bending. The present study focussed on the localized heat treatment (Joule heat effect, reaching 300°C, 350 and 400°C pulses for 10 minutes) of NiTi wires, using an equipment that allows a large variety of localized heat treatments. Structural, mechanical and thermomechanical characterization is presented in order to get a perspective of the optimization parameters for the adequate graded functionality. Experimental results put in evidence the structural gradient resulting from the localized heat treatments. The analysis of the in-situ XRD provide detailed information about the phase transformations in different regions of the wire, at different steps of the cyclic load/unload, giving a better understanding of the overall mechanical behavior of a functionally graded material.

Beyond the strain recoverability limits of martensitic transformations in NiTi

30 Aug
11:30am
Amphi 1

Šittner P.[1],Heller L.[1,2],Sedlák P.[2,3],Seiner H.[3],Tyc O.[1,4],Kadeřávek L.[1,2,4],Vronka M.[1],Sedmák P. [5]

[1]Institute of Physics of the CAS, Na Slovance 1992/2, 18221 Prague, CZECHIA;
[2]Nuclear Physics Institute of the CAS, Husinec - Řež 130, 250 68, Řež, CZECHIA;
[3]Institute of Thermomechanics of the CAS, Dolejskova 5, 18200 Praha, CZECHIA;
[4]Faculty of Nuclear Sciences and Physical Engineering, CTU Prague, CZECHIA;
[5]ESRF, Grenoble, FRANCE

NiTi shape memory alloy wires exhibit shape memory effect and superelasticity in cyclic thermo-mechanical loads when the applied stress, strain and temperature fall within certain limits - for high quality superelastic NiTi wire roughly tensile stress < 1GPa, tensile strain < 10% and temperature <100°C. When these limits are exceeded in cyclic thermomechanical loads, plastic deformation ac-companies martensitic transformation, external strains become unrecoverable, shape memory effect is lost but both forward and reverse martensitic transformations between austenite and martensite phases still proceed until completion in thermomechanical loading cycles. Only, as the external strain cannot be directly linked to the variation of the phase fraction of austenite and martensite phases, it becomes difficult to track down the progress of martensitic transformation during ther-momechanical load cycles. The deformation mechanism acting in NiTi under such extreme condi-tions will be elaborated in the related conference presentation [1].
In order to explore this deformation process involving coupled martensitic transformation and plas-ticity, NiTi wires were subjected to complex thermomechanical loading tests in tension from -100°C to 350°C with supplemental in-situ electrical resistance, in-situ synchrotron x-ray diffraction and ex-situ TEM observations of microstructures in deformed wires. Based on the obtained results, it is claimed that, under high stress - high temperature conditions, martensitic transformation may proceed into twinned austenite B2T as the B2→B19$^{'}$→B2T two step transformation. Dislocation slip always accompanies the latter process. Since both the dislocation slip and B19$^{'}$→B2T reverse transformation bring about irrecoverable plastic strains with respect to the original parent B2 phase, the superelastic and/or actuation functionalities of NiTi wires in such thermomechanical loading tests are incomplete or not observed at all. However, the acting deformation process [1] renders NiTi excellent ductility, causes refinement of its microstructure and allows for its low temperature processing and shape setting.

Residual stress-induced martensite process in NiTi shape memory alloy during and after different training methods studied by electric resistivity measurements

30 Aug
11:50am
Amphi 1

Barati M.,[1]Arbab Chirani S.[1],Kadkhodaei M. [2]

[1]Ecole Nationale dIngénieur de Brest, ENIB, FRE CNRS 3744, IRDL, F-29200 Brest, FRANCE;
[2]Department of Mechanical Engineering, Isfahan University of Technology, Isfahan, 84156-83111, IRAN

In most of the applications, the structural components and devices made of shape memory alloys (SMAs) work under cyclic thermomechanical loading (i.e. training) and, therefore, are susceptible to functional fatigue. Fatigue of SMA is a big challenge for this material and should be solved to promote their engineering applications and to utilize their unique shape memory effect (SME) as well as pseudoelasticity (PE) more effectively. On the other hand, thermomechanical cycling is an important procedure to develop two-way shape memory effect (TWSME) in SMAs. Study of TWSM strain is important because it can be an indicator of the residual stress-induced martensite (RSIM) in an SMA after the training procedure. In this study, degradation in the functional properties of NiTi SMAs during different thermomechanical training cycles and the resultant two-way shape memory effect are studied using in-situ electric resistivity measurement. Three different training methods are employed to induce two-way shape memory behavior in the specimens. The combined effects of plasticity (dislocation slip) and RSIM on development of two-way shape memory effect are investigated. Particular attention is paid to the origin of residual strain during different training methods. It is found that these two mechanisms have very different influences on the thermomechanical behaviors of the alloy, including evolutions in the residual strain, development of two-way memory effect and variations in the amount of electric resistivity, during and after training. The results of in-situ electric resistivity measurements can hence be beneficially used to better understand the deformation mechanism of simultaneous plasticity and RSIM during different training procedures.

Obtained results show that, after all training procedures, the electric resistivity of the material increases at low and high temperatures. Plasticity-induced increase in electric resistivity for low and high temperatures is the same; however, the effect of RSIM on the electric resistivity are more pronounced at low temperatures than those at high temperatures. For one-way shape memory effect and assisted two-way memory effect training methods under low stress or strain levels, the "negative" TWSM strain is observed after training. In these cases, similar to the untrained samples, the materials electric resistivity increases during the reverse transformation (M to A; upon heating); however, for the other cases (with "positive" TWSM strain), it decreases. Increase in electric resistivity during the reverse transformation is due to insufficient RSIM induced during the training process.

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31 Aug
09:00am
Amphi 1

Influence of the heat treatment of nanostructured NiTi wires on the specific latent heat and martensite fraction during superelastic tensile tests.

Martinni Ramos De Oliveira H.[1],Louche H.[2],Favier D. [1]

[1]*Laboratoire TIMC-IMAG/ Biommat - UGA FRANCE;*
[2]*Laboratoire LMGC - Montpellier 2 FRANCE*

Superelastic (SE) deformation of Shape memory alloys (SMA) can induce large temperature variations. This phenomenon is due to the martensitic phase transformation responsible of the superelastic behavior which causes an important thermal-mechanical coupling. The temperature variations are dependent on the thermal conditions of the deformation (strain rate, convection, conduction,..) and on the specific latent heat of the transformation Htr and the fraction of transformed material fm.
In this context, this work presents a study on the investigation of ΔHtr and of fm during superelastic tensile tests of nanostructured Ti-50.8 at.% Ni wires. The initially cold worked wires were heat treated at low temperatures and deformed in tension at room temperature. It is shown that the measurement of temperature and strain fields during these tests allow to study the influence of the heat treatment on ΔHtr and fm.
For well annealed SMA, the specific latent heat (ΔH) is measured by differential scanning calorimetry (DSC). This method cannot been used for cold worked NiTi SMA heat treated at low temperature, for which the DSC peaks are not well defined and very broad.
When deformed in tension, these materials exhibit uniform superelastic behavior without any stress plateau and localization phenomenon; this behavior is due to a uniform stress induced phase transformation (SIPT) accompanied by temperature change. Temperature and strain fields were measured during superelastic tensile tests performed on cold worked wires of diameter 0.5mm heat treated at 523K, 548K, 573K and 598K for 30min. From these experimental data and a theoretical approach, thermal and mechanical power and energies were determined during the SIPT. The specific latent heat ΔHtr for all the tested materials were determined by analyzing these results through a thermodynamic framework based on the Gibbs free energy. Furthermore, the martensitic fractions fm were estimated and analyzed in function of stress and strain in regard to the superelastic behavior.
The study shows that ΔHtr is not dependent on the heat treatment temperature whereas fm is dependent on strain and decreases for decreasing heat treatment temperatures.

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31 Aug
09:20am
Amphi 1

R-phase and Intermediate-phase Transitions in Ti50.0-xNi47.0 +xFe3.0 Shape Memory Alloys

Kimura Y.,Han K.,Niitsu K.,Xu X.,Omori T.,Kainuma R.

Department of Materials Science, Graduate School of Engineering, Tohoku University JAPAN

Ti50.0Ni50.0-xFex alloys are known to show a two-step martensitic transformation from B2 to B19$'$-phase via R-phase. The R-phase transformation is first-order transformation with a small latent heat and a small thermal hysteresis in the forward/reverse martensitic transformations. Fukuda and al. recently reported that Ti50.0Ni50.0-xFex alloys with a relatively high Fe composition shows a negative temperature dependence of electrical resistivity (ER) in the parent phase region, and in Ti50.0Ni44.0Fe6.0 alloy where no R-phase transformation is detected, a broad peak due to Commensurate-Incommensurate (C-IC) transition appears in the specific heat (SH) [1]. On the other hand, a similar broad peak due to the C-IC transition, that affects the B2/B19 martensitic transformation, was recently reported in Ti-poor Ti50.0-xNi40.0+xCu10.0 alloys, where the commensurate state was called "Intermediate (I)-phase" [2].

In the present study, Ti-poor Ti50.0xNi47.0+xFe3.0 alloys are selected in order to examine the I-phase transition and its influence to R-phase transformation. In alloys quenched from 1273 K, B2 single-phase condition was kept to 48.0 at. % Ni. While gradually decreasing with increasing Ni content, R-phase transformation temperature, TR, declines suddenly at around x = 0.35. In some alloys with x > 0.4, in which R-phase transformation perfectly disappears, a broad peak in SH due to the I-phase transition was detected near 195 K as well as in the Ti-Ni-Cu alloys. It should be emphasized that the drastic decrease of the TR occurs in the composition region, where the TR curve intersects the I-phase transition temperature, TI, at about 195 K. This result suggests that the I-phase transformation significantly stabilize the parent phase. By in-situ TEM observations, the ordinary twin microstructure of R-phase was confirmed at T=87K in the stoichiometric x = 0.0 alloy. On the other hand, in the off-stoichiometric x = 0.4 alloy, the fine domain structure, that is very similar to that of the I-phase, was formed with no twin microstructure even in the R-phase region at T=87K. This means that the microstructure of R-phase is strongly influenced by existence of the I-phase.

[1] T. Fukuda and al., Mater. Sci. Eng. A, 481-482,235-238, 2008.

[2] Y. Kimura and al., Mater. Trans., JIM 57 269-277,2016.

Influence of grain size on shape memory effects in NiTi alloy

31 Aug
09:40am
Amphi 1

Glazova D.[1], Resnina N.[1], Belyaev S.[1],Saveleva A.[1],Zeldovich V.[2],Pilyugin V.[2], Frolova N. [2]

[1]*Saint Petersburg State University RUSSIAN FEDERATION;*
[2]*M.N. Mikheev Institute of Metal Physics of the Ural Branch of the Russian Academy of Sciences, Ekaterinburg, RUSSIA*

The aim of this work was to study the influence of grain size and stress on the transformation plasticity and shape memory effects in nanostructured NiTi alloy. To obtain nanostructured state first, plate samples of the initially coarse grained NiTi alloy (50,2 at.% Ni) were amorphyzed by high-pressure torsion at P = 8 GPa and n = 3,5 turns at T = 25 . Then, bone-shaped samples were stamped out from amorphous discs and heated up to different temperatures at a heating rate of 10 ˚C/min to obtain samples with different grain sizes varied from 20 to 500 nm. Heating was carried out in the differential scanning calorimeter camera, heating temperatures varied from 359 to 550 ˚C. The transformation plasticity and shape memory effects were studied on cooling and heating under tensile stress using the following procedure. The sample was heated up to 130 ˚C, loaded up to a stress in the range of 100 to 600 MPa, cooled down to -50 ˚C and then, heated up again. The strain accumulation on cooling was the transformation plasticity value, the strain recovery on heating was the shape memory value and the difference was the irrecoverable strain. It was found that NiTi alloy with a grain size of 80 nm or more demonstrates the transformation plasticity and shape memory effects on cooling and heating under all stresses, while NiTi alloy with a grain size of less than 80 nm shows these effects only when stress exceeds the critical value, which increases on a decrease in grain size. The critical stress is 200 MPa in NiTi alloy with 40 nm grain size, and 400 MPa in NiTi alloy with 20 nm grain size. The transformation plasticity and shape memory values increase on an increase in stress, and the larger the grain size, the greater the influence of stress on strain variation. An increase in grain size leads to a monotonic increase in the transformation plasticity value, whereas the shape memory value depends on grain size in non-monotonic way. The maximum value of the shape memory effect is observed in the NiTi alloy with 130 nm grain size and it is equal to 15 %. The transformation temperatures linearly increase on an increase in stress and decrease on a decrease in grain size. A decrease in grain size does not affect the slope of Rs(σ), Ms(σ), or Af(σ) curves.

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31 Aug
10:00am
Amphi 1

On the growth mechanism of hetero epitaxial {100} Nickel Titanium shape memory alloy thin films

Hahn S.[1],Klemm V.[2],Rafaja D.[2],Kaufmann-Weiß

S

.[31],Fähler S.[4],Wagner M.F.-X. [5] [1]TU Chemnitz, Institute of Materials Science and Engineering GERMANY;
[2]TU Freiberg, Institute of Materials Science, 09599 Freiberg GERMANY;
[3]KIT Karlsruhe, Department Superconducting Materials and Energy Applications, 76131 Karlsruhe GERMANY;
[4]IFW Dresden, Institute for Metallic Materials, 01069 Dresden GERMANY;
[5]Institute of Materials Science and Engineering, 09125 Chemnitz GERMANY
S.,Fähler S.,Wagner M.F.-X. Epitaxial Nickel Titanium (NiTi) films have the potential to be used as model systems to provide new insights on phase formations during crystal growth. [1] We deposited Nickel rich NiTi thin films in order to obtain the cubic B2 phase after crystallization at room temperature. Thin films in the range of 100 to 120 nm were deposited on {100} single crystalline MgO substrate materials using a PVD magnetron sputter process. During deposition, the substrates were heated at 250 °C and the deposition target was an alloyed Ti-rich NiTi target. Subsequently, one- or two-step annealing treatments were performed (one-step at 650 °C: sample series A; for two-step, an additional temper step at 300 °C was used: sample series B). All heat treatments were performed in the deposition chamber in order to avoid contamination. We studied the crystal growth and structure of the NiTi thin films by AFM, SEM, XRD and TEM investigations. The orientation relationship of the films investigated here is determined by the {100} single crystalline MgO substrate. Because of the lattice misfit between NiTi and MgO, the as-prepared films are intrinsically stressed; these stresses can be reduced by the two-step heat treatment routine. The stresses do also effect the surface roughness and morphology of the crystalline thin films. To characterize the surface, AFM measurements were used. The resulting phase in the films and the crystal orientation relative to the substrate is determined by XRD and pole figure measurements. TEM investigations of $< 100 >$ and $< 110 >$ cross-sectional FIB lamellae are used for interface studies. We discuss how the microstructure is influenced by the different heat treatments, and particularly how forest dislocations and well-defined dislocation networks located in the film influence the film formation itself. Furthermore, we show that forest dislocations can be replaced by misfit dislocations close to the substrate-film interface by changing the heat treatment procedure. These microstructural investigations enable a deeper understanding of microscopic growth mechanisms related to macroscopically observed microstructures in epitaxial NiTi films.

This work is supported by DFG (WA 2602 and FA 453/13).

[1] S. Kauffmann-Weiss, S. Hahn, C. Weigelt, L. Schultz, M.F.-X. Wagner, S. Fhler, Acta Mat. 132,255, 2017.

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28 Aug
02:00pm
Poster
Session B

Effect of aging at room temperature after hydrogenation on the martensitic transformation and superelasticity in the TiNi alloys with coarse-grained and ultrafine-grained structure

Baturin A.[1,2],Lotkov A.[1],Grishkov V.[1],Rodionov I. [1]

[1]Institute of Strength Physics and Materials Science SB RAS, 2/4, Akademicheskii pr., Tomsk, 634055, RUSSIA;
[2]National Research Tomsk Polytechnic University, 30, Lenin pr., Tomsk, 634050, RUSSIA

At present, more and more attention has been paid to fundamental studies of the effect of diffusion redistribution of hydrogen on the properties of nickel-titanium alloys. The relevance of the study of the effect of hydrogen on the properties of TiNi-based alloys is determined by the expanding use of products from TiNi (in particular implants) in medical practice. Hydrogenation of implants occurs either during their preparation in the process of chemical polishing, or in the process of interaction with the physiological environment in the body at the temperature of the human body. This leads to chemical destruction of the oxide film on the surface of samples or implants, followed by the penetration of hydrogen atoms into the near-surface layer. Because of low diffusion mobility at temperatures close to room temperature or the temperature of a human body, hydrogen accumulates in a significant concentration, especially in the near-surface layer. Later, it diffuses into the interior of the material, leading to a significant degradation of the superelastic properties and even to brittle fracture of the material. The main attention is paid to the influence of hydrogen on the superelasticity and mechanical properties. However, the possible effect of hydrogen on martensitic transformations (MT) is lost, which determine the basic functional properties of these alloys. In this report, on the basis of our own results and published data, we analyze the effect of hydrogen on martensitic transformations as a function of the initial structural-phase state and the aging time at room temperature in binary TiNi-based alloys. It is shown that the influence of hydrogen at its diffusion redistribution depends very strongly on the initial structural state at which hydrogenation takes place, in particular, it is shown that during hydrogenation in the B2 phase, the Ms temperature of the direct MT $R \longleftrightarrow 19'$ or $B2 \longleftrightarrow B19'$ decreases by more than one hundred degrees Kelvin.

The second aspect, which is also not currently being studied in the world for this class of materials, is the effect of grain size on the processes of interaction with hydrogen. At present, ultrafine grained materials are finding increasing applications, including in medicine. Therefore, this report presents the first studies comparing inelastic properties of samples of a TiNi binary alloy with coarse-grained and nanocrystalline structures under the influence of hydrogen from a physiological solution. The possible mechanisms of the influence of hydrogen on the temperature of the MT and the superelasticity are analyzed.

The work was supported by Fundamental Research Program of the State Academies of Sciences for 20132020 (III.23.2.2) and Russian Foundation for Basic Research (grant No. 15 - 08 - 99489).

Pre-martensitic phenomena, martensitic transformation and strain variation in quenched Ni-rich NiTi alloy

28 Aug
02:00pm
Poster
Session B

Belyaev S.[1], Resnina N.[1],Ivanov A.[1],Demidova E.[1],Andreev V. [2]

[1]Saint-Petersburg State University RUSSIAN FEDERATION;
[2]MATEK-SMA Ltd., Moscow, RUSSIA

Previously it was shown that the NiTi-based alloy underwent the martensitic transformation during holding at constant temperatures within or outside the temperature range of the athermal martensitic transformation. It was assumed that this phenomenon was caused by the variation in the strain nanodomains structures in pre-martensitic temperature range that led to a decrease in elastic energy and change in the thermodynamic equilibrium that made possible the realization of the martensitic transformation at constant temperature. Moreover, the formation of the strain nano-domains and martensitic transformation should be accompanied by the strain variation if cooling and heating occurs under a stress. The relation between the pre-martensitic phenomena, the isothermal martensitic transformation and strain variation in Ni-rich NiTi alloy has not been found and it is the aim of the present work. The pre-martensitic phenomenon and martensitic transformation was studied by resistivity measurement and differential scanning calorimetry on cooling and heating in a temperature range of 100 to -100 ˚C with different cooling/heating rate. The strain variation was measured on cooling and heating under a constant stress of 50 to 300 MPa in a temperature range of the pre-martensitic state and phase transformation. The results obtained show that the formation of the strain nanodomains is accompanied by the resistivity variation without any heat effects on calorimetric curve. A variation on cooling/heating rate influences the temperature ranges of the pre-martensitic states and transformation temperatures. The isothermal holding of the alloy in pre-martensitic temperature range leads to the formation of the martensitic phase. The strain variation on cooling and heating depends on the cooling/heating rate and the less the rate the larger the strain variation and the larger the plastic strain.

On the annealing temperature effect on the thermomechanical behavior of the Ni-rich NiTi SMA

28 Aug
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Poster
Session B

Ben Fraj B.[1],Zghal S.[2],Gahbiche A.[3],Tourki Z. [4]

[1]Mechanical laboratory of Sousse, National Engineering School of Sousse TUNISIA;
[2]Laboratoire des Matériaux Multifonctionnels et Applications LaMMA,Faculté des sciences de Sfax, Université de Sfax, 3000 Sfax, TUNISIA;
[3]Laboratoire de Génie Mécanique LGM, Ecole Nationale d'Ingénieurs de Monastir ENIM, Université de Monastir, 5019 Monastir, TUNISIA;
[4]Laboratoire de Mécanique de Sousse LMS, Ecole Nationale d'Ingénieurs de Sousse ENISo, Université de Sousse, 4023 Sousse,TUNISIA;

In the present research work, the relationship between the phase transformation behavior and the annealing temperature in the Ni-rich NiTi SMA was investigated. In view of the high dependence of this alloy to the heat treatment conditions, an experimental study was carried out in order to identify the optimal thermal conditions that produce later the shape memory and the superelastic effects of this material. The thermal characterization was performed at zero stress using Differential Scanning Calorimetry (DSC) experiments. The calorimetric results showed that the thermal properties, mainly the transformation temperatures, and the R phase presence level are strongly influenced by the increase of the annealing temperature in the range temperature between 450°C and 650°C. This result was proved by a hardness measurements which described in their turn the effect of the annealing temperature on the mechanical behavior of the examined alloy. Moreover, the interpretations of the obtained findings were also argued and supported by a microstructural study through scanning electron microscopic (SEM) observations and X-Ray diffraction (XRD) analysis. These analyzes allowed an integral estimation of the material structure and phase composition for each proposed annealing temperature. The influence of the precipitation process, occurred during the NiTi phase transformation, was discussed and correlated with the results of the thermal analysis. The mechanical characterization was extended to an experimental compression tests at various temperatures where both superelastic and shape memory effects were highlighted. It was found that numerous mechanical properties such as transformation stresses, strain and dissipated energy are considerably affected by the variation of the annealing temperature. The critical stress-temperature diagrams were constructed based on both thermal and mechanical studies and showed the great effect of the annealing temperature on the transformation lines that govern the overall NiTi thermomechanical behavior and define the limits of the areas in where the different aspects of superelasticity and shape memory can be occurred.

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28 Aug
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Session B

In situ study of thermomechanical processing of NiTi shape memory alloy

Braz Fernandes F. M.[1],Camacho E.[1], Paula A. S.[2],Schell N. [3]

[1] *CENIMAT/I3N, Department of Materials Science, Faculdade de Ciências e Tecnologia, Universidade Nova de Lisboa, Monte da Caparica, PORTUGAL;*
[2] *Mechanical Engineering and Materials Department-SE-4, Instituto Militar de EngenhariaIME, BRAZIL;*
[3] *Institute of Materials Research, Helmholtz-Zentrum Geesthacht, Max-Planck-Str. 1, D-21502 Geesthacht, GERMANY*

The combined efect of solubilization, hot deformation and precipitation phenomena affect the mechanical behavior and functional properties of shape memory alloys. In order to evaluate the hot workability and processing parameters, in situ deformation experiments were conducted with the high energy X-ray diffraction (HEXRD) setup of the HZG beamline (EH3 - P07) at Petra III, DESY, Hamburg. The uniaxial compression tests were carried out under controlled atmosphere in a modified dilatometer Bähr DIL 805 A/D with tensile/compressive stress units. Solubilization temperatures of 800, 850, 900 and 950°C, followed by 500°C annealing, combined with compressive deformation, were analyzed in order to optimize the processing parameters.

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Session B

Martensitic Transformations and Shape Memory Characteristic of Ti-rich Ti51.9Ni48.0Si0.1 Shape Memory Ribbons

Chen C.-H.[1],Wu S.-K.[1],Wang Y.-C. [2]

[1] *Dept. Mechanical Engineering, National Taiwan University and Dept. Materials Science and Engineering, National Taiwan University, PROVINCE OF CHINA;*
[2] *Dept. Materials Science and Engineering, National Taiwan University CHINA*

Ti51.9Ni48.0Si0.1 Shape Memory Ribbons Ti-rich (50.1∼50.4 at. %Ti) TiNi shape memory ribbons with a slight amount of Si content (0.1∼0.2 at. %) have been reported to make the matrix Ni-rich due to extensive precipitation of Ti2Ni phase stabilized by Si. As a consequence, Ti-rich Ti2Ni and Ni-rich Ti3Ni4 precipitates coexist to improve the hardness and shape memory characteristics of these ribbons. However, the properties of TiNi binary shape memory ribbons with still higher Ti content remain unclear. In the present study, a Ti-rich Ti51.9Ni48.0Si0.1 ribbon was investigated to understand their martensitic transformation behaviors, phase constitution, nanoindentation hardness, microstructure, precipitation process and the shape memory effect. Our investigations show that the Ti51.9Ni48.0Si0.1 ribbon can

still precipitate out Ni-rich Ti3Ni4 precipitates even with Ti content about 52 at. %. The existence of Ti3Ni4 precipitate is confirmed by XRD and EDS measurements. With the aids of the nanoscale Ti2Ni and Ti3Ni4 precipitates, nanoindentation hardness reveals the precipitation hardening process in both ribbons. The Ti51.9Ni48.0Si0.1 ribbon shows a two-stage hardening process. Firstly, the hardness of Ti51.9Ni48.0Si0.1 ribbon decreases from 4.2 GPa to 3.2 GPa when aged at 500 °C for less than 3 hr. This is a result of annealing effect, which eliminates defects introduced during the melt-spinning process. Secondly, the precipitation process of nanoscale Ti2Ni improves it hardness to 3.8 GPa, when the aging time reaches 5 hr. The strengthening effect of Ti2Ni becomes less pronounced when the aging time increases to 24 hr and the hardness drops to 3.0 GPa. Finally, the precipitation of Ni-rich Ti3Ni4 precipitates from the Ni-rich matrix attributes to the second hardening effect and the hardness raised to 3.9 GPa again. Tests of shape memory effect shown that the 5 hr-aged ribbon (with the first peak hardness of 3.8 GPa) exhibits larger reversible strain (4.8 %) than that of the as-spun one (4.6 %) under all the applied stress of 20-160 MPa. These results show the hardening effect of nanoscale precipitate in a Ti-rich ribbon with Ti content reaches nearly 52 at.%, which also promote its shape memory function. This study demonstrates that prepared by the melt-spinning technique, Ti3Ni4 precipitates can still form in the Ti-rich (about 52 at.% Ti) ribbons due to the assistance of Si content and the rapid cooling rate. These special characteristics exhibited in Ti-rich TiNi ribbons can effectively improve the mechanical and functional properties of the ribbons over the conventional bulk counterparts.

Strain variation during isothermal martensitic transformation in the Ti40.7 Hf9.5 Ni44.8 Cu5 shape memory alloy

28 Aug
02:00pm
Poster
Session B

Demidova E.[1],Shelyakov A. [2]

[1]*Saint Petersburg State University, Saint-Petersburg, Russia RUSSIAN FEDERATION;*
[2] *National Research Nuclear University MEPhI (Moscow Engineering Physics Institute), Moscow, RUSSIA*

Recent years it was found that thermoelastic martensitic transformations might occur on holding of NiTi-based shape memory alloys at constant temperature within or outside the forward transformation temperature range. However, strain variation during isothermal martensitic transformations has not been investigated and it is the aim of the present work. The Ti40.7Hf9.5Ni44.8Cu5 alloy that underwent the B2$\longleftrightarrow$B19$^{'}$ martensitic transformations was used. The transformation temperatures were determined using calorimetric curve that was obtained on continuous cooling and heating of the sample and found to be Ms= -7°C, Mf = -13°C, As = 29°C, Af= 52°C. To study the strain variation on isothermal holding, the experiments were carried out in three steps. First samples were cooled and heated in a full martensitic transformation temperature range under a constant stress σ. Then, samples were cooled under a stress σ down to the holding temperature T*, held for 60 min, and heated up to a temperature that was greater than Af. On the third step samples were cooled under a stress σ down to the holding temperature T* and immediately heated up to a temperature that was greater than Af. The strain variation on isothermal holding was determined as the difference between strain variation on the second and on the third steps. The constant stress was equal to 100 MPa andthe holding temperature T* was chosen to be outside and within the temperature range of the strain variation on continuous cooling under a constant stress σ through a full temperature range of the forward transformation. The results of this study showed that an additional strain accumulated on isothermal holding. This strain was completely reversible on further heating thus, one may conclude, that the strain variation on holding at constant temperature was caused by the isothermal martensitic transformation. It was found that in the Ti40.7Hf9.5Ni44.8Cu5 alloy strain increased on holding up to the saturation, which value depended on holding temperature.

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28 Aug
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Session B

The effect of the isothermal mechanical cycling on superelasticity and shape memory of Ti49.3Ni50.7 (at.%) alloy

Grishkov V.,Lotkov A.I., Baturin A., Zhapova D.Yu., Timkin V.N.

Institute of Strength Physics and Materials Science of Siberian Branch Russian Academy of Sciences (ISPMS SB RAS) RUSSIAN FEDERATION

The experimental data about the effect of isothermal bending cycling on superelasticity (SE) and shape memory effect (SME) in samples of Ti49.3Ni50.7(at.%) alloy are presented. The cycling temperature (293K) is higher then the start temperature of martensitic transformation from cubic B2 phase into the monoclinic $B19^{'}$ martensitic phase (252K). The SE, ϵSE, was determined in the isothermal "loading-unloading" cycles. The SME, ϵSME, equels to the strain recovery during subsequent heating of unloaded samples. The total deformation ϵt was constant in each bending cycles (from 1 to 11). The series of cycling tests with different ϵt were conducted. The maximum ϵt value was 24%. The changes of ϵSE and ϵSME in depend on the number of cycles were obtained for all series of mechanical cycling with different ϵt. It is shown that the effect of isothermal mechanical cycling on ϵSE is determined by the ϵt value. It is observed only ϵSE decreasing (from 6% to 4-5%) during bending cycling when the ϵt is less than 12%. When ϵt changes from 12% to 24%, the increase of ϵSE take place in the second cycle, but ϵSE decreases again during subsequent cycling. However, the ϵSE degradation either absent or the ϵSE increases slightly (about 1%) when ϵt had been varied from 12% to 24%. The stabilization of ϵSE value (4-7 %) was observed after 6 mechanical cycles with $\epsilon t \geq 24\%$. The ϵSME value increases after first bending cycle at 293K from zero level to 4-5% with ϵt increasing from 4% to 24%. But ϵSME decreases sharply during mechanical cycling independently of ϵt (ϵSME=0.5% after 11 cycles). The comparative analysis of former data about the effect of tension cycling at 293K on SE of TiNi-based alloys and experimental data of present work, obtained under bending, will be conducted.
The work was supported by the Fundamental Research Program of the State Academies of Science for 2013-2020 (project No.III.23.2.2).

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28 Aug
02:00pm
Poster
Session B

Unrecovered strains generated during thermomechanical loading of NiTi wires - why, when and how ?

Heller L.[1,2],Šittner P.[1,2],Tyc O.[1,3],Kadeřávek L.[1,2,3],Seiner H.[4],Sedlák P. [4]

[1]Institute of Physics of the CAS, Na Slovance 1992/2, 18221 Prague, CZECHIA;
[2]Nuclear Physics Institute of the CAS, Husinec - Řež 130, 250 68, Řež, CZECHIA;
[3]Faculty of Nuclear Sciences and Physical Engineering, CTU Prague, Prague 2, CZECHIA;
[4]Institute of Thermomechanics of the CAS, Dolejskova 5, 18200 Praha, CZECHIA

The cyclic response of either superelastic or actuator NiTi wires is accompanied with accumulation of unrecovered strains. It is generally accepted that the unrecovered strain is generated by martensitic transformation (MT) induced under stress. The unrecovered strain tends to stabilize over cycling and the final magnitude is considered as one of the measures of the quality of NiTi wires. The unrecovered strain is believed to depend on the loading regime; it is thus usually evaluated for loading regimes typical to the NiTi material in question; the superelastic NiTi wires are subjected to full superelastic cycles at near ambient temperatures while the actuator NiTi wires are subjected to thermal cycles under constant loads or against bias springs. Moreover, the actuator wires are usually subjected to lower stresses and higher temperatures compared to plateau stresses of superelastic wires at usually applied ambient temperatures. Therefore, it is unclear from the current state of the art when, how, and why the unrecovered strain appear. It remains unclear what parameters determine the magnitude of unrecovered strain and whether the unrecovered strain of a NiTi wire is a unique function of loading parameters. To answer these questions we performed a large set of thermomechanical experiments in wide stress and temperature ranges (-100 ˚C 100 ˚C, 0 1300 MPa) on superelastic NiTi wires with a selected microstructure that preserved stable superelastic response and yield stress above 1 GPa but showed slightly larger thus accurately measurable unrecovered strains.
The experiments clearly demonstrate that the unrecovered strains accumulate in medical graded NiTi wires at stresses that are fractions of yield stresses of austenite and martensite. The unrecovered strains are, hence, without any doubt coupled with the MT. Unique thermomechanical experiments deconvolved the unrecovered strains generated in forward and reverse MT. It is shown that the unrecovered strains are generated in both the reverse and forward MT and this holds for both the superelastic and actuator loading regimes. A number of experiments was used to correlate the unrecovered strains with the stress

and temperature at which the MT proceeds. Based on these experiments we claim that the unrecovered strains generated by forward and reverse MT are unique functions of the stress and temperature at which MT proceeds. The unrecovered strains can thus be represented by two exponentials (forward/reverse MT) in the stress-temperature space. Such a diagram can be used as a tool for prediction of the unrecovered strain arising from any thermomechanical path. Additional experiments were performed to investigate the effect of martensite stabilisation by deformation on the unrecovered strains in reverse MT and the effect of stress-free cycling on the unrecovered strains generated in subsequent MT under stress.
To sum up, we will discuss the unrecovered strains generated in strong NiTi wires during the MT under stresses well below the yield stresses of both the phases. We will present experimental results supporting our claim that the forward as well as reverse MT under stress generate the unrecovered strains that are unique functions of the stress and temperature at which MT proceeds.

Thermomechanical characterisation and modelling of NiTiFe and NiTiNb for design of couplings

28 Aug
02:00pm
Poster
Session B

Jury A.[2],Heller L.[2,1],Alarcon E.[2,1],Šittner P.[2,1],Sedlák P.[3],Frost M.[3],Stalmans R. [4]

[1]Nuclear Physics Institute of the CAS, Husinec - Řež 130, 250 68, Řež, CZECHIA;
[2]Institute of Physics of the CAS, Na Slovance 1992/2, 18221 Prague, CZECHIA;
[3]Institute of Thermomechanics of the CAS, Dolejskova 5, 18200 Praha, CZECHIA ;
[4]FLEXMET BVBA, Rillaarsebaan 233, BE-3200 Gelrode (Aarschot),BELGIUM

NiTiNb and NiTiFe Shape memory alloys (SMA) have been applied in couplings for their large hysteresis and low transformation temperatures. To optimize the design of couplings the finite element modelling (FEM) is often considered, which, however, requires a reliable SMA material model fed with material parameters identified from experiments that are relevant for the targeted application. In this work, we performed dedicated experiments on selected NiTiFe and NiTiNb SMAs aimed at identifying the key features of the functional behaviour that are critical for their use in couplings. Furthermore, we tuned parameters of SMA model [1] on the experimentally observed responses in order to setup a FEM tool for the optimization of the geometrical parameters of SMA couplings.
The transformation behaviour of SMA dog-bone samples in tension was mapped in the stress-temperature space by thermomechanical experiments at temperatures ranging from -180 °C up to 100 °C. As a result, non-equilibrium stress-temperature diagram [2] was constructed serving as a basic input for the SMA model. Further experiments were carried out in order to evaluate the performance of the shape memory effect and recovery stresses that are critical for couplings. Accordingly, the recovery stresses were evaluated for a constant external bias stress and for linearly increasing bias stress mimicking the elastic resistance of the tubing to be coupled. The increase of the plastic strain with increasing bias stresses was evaluated and correlated with the trend of temperature evolutions of recovery stresses that tend to reach a maximum at a critical temperature. Finally, the effect of martensite stabilization by deformation was evaluated in terms of the shift in austenite finish temperature with respect to maximum deformation of the martensite phase.
The experiments provided data for tuning the SMA model that was systematically subjected to experimental conditions while the model parameters were updated until a reasonable agreement was reached between the experimental and simulated responses. Finally, preliminary simulations of a simple couplings design was performed considering an elastoplastic model for the material of tubing.

[1] P. Sedlák , M. Frost , B. Benešová , T. Ben Zineb,P. Šittner, Thermomechanical model for NiTi-based shape memory alloys including R-phase and material anisotropy under multi-axial loadingsInternational Journal of Plasticity, 39, pp. 132-151, 2012 .

[2] Šittner, P., Novák, V.,Anisotropy of martensitic transformations in modeling of shape memory alloy polycrystals. Int. J. Plast. 16, 12431268, 2000. .
https://doi.org/10.1016/S0749 - 6419(00)00009 - 7

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28 Aug
02:00pm
Poster
Session B

How localized superelastic deformation of NiTi wires in tension affects its fatigue performance

Kadeřávek L.[1,2], Heller L.[1], Klimša L[1].,Šittner P. [1]

[1]*Institute of Physics of the CAS, Na Slovance 1992/2, 18221 Prague, CZECH REPUBLIC;*
[2]*Faculty of Nuclear Sciences and Physical Engineering, CTU Prague, Prague 2,CZECH REPUBLIC*

Although superelastic applications of shape memory alloys, particularly NiTi in medical devices, are already well established, fatigue remains to be an issue. This work deals with tensile superelastic fatigue of thin NiTi wires having different starting microstructure due to different heat treatment. Since NiTi wires cycled in tension may or may not deform via propagation of martensite bands fronts in which the forward and reverse martensitic transformation is localized, any analysis of fatigue performance has to account for that. Nevertheless, until recently there was very little systematic experimental evidence on the impact of localized deformation on fatigue performance of NiTi available in the literature.
In this work, we will present results of the systematic analysis of localized cyclic deformation of thin NiTi wires in tension. The localized deformation of a thin wire is beneficially detected by a Digital image correlation /DIC/ method. The effect of starting microstructure, temperature, tensile test parameters, material evolution on the localized deformation during cyclic loading will be discussed. It is proposed that the amount of plastic deformation, which accompanies the martensitic transformation within the propagating martensite band front, has impact on fatigue performance. It controls the accumulation of material damage, which ultimately leads to crack nucleation and fatigue failure. This was confirmed by the results of fatigue testing in the SEM carried out to evaluate the accumulation of material damage upon cycling. A question whether and how the localized deformation affects the fatigue performance of superelastic NiTi wires will be discussed based on the experimental results.

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28 Aug
02:00pm
Poster
Session B

Deformation in the bend of rectangular plate from titanium nickelide

Dyomina M.[1],Andronov I. [2]

[1]*Syktyvkar Forest Institute RUSSIAN FEDERATION;*
[2]*Ukhta Technical University RUSSIA*

Theoretical and experimental studies on the change and design of the shape, the size of the parts in specific temperature-strength operating conditions are relevant. One of the possible forms of working elements with shape memory is a flat rectangular plate made of titanium nickelide, and the deformation properties of such products depend on the preliminary thermal and plastic processing of the metal. An experimental study of the plane bending of a rectangular plate made of equiatomic TiNi, preliminarily annealed at a temperature of 500°C, was carried out in this work. The experiment was carried out in the following sequence: the plate was plastically deformed at a temperature of 20°C, pressing against a cylindrical surface. After removing the force, i.e. under isothermal (pseudoelastic) unloading, the radius of curvature of the long side of the plate changed. As a result of isothermal deformation with gradually increasing curvature, a low-temperature curvilinear shape and a high-temperature flat shape were formed at the plate. The plate was then heated in a free state through an interval of reverse martensitic transformation and the final radius of curvature of the plate after heating was determined. After isothermal deformation, the plate was subjected to flat bending under the action of a constant force with a change in temperature in the intervals of the martensitic transformations. The plate was clamped rigidly along one of the short sides, heated to a temperature of 90°C, loaded with a constant concentrated force applied to the opposite unattached end, and cooled under load to a temperature of $\sim$ 24°C, then heated to 90°C. When cooling under load, the plate bent in the vertical plane, changing the curvature, when heated under load, the flat plate shape was restored. The plate bent in the vertical plane, changing the curvature, when cooling under load, the flat plate shape was restored when heated under load. Based on the results of the experiments, the radius of curvature was determined and the deformation of the stretched plate surface was calculated. The amount of deformation due to the effect of shape memory, realized when the rectangular plate of titanium nickelide is heated after isothermal bending, increases with decreasing radius of curvature. The curvature of the longitudinal fibers in the bending of the clamped plate during thermal cycling under the action of a vertical force varies in a complex manner. Calculations showed that the linear deformation of the surface does not exceed 1.5% with a decrease in the radius of curvature of the longitudinal fiber by 56%.

Nanohardness and modulus of elasticity of single crystals of the TiNi-TiFe system

28 Aug
02:00pm
Poster
Session B

Muslov, S.

MSMSU RUSSIAN FEDERATION

The study of the physical and mechanical properties of materials has always been and is of interest to researchers dealing with the problems of phase transformations in solids. In this case, the hardness and elastic moduli of materials are important and very informative characteristics. In this message the hardness and modulus of elasticity of single crystals of Ti50Ni50-xFex alloys (x = 50, 25, 10, 5, 2) and TiNi (Ti49Ni51), which gradually lose stability to one B2-R, and then to two martensitic transformations B2-R-$B19^{\prime}$ (TiNi and TiNiFe with an iron content of less than 10%).
The level of elastic properties of single crystals of alloys depends on the concentration of iron and decreases smoothly with decreasing Fe content in alloys and gradual loss of their resistance to martensitic transformations. The greatest value of the elastic modulus E (190 GPa) was observed in the TiFe intermetallide, the farthest from alloys with martensitic transformations, the smallest (68 GPa) in the TiNi polycrystal undergoing the B2-R-$B19^{\prime}$ transformation chain, the average Young's modulus of the TiNiFe alloys was 112, 43 GPa.
The hardness H of alloys Ti50Ni50-xFex also decreased with decreasing x, although not so monotonously. The deviation of the concentration dependence of hardness from the monotonic one can be due to the orientation dependence (anisotropy) of the physical characteristics of the crystals. Nevertheless, the maximum value of H (6.90 GPa) was noted for the intermetallide Ti50Ni25Fe25, the smallest (3.27 GPa) for Ti49Ni51 in the polycrystalline state, the average value was 4.25 GPa.
It is known that the moduli of elasticity of polycrystals can be calculated from the elastic characteristics of a single crystal by averaging them over all possible orientations of the crystallographic axes. We used this fact to compare the experimental data obtained with indentation with known data on single crystals. The average deviation of the averaged values of the modules from the empirical data was 11.55%.
Also, the ratio of H/E Ti50Ni50-xFex alloys with and without martensitic transformations was of interest. According to known data this ratio for most metals is less than 0.02-0.1, and for NiTi it is 0.45. These data are in good agreement with the values obtained in this paper, including 0.055 for single crystals of Ti49Ni51 and 0.06 for the Ti49Ni51 alloy in the polycrystalline state. As for the general appearance (rise) of the H/E curve as a function of the iron concentration in the alloys, it is most likely related to the softening of the elastic moduli of the alloys as they approach the martensitic transformation points. For alloys experiencing martensitic transformations H/E>0.035.

The influence of high-strain rate compression on the functional properties of the TiNi alloy

28 Aug
02:00pm
Poster
Session B

Ostropiko E.

Saint Petersburg University RUSSIAN FEDERATION

The work is devoted to studying the influence of the compression strain rate on the functional properties of the TiNi alloy. ylindrical specimens made of TiNi alloy were compressed at different rates. High-strain rate compression was performed using split Hopkinson pressure bar at 1000s$^{-}1 strainrate.Quasi - staticcompressionwasperformedusinguniversaltestingmachineat0.001s^{-}1$ strain rate. The compressions were conducted at various temperatures in the range from 20 to 300 degrees Celsius. After deformation samples were subjected to two thermocycles in the temperature range of martensitic transformation. During the thermal cycling, strain and temperature were measured. This allowed calculating the values of one-way and two-way shape memory effect. Thus, dependences of the one-way and two-way shape memory effects on the pre-deformation temperature were obtained. It is shown that the value of shape memory effect after high-strain rate compression in the temperature range from 20 to 60 degree Celsius is higher than after quasi-static compression. One-way shape memory effect initiated by the high-strain rate compression decreases faster with increasing test temperatures than one-way shape memory effect after quasi-static compression. Two-way shape memory effect of martensitic type behaves like shape memory effect: the value in the temperature range from 20 to 60 degree Celsius is higher in case of high-strain rate compression. The value of two-way shape memory effect of austenitic type after high-strain compression is always greater than after quasi-static compression, independent of the pre-deformation temperature. Results are compared with previous similar investigations devoted to tension tests. The previous studies did not show any improvement in functional properties of TiNi alloy after high-strain rate tension.

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Poster
Session B

The Effect of Stress Risers on Superelasticity of NiTi Components

Shayanfard P.[1],Heller L.[2],Alarcon E.[2],Šittner P.[2],Sandera P.[1],Hornikova J.[1],Pokluda J. [1]

[1]*Faculty of Mechanical Engineering, Brno University of Technology, Technická 2, CZ61669 Brno, CZECH REPUBLIC ;*
[2]*Institute of Physics of the Academy of Sciences, Na Slovance 2, Prague 18221,CZECH REPUBLIC*

The mechanical response, its stability, and fatigue performance of superelastic NiTi components is affected by peak stresses and multiaxial stress states that develop around stress risers present in components with complex shapes such as stents and orthodontic files. Upon loading the geometrical irregularities lead to stress concentrations triggering locally the martensitic transformation (MT) that gradually extends further to the bulk thus providing required macroscopic superelasticity. The material in the stress risers is subjected to a triaxial stress-strain state while it must sustain high transformation strains of the surrounding bulk that deforms preferentially in the direction of the external loadings. These aspects makes from the stress risers critical locations where irreversible processes may preferential occur and degrade the fatigue performance of NiTi components.
In order to understand the interaction between deformation processes around the stress risers and in the bulk we analyzed cyclic superelastic response of a thin NiTi ribbon with a semi-circular notch at the edge. We used Digital Image Correlation to track localization of MT and accumulation of irreversible strains. Furthermore, we used the micro diffraction to identify the accumulation of residual martensite during cycling. The interpretation of experimental results was assisted by Finite Element Modelling with the use of MSC Marc software and implemented thermomechanical SMA model including plasticity. The experiments clearly showed that the MT is triggered at the notch and further extends to the bulk in a localized manner upon further loading. The localization of MT persists over cycling until the final rupture. The accumulation of unrecovered strains and retained martensite at the notch tip saturates within the first ten cycles. Upon further loading the saturated zone advances further from the notch to the bulk. The modelling shows that the fully transformed notch is subjected to monotonically increasing triaxial stress state while the MT extends into the bulk. As soon as the MT band extends over the whole cross section the macroscopic plateau appears saving the notch zone from further loading. However, before the macroscopic plateau appears the material at the notch zone yields and plastic deformation takes place. The modelling combining SMA behaviour with simple plasticity of martensite was able to predict the appearance of residual martensite induced in the notch zone by compressive internal stresses arising from the local plastic deformation.
However, this simplified picture of mechanics of the notch zone does not correlate with the generally observed poor fatigue behaviour of NiTi in cyclic superelasticity as the predicted compressive stresses would tend to protect the notch zone from further loading and from further fatigue damage. In fact, according to recent findings it seems that the martensite loaded into its apparent plastic regime does not deform through regular slip. Therefore, the obtained results will be discussed with regard to these recent findings.

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28 Aug
02:00pm
Poster
Session B

Effect of annealing conditions on transformation and shape memory characteristics of melt-spun TiNiCu alloys with high Cu content

Shelyakov A.,Sitnikov N.,Borodako K.,Menushenkov A.

National Research Nuclear University "MEPhI" RUSSIAN FEDERATION

A series of the alloys of the quasi-binary TiNi-TiCu system with a copper content of 26 to 38 at.% was obtained by the melt-spinning technique at a cooling rate of 106 K/s in the form of ribbons with a width of 1 to 2 mm and a thickness of 30 to 50μm. X-ray diffraction, transmission and scanning electron microscopy, differential scanning calorimetry (DSC) and mechanical tests were performed to characterize microstructure, phase transformations and shape memory effect (SME) in the ribbons. It was established that in the initial state after quenching the alloys are amorphous. The temperature intervals of the crystallization of the alloys from the amorphous state were determined by the DSC, the alloys with Cu content of 36 and 38 at.% showed two peaks of crystallization. The isothermal crystallization of amorphous alloys was carried out at a temperature of 500°C with different annealing time in the range from 100 to 300 seconds. It was revealed that isothermal annealing leads to the formation of a crystal structure with an average grain size of 0.6-1.0 μm. Structural analysis showed that the alloys with Cu content from 26 to 32 at.% crystallize with the formation of single-phase structure 2 (TiNiCu), which transforms into martensitic phase B19 at the cooling. The critical temperatures and enthalpies of martensitic transformation B2-B19 decrease with increasing Cu content. In the alloys with Cu content from 34 to 38 at.% a two-phase structure consisting of B2-phase and B11-phase (TiCu) is formed and no martensitic transformation is observed, the alloys are brittle and do not exhibit the SME. A decrease in the annealing time to 100 s results in a shift of the martensitic transformation intervals in the region of higher temperatures, a slight increase in the SME in the alloy with 32 at.% Cu and the appearance of small SME in the alloy with 34 at.% Cu.

Influence of holding temperature on recovery in martensitic transformation temperatures in TiNi alloy after preliminary thermal cycling

28 Aug
02:00pm
Poster
Session B

Sibirev A.,Resnina N.,Belyaev S.

Saint-Petersburg State University RUSSIAN FEDERATION

The aim of the present work was to investigate the peculiarities of TiNi alloy properties recovery on isothermal holding after thermal cycling through a martensitic transformations temperature range. The Ti50Ni50 alloy wire samples were water quenched from 700 °C and annealed at a temperature of 500 °C for two hours, after annealing alloy underwent B2-B19$'$ transformation on cooling and B19$'$-B2 transformation on heating. The samples were subjected to 20 thermal cycles in a temperature range of 0 °C ÷ 200 °C and it led to a change of the forward transformation sequence from B2 B19$'$ to B2-R-B19$'$. After thermal cycling the samples were subjected to holding at constant temperature that was belong to a range from 100 °C to 300 °C for a duration from 20 to 180 minutes. Four-point resistivity measurement method was used to determine parameters of martensitic transformations after thermal cycling and holding. As it was expected, thermal cycling resulted in a decrease in transformation temperature due to an increase in dislocation density. It was found that isothermal holding influenced the temperatures of martensitic transformations in different way. If holding temperature was less than 200 °C then transformation temperatures additionally decreased after holding. If the holding temperature was equal to 200 °C then no variation in martensitic transformations temperatures was found despite the holding duration. If holding temperature was more than 200 °C, then transformation temperatures increased after holding hence the recovery of martensitic transformation temperatures to the parameters that were measured in annealed alloy was found. The higher the holding temperature or longer duration the more intensive the recovery in transformation temperatures. As the variation in martensitic transformation temperatures is caused by a variation in defect density, then it may be concluded that holding alloy at temperature that is higher than the thermal cycling maximum temperature (200 °C) resulted in redistribution or annihilation of dislocation structure in alloy and it is the nature of recovery in martensitic transformation temperature.

Gradient of hardness and Youngs modulus over crosssection of laser annealed NiTi wires

28 Aug
02:00pm
Poster
Session B

Samal S.[1],Heller L.[1],Brajer J.[2],Manak J.[1],Šittner P. [1]

[1]*Institute of Physics of Czech Academy of Science, Prague 182 21, CZECH REPUBLIC;*
[2]*Czech Technical University, CVUT, Prague, CZECH REPUBLIC*

Superelastic NiTi wires (diameter $100\mu m$) were laser annealed with the aim to modify its surface and subsurface microstructure and improve thus its fatigue performance. The laser annealing was carried out keeping the wire prestrained at constant length ($\epsilon \sim 5$ %) in stress induced martensitic phase. The continuous laser scanning was performed at the speed of 300 mm/min along the length of the NiTi wire at longitudinal axis with pulse frequency of 500 Hz at interval time of 0.5 ms. The laser scanning of the wire was performed twice with 180°rotation along the wire axis. When heating the constrained martensite, it undergoes reverse transformation coupled with plastic deformation [1]. As this is most pronounced at the hottest wire surface, gradient of microstructure and hardness is expected to exist from the center to the wire surface.

The gradient of hardness on wire cross section was examined by a nanoindentation technique. The hardness and Youngs modulus of the laser annealed sample were calculated from the load versus displacement curve that obtained by Berkovich indenter impacted at the individual locations on the wire cross section from the surface towards the center. A gradient of hardness and Youngs modulus is observed from the surface towards the center, which can be controlled by the parameters of laser heat treatment and applied prestrain. It is proposed that these gradients reflect compressive internal stresses introduced into the wire surface by the laser treatment that may contribute towards improve in fatigue life.

[1] P. Šittner and al, Esomat 2009

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28 Aug
02:00pm
Poster
Session B

Cryogenic Treatment Effect on NiTi Wire Under Thermomechanical Cycling

Da Silva T. C., Contaifer de Moraes L.,Pinheiro Barcelos A.,Da Silva E. P.

University of Brasilia, Federal District, BRAZIL

Shape Memory Alloys (SMA) have been explored for their capacity of recovering great strains through heating in several engineering applications since the discovery of the shape memory effect on NiTi. This material was thoroughly used in actuators and several papers were dedicated to understand the shape memory effect behavior under thermomechanical cycling to improve SMA actuator performance. This work investigated the effect of cryogenic treatment in Ni54Ti wires under constant stress and thermal cycling. An analysis was done by comparing the number of cycles until failure and strain behavior of treated and as-received samples. Results showed a decrease in the plastic strain, but also a loss of recoverable strain after the treatment and about the same fatigue life when compared to as-received wires.

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28 Aug
02:00pm
Poster
Session B

The features of the orientation dependence of reversible strain in aged TiNi single crystals at the variation of Ni concentration

Timofeeva E. E.,Panchenko E.Yu., Tagiltsev A.I., Chumlyakov Yu.I.

Tomsk State University, 634050, Lenina str., 36, Tomsk, RUSSIAN FEDERATION

In heterophase Ti50-xNi50+x (x = 0.6; 0.8; 1.5; 1.8) single crystals containing Ti3Ni4 particles with size of d $\sim$ 400 nm, the value of reversible strain ϵtr at B2-B19$^{'}$ martensitic transformation is determined by the crystal orientation and the microstructure parameters (volume fraction and interparticle distance of dispersed particles). It has been experimentally shown that a strong orientation dependence of the reversible strain ϵtr is observed for a small volume fraction f = 3 % and a large interparticle spacing $\lambda >$ 500 nm in the aged Ti50-xNi50+x (x = 0.6; 0.8) single crystals at shape memory effect and superelasticity under tensile load. Such orientation dependence is in accordance with the theoretical lattice strain at B2-B19$^{'}$ martensitic transformation. In contrast, the orientation dependence of the reversible strain ϵtr degenerates in the aged Ti50-xNi50+x (x = 1.5; 1.8) single crystals with a high volume fraction of dispersed particles Ti3Ni4 f $>$ 16 % and a small interparticle distance λleq 250 nm. Such effect is associated with the influence of Ti3Ni4 particles with size of d $\sim$ 400 nm on the nucleation and growth of martensite crystals, and on the detwining processes of B19$^{'}$-martensite variants under load. The basic type of twinning in the B19$^{'}$-martensite of aged TiNi crystals is $< 100 >$ compound twinning. Compound twins appear for keeping the continuity during the martensitic deformation of the matrix and the elastic deformation of the particles. The significant (more than in 2 times) decrease of reversible strain in comparison with the theoretical resource of the lattice strain occurs in aged Ti50-xNi50+x (x = 1.5; 1.8) single crystals (f $>$ 16 %) in orientations near the $< 111 >$ direction. In these orientations the detwinning makes a significant contribution to the transformation strain ϵtr. The reason for this is the impediment of the detwining during the stress-induced martensitic transformation in the case of high twins density and small interparticle distances. On the contrary, the reversible strain slightly changes in orientations near the $< 001 >$ direction of Ti50-xNi50+x (x = 1.5; 1.8) single crystals. In these orientations the detwinning has no contribution to the transformation strain ϵtr. As a result this leads to a degeneracy of the orientational dependence of reversible strain. The models describing the process of martensitic transformations in heterophase Ti-Ni single crystals were proposed on the basis of experimental data. These models take into account the effect of local stress fields around the particles on the nucleation and growth of martensite crystals, the formation of the multivariant structure of B19$^{'}$ martensite, and the orientation dependence of the detwinning of martensite crystals under load.
This work was supported by the Russian Science Foundation (grant No. 18-19-00298).

Thinner and thick wires in NiTi SMA alloy and their applications

28 Aug
02:00pm
Poster
Session B

Torra V.

Polytechnic University of Catalonia (retired) SPAIN

Applicability of SMA NiTi wires requires 1) predictable and reproducible wire from a qualified industrial manufacturer; 2) appropriate experimental measurements establishing the eventual evolutions of alloys and their thermo-mechanical behavior and 3) reliable simulation of their behavior. In general, the flag model, reliable for thinner wires, was suggested for different authors but the technical requirements could be hardly different. For instance, in applications associated to magnetic disks, the force remains under several Newton but to damp civil structures, i.e., as required in damping of stayed cables in bridges the force overcomes the kN. Eventually, when the target was related the smoothing of oscillations induced by earthquakes the forces can overcome the MN, i.e., different wires diameter were required for the applications.
Wires of NiTi were buying from Special Metals (USA) and, later, from SAES Getters (Italy) in several years with similar macroscopic properties. The experimental behavior of NiTi wires shows particular aspects for different diameters. The thinner wire shows flat cycles that, progressively go down to martensite and in the thick wires, cycling induces an S-shaped cycles. The particular shape effect seems clearly intrinsic and related with of the diameter. In fact, strain aging increases the effects. Thinner wires convert in martensite and thick wires remain in Sshaped cycles with an increase of the maximal stress, i.e., eventually up to 50 %. Thick wires were used in experimental measurements in facilities and the S-shaped cycles were simulated in transformation and in retransformation by a cubic model.

Session 10: Elasto-caloric cooling

Invited Lecture: Friday August 31	
8:30 Amphi 4	**S. Seelecke** "Elastocaloric cooling: From basic concepts towards novel air cooling devices"

Oral Presentations: Friday August 31	
9:00 Amphi 4	**I. Aaltio**, T. Fukuda, T. Kakeshita "Elastocaloric effect in hot rolled Ni-Ti-Fe alloys "
9:20 Amphi 4	**H. Dufour**, D. Bourgault, L. Porcar, P. Courtois, S. Pailhès, F. Porcher, J. Debray, B. Ouladdiaf, C. Colin "Study of the multicaloric effects induced during the structural phase transformation in Heusler compounds"
9:40 Amphi 4	**W. Maziarz**, A. Wójcik, P. Czaja, R. Chulist, M.J. Szczerba, M. Kowalczyk, J. Dutkiewicz, E. Cesari "Heat treatment of sintered Ni-Mn-Sn-In metamagnetic shape memory alloys"
10:00 Amphi 4	**J. Tušek** "Elastocaloric cooling: will your next cooling device be driven by martensitic transformation? "

Poster Session C: Wednesday August 29
A. Aliev, A.G. Gamzatov, A.B. Batdalov, L.N. Khanov, A.A. Mukhuchev, A.P. Kamantsev, A.V. Mashirov, V.V. Koledov, V.G. Shavrov "Peculiarities of the magnetocaloric effect in Heusler alloys in the martensitic transformation region in cyclic magnetic fields "
A. Gamzatov, A.M. Aliev, A. Ghotbi, P. Kameli, I. Abdolhosseini " The behavior of the magnetocaloric effect near the martensitic transition in the Heusler alloy Ni47Mn40Sn12.5Cu0.5 in cyclic magnetic fields "
F. Bruederlin, L. Bumke, H. Ossmer, C. Chluba, E. Quandt, M. Kohl " Advanced SMA-Film Based Elastocaloric Cooling Devices "
J. Frenzel, A. Wieczorek, A. Paulsen, G. Eggeler " Optimizing Ni-Ti for Ferroic Cooling "

Contents

31 Aug
8:30am
Amphi 4

Elastocaloric cooling: From basic concepts towards novel air cooling devices

Seelecke S.

Department of Systems Engineering, Department of Materials Science and Engineering, Saarland University, Saarbrücken, GERMANY

The talk gives an overview of recent results concerning the development of a tensile NiTi-based elastocaloric air cooling device. First, a specifically designed test rig for conductive heat transfer concepts between solid copper blocks has been built that allowing for a systematic investigation of process parameters [1,2]. An analysis of training effects, strain increment, strain rate, or phase angle between loading/unloading and heat transfer on cooling power, temperature span and COP [3,4] identifies optimal processes implementing a hybrid adiabatic/isothermal concept.
The test rig has subsequently been modified to enable experiments with NiTi wires under convective air cooling, which then serve as basis for the design of a novel continuously operating machine. The machine design is presented along with an illustrative simulation tool that assists the design by predicting mechanical and thermal output parameters as a function of, e.g. wire number and diameter, rotational frequency, air flow rate, inlet temperature and loading/unloading profile and hence cooling power and COP [5,6]. Finally, first pictures of the assembled air-cooling device are presented together with infrared measurements to verify the concept.

[1] M Schmidt, A Schütze, S Seelecke, Scientific test setup for investigation of shape memory alloy based elastocaloric cooling processes, International Journal of Refrigeration 54, 2015, 88-97

[2] M Schmidt, A Schuetze, S Seelecke, Experimental investigation of elastocaloric cooling processes, TM-TECHNISCHES MESSEN 83 (4), 2016, 208-218

[3] M Schmidt, J Ullrich, A Wieczorek, J Frenzel, A Schütze, G Eggeler, S Seelecke, Thermal stabilization of NiTiCuV shape memory alloys: Observations during elastocaloric training, Shape Memory and Superelasticity 1 (2), 132-141, 27, 2015

[4] M Schmidt, A Schütze, S Seelecke, Elastocaloric cooling processes: The influence of material strain and strain rate on efficiency and temperature span, APL Materials 4 (6), 2016, 064107

[5] Susanne-Marie Kirsch, Marvin Schmidt, Felix Welsch, Nicolas Michaelis, Andreas Schütze, Stefan Seelecke, Development of a shape memory based air conditioning system, Engineering for a Changing World: Proceedings; 59th IWK, Ilmenau Scientific Colloquium, Technische Universitt Ilmenau, September 11-15, 2017

[6] Susanne-Marie Kirsch, Felix Welsch, Nicolas Michaelis, André Wieczorek, Marvin Schmidt, Jan Frenzel, Gunter Eggeler, Andreas Schütze, and Stefan Seelecke, NiTi-based elastocaloric cooling on the macroscale - from basic conceptsto realization, Energy Technol. 10.1002/ente.201800152

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31 Aug
9:00am
Amphi 4

Elastocaloric effect in hot rolled Ni-Ti-Fe alloys

Aaltio I.,Fukuda T.,Kakeshita T.

Osaka University JAPAN

Elastocaloric effect has received increasing research attention for its promising potential for sustainable solid state cooling applications. Recent reviews of mechano - caloric materials have stated that requirements for their industrial application are that the elastocaloric effect should have high enough repeatability and the material should have a long lifetime, of which features there seems to be presently lack of both alloy candidates and data. In search of such kind of alloys, we selected the Fe-alloyed Ni-Ti, which typically exhibits the two-step B2 to R phase and R-phase to B19´ martensitic phase transformation at cooling. It is known that Fe substitutes Ni atoms of the Ni-Ti lattice and at the same time Fe lowers the martensitic transformation temperature. The hysteresis of the B2 - R phase transformation is smaller than that of the R phase B19´ transformation, which suggests that its fatigue performance related to the former transformation would be better than that of the latter which also supports our selection criteria. We studied experimentally the former transformation behaviour of Ni(50-X)Ti50FeX alloys with X=2 and X=4 in the hot rolled state. The stress-strain-temperature dependency of the test materials was investigated by temperature controlled loading experiments and by calorimetry. Relative magnitude and temperature dependency of the elastocaloric effect was determined from the measured results. The results show that the Ni48Ti50Fe2 alloy could be used as an elastocaloric material near room temperature, while the Ni46Ti50Fe4 alloy seems applicable at a lower -30 to -50 °C temperature region.

Study of the multicaloric effects induced during the structural phase transformation in Heusler compounds

31 Aug
9:20am
Amphi 4

Dufour H. , Bourgault D., Porcar L., Courtois P., Pailhès S.,Porcher F. , Debray J., Ouladdiaf B., Colin C.

CNRS - Institut Néel FRANCE

The aim of this work is to evaluate magnetocaloric and elastocaloric effects that may be coupled in Ni-Mn-In crystal. Our research line focuses on the understanding of the microscopic mechanism behind the structural transition at the origin of their properties and on the measurement of the entropy variation during the first order martensitic transition. Starting from stoichiometric alloys prepared by induction melting, Ni50Mn34.5In15.5 and Ni45Co5Mn37.5In12.5 single crystals were grown with success using the Bridgman technique. Electrical resistivity and deformation were measured at variable temperatures with two home made devices enabling continuous variation of uniaxial stress (up to 3kN, 8T between 77K and 450K), thus taking into account deformation, thermal contraction and volume discontinuity. Single crystals were cut in some specific crystallographic directions.
The low temperature nuclear structure by means of neutrons diffractions studies on powder and single crystals Ni50Mn34.5In15.5 was refined as a function of temperature and compared with literature. The transformation was also followed-up in real time and under magnetic field. Structure refinement of Laue picture was carried out on a single crystal neutron Laue diffractometer with an ultra-large set of area detectors (CYCLOPS, CYlindrical Ccd Laue Octagonal Photo Scintillator). The anisotropic effects and the modulation of the low temperature phase was measured in CYCLOPS with the application of a uniaxial strain on the M-A transition using a home-made piston cylinder cell compatible with neutrons. Deformation as a function of uniaxial stress was measured at different temperatures inside the hysteresis. Deformation in constant uniaxial stress along the phase transformation was also measured. The entropy variation under uniaxial stress was calculated using the Clausius Clapeyron equation and Maxwell relation from the stressstraintemperature characteristics. Efficiency of the elastocaloric properties is discussed and its relation to the transformation arrest during heating under uniaxial stress is analysed. Indeed, a study of the kinetics of the transformation under uniaxial stress shows that a supplementary buildup of compressive strain energy consequent to uniaxial pressure can distort the hysteretic cycle while heating leading to an austenite avalanche transformation.

IHeat treatment of sintered Ni-Mn-Sn-In metamagnetic shape memory alloys

31 Aug
9:40am
Amphi 4

Wojcik A.,Maziarz W. , Wójcik A., Czaja P., Chulist R., Szczerba M.J., Kowalczyk M.,Dutkiewicz J. , Cesari E

Institute of Metallurgy and Materials Science, Polish Academy of Sciences POLAND

Ni-Mn-Sn-based metamagnetic shape memory alloys show great potential as magnetocaloric materials since the discovery of inverse magnetocaloric effect by Krenke et al. [1]. It is well known that magnato-structutal behavior and magnetocalorioc properties depend on factors such as chemical composition and fabrication method. Among several fabrication methods (arc-melting, induction casting, rapid solidification process), sintering of powders is a practical solution due to geometrical advantages, which may increase efficiency in magnetic refrigeration. The approach is also rational due to aspects such as environmentally friendliness and economy. The economic factor is linked to a low-cost fabrication method of the materials, which can be fabricated in powder form in order to obtain net-shape final product. Alloys with composition Ni50Mn37.5Sn12.5-xInx (In= 0, 2, 4 at.%) were obtained from milled powders and sintered by hot pressing at 920 K for 30 min under an uniaxial pressure of 320 MPa. Subsequently, disk-shaped materials were sliced into smaller parts and heat treated under various conditions, in order to control the homogeneity and various atomic order degree. Crystal structure of alloys were determined by X-ray diffraction patterns. Then, detailed microstructural investigation was carried out by scanning and transmission electron microscopy.
Based on differential scanning calorimetry and magnetic measurements, martensitic and magnetic transformation temperatures were estimated. Moreover, thermal effects related to phase transformation were also calculated. Using X-ray and electron diffraction it has been shown that both chemical composition as well as heat treatment influence the crystal structure of the alloys. Also the microstructure is affected since larger grains are detected in heat treated bulk. Moreover, the heat treated alloys appear to have

more homogenous chemical composition than those obtained by sintering. Martensitic transformation temperatures slightly decrease with decreasing temperature of heat treatment being connected with various atomic order degree. Enthalpy and entropy changes strongly depend on the thermal treatment as well.

Acknowledgements
This work has been carried out within the statutory activity of IMMS PAN (Z-10).

[1]T. Krenke, E. Duman, M. Acet, E.F. Wasserman, X. Moya, L. Mañosa A. Planes,Nat., 4 , p. 450, 2005.

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31 Aug
10:00am
Amphi 4

Elastocaloric cooling: will your next cooling device be driven by martensitic transformation?

Tušek J.

Faculty of Mechanical Engineering, University of Ljubljana SLOVENIA

Elastocaloric cooling based on the elastocaloric effect (eCE) in shape memory alloys (SMA) is emerging as one of the most promising alternatives to the widely used vapour-compression cooling technology, which is relatively inefficient and environmentally harmful. The eCE can be characterized as positive temperature changes during stress-induced forward martensitic transformation and as a negative temperature changes during reverse martensitic transformation. Generally, all SMAs can be considered as potential elastocaloric materials when they undergo the stress-induced transformation, and their transformation temperatures are below the operating temperature for the desired application. Theoretical studies based on experimental characterisation of the eCE show that elastocaloric cooling can be potentially significantly more efficient than vapour-compression technology as well as environmentally harmless. Recently, the first prototypes demonstrating a high potential of the eCE as a cooling or heat-pumping mechanism have been developed and tested.
Here, the most interesting SMAs for elastocaloric applications and the state-of-the-art prototypes of elastocaloric devices will be reviewed. The basic operational principles of elastocaloric cooling cycle will be explained and discussed. In general, one can distinguish between a single-stage elastocaloric cycle, with contact heat transfer between the elastocaloric material and heat sink/source, and a regenerative elastocaloric cycle, with a porous elastocaloric material, through which a fluid that transfers heat between the elastocaloric material and heat sink/source, is pumped in a counter-flow direction. In addition to positive aspects of this novel cooling technology, future challenges that need to be addressed and solved to bring elastocaloric technology closer to the market will be discussed. The major obstacle that prevents practical elastocaloric applications is structural fatigue of SMAs, in particular for tension loading when high strains (related with high temperature changes in SMA) are applied. It is known that fatigue life can be enhanced if compression loading is applied instead of tension, but it is a big challenge to design an elastocaloric structure (regenerator) that is geometrically stable during compression and shows sufficient heat transfer properties (high specific heat transfer area per volume unit). Different geometries of elastocaloric structures to be loaded in compression, such as block with holes, set of tubes in a holder, shell-and-tube-like regenerator, etc., will be shown and the main challenges associated with those geometries will be discussed.

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29 Aug
02:00pm
Poster
Session C

Peculiarities of the magnetocaloric effect in Heusler alloys in the martensitic transformation region in cyclic magnetic fields

Gamzatov A.G.[1], Batdalov A.B.[1], Khanov L.N.[1], Mukhuchev A.A.[1], Kamantsev A.P.[2], Mashirov A.V.[2],Koledov V.V.[2] , Shavrov V.G.[2]

[1]Amirkhanov Institute of Physics of Daghestan Scientific Center RAS, Makhachkala, RUSSIA;
[2]Kotelnikov Institute of Radio Engineering and Electronics, RAS, Moscow, RUSSIA

Presently, one of the major requirements for a material to be used as a prospective magnetocaloric material is its capability to exhibit a giant magnetocaloric effect. From a practical point of view there is a substantial need for studying the magnetocaloric effect (MCE) in promising magnetocaloric materials under prolonged exposure to the cyclic magnetic fields. Magnetocaloric properties of the materials exposed to cyclic and constant magnetic fields may show significantly different behavior for a variety of reasons. Recent researches have shown that Ni-Mn-X Heusler alloys are the perspective materials as

refrigerants in cooling devices. Near martensitic transformations these alloys exhibit approximately the same properties as the best MCE materials.
The aim of the work is the direct study of the magnetocaloric properties of Ni-Mn-In Heusler alloys of various compositions in cyclic magnetic fields. MCE, magnetostriction, thermal expansion and specific heat of Ni-Mn-In alloys are measured in magnetic fields up to 8 T. The influence of hysteresis on the magnetocaloric properties of materials, the dependence of the MCE on the frequency of alternating magnetic field, the effect of prolonged action of cyclic magnetic field in the Heusler alloys are studied in detail. For some compositions, the ratio of magnetic and lattice contributions to the total magnetocaloric effect are estimated. The explanation of differences in the values of MCE in the heating and cooling runs, as well as the effect of degradation of MCE under prolonged action of cyclic magnetic field are given. Various mechanisms for MCE reduction with a magnetic field frequency are proposed.
This work is supported by RFBR grants No. 17-02-01195 and 18-08-01434.

The behavior of the magnetocaloric effect near the martensitic transition in the Heusler alloy Ni47Mn40Sn12.5Cu0.5 in cyclic magnetic fields

29 Aug
02:00pm
Poster
Session C

Gamzatov A. [1],Aliev A.M. [1], Ghotbi A.[2], Kameli P.[2], Abdolhosseini I.[2]

[1]*Amirkhanov Institute of Physics of Daghestan Scientific Center of RAS, Makhachkala, Russia;*
[2]*Department of Physics, Isfahan University of Technology, Isfahan, 8415683111, IRAN*

In this paper we present the results of direct measurement of the magnetocaloric effect (MCE) in the Ni47Mn40Sn12.5Cu0.5 Heusler alloy in a magnetic fields of 1.8 T at the heating and cooling protocols. The maximum value of direct effect at the field change of 1.8 T is 1.24 K and revealed at T=314 K. The inverse MCE accompanied with a wide hysteresis (14-20 K) is observed near the martensitic transition (at T=275 K at heating). The value of the inverse effect depends on the rate of a sample temperature change (heating or cooling), the higher the rate is, the higher the inverse MCE. At the fixed temperature a gradual crossover ¨inverse MCE-direct MCE¨ takes place under action of cyclic magnetic fields.
Such a behavior is explainable if to follow a detail change in the MCE under the cyclic magnetic field. The time dependence of MCE was measured at two temperatures (T=275 and 277 K) in the region of the temperature hysteresis. In the course of the experiment the sample first was heated above the Curie point of the austenitic phase, next was cooled well below austenite-martensite transition temperature, and after that was heated up to the required temperatures. All this process was performed under zero magnetic fields. The observed effects can be explained in terms of an example of deltaT(t) dependence at T=275 K. First application of the magnetic field results to the inverse MCE caused by a transition from low-temperature antiferromagnetic martensite to high-temperature ferromagnetic austenite. However, the transition into the ferromagnetic phase occurs not in the whole sample, only a part of the martensite transforms into the austenite. What part of the martensite phase transforms into the austenite one depends both on a field value (in this case 1.8 T is not sufficient to provide the transformation of the whole sample), and on a temperature. Because of the hysteresis effect the ferromagnetic phase doesn‘t revert into martensite at the field switching off. Due to such irreversibility, after several cycles of application of the magnetic field, the martensitic phase completely disappears. From the obtained results it can be concluded that in order to obtain reversible martensite-austenite transitions and, correspondingly, a time-stable giant MCE, it is necessary to apply higher fields that shift the martensite-austenite transition temperature in the heating regime beyond the hysteresis loop.
This work is supported by RFBR grant No. 17-02-01195.

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29 Aug
02:00pm
Poster
Session C

Advanced SMA-Film Based Elastocaloric Cooling Devices

Bruederlin F.[1], Bumke L. [2], Ossmer H.[1], Chluba C.[2], Quandt E.[2], Kohl M.[1]

[1] *Karlsruhe Institute of Technology (KIT), Karlsruhe, GERMANY;*
[2] *University of Kiel (CAU), Kiel, GERMANY*

Elastocaloric cooling is a promising new technology with proven potential to provide efficient, environmentally friendly cooling. The following challenges need to be addressed: (1) Development of elastocaloric shape memory alloys (SMA) with large latent heat, high elastocaloric efficiency and ultra-low fatigue; (2) the engineering and characterization of elastocaloric cooling devices.
Here, we design and demonstrate elastocaloric cooling using TiNiCuCo films, which are currently the most prominent elastocaloric materials due to their ultra-low fatigue properties and high coefficient of performance of the material (COP_ mat) up to 15 [1]. Their large surface-to-volume ratio enables rapid heat transfer. The SMA films are fabricated by magnetron sput-tering, which allows for tuning of chemical composition and microstructure to tailor elastocaloric effect, fatigue properties and transformation temperatures. In the present device concept, mechanical loading/unloading of the SMA film and separation of caloric heating and cooling power is accomplished by periodic mechanical contact of a free-standing elastocaloric SMA film to solid heat sink and source elements [2]. Devices based on a single SMA film are limited by the adiabatic temperature span of the elastocaloric material. In order to achieve higher temperature spans, we propose the advanced concept of cascaded elastocaloric cooling systems. In this case, several cooling units are thermally coupled in series,where the heat sink of a given unit represents the heat source of the neighboring unit.
First demonstrators consisting of up to three units are investigated. The dimension of the cascaded setup without the actuator is 37*22*30 mm^3, used SMA films are 30 μm thick. The maximum device temperature span ΔT measured under cyclic operation increases with the number of units and operation frequency. For the current design of three units, a maximum ΔT of 14.8 K is reached at a frequency of 2 Hz, which corresponds to 85 % of the adiabatic temperature span. The corresponding single unit device reaches 8.4 K under similar conditions. The cooling power of the cascaded device reaches 5 J/g, the maximum Coefficient of Performance of the overall device without the actuator (COP_ dev) is 2.7. These results show that the concept of cascading is a promising approach to enhance elastocaloric cooling and to ultimately overcome the adiabatic limit. This may be achieved in the near future by implementing SMA films with tailored transition temperatures.

[1] H. Ossmer and al., Shap. Mem. Superelasticity 1:142, 2015.

[1] F. Bruederlin and al., J. Phys. D: Appl. Phys. 50.42:424003 , 2017.

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29 Aug
02:00pm
Poster
Session C

Optimizing Ni-Ti for Ferroic Cooling

Frenzel J.,Wieczorek A.,Paulsen A.,Eggeler G.

Ruhr University Bochum GERMANY

Pseudoelastic shape memory alloys (SMAs) on the basis of Ni-Ti are attractive candidate materials for ferroic cooling, where elementary solid-state processes like martensitic transformations yield the required heat effects. The present work aims for a chemical and microstructural optimization of Ni-Ti for ferroic cooling. The goal is to identify new alloy compositions which allow for a higher elastocaloric cooling performance. A large number of Ni-Ti-based SMAs was evaluated in terms of phase transformation temperatures, latent heats, mechanical hysteresis widths and functional stability. Different material states were prepared by arc melting, various heat treatments, and thermo-mechanical processing. Differential scanning calorimetry and uniaxial tensile testing in combination with thermography analysis were used to assess the cooling performance of selected material states. The results show that a fundamental dilemma exists. The key parameters latent heat and hysteresis width correlate. Therefore, it is difficult to select alloys which simultaneously combine low a hysteresis width and large latent heats, which is required for highly efficient ferroic cooling processes. Nevertheless, a good compromise was found in a Ni45Ti47.25Cu5V2.75 SMA. This material exhibits an extremely stable elastocaloric effect at room temperature and a very low hysteresis width. The ferroic cooling efficiency of this material is close to four times higher as compared to binary Ni-Ti. Furthermore, this material provides sufficient ductility such that sheets and wires can be prepared by thermo mechanical processing.

Social Events, Conference excursions & Accompanying persons program

Social events

- **Opening ceremony**: Monday August 27
  1:00 pm Ile du Saulcy, Metz
- **Welcome reception**: Monday August 27
  07:00 pm Metz Town Hall, Metz
- **Gala evening**: Thursday August 30
  08:00 pm Le Royal, Metz
- **Closing ceremony**: Friday August 31
  12:40 pm Ile du Saulcy, Metz

Conference excursions: Thursday August 30

Option 1: Centre Pompidou (60 people)

03:00 pm: Guided tour in English of the Centre Pompidou

The Center Pompidou-Metz is the first decentralization of a national public cultural institution. The Pompidou Center brings its model to the region and provides its know-how and collections, in an unprecedented partnership with the local authorities that provide funding while ensuring the autonomy of scientific and cultural choices. True to the Pompidou Center's values of generosity, openness to all audiences and to all forms of contemporary creation, the Center Pompidou-Metz illustrates, through its societal and cultural dimension, the renewal of the Center Pompidou's strategy. refocus on its primary vocation: to be a platform for exchanges between French society and creation.

Option 2: Visit of the Historical centre of Nancy (50 persons)

A bus will take the participants to visit Nancy located 60 km south from Metz

03:00 pm to 05:00 pm: Guided tour in English of the Historical centre of Nancy

A pedestrian guided tour of 2h is proposed to introduce the historical center of Nancy. Is included the visit of Place Stanislas and Place Carrière, which allows to highlight the mark that the last Duke of Lorraine let on the town, the old medieval and renaissance city (Palais Ducal, Basilique Saint Epvre, Porte de la Craffe, Hôtel d'Haussonville...).

Option 3: Cour d'Or Museum (25 people)

02:30 pm to 04:30 pm: Guided tour in English of Cour d'Or Museum

Located in the centre of town, the golden court museum is one of the city's cultural jewels. Gallo-Roman treasures, mediaeval painted ceilings, paintings from the Metz school and abstract art form the basis of the wealth of our Gallo-Roman, mediaeval and Fine Art collections. It doesn't matter how old you are - come and be surprised by our 5000 square meters of exhibition space, and set off on a journey of discovery through 2000 years of history of the city of Metz...

Option 4: Quartier Impérial (30 people)

02:30 pm to 04:30 pm: Guided tour in English of the Quartier Impérial

This picturesque district, built around the station, is a remarkable illustration of early 20^{th} century Germanic urbanism. William II took the decision to destroy the walls and build "a new city", south of the historic heart on a free zone of any construction. The station, inaugurated in 1908, modern and monumental, looks like a Romanesque church with its massive belfry. The water tower, next to the station, looks like a dungeon and once powered the steam engines. The post office, in pink sandstone and neo-Romanesque style, looks like an imposing castle reminiscent of the monuments of Western Prussia. Avenue Foch is built on the embankment of the old medieval ramparts of which remains only remnant, the Camoufle tower. This prestigious avenue is surrounded by public buildings, private and immovable villas, offering an extraordinary encyclopaedia of historical and new styles.

Option 5: Cathedral, Place de la comédie, Metz (30 people)

02:30 pm to 04:30 pm: Guided tour in English of the Cathedral and Place de la Comédie

St Stephen's Cathedral, a pure gem of Gothic art, was built between 1220 and 1520, and resulted from the meeting under one roof of two churches. We owe the architect messin, Pierre Perrat, this fusion and the slender vault rising to 42 m in height. The imposing silhouette of St Etienne Cathedral dominates the city and offers magnificent stained glass windows, executed between the 13th and 20th centuries by renowned artists (including Chagall). With its $6500m^2$ of stained glass, it is aptly named "Lantern of the God". Continuation to the Place de Chambre, the Place de la Comédie (where the Opera-Theater is located, the oldest theater in France still in activity, and the Neuf Temple, built during the German annexation, neo-Romanesque and in gray sandstone).

Registration

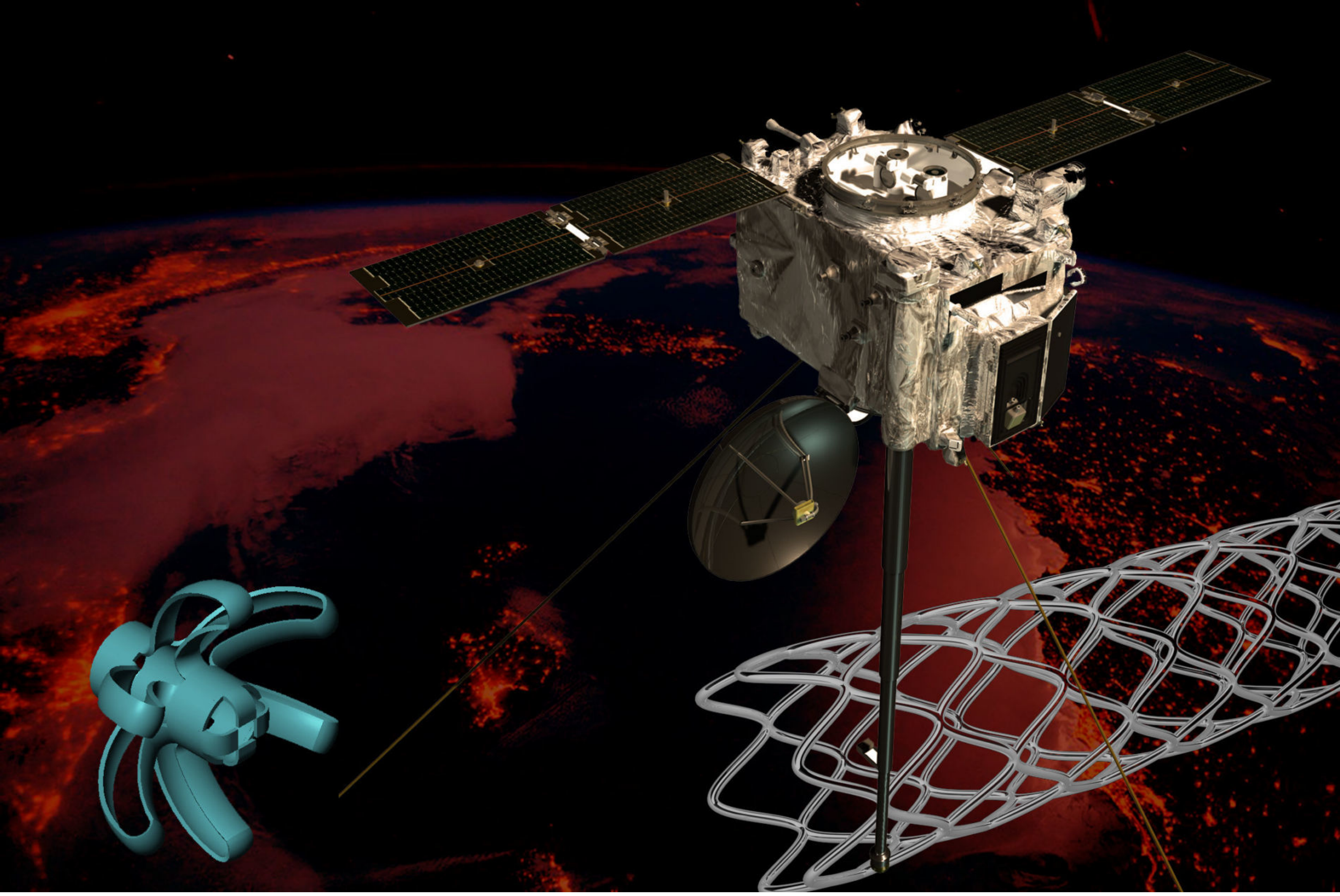
The registration for conference excursion is required and available on the conference website. Registrations are accounted until the maximum number of participants for the option is reached.

Accompanying persons program

A bus will leave in front of the conference locaton, at "UFR Droit, Economie, Administration" of Lorraine University

Monday 27^{th} afternoon
03:00 pm to 05:15 pm Little train tour and guided tour of Metz Cathedral and Place de la Comédie
Tuesday 28^{th} morning
09:30 am to 12:00 pm Metz-Sillegny-Gorze
Tuesday 28^{th} afternoon
02:00 pm to 04:00 pm Guided tour of the Quartier Impérial and visit of a glass craftsman shop
Wednesday 29^{th} morning
09:30 am to 12:00 pm Guided visit of Robert Schuman house
Wednesday 29^{th} afternoon
02:30 pm to 05:00 pm Visit of Gravelotte Museum
Thursday 30^{th} morning
10:00 am to 11:30 am Boat ride at "Plan d'eau"
Thursday 30^{th} afternoon
The accompanying persons will join the conference excursions (5 options)
Friday 31^{st} morning
Shopping in Metz

Author Index



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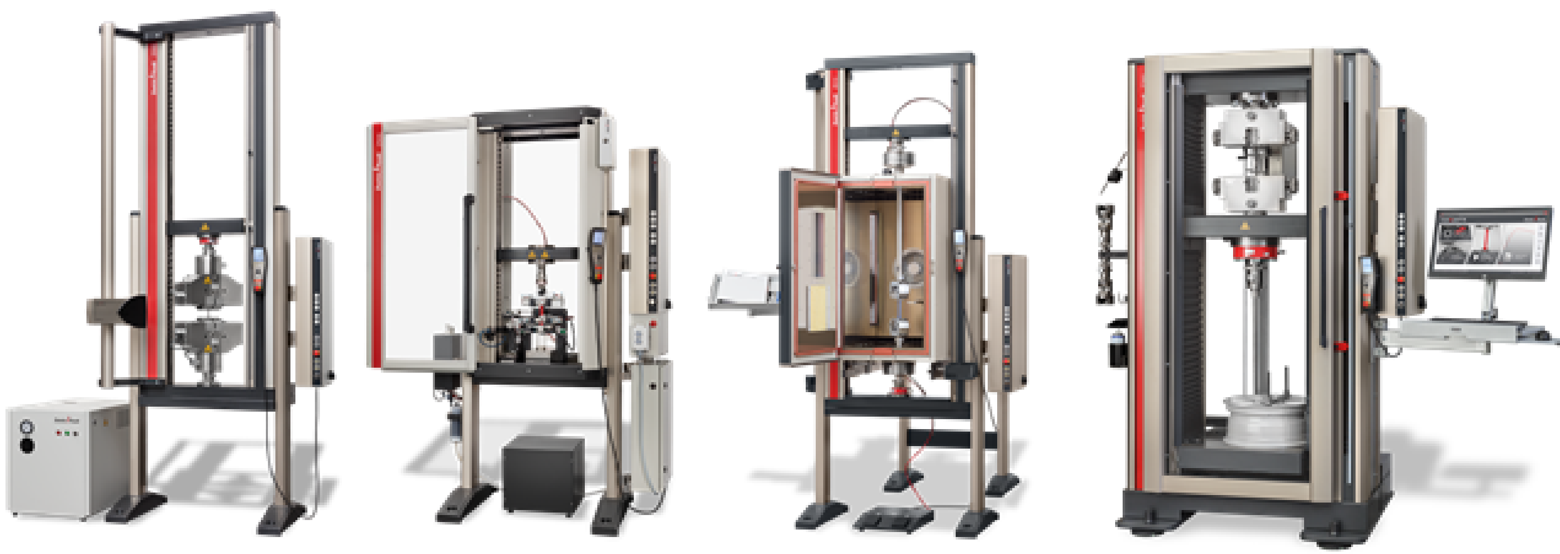

Zwick / Roell