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### Features of the surface relief of TiNi alloy in coarse-grained and ultrafine-grained states at room and elevated temperatures

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Abstract. The effect of coarse-grained (CG) and ultrafine-grained (UFG) structures processed by equal channel angular pressing on the formation and features of the surface relief during mechanical tensile tests at room and elevated temperatures was studied. The formation of the UFG structure leads to a significant increase in strength, but the ductility decreases. Coarsegrained samples in the quenched state after tensile tests have a developed relief and microcracks. After tensile testing of the annealed CG sample, a similar situation is observed. On samples in the UFG state, no developed relief and cracks were found. The surface of the UFG sample in the annealed state at a temperature of 500 °C shows a very weak relief, but no cracks are observed. Possible reasons of these effects are discussed.

#### **1. Introduction**

Alloys of titanium nickelide (TiNi) belong to the class of functional materials with shape memory effects (SME) and superelasticity (SE), caused by thermoelastic martensitic transformations. These alloys are widely used as structural and functional materials in engineering and medicine [1]. A promising trend in improving the service properties of metallic materials, including TiNi alloys, is the formation of an ultrafine-grained (UFG) state in them by methods of severe plastic deformation (SPD) [2-3]. As a result of equal channel angular pressing (ECAP), an ultrafine-grained (UFG) structure with a grain size of about 400 nm is formed in TiNi alloys [2], which leads to a significant increase in the mechanical and functional properties of shape memory alloys. However, the mechanisms of mechanical behavior and deformation of the UFG TiNi are not sufficiently studied, despite years of research by various scientific groups. The aim of this work was to study the influence of the method of ECAP and heat treatment on the mechanical properties, structure and morphology of the alloy surface after tensile testing.

#### 2. Material and experimental methods

The material of the study was a two-component Ti<sub>50.2</sub>Ni<sub>49.8</sub> alloy. The Ti<sub>50.2</sub>Ni<sub>49.8</sub> alloy is enriched in Ti

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with respect to stoichiometry at room temperature possesses the B19' crystal lattice of the monoclinic martensite and the Ti<sub>2</sub>Ni with the FCC lattice [1]. The temperatures of the martensitic transformations B2-B19' are  $M_s = 65 \text{ °C}$ ,  $M_f = 38 \text{ °C}$ ,  $A_s = 79 \text{ °C}$ , and  $A_f = 107 \text{ °C}$ .

The TiNi alloy was homogenized at a temperature of 800 °C for one hour, and then quenched in water to form a supersaturated solid solution of Ti in Ni. The state after quenching was taken as the initial coarse-grained. Thermal treatment of the TiNi alloy was carried out at 500 °C for one hour, followed by cooling in air. The ultrafine-grained structure was formed by the method of equal-channel angular pressing (ECAP) in the regime: n = 6 passes, T = 450 °C on rods with a diameter of 10 mm. The microstructure and surface relief of the alloys were studied using an OLYMPUS GX5 optical microscope and a JEOL JSM 6395 scanning electron microscope. Mechanical tensile tests of small samples were carried out at room temperature at a rate of 0.04 mm/min in a machine for mechanical tensile testing of small specimens developed in IPAM USATU.

#### 3. Results

According to the results of optical microscopy, the initial alloy in a coarse-grained state is characterized by a structure with a grain size of the original austenite of about 170  $\mu$ m with martensite twins inside the grains (figure 1a). The Ti<sub>50.2</sub>Ni<sub>49.8</sub> alloy after ECAP is also in the martensite state. The presence of martensitic boundaries makes it difficult to estimate the austenite grain size after ECAP, but the estimated austenite grain size is about 0.7  $\mu$ m (figure 1b).



Figure 1. (a) Microstructure of the quenched  $Ti_{50.2}Ni_{49.8}$  alloy, optical microscopy; (b) Microstructure of the  $Ti_{50.2}Ni_{49.8}$  alloy after ECAP, TEM.

Tensile tests showed that the " $\sigma$  -  $\varepsilon$ " curves have the form characteristic of the deformation of Ti-Ni alloys [3]. During the tensile test at room temperature, when the critical stress ( $\sigma_m$ ) is reached, an area (5-10% of deformation) appears where the material is deformed due to the deformation-induced martensitic transformation. After this, an elastic deformation of martensite occurs, and then a plastic deformation of martensite takes place. The alloy in a quenched state has the following characteristics: the stress of the beginning of martensitic transformation ( $\sigma_m$ ) is 220 MPa, the yield stress (YS) is 580 MPa, the tensile strength (UTS) is 850 MPa, and the elongation is 21%. Mechanical tests at room temperature led to the destruction of samples without localizing the deformation and the formation of a neck. ECAP leads to a significant increase in strength and yield stress (up to 1070 MPa and 930 MPa, respectively), and the elongation decreases to 18%. The ductility of the CG alloy after annealing at 500 °C increases noticeably, possibly due to the annihilation of the quenching stresses. Annealing at 500 °C of the UFG specimens leads to a decrease in the yield stress to 870 MPa, while the ductility varies insignificantly (figure 2).

One of the few effective methods for the direct study of the features of deformation-induced martensitic transformations is the metallographic observation of the surface relief of a sample during tensile testis. The surface was studied in four structural states: a coarse-grained state with a grain size of 170  $\mu$ m, a coarse-grained state after annealing at 500 °C (d=170±15  $\mu$ m), an ultrafine-grained state

with a grain size of 700 nm, and a UFG state after annealing at a temperature of 500 °C (d=0.8-1.0  $\mu m).$ 



**Figure 2.** The «stress-strain» curves of the  $Ti_{50,2}Ni_{49,8}$  alloy in various states: CG; CG and annealing at 500 °C; UFG; UFG and annealing at 500 °C.

Figure 3a shows a typical image of the surface relief of a quenched alloy. On the surface of the sample there is a developed relief. In addition, there are numerous cracks on the working gage. Cracks are also observed in SEM images. After tensile testing of the annealed sample, a similar pattern is observed (figure 3c). Deformation bands are also detected by scanning electron microscopy (figure 3d), which indicates that the relief has become even more developed.



**Figure 3.** The structure of the surface relief of the  $Ti_{50.2}Ni_{49.8}$  alloy in the CG state (a, b) and the annealed state at T = 500 °C (c, g): OM (a, c), SEM (b, d). The arrows indicate the direction of stretching.

The study of the deformation relief on the surface of the samples after the tensile tests of the UFG alloy by the OM method did not reveal a developed relief (figure 4), no cracks were observed. Perhaps this is due to the grain refinement after ECAP. In SEM, the relief is also not observed (figure 4b). The surface of the UFG specimen, annealed at a temperature of 500 °C, has a weak relief. Perhaps this is due to the coarsening of grains and an increase in ductility after annealing.



**Figure 4.** Structure of the surface relief of the  $Ti_{50.2}Ni_{49.8}$  alloy in the UFG state (a, b) and annealed state at T = 500 °C (c, g): OM (a, c), SEM (b, d). The arrows indicate the direction of stretching.

In the initial state, the alloy has a high ductility and under tension undergoes a large deformation, but it breaks down without the forming of a neck. Hence, we observe expressed deformation bands and considerable work hardening of the material under tension in the martensitic structure, which leads to the formation of a developed relief and cracks during tensile testing of the sample in the initial state. ECAP leads to a decrease in ductility, and the material is not so significantly deformed and is work hardened when elongated to failure. Martensitic deformation is localized in individual small grains. Therefore, the developed relief and cracks are not formed.

#### 4. Conclusion

The results of mechanical tests showed that annealing of the quenched state leads to an increase in ductility and strength. ECAP leads to an increase in the yield stress by about 40%. Annealing of ECAP states leads to a slight decrease in strength and yield stress. Samples after tension in the quenched state have a developed relief and microcracks. ECAP samples after tensile tests do not demonstrate such relief.

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