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About formability of ultra-fine grained metallic materials

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Abstract. Ultra-fine grained (UFG) and nanostructured metallic materials obtained via severe plastic deformation typically show very high mechanical strength but low tensile ductility, which dramatically limits their practical utility. Significant efforts were made to improve uniaxial tensile ductility of ultra-fine grained and nanostructured metallic materials. The developed strategies can be divided into two main groups. (1) The ‘mechanical’ strategies employ the mechanical characteristics of these materials, such as their work hardening ability and/or strain rate sensitivity. These mechanical characteristics can be varied via changing testing parameters, such as temperature and/or strain rate. (2) The ‘microstructural’ strategies are based on idea of intelligent microstructural design to suppress necking at early stages of plastic deformation thus improving ductility. However, not much attention was paid to the fact, that in metallforming operations, metallic materials are not deformed uniaxially, but have to undergo deformation under complex strain paths. This work aims to demonstrate that despite UFG metallic materials have low tensile ductility, they can show enhanced formability during plastic deformation in complex stress state (such as formability under biaxial stretch, which is sufficient for metalforming operations.

Introduction

Research on ultra-fine grained (UFG) and nanostructured metallic materials has been a main stream topic for over two last decades [1]. It has been well established that grain refinement down to ultra-fine or nanoscale can dramatically increase mechanical strength of the metallic materials [2, 3] according to the well-known Hall-Petch law [4, 5]. However, this effect is accompanied by significant drop of uniaxial tensile ductility, which is often reduced nearly by an order of magnitude [2, 3]. This drawback could be insurmountable hurdle in bringing the UFG materials from laboratory to commercialization. Therefore, significant research activities focused on improvement of tensile ductility of UFG metallic materials. Various strategies have been developed to increase their uniaxial tensile ductility and they have been considered in details in several comprehensive review articles [6-8]. All these strategies can be divided into two main groups:

1) The ‘mechanical’ strategies employ the mechanical characteristics of these materials, such as their work hardening ability and/or strain rate sensitivity. These mechanical characteristics can be varied via changing testing parameters, such as temperature and/or strain rate. For example, it was demonstrated that plastic deformation of UFG materials at cryogenic temperatures suppresses the recovery processes, thus promoting dislocation accumulation in the interior of ultra-fine grains, which in turn improves strain hardening and uniform elongation [9]. Similar effect was also observed during tensile testing of UFG materials at high strain rates [9].

2) The 'microstructural' strategies are based on idea of intelligent microstructural design to suppress necking at the early stages of plastic deformation thus improving tensile ductility. Formation of bimodal microstructures [10], introduction of nanoprecipitates [11] or nanotwins [12] into ultra-fine grains, grain boundary engineering [13], manipulation with stacking fault energy [14] and many other approaches have been successfully applied to increase tensile ductility of UFG metallic materials. All these strategies result in activation of deformation mechanisms that lead to increase in ductility and/or suppression of deformation mechanisms degrading ductility. Some of these strategies even allowed to reach superior tensile ductility at room temperature. For example, in [15], an UFG Al-30%Zn alloy showed tensile ductility of ~160 % at room temperature. This was attributed to the development of very thin layers of a Zn-rich grain boundary phase, which leads readily to the occurrence of enhanced grain boundary sliding at room temperature.

However, not much attention was paid to the fact, that in metalforming operations, metallic materials are not deformed uniaxially, but have to undergo deformation under complex strain paths. As is well known, plastic deformation of the UFG materials under complex strain path might lead to activation of other deformation mechanisms, such as grain boundary sliding, which are usually not active during uniaxial tensile deformation of these materials [16]. Activation of these mechanisms during plastic deformation in the multiaxial mode may also lead to improved formability of the UFG and nanostructured metallic materials. The main objective of the present work is to demonstrate that UFG metallic materials can have enhanced formability in biaxial stretching even if their uniaxial tensile ductility is low.

Materials and Processing

Commercially pure (CP) copper (99.9% purity) and an Al 6063 alloy with chemical composition Al–0.60Mg–0.45Si (wt. %) were chosen as materials for this investigation.

The as-received CP Cu was subjected to annealing at 600 ^{o}C for 2 h. Homogeneous coarse-grained (CG) microstructure with an average grain size of 50 μm was observed in the annealed material. Billets having a diameter of 18 mm and a length of 100 mm were machined. The billets were subjected to equal channel angular pressing (ECAP) at room temperature in a die with the internal channel angle φ = 90 ^{o} and the outer angle ψ = 0 ^{o} using route Bc (bar is rotated by 90 deg around the pressing direction after each ECAP pass) for 2 and 12 ECAP passes [17]. The strain produced in each pass was about 1, so the cumulative strain the specimens underwent was about 2 and 12. Hereafter, these material conditions will be referred to as 2P Cu and 12P Cu.

Billets with the same dimensions were machined from the as-received Al 6063 alloy. They were subjected to solution treatment at 535^{o}C for 2 h followed by water quenching. The as-quenched billets were subjected to equal channel angular pressing with parallel channels (ECAP-PC) at 100 ^{o}C for 4 passes, so the accumulative total strain induced into billets was 6.4. The internal angle of the ECAP-PC die was 100^{o}. Half of the billet after ECAP-PC processing was additionally artificially aged at 130 ^{o}C for 24 h.

Experimental Procedures

To study the microstructure, transmission electron microscopy (TEM) studies were carried out using a JEOL-2100 microscope (JEOL, Tokyo, Japan) operating at 200 kV. The CP Cu samples for TEM study were prepared by twin jet electropolishing with electrolyte (25 % of orthophosphoric acid, 25 % of ethanol, and 50 % of distilled water) at room temperature at a voltage of 9-10 V. Samples of the Al 6063 alloy were prepared by twin jet electropolishing using 1:4 solution of nitric acid in methanol. Electropolishing was performed at -25 ^{o}C at voltage of 12 V. Observations were made in both bright and dark field imaging modes. Selected area electron diffraction (SAED) patterns were recorded from areas of interest using an aperture of 1 μm nominal diameter.

Tensile specimens with a gage length of 7.5 mm, a gage width of 1.5 mm and a thickness of ~1 mm were machined from the as-received materials and processed billets. The tensile axis of specimens was perpendicular to the bar axis. Tensile tests were carried out at room temperature using an INSTRON 3384 universal testing machine (Instron, MA) with a 2 kN load cell. Tensile specimens were deformed to failure with constant cross-head speed corresponding to the initial strain rate of 10^{-3} s^{-1}.

Specimens for small punch testing were cut in transverse section of the processed billets. Both sides of the obtained discs were ground and polished to mirror-like surface using colloidal silica at the final stage. The final thickness of specimens was 0.40 mm. The specimens were clamped between upper and bottom dies. They were deformed at room temperature by a well lubricated hemi-spherical rigid punch having a diameter of 2.4 mm. The punch speed was 0.5 mm/min. Data on load F and central deflection h were recorded during small punch testing. Small punch tests were stopped at the moment of onset of plastic instability on the F-h curve. To estimate the equivalent strain induced in the small punch specimens, final thickness was measured over areas deformed under membrane (bi-axial) stretching located at 20-45º with respect to the vertical axis of dome as illustrated in Fig. 1. The true strain was estimated as

$$\boldsymbol{\varepsilon} = \mathbf{ln}\left(\frac{t_0}{t}\right), \tag{1}$$

where t_o is the initial thickness and t the final thickness of the punch specimen.

Results and discussions

Microstructure of the ECAP processed Cu is presented in Fig. 1. Inhomogeneous microstructure consisting of coarse grains with equiaxed subgrains having a size of 250-650 nm, dislocation walls and small areas of ultra-fine grains having a size of 150-300 nm is developed after 2 ECAP passes (Fig. 1a). Shear bands with average width of 200 nm and well defined boundaries are also present in the microstructure. ECAP processing for 12 passes leads to formation of a homogeneous UFG microstructure with equiaxed grains having a size in the range of 100-250 nm and well defined grain boundaries is formed after 12 ECAP passes (Fig. 1b). Most of grains are free of dislocations due to dynamic recovery by dislocation rearrangement during processing to large strains [9, 10]. It should be noted that very similar microstructures were observed in ECAP processed CP Cu in the earlier works [18, 19].

Microstructure of the Al 6063 alloy after ECAP-PC processing is illustrated in Fig. 2. Homogeneous UFG microstructure with elongated grains having a length of 515 μm and aspect ratio of ~1.5 (Fig. 2a) is observed. Nanosized spherical second phase precipitates having size of ~10 nm are observed (marked by arrows on Fig. 2b). According to the earlier study, these nanosized precipitates were identified as β'-Mg_2Si precipitates [20]. Artificial aging leads to formation of nanoscale needle-type precipitates β''-Mg_2Si precipitates in the grain interior (Fig. 2c).

Mechanical properties of the studied materials are listed in Table 1. One can see that SPD processing of both metallic materials results in significant increase of mechanical strength, whereas their tensile ductility dramatically decreases. An artificial aging of the Al 6063 alloy results in a further increase of its mechanical strength and a slight increase of its ductility.

Figure 3a illustrates the appearance of some specimens after small punch testing. It is seen that specimens are deformed into a dome shaped cap, and the CG Cu and Cu after ECAP processing for 2 passes have very similar appearance and similar dome height. Analysis of load - central deflection curves from small punch testing (Fig. 3b) allows to determine the maximum load reached during small testing as well as the maximum central deflection before sample cracking. These data along with the values of equivalent strain $\boldsymbol{\varepsilon_{eq}}$ measured in the areas of biaxial stretching in the small punch specimens are listed in Table 2. It is seen that the $\boldsymbol{\varepsilon_{eq}}$-values are similar for the CG Cu and Cu after 2 ECAP passes. Analysis of deformation mechanisms during biaxial stretching of Cu after 2 ECAP passes showed significant activity of microshear banding, that was not been observed in Cu after 12

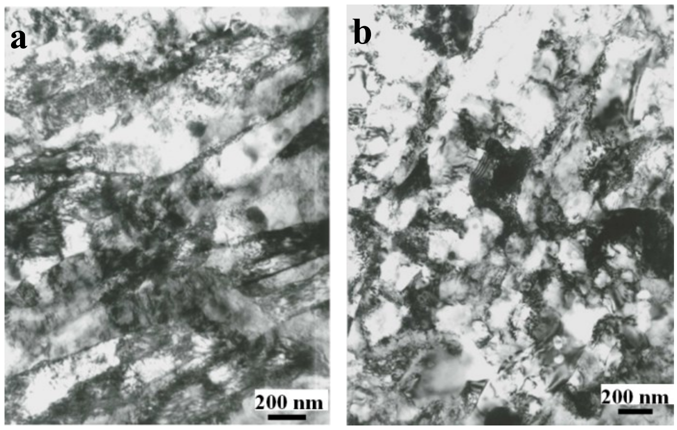


Fig. 1. Microstructure of (a) CP Cu after 2 ECAP passes, (b) CP Cu after 12 ECAP passes.

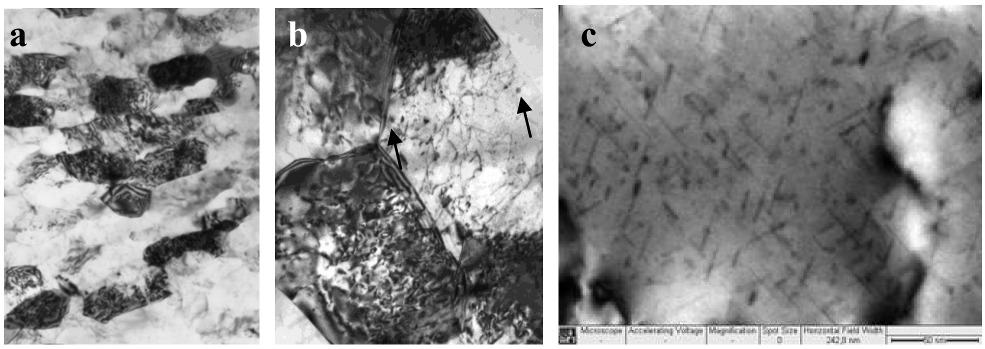


Fig. 2. Microstructure of Al 6063 alloy (a, b) after 4 ECAP-PC passes, (c) after 4 ECAP-PC passes followed by artificial aging at 130 ^{o}C for 24 h.

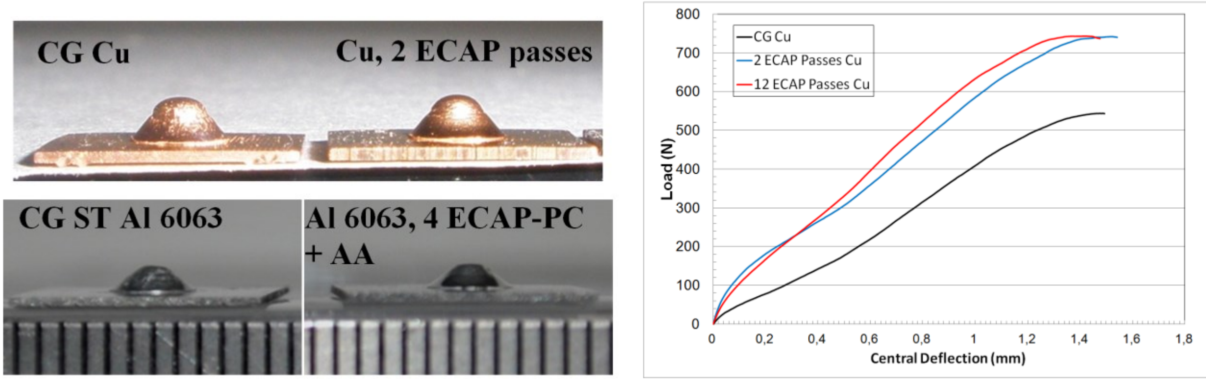
Table 1. Mechanical properties of studied materials before and after SPD processing.

Material	Condition	$\sigma_{0.2}$ [MPa]	σ_{UTS} [MPa]	ε_u [%]	ε_f [%]
Cu	CG	61	206	44	47
	ECAP, 2 passes	363	400	1	10
	ECAP, 12 passes	359	410	3	12
Al 6063 alloy	CG (T4)	90	165	14	22
	ECAP-PC, 4 passes	260	275	2	10
	ECAP-PC, 4 passes + AA	285	300	2.5	14

ECAP passes [21]. Such inhomogeneous plastic flow can play an important role in biaxial stretching of the ECAP processed Cu providing significant amount of plastic deformation and, therefore, formability. A significant increase of the $\boldsymbol{\varepsilon_{eq}}$-value after aging treatment of the ECAP-PC processed Al 6063 alloy is observed (Table 2). It becomes similar to the $\boldsymbol{\varepsilon_{eq}}$-value of the CG alloy, indicating their similar formability in biaxial stretching, whereas in uniaxial tensile mode the CG alloy shows much higher ductility (Table 1). This is related to interaction of gliding dislocations with nanoprecipitates resulting in increased strain hardening and, therefore, in enhanced formability [11]. It is suggested that application of standard strategies to improve uniaxial tensile ductility can significantly improve formability in biaxial stretching of UFG metallic materials.

Summary

CP Cu and Al 6063 alloy were subjected to ECAP and ECAP-PC processing for varying number of passes. The SPD processing of these materials led to significant increase of their mechanical strength and degradation of their uniaxial tensile ductility. Small punch testing of specimens showed



a) b)

Fig. 3. a) Specimens after small punch testing. b) typical load - central deflection curves for Cu before and after ECAP processing.

Table 2. Results of small punch testing of studied materials before and after SPD processing.

Material	Sample	F_{max} (N)	h_{max} (mm)	ε
Cu	CG	540	1.48	0.55
	ECAP, 2 passes	740	1.52	0.66
	ECAP, 12 passes	740	1.40	0.48
Al 6063 alloy	CG, ST	370	1.25	0.47
	ECAP-PC, 4 passes	420	0.93	0.23
	ECAP-PC, 4 passes, aging at 130 ^{o}C for 24 h	500	1.18	0.40

that high strength ECAP processed Cu can show similar formability in biaxial stretching as its low strength coarse-grained counterpart. Low formability in biaxial stretching of the ECAP-PC processed Al 6063 alloy can be enhanced through introduction of nano-precipitates into grain interior via artificial aging. It is suggested that strategies developed for improvement of uniaxial tensile ductility of the UFG metallic materials can be effectively used to improve their formability in biaxial stretching.

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