Volume 62 • Issue 2 • February 2014

Journal of Achievements in Materials and Manufacturing Engineering

International Scientific Journal published monthly by the World Academy of Materials and Manufacturing Engineering

In-situ testing and heterogeneity of UFG Cu at elevated temperatures

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Received 30.11.2013; published in revised form 01.02.2014

ABSTRACT

Purpose: The motivation of present investigation is the study of deformation-induced processes during in-situ tensile and compression test at elevated temperature in order to elucidate the role of the microstructure changes during creep testing.

Design/methodology/approach: Experiments were conducted to investigate deformation-induced processes during in-situ tensile test at elevated temperature.

Findings: It was found that creep resistance of UFG pure Al and Cu is considerably improved after one ECAP pass in comparison with coarse grained material, however, further repetitive pressing leads to a noticeable deterioration in creep properties of ECAP material.

Researches limitations/implications: In the present work was found that ultrafine-grained microstructure is instable and significant grain growth has already occurred during heating to the testing temperature.

Originality/value: The experiments conducted on pure Al and Cu found that their creep resistance is considerably improved after one ECAP pass in comparison with unpressed material.

Keywords: In-situ testing; Heterogeneity; Creep behavior; EBSD

Reference to this paper should be given in the following way:

M. Petrenec, P. Král, J. Dvořák, M. Svoboda, V. Sklenička , In-situ testing and heterogeneity of UFG Cu at elevated temperatures, Journal of Achievements in Materials and Manufacturing Engineering 62/2 (2014) 69-74.

PROPERTIES

1. Introduction

The creep behaviour of materials processed by severe plastic deformation [1,2] is influenced by amount of imposed deformation and temperature stability of microstructure. The experiments conducted on pure Al and Cu found that their creep resistance is considerably improved after one ECAP pass in comparison with unpressed material. However, successive pressing leads to a noticeable decrease in the creep properties of the ECAP material [3-8]. Based on determined values of the stress exponents it can be suggested that the operating creep mechanisms for the unpressed and SPD pure metals are the same. However, grain boundary sliding is probably more important flow process in creep of the pressed samples [3,5,7]. Recently, Kawasaki et al. [9,10] examined pure Al processed by

ECAP. Based on the texture measurement they demonstrated that creep occurs through an intragranular dislocation process with no significant contribution of diffusion creep.

Despite of a considerable interest in ECAP processing method, there are not many works documenting microstructure evolution and changes during creep testing and determining creep mechanisms of ultrafine-grained materials processed by ECAP. The coarsening of the grains in microstructure of ECAP copper during creep at elevated temperature [11] was recently observed. It was suggested that creep behaviour is controlled by storage and dynamic recovery of dislocations at high-angle boundaries [11,12].

The creep fracture can be associated with nucleation of cavities on grain boundaries, particularly on boundaries transverse to tensile stress or on particles of second-phase. The cavities can be also nucleated at ledges which are formed as a result of slip along planes intersecting the grain boundaries. The nucleation of cavities is followed by their growth and interlinkage, leading to the final failure [13]. Creep fracture can occur by formation of wedge cracking at triple point when grain boundary sliding (GBS) is not accommodated. The GBS can induce local stress concentration at triple points and particles on the grain boundaries. Cavitation was observed in bicrystals where the boundary was perpendicular to the applied stress, therefore there is no resolved shear and an absence of sliding [14]. The damage near grain boundaries is one of the key parameters because many cracks are initiated from grain boundaries which are major degradation phenomena in materials subjected to the creep exposure. In particular grain boundaries influence creep behaviour of ultrafine-grained materials because the increasing contribution of grain boundary sliding to the total creep deformation can be expected. The investigation of the fracture and strain deformation can contribute to the better understanding of the creep behaviour in materials processed by ECAP.

The motivation of present investigation is the study of deformation-induced processes during in-situ tensile and compression test at elevated temperature in order to elucidate the role of the microstructure changes during creep testing.

2. Experimental material and procedures

The experimental material used in this investigation was a coarse-grained copper. The initial material was cut into the billets with cross-sections of 10x10 mm^2. The ECAP pressing was conducted at room temperature using a die that had a 90° angle between the channels. The pressing speed was 10 mm/min. To obtain an ultrafine-grained (UFG) material, the billets were subsequently pressed by route B_c [1] up to 8 ECAP passes to give the mean grain size ~0.7 µm. The constant strain-rate test in tension and compression were performed at 473 K using testing module Microtest 2000EW (Fig. 1) developed by GATAN which is configured for in-situ electron back scatter diffraction (EBSD) observations. Microstructure was examined by TESCAN FEG-SEM MIRA3 XM equipped by EBSD detector Nordlys NordlysMax from OXFORD INSTRUMENT. The tests were interrupted by fast stress reductions after different deformation steps and observation of microstructure changes was performed.

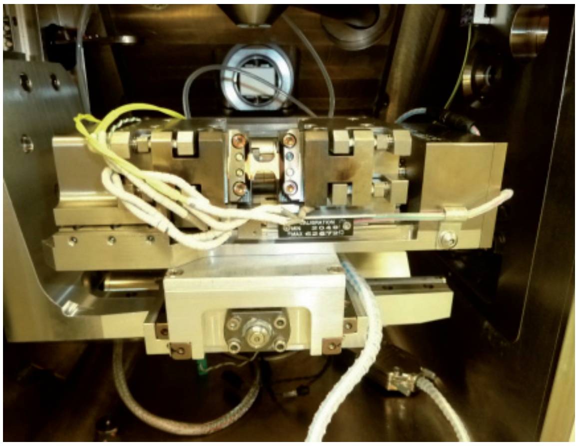

Fig. 1. Testing module GATAN Microtest 2000EW with specimen both inserted in the TESCAN SEM-FEG

The 3D EBSD analyses were performed in the XZ section [13]. The milling was conducted at 30 kV in the SEM-FIB LYRA 3 microscope with the beam current about 800 pA. At first, the surrounding of the micro block on the edges of specimens was milled by FIB procedure. The FIB procedure ensured sufficient space around the block which is necessary for good-quality of EBSD patterns from the depth of the investigated volume of block. Before FIB milling the surface of block was covered by platinum protective layer to minimize the surface damage by Ga ions. The step size and depth for 3D EBSD maps was 150 nm for UFG Cu after creep testing at 373 K.

3. Experimental results

In the Fig. 2 the curve of interrupted tensile test of UFG copper deformed at 473 K is shown. At the beginning of the tensile test the interruptions were performed after deformation step ~0.006 in order to find significant microstructure changes occurring up to and around yield point.

After the yield point was reached the deformation step was increased up to ~0.025. Investigation of tensile interrupted curve found that yield point of pure copper tested at 473 K was about 110 MPa and ultimate tensile strength was ~185 MPa. The fracture elongation was approximately 0.35.

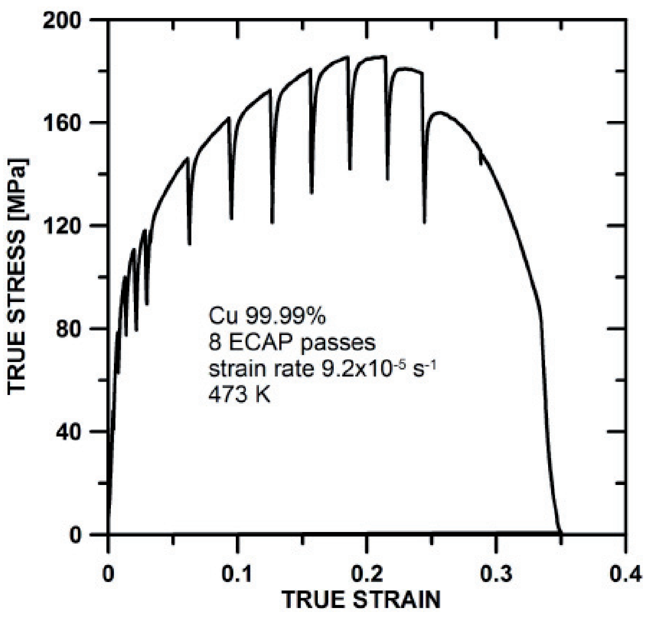

Fig. 2. The dependence of true stress vs. true strain for interrupted test with interruptions

The microstructure changes occurs and individual deformation steps were monitored by EBSD analysis after unloading in order to describe the evolution of the grain structure in the interruption periods.

Microstructure of UFG copper contained more or less equiaxed grains with mean size ~700 nm (Fig. 3a). We measured about 60% of high angle-grain boundaries (HAGBs) in the UFG microstructure which had random character (Fig. 3d). Heating to the testing temperature (~20 min.) led to the grain growth. The microstructure after heating consists of large grains with mean size ~8 µm. However, groups of fine grains with mean size ~1 µm can be found in the microstructure. It is generally accepted that the co-existence of larger recrystallized grains in the bimodal structure can improve deformation behaviour of UFG material by relaxation of the stress concentration through plastic deformation inside of larger grains [15]. The high number of HAGBs in the microstructure in the connection with relatively small grain size probably lead to the higher activity of additional creep mechanisms like GBS, cavitation and more intensive diffusion processes.

It was observed that frequency of HAGBs increases significantly in the microstructure after heating to the testing temperature and microstructure contained ~97% of HAGBs predominantly special boundaries such as ∑9 (<110>/38.94°) and twins with ∑3 (<111>/60°).

The observation of high frequency of twins is fully consistent with the investigation of the role of shear stress in formation of annealing twin boundaries in copper [16]. It was found that the twin content in rolled copper (92% rolled) is significantly lower than that in any copper deformed by ECAP, regardless of annealing temperature. Molodova et al. [17] found a very low thermal stability of pure copper processed by ECAP. They observed that in the microstructure of pure copper processed by 12 ECAP passes can be already found large recrystallized grains even after annealing at 393 K for 10 min. and 423 K for 2 min.

Creep deformation led to the increase of ∑3 boundaries and in the microstructure was found about 57% of twin boundaries. The occurrence of high frequency of twin boundaries in the microstructure can significantly influence creep behaviour of UFG copper. It was found [18] that a high fraction of strong low-∑boundaries is a key factor controlling intergranular brittleness. The control of intergranular fracture and intergranular brittleness can be achieved by reduction of random boundaries or conversely by increasing the fraction of LAGBs or special low ∑ coincidence boundaries resistant to fracture. It is generally accepted that the damage near grain boundaries is one of the key factor controlling creep life because many cracks are initiated at grain boundaries and frequently are major degradation phenomena in materials subjected to the creep exposure. Furthermore, grain boundaries can influence creep behaviour of ultrafine-grained materials due to synergetic effect of additional operating creep mechanisms like grain boundary sliding (GBS), intergranular cavitation or more intensive grain boundary diffusion [19]. Nevertheless the ability of GBS takes place more significantly at a random grain boundary compared with low-∑ boundaries [20,21].

Fig. 3c shows microstructure after tensile deformation of 0.03. Microstructure changes caused by plastic deformation are defined by circles. It was found that plastic deformation led mainly to the dynamic coarsening (black circles). However the formation of new grains (dynamic recrystallization) was also observed (white circles). Recently [22] was found that the dynamic coarsening in UFG Ti64 alloy resulted in flow hardening at different temperatures and strain rates for a short preheat time (15 min.) but was noticeably reduced when a longer preheat time (1 h) was used prior to testing at 10^{-3} s^{-1}. The latter behaviour was largely attributed to noticeable static coarsening during preheating. Ivanov et al. [23] showed that high values of strength, weak strain hardening, pronounced localization of deformation and limited elongation up to failure are characteristic features of deformation behaviour of ultrafine-grained copper processed by equal-channel angular pressing in the temperature interval of 293-523 K at strain rates of $1.3x10^{-2}$-$3.0x10^{-5}$ s^{-1}. At high temperatures and low strain rates these features are disappeared due to dynamic recrystallization.

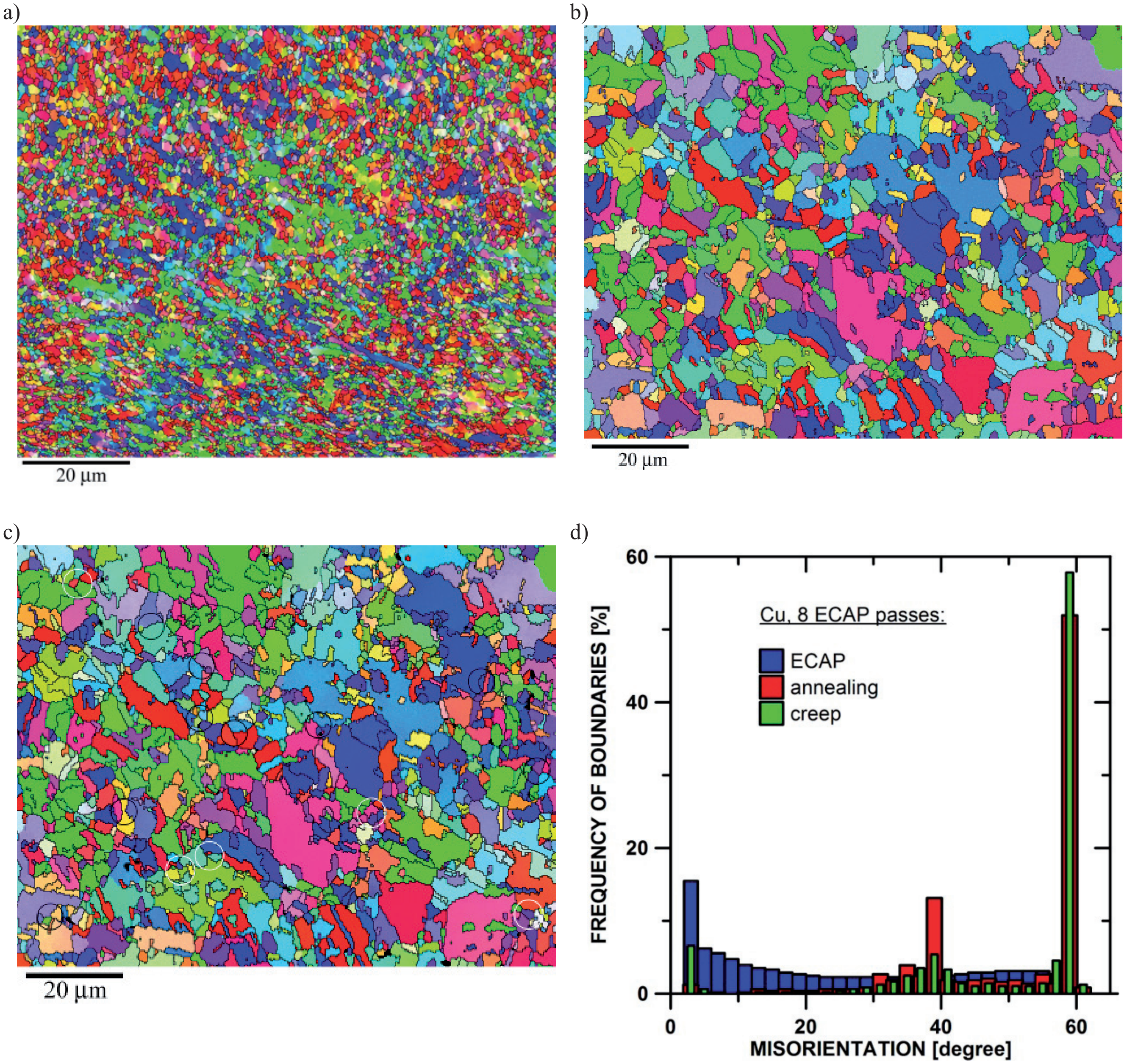

Fig. 3. Microstructure of UFG copper after: a) 8 ECAP passes, b) heating to the testing temperature, c) tensile creep deformation 0.03 at 473 K, d) misorientation distributions

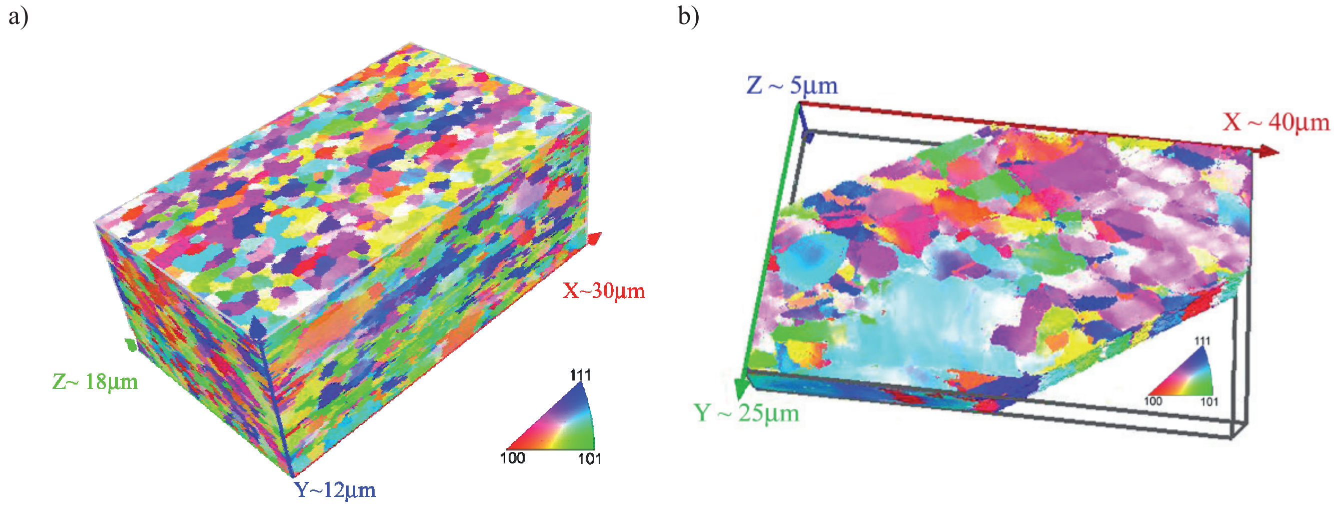

Fig. 4. The 3D microstructure of UFG Cu after creep at 373 K and a) 260 MPa and b) 130 MPa

3D EBSD orientation map revealed that grain size and heterogeneity of microstructure is significantly influenced by value of applied stress. The dynamic coarsening of the grains in microstructure of UFG copper occurred especially after creep at 373 K and higher stress (Fig. 4a) was observed. It was measured about 5% of twin boundaries in the microstructure. It can be suggested on the basis of simple dislocation model [12] that creep behaviour is controlled by storage and dynamic recovery of dislocations at high-angle boundaries. However, large grains were observed in the microstructure of UFG copper after creep test at 373 K under low stress (Fig. 4b). Observation of large grains can be the result low thermal stability of UFG microstructure during longer creep exposure. However, some kind of dynamic recrystallization cannot also be excluded because in the low stress was measured more than 15% of twin boundaries.

4. Conclusions

ECAP method led to reduction of grain size in pure Cu down to 0.7 μm. The short annealing of UFG Cu alloy at 473 K caused a noticeable grain growth. Microstructure observations showed deformation-induced grain growth and the formation of new grain during creep exposure. Thermal stability of UFG microstructure is influenced not only by temperature but also significantly by applied stress.

Acknowledgements

Financial support for this work was provided by the Czech Science Foundation under Grant P108/11/2260.

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