CREEP BEHAVIOUR AND MICROSTRUCTURE OF ULTRAFINE GRAINED IRON PROCESSED BY ECAP

Petr KRÁL a, Jiří DVOŘÁK a, Marie KVAPILOVÁ a, Milan SVOBODA a, Václav SKLENIČKA a

a INSTITUTE OF PHYSICS OF MATERIALS, ASCR, Žižkova 22, 616 62 Brno, Czech Republic
pkral@ipm.cz

Abstract

Pure iron (99.99%) was processed by equal-channel angular pressing (ECAP) with a die having an internal channel of 90°. The subsequent extrusion passes were performed by route A up to 4 passes. The microstructure of ultrafine-grained (UFG) iron was investigated using electron back scatter diffraction (EBSD) and transmission el. microscopy (TEM). Tensile creep tests were conducted on the as-pressed samples at temperature of 773 K and under applied load 60 MPa. For comparison purposes, the creep test under the same loading conditions was performed also on the unpressed material. The effect of number ECAP passes on creep behaviour of pure Fe was investigated. Results showed that the creep resistance of pure Fe after ECAP is improved in comparison with those for the unpressed as-received state.

Keywords: ECAP, electron back scatter diffraction, creep behavior

INTRODUCTION

The methods of severe plastic deformation (SPD) enable to reduce the grain size of metals to the submicrocrystalline or even nanocrystalline region. Materials processed by SPD exhibit high strength at room temperature and superplasticity at high temperatures [1,2]. However, creep behaviour probably belongs to the fewest examined properties of the materials processed by ECAP. Creep in materials after SPD was investigated on pure FCC materials [3,4,5,6], alloys [7,8,9] and Cu-Al2O3 composite [4,6] but the influence of SPD on creep behaviour of metals is still under discussion. It is not quite obvious whether application of SPD improves or deteriorates the creep properties of SPD materials.

The most reports describe creep behaviour of materials subjected only certain number of ECAP passes. There are only a few reports [3,7,8,10] documenting the creep properties and creep mechanisms of the UFG materials processed by different number of ECAP passes. In our earlier experiments conducted on pure Al it was found that its creep resistance at 473 K is considerably improved after the first ECAP pass in comparison with coarse grained material, however, repetitive pressing leads to a noticeable decrease in the creep properties of the processed material. Nevertheless, the creep resistances of the processed pure Al was higher even after 12 ECAP passes [12]. On the basis of measuring of the values of the stress exponent of the creep rate it was suggested the same operating creep mechanism for the unpressed and ECAP aluminium based on generation and movement of dislocation and by grain boundary sliding [3,7,12]. Kawasaki et al. [13] examined pure Al after 4 ECAP passes. They demonstrated, on the based of the texture measurements, that creep occurs through an intragranular dislocation process with no significant contribution of diffusion creep. Very recent results [10,14] showed that pure Cu after SPD exhibits similar creep behaviour as pure Al. In the case of pure Al we suggested, on the basis of analyses of number of high angle grain boundaries (HAGBs) that grain boundary sliding is important process in creep of Al after ECAP. However, the creep behaviour of UFG Cu is very difficult to explain by this way because HAGBs are present in microstructure in reasonable amount even at low number of ECAP passes. Further, our numerous creep tests found that samples of high purity aluminium with the same ECAP history exhibited the high scatter of creep behaviour [15,14]. The ranges of observed times to fracture were 260-1090 hours for samples after 1 ECAP pass and 18-40 for samples after 12 ECAP passes [14]. On the other hands, the investigation of creep in precipitation strengthened aluminium alloys processed by ECAP showed a deterioration of the creep properties. These alloys after ECAP exhibited faster minimum creep rates by two or three orders of
magnitude than unpressed ones [8,11,16]. The very recent investigation of creep in Cu-0.2wt.%Zr alloy showed that creep resistance at 673 K is considerably improved after 1 ECAP pass in comparison with unpressed state [17,18]. However, second ECAP pass resulted in an unexpectedly further significant increase in creep resistance of this alloy but after 8 ECAP passes were creep properties deteriorated in comparison with the coarse-grained state. Such creep behaviour is not consistent with none of previous results of creep behaviour of materials after ECAP and thus the creep strengthening mechanism in Cu-0.2wt.%Zr alloy is unclear. From previous studies results that creep resistance can be improved even at high number of ECAP passes in comparison with unpressed state but only in pure FCC metals [3,7,10]. The aim of this work is to evaluate whether the improvement of creep resistance can be reached in BCC metals like in pure iron.

EXPERIMENTAL MATERIAL AND PROCEDURES

The experiments were conducted on cast iron 99.99%. The ingots were cut into billets having a length of ~ 60 mm and cross section 10 x 10 mm². The ECAP was conducted at room temperature using a die containing the channels which formed the angle of 90°. This angle led to the equivalent true plastic strain ~ 1 after each ECAP pass without change of the billet cross-section. The billets were subsequently pressed by route A up to 4 ECAP passes.

The microstructure of specimens after ECAP was examined by scanning electron microscope Jeol 6460 equipped with an electron back scattering diffraction (EBSD) unit and using transmission electron microscope Philips CM12. Tensile creep tests were performed at 773 K and under constant applied load of 150 MPa in the argon atmosphere. The creep tests were running up to final fracture of creep specimens. The hardness was measured on the polished surfaces of samples in the plane denoted as XZ (see 19).

EXPERIMENTAL RESULTS AND DISCUSSION

1.1 Mechanical behaviour

The dependence of hardness on the number of ECAP passes is shown in the Fig. 1. The inspection of Fig. 1 showed that the hardness increases with increasing number of ECAP passes. The increase in hardness is probably related with the reduction of grain size during ECAP deformation.

In the Fig. 2 a,b are shown creep curves. From the creep curves results that the creep resistance decreases with increasing number of ECAP passes. The creep resistance of sample after 1 ECAP passes is improved in comparison with the creep resistance of the unpressed state. However, the material processed by 4 ECAP passes exhibited the deterioration of the creep resistance in comparison with the unpressed one.

![Fig. 1](image1.png) **Fig. 1** The effect of number of ECAP passes on the hardness of pure Fe
1.2 Microstructural observations

Fig. 2 Creep curves for samples processed by 1 and 4 ECAP passes and for unpressed state: a) standard creep curves, b) creep rate vs. time

Fig. 3 Microstructure of pure Fe processed by 1 ECAP pass.

Fig. 4 Microstructure of pure Fe processed by 4 ECAP passes.

Microstructure processed by 1 ECAP pass (Fig. 3) contains (sub)grains with mean size about 850 nm. The size of (sub)grains was reduced to the mean size about 400 nm in the microstructure after 4 ECAP passes (Fig. 4). EBSD analyses of samples after ECAP and subsequent creep exposure at 773 K showed that number of high-angle grain boundaries (HAGB) increases with increasing number of ECAP passes (Fig. 5 a,b). The microstructure after 1 ECAP pass contained about 37 % of HAGB and UFG microstructure after 4 ECAP passes contained about 81 % of HAGB.

The creep behavior of pure Fe is different in comparison with pure Al and Cu. Creep resistance of UFG Al and Cu is improved in comparison with their unpressed states although the grain sizes in the materials processed by 8 ECAP passes were reduced to the microcrystalline level and microstructures contained large number of HAGB [see 3,7,10]. Different creep behaviour of pure Fe can be the function of material and can
be influenced by different number of active slip systems in BCC lattice during ECAP deformation at room temperature and creep exposure in comparison with FCC metals. Nevertheless, similar creep behaviour has been observed in creep of Cu-0.2%Zr alloy at 673 K and 150 MPa [17,18]. The Cu-0.2%Zr alloy processed by low number of ECAP passes exhibited longer creep lifetime than the unpressed state. However, the alloy after 8 ECAP passes exhibited a deterioration of the creep resistance. Recently it was found that the decrease of creep temperature to 573 K in creep of Cu-0.2%Zr alloy changed the creep behavior of UFG material in comparison with uppressed one. The creep resistance of UFG Cu-0.2%Zr alloy was improved in comparison with the unpressed state [20]. We can speculate that creep behaviour of materials processed by ECAP is influenced not only by kind of material but by creep temperature and applied stress, too.

![Histograms showing number of boundaries vs. misorientation](image)

**Fig. 5** Number of boundaries vs. misorientation a) microstructure after 1 ECAP and b) 4 ECAP passes after creep exposure at 773 K and 50 MPa

**CONCLUSIONS**

The pure Fe processed by ECAP exhibits markedly improved hardness at room temperature than an unpressed material. Further, the creep resistance is improved after one ECAP pass. However, creep behaviour of material after 4 ECAP passes showed some detrimental effect of ECAP causing a deterioration of creep resistance in comparison with the unpressed Fe.

**ACKNOWLEDGEMENTS**

Financial support for this work was provided by the Czech Science Foundation under Grant 108/10/P469 and by the Academy of Sciences of the Czech Republic under the Institutional Research Plan AV0Z20410507.

**LITERATURE**


[20.] P. Kral, M. Svoboda, J. Dvorak, M. Kvapilova, V. Sklenicka, Microstructural mechanisms governing the creep life of ultrafine-grained Cu-0.2wt.%Zr alloy, 12th International Symposium on Physics of materials (ISPMA 12), Prague, September 4 - 8, 2011.