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Microstructure Mechanisms Governing the Creep Life of Ultrafine-Grained Cu–0.2 wt%Zr Alloy

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Equal-channel angular pressing was conducted at room temperature and extrusion was performed up to 12 passes using route where the billets were rotated by 90° in the same sense between consecutive passes. Tensile creep tests were performed at 473, 573 and 673 K at different constant applied stresses. It was observed that the original coarse grain size of unprocessed alloy was reduced to 0.3 μm after 8 equal-channel angular pressing passes and the grain growth during creep was restricted by precipitates with the mean diameter $\approx$ 4.0 nm. No significant effect on creep resistance was found after one equal-channel angular pressing pass at 473 and 573 K. However, the longest time to fracture was exhibited by alloy after 2 equal-channel angular pressing passes at 573 and 673 K but with further increasing number of equal-channel angular pressing passes a decrease in the time to fracture was observed. Nevertheless, the beneficial effect of equal-channel angular pressing on creep resistance was still documented after 8 passes for temperatures of 473 and 573 K. By contrast, creep tests performed at 673 K showed that the time to fracture of ultrafine-grained material is shorter as compared with that for as-received state. The 3D laser measurement of surface showed that the creep fracture process is accelerated by formation of vertical surface step relief and cavitation at the intersection of the shear bands during creep.

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1. Introduction

It is generally known that the reduction of grain size to the nanocrystalline region may lead to high strength at the room temperature and superplasticity at high temperatures [1, 2]. However, creep experiments carried out on the ultrafine-grained (UFG) materials processed by severe plastic deformation have exhibited contradictory results. It was found that certain UFG materials (mostly pure fcc metals) exhibit improved creep resistance [3, 4]. Generally, the creep resistance of pure Al and Cu increases considerably after the first equal-channel angular pressing (ECAP) pass, but decreases in subsequent passes. The decrease of creep resistance with increasing ECAP passes can be explained by microstructure changes and instability of microstructure and by increasing contribution of grain boundary sliding to the total creep deformation. By contrast, some alloys [5–8] showed a detrimental effect of ECAP on the creep resistance. The investigation of the creep behavior in precipitate strengthened aluminum alloys (Al–0.2%Sc, Al–3%Mg–0.2%Sc) showed deterioration of the creep properties at 473 K even after one ECAP pass. Recently Dvorak et al. [9] found that Cu–0.2%Zr alloy after 1 and 2 ECAP passes and creep at 673 K exhibits lower values of the minimum creep rate and longer times to fracture than those for the unpressed alloy. However, ECAP for 8 passes leads again to faster creep rates and shorter times to fracture. The aim of this paper is to present and discuss in more detail some new results and to identify and clarify microstructural mechanisms governing the creep resistance of the Cu–0.2%Zr alloy processed by ECAP.

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2. Experimental materials and procedures

The experimental material used in this investigation was a coarse-grained Cu–0.2%Zr. The material was homogenized at 1073 K for 24 h and hot-rolled. The as-received material was cut into billets with cross-sections of 10×10 mm^2. Before that ECAP billets were solution treated at 1233 K for 1 h to establish a mean grain size of 350 μm. The ECAP pressing was conducted at room temperature using a die that had a 90° angle between the channels. The subsequent extrusion passes were performed by route B_c [1] up to 12 passes to give grain size $\approx$ 400 nm. The grain size was stable up to 673 K. After ECAP the billets were annealed at 473 K for 100 h to eliminate the possible influence of non-equilibrium grain boundaries on the creep behavior. Constant load tensile creep tests were conducted at 473, 573 and 673 K and under different applied stresses. Microstructure investigations were performed using transmission electron microscope Philips CM 12 and scanning electron microscope Tescan Lyra 3. TEM foils were prepared by ion polishing using microscope Tescan Lyra 3 XMU FEG/SEM-FIB. The surface of samples was analyzed by laser confocal microscope Olympus LEXT OLS3100.

3. Results and discussion

3.1. Creep behavior and creep resistance of Cu–0.2%Zr alloy

Creep tensile tests at 673 K revealed that the time to fracture increases during the first two ECAP passes (Fig. 1). The sample processed by 4 ECAP passes exhibited a considerable decrease of the creep resistance. Nevertheless, the creep resistance of sample after 4 ECAP passes was still higher in comparison with unpressed state. The time to fracture of UFG alloy after 8 ECAP passes was lower in comparison with its unprocessed coarse-grained state at 673 K. The dependence of time to fracture on number of ECAP passes obtained at 573 K showed unexpected creep behavior of samples processed by ECAP. The creep resistances of the unprocessed material and sample after 1 ECAP pass were similar and no significant influence on creep resistance was observed after 1 ECAP pass. However, after two ECAP passes an enormous increase of creep resistance was observed. With further increase of ECAP passes the creep resistance gradually decreased. Nevertheless, the creep resistance of sample after 8 ECAP passes was still considerably higher in comparison with unpressed alloy and state after 1 ECAP pass. Such creep behavior has never been observed in other materials processed by ECAP. In general, in previous studies of creep behavior it has been found that the creep resistance decreases with increasing number of ECAP passes [4, 6, 7]. The maximum creep resistance has been experimentally found in materials processed by 1 ECAP pass in many previous works [3, 7].

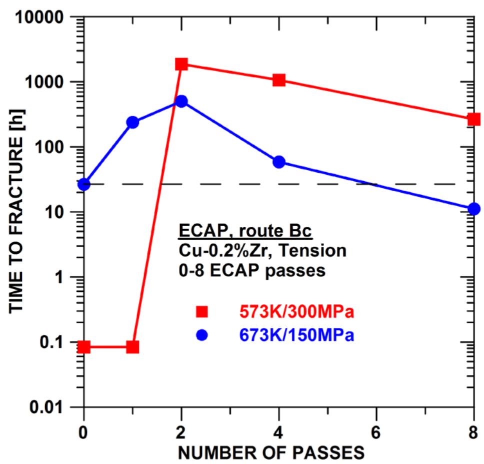

Fig. 1. Influence of number of ECAP passes on the creep resistance of Cu–0.2%Zr alloy at 573 K and 673 K.

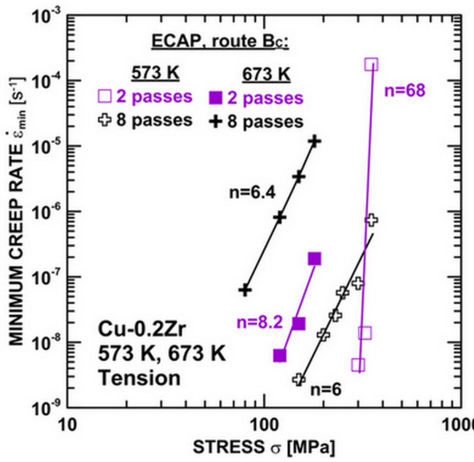

Fig. 2. Stress dependences of the creep rate at 573 K and 673 K for Cu–0.2%Zr alloy after eight and two ECAP passes.

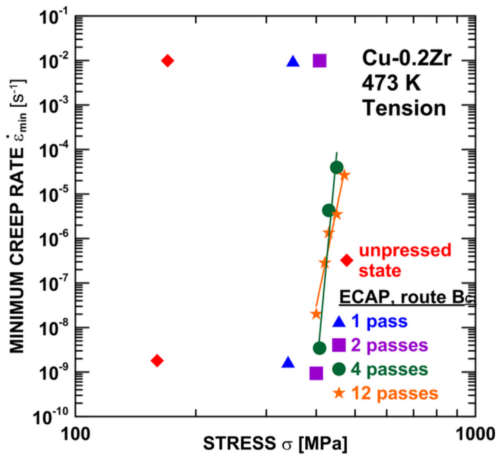

Fig. 3. Stress dependences of the creep rate at 473 K for Cu–0.2%Zr alloy initial state and state processed by one, two and twelve ECAP passes.

The stress dependences of the minimum creep rate for ECAP material after 2 and 8 ECAP passes are illustrated in Fig. 2. The results demonstrate that the values of stress exponent n of the creep rate determined at 673 K decreases from the value of $n = 8.2$ in material after 2 ECAP passes to the value of $n = 6.4$ in material after 8 ECAP passes. The same trend was observed at 573 K when the value of stress exponent n decreases from the value of $n = 68$ in material after 2 ECAP passes to the value of $n = 6$ in material after 8 ECAP passes. The values of stress exponent $n = 6$ for material after 8 ECAP passes are consistent with an intragranular dislocation process involving the glide and climb of dislocations. However, the decrease of value of the stress exponent n in the material after 8 ECAP passes can be caused by synergetic effect of additional creep mechanisms as co-operative grain boundary sliding (GBS), cavitation and more intensive diffusion processes. The high growth of n in material after 2 ECAP passes when the creep temperature is reduced from 673 to 573 K may be explained by an occurrence of power law breakdown at higher stresses and low amount of diffusion processes. Figure 3 shows the plots of minimum creep rate for ECAP materials and unprocessed state at 473 K and different applied stresses against the number of ECAP passes. The results demonstrate that materials after low number of ECAP passes and initial state are very sensitive to a change of the applied stress. The minimum creep rate increased by about 7–8 orders of magnitude when the applied stress was increased only by about 10 MPa. This creep behavior caused that alloy after 2 ECAP passes and creep at 473 K and 400 MPa has lower minimum creep rate (higher creep resistance) in comparison with state after 4 and 12 ECAP passes. However opposite results were found in the samples tested at the same temperature but at higher stresses. The similar creep behavior can be also observed at 573 K and 300÷350 MPa (Fig. 2).

3.2. Microstructural observations

In our recent work [6, 10] the formation of mesoscopic shear bands near the plane corresponding to the shear plane of the last ECAP pass was observed. In mentioned work mesoscopic sliding was documented by displacement of marker lines at their intersections with the mesoscopic shear bands. In present work the surface of the samples after creep exposures was investigated by laser confocal microscope. It was found that in the interfaces of the mesoscopic shear bands cavities can be nucleated (Fig. 4). The formation of cavities along the

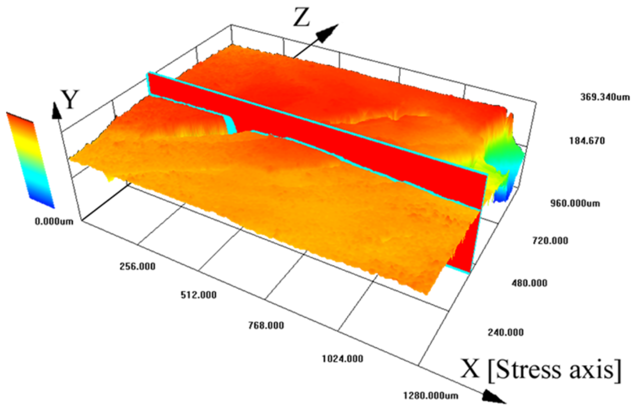

Fig. 4. Measurement of surface relief of sample after 4 ECAP passes and creep at 673 K and 150 MPa using laser confocal microscope.

mesoscopic shear bands can be caused by an insufficient accommodation of grain boundary sliding which is concentrated at the interface of the bands. From this reason transmission electron microscopy (TEM) foils perpendicular to the intersection of the mesoscopic shear bands on the surface of the samples after creep were prepared.

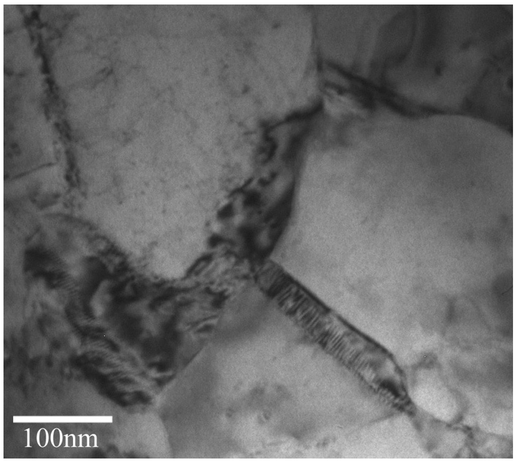

Fig. 5. Local plastic deformation in the interior of the grain (alloy after 8 ECAP passes and creep at 573 K and 350 MPa).

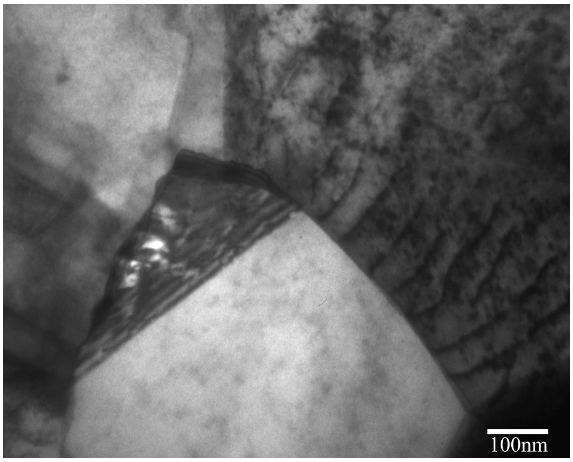

Fig. 6. Local plastic deformation at the triple junction (alloy after 4 ECAP passes and creep at 573 K and 350 MPa).

Microstructure analyses in the intersections of the mesoscopic shear bands in the samples after 12 ECAP passes revealed the formation of high local concentration of dislocations at the triple junctions (Fig. 5).

Figure 6 shows the microstructure of material after 4 ECAP passes and creep at 573 K. The inspection of Fig. 6 showed that in the triple junction local migration of the grain boundary can be observed. The local migration of the grain boundary was often observed in conjunction with grain boundary sliding [11]. The microstructure results show that the grain boundary sliding is probably accommodated by high local plastic deformation at the triple junction known as fold-formation [11].

Creep behavior of UFG materials is usually studied, due to thermal stability of the grain size, at relatively low temperatures ($T \leq 0.5T_{\rm m}$). The dislocation (power-law) creep very often plays dominant role at these temperatures. The presence of GBS in the creep of ECAP material is indicated by creep results. The decrease of stress exponent n with increasing number of ECAP passes may be connected with increasing number of number of high-angle grain boundaries in the microstructure where the GBS operates on.

4. Conclusions

The Cu–0.2%Zr alloy processed by ECAP can exhibit considerably improved creep resistance in comparison with its unprocessed state. The creep behavior of alloy is influenced by number of ECAP passes and by conditions of creep testing. The measured value of stress exponent $n \approx 6$ suggests an intragranular deformation mechanism as the rate-controlling process.

Acknowledgments

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