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Effect of Equal-Channel Angular Pressing on the Creep Resistance of Precipitation-Strengthened Alloys

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Abstract. Despite intensive research efforts in precipitation-strengthened alloys processed by equal-channel angular pressing (ECAP), so far only a few investigations have been conducted on their creep behaviour and the results from these studies are controversial. This paper examines these differences and evaluates some factors influencing the creep resistance of the binary Al-0.2%Sc and Cu-0.2% Zr alloys processed by ECAP.

Introduction

The mechanical properties of bulk ultrafine-grained (UFG) materials at elevated and/or high temperatures are a new and important area of research [1,2]. However, there have been only a few investigations on the creep behaviour of bulk UFG materials processed by equal-channel angular pressing (ECAP) [3,4]. By comparison with the unpressed (coarse-grained) state, processing by ECAP may lead to considerable changes in the creep properties in bulk UFG materials including a decrease [4 -7] and/or an increase [8 - 11] in the minimum creep rate and the creep life. It is important to note that these trends may be noticeably dependent on the number of ECAP passes [5-7]. Recent studies have revealed [3,5-7] that the creep resistance of pure aluminium and copper increases considerably after the first ECAP pass but the repetitive pressing leads to a gradual degradation of creep properties.

The results from an investigation of a commercial spray-cast aluminium 7034 alloy containing rod-like $MgZn_2$ and spherical Al_3Zr precipitates [9] and the results for Al-0.2 wt.%Sc and Al-3wt.%Mg-Sc [8,10] alloys containing Al_3Sc precipitates failed to confirm a general validity of the studies on pure aluminium and copper. By contrast, a very recent report of creep behaviour of a dispersion-strengthened Cu-0.2wt.%Zr alloy has shown [12] that the alloy after up to 4 ECAP passes exhibited a considerable improvement in creep properties in comparison with the behaviour of the unprocessed alloy. Accordingly, the present analysis was undertaken to examine the reason for such different behaviour of precipitation-strengthened alloys.

Experimental Materials and Procedures

Full details on the fabrication of the experimental materials, ECAP technique, creep testing and microstructural investigations are given elsewhere [8,11,12]. An Al-0.2wt.%Sc alloy with an initial grain size of 5-10 mm processed by ECAP for 8 passes at room temperature to give a grain size of

0.55 μm was tested in tensile creep at 473 K [11,13]. A coarse-grained Cu-0.2wt.%Zr alloy was homogenized for 24h at 1073 K and hot-rolled. Before ECAP, the billets were solution treated at 1233 K for 1 h to give an initial grain size of 350 μm. The pressing was conducted at room temperature up to 12 passes to give a grain size of ~0.35 μm. The ECAP of both alloys was performed at the IPM ASCR Brno using a die having a 90° angle between the channels [5]. Processing by ECAP was performed by route B_C in which the billets are rotated by 90° in the same sense [14]. Additional microstructural investigations in this work to explain the observed different creep behaviour of both alloys were performed using a Philips CM12 transmission electron microscope and a Jeol 6460 scanning electron microscope equipped with an electron back scattering diffraction (EBSD) unit.

Results and Discussion

Two sets of creep data for an Al-0.2Sc alloy and a Cu-0.2Zr alloy are examined in the following analysis.

First, the earlier published data were used for an Al-0.2Sc alloy processed by ECAP for 1 and 8 passes [8,10]. This alloy was creep tested in tension at 473 K (~ 0.49 T_M, where T_M is the melting temperature) and the results are shown both for the unprocessed material and the material after processing by ECAP in Figs. 1(a), 2(a) and 3(a). The differences in creep behaviour of both states of the alloy are easily demonstrated by logarithmically plotting the minimum creep rate, $\dot{\varepsilon}_m$, against the stress as shown in Fig. 1(a). Thus, the pressed alloy exhibits more than two and/or three orders of magnitude faster minimum creep rates after 8 ECAP passes by comparison with the unpresssed material. Even 1 ECAP pass leads to an increase in the creep rate. Fig. 2(a) shows the variation of the time to fracture, t_f, with the applied stress, again for both states of the alloy. The results demonstrate the creep lifetimes of the pressed material (after 8 ECAP passes) may be up to one and/or two orders of magnitude shorter than those for the unpressed alloy. Thus, the pressed alloy exhibits deteriorated creep resistance by comparison with the unpressed alloy and, furthermore, the trends in these plots is different because the pressed alloy exhibits a decreasing value of the stress exponent, n, of the creep rate in comparison with the value of n for the unpressed alloy. An increase in the fracture strain of the pressed alloy is clearly seen in Fig, 3 (a).

The second set in Figs.1(b), 2(b) and 3(b) is for a Cu-0.2 Zr alloy with initial grain size of ~ 350 μm which was processed also by ECAP for up to 12 passes at room temperature to give grain sizes of ~ 300 – 400 nm and then tested in tensile creep at 673 K (~ 0.49 T_M). By contrast to the Al-0.2 Sc alloy, the pressed Cu-0.2 Zr alloy for 1 and 2 passes exhibits slower values of the minimum creep rate and longer times to fracture than those for the unpressed alloy (Figs. 1(b) and 2(b)). However, ECAP for 8 passes leads again to faster creep rates (Fig. 1(b)) and shorter times to fracture (Fig. 2(b)). As demonstrated by Fig. 3(b) the values of strain to fracture in the pressed alloy are again higher than those for the unpressed alloy.

Since the results from both sets of experiments fail to exhibit a general consistency, it is necessary to discuss some factors which may arise in the observed differences in creep behaviour of both alloys under investigation.

We start with the problem of the thermal stability of ultrafine-grained materials because grain growth occurs easily at the elevated temperatures used in creep experiments [10]. The variation of the grain size with the annealing temperatures is illustrated in Fig. 4 for the Al-0.2Sc [15] and Cu-0.2Zr alloys. It is apparent from Fig. 4 that both creep testing temperatures (473 K for Al-0.2Sc and 673 K for Cu-0.2Zr) will not cause considerable grain growth during creep exposures which was confirmed by detailed microstructural evolutions of crept specimens [11,12]. Thus it is reasonable to conclude that the precipitates in both alloys exhibit an effective pinning effect on the grain boundaries which inhibits and/or restricts grain growth during creep exposures.

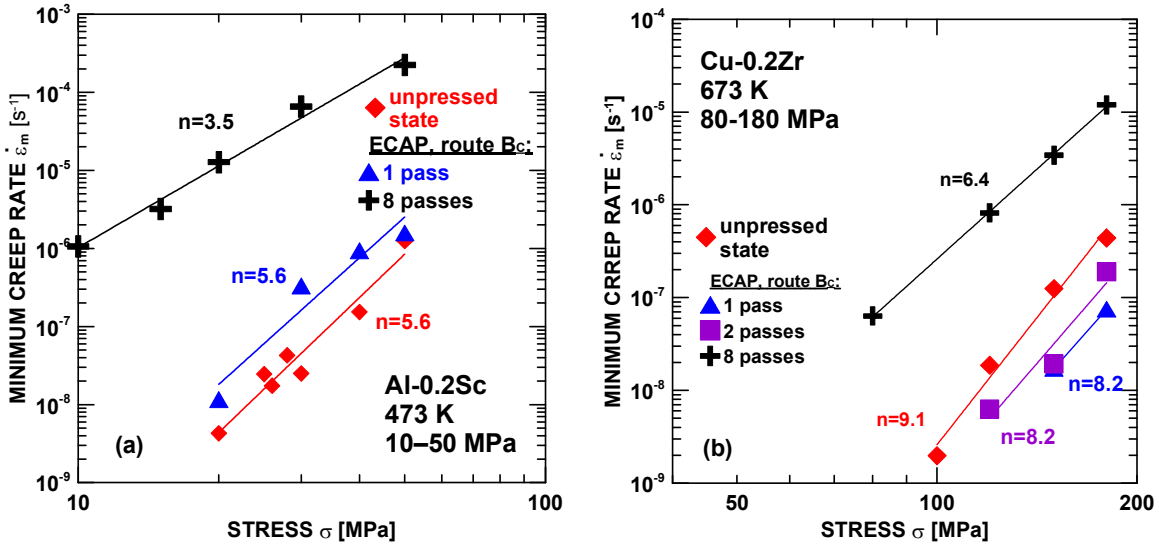


Figure 1. Stress dependence of the minimum creep rate for (a) Al-0.2Sc alloy, and (b) Cu-0.2Zr alloy.

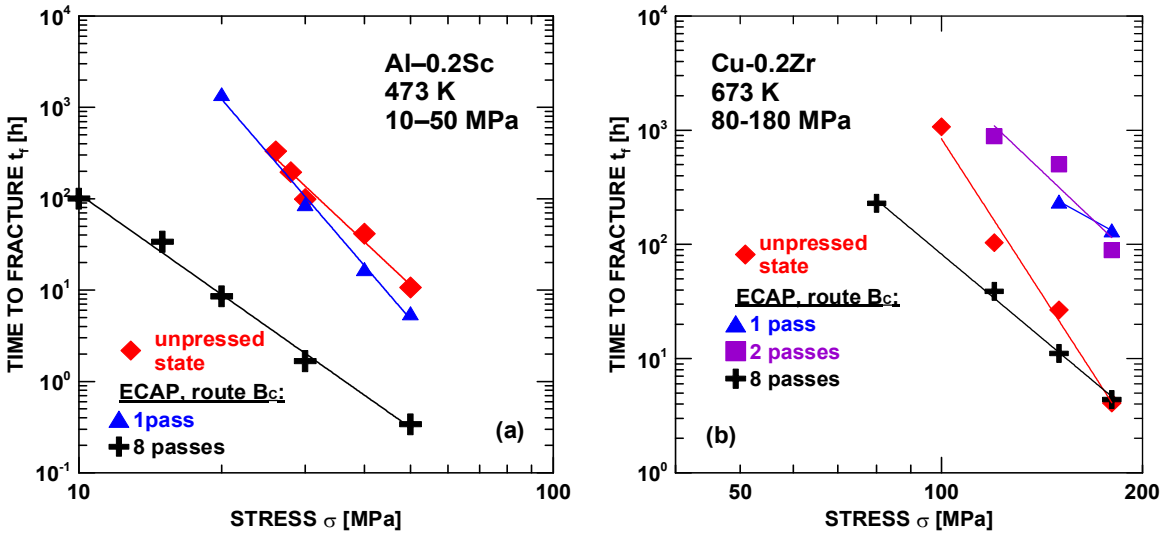


Figure 2. Stress dependence of the time to fracture for (a) Al-0.2Sc alloy, and (b) Cu-0.2Zr alloy.

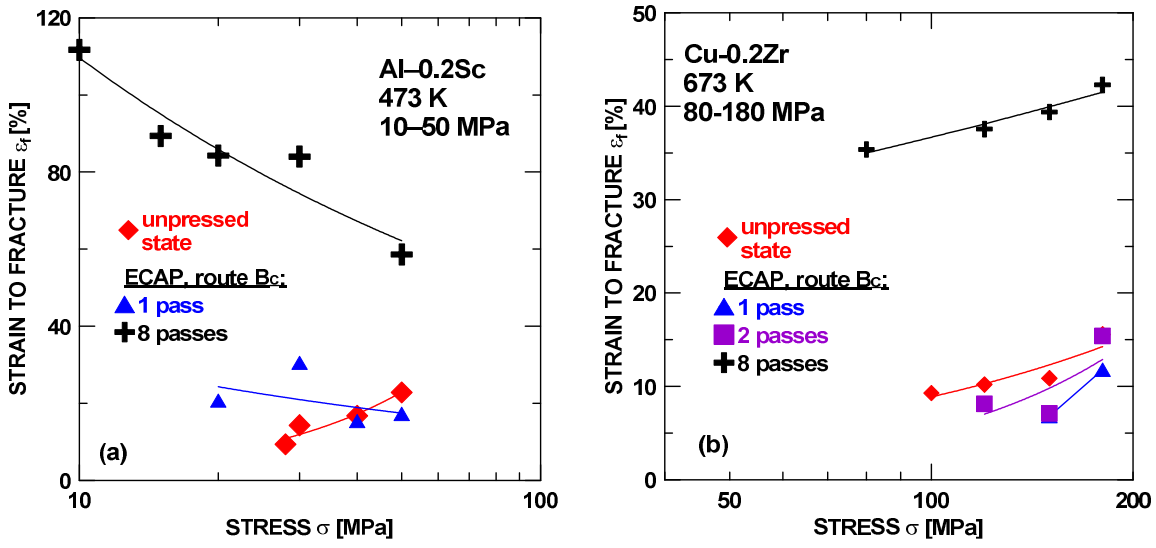


Figure 3. Stress dependence of the strain to fracture for (a) Al-0.2Sc alloy, and (b) Cu-0.2Zr alloy.

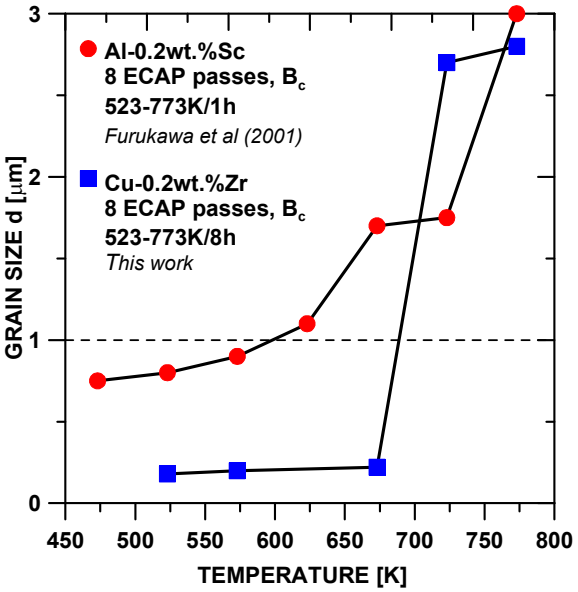


Figure 4. Thermal stability of grain size for Al-0.2Sc and Cu-0.2Zr alloys .

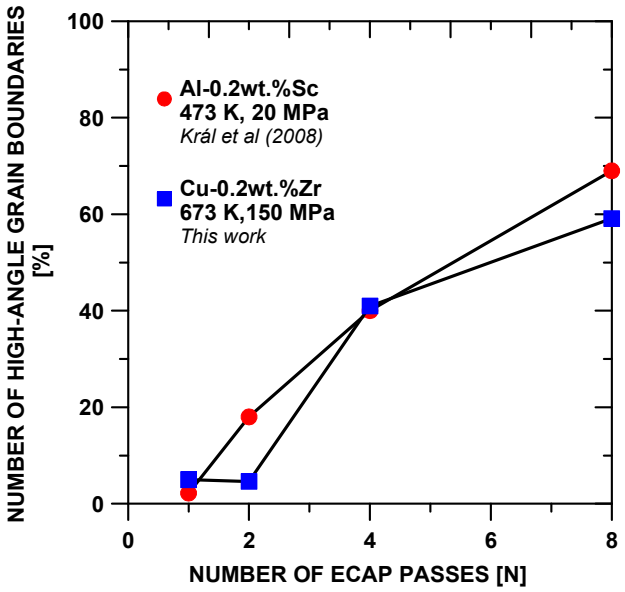


Figure 5. Dependence of HAGBs on the number of ECAP passes.

Detailed microstructural information is given in Figs 5 and 6. Investigations of the Al-0.2Sc alloy revealed very fine spherical and homogeneously distributed coherent Al_3Sc precipitates having sizes in the range ~ 6 nm (Fig. 6(a).) which appeared to be unaffected by the ECAP and the creep exposures [8,11]. The grain growth in the Cu-0.2Zr alloy is effectively restricted by very tiny precipitates Cu_9Zr_2 (Fig. 6(b)) having a size of ~ 4 nm which were also homogeneously distributed in the interiors of grains and at grain boundaries. These precipitates are most probably semicoherent, however, further investigation by high-resolution TEM is needed. There was no evidence for any breaking of the particles. Although particle cracking may occur in ECAP [16], cracking becomes less likely when the particle size is reduced.

The boundary spacing saturates after the first few passes but the fraction of high-angle boundaries (HAGBs, misorientation > 15°) continues to increase with increasing ECAP passes. The boundary misorientation distribution results obtained from EBSD analysis for both alloys are summarized in Fig. 5. This figure clearly shows an increase in the population of HAGBs associated with repetitive ECAP passes. The boundaries were predominantly low-angle grain boundaries in character up to the first four passes, which is more pronounced in the Cu-0.2Zr alloy. The fraction of HAGBs considerably increased in the alloys following four ECAP passes. Qualitatively, the same trend was also observed in earlier investigations of pressed pure aluminium and copper [13].

It was shown that successive ECAP passes leads to a noticeable deterioration in the creep properties (Figs 1 and 2). This softening may be related to the increase in the spacing of HAGBs at approximately constant subgrain size with increasing number of ECAP passes, resulting in the fraction of low-angle grain boundaries decreasing considerably (Fig. 5).

Both alloys show increased ductility with increasing number of ECAP passes (Fig. 3). This indicates that HAGBs have a lower strengthening effect under creep than low-angle boundaries. The softening by HAGBs may be explained in terms of the indirect effect which grain boundaries exert on the creep resistance by influencing the evolution of the dislocation microstructure in modifying the rates of generation and annihilation of dislocations. Further, a progressively increasing contribution of grain boundary sliding along HAGBs to the overall creep strain can be expected as a consequence of the accompanying transformation of low-angle boundaries. Although it seems acceptable to associate faster creep rates with the smaller grain size after ECAP due to more intensive grain boundary sliding, there are probable further reasons strongly influencing the creep deformation mechanism.

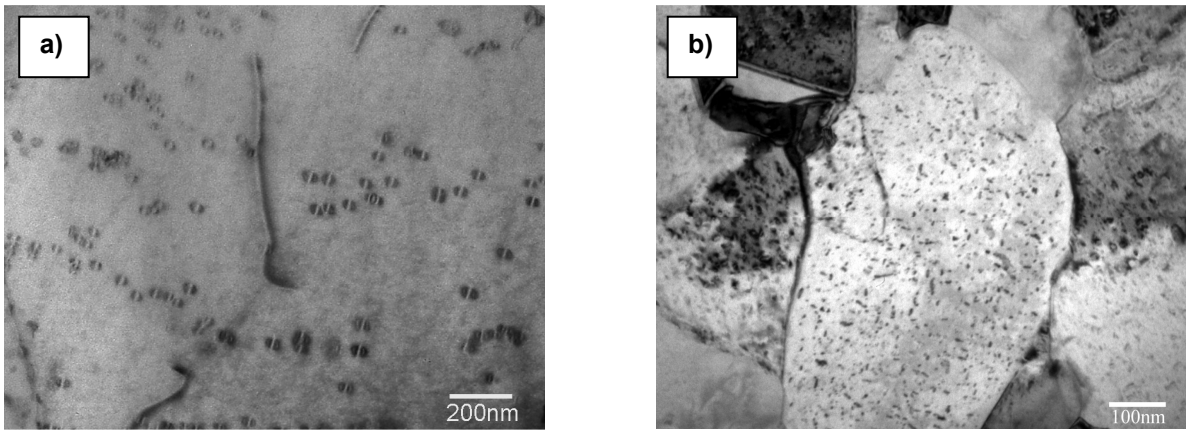


Figure 6. TEM micrographs of precipitates after ECAP and creep in the microstructure of: a) Al-0.2Sc alloy after 8 ECAP passes and b) Cu-0.2Zr alloy after 12 ECAP passes.

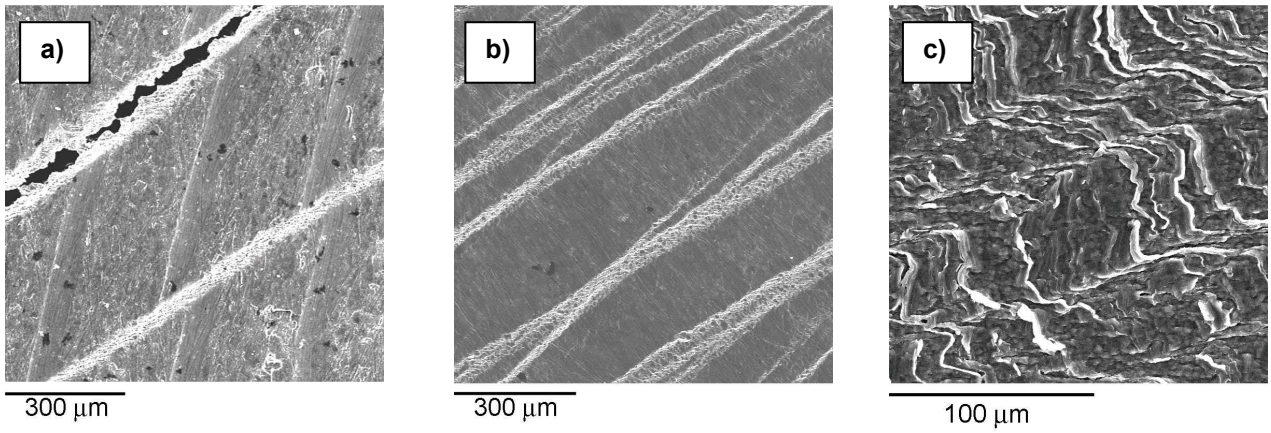
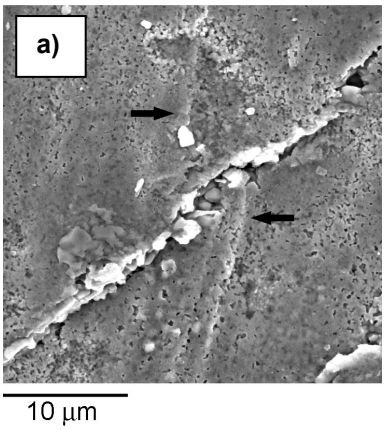


Figure 7. Mesoscopic shear bands on the creep fracture surface of the Al-0.2Sc alloy after: a) 2, b) 4 and c) 8 ECAP passes

Examination of polished but unetched surfaces of pressed and crept specimens of both alloys revealed long mesoscopic shear bands exceeding considerably the average grain dimension (Figs. 7 and 8). For this alternative intergranular sliding process the term "mesoscopic grain boundary sliding" was proposed [17]. Detailed observations of the mesoscopic shear plane by EBSD revealed (Fig. 9), that the microstructure of mesoscopic shear bands is created by HAGBs. This finding may be consistent with a model of an alternative sliding mechanism for nanocrystalline materials proposed by Hahn and Padmanabhan [18] in which, in order to produce substantial sliding on a mesoscopic scale, two or more grain boundaries must cooperate to form a planar interface which by further interconnection with other planar interfaces leads to long-range sliding.

The observed shear bands nucleate and propagate primarily in the plane corresponding to the shear plane of the last ECAP pass. Mesoscopic sliding is well documented in Figs. 7 (a) and 8 (a) by longitudinal displacements of the marker lines at their intersections with the mesoscopic shear bands. It is clear that such mesoscopic sliding can represent an important contribution to the creep strain. Contrary to the Al-0.2Sc alloy, where the mesoscopic shear bands are observable already after the first two ECAP passes, the nucleation of the mesoscopic shear bands in the Cu-0.2Zr alloy was postponed and the first bands were clearly observed after 4 ECAP passes. It is reasonable to assume that such mesoscale-banded structure may have important consequences for the fracture process in creep. Indeed, cavities and microcracks nucleate along shear bands due to the development of the necessary high local stress concentrations after considerable amounts of mesoscopic sliding as a consequence of insufficient activity of accommodation processes which can be different for both alloys.

Thus, while the mesoscopic sliding in the crept specimens of the Al-0.2Sc alloy after 8 ECAP passes is fully relaxed by plastic deformation (Fig. 7 (c)), an intensive cavitation connected with shear bands after 12 passes is clearly seen in Fig. 8 (b). These evident differences in the kinetics and extent



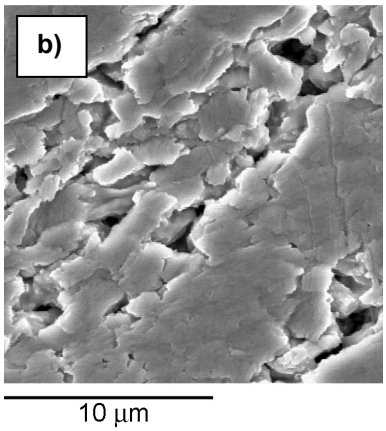


Figure 8. Mesoscopic shear bands in the Cu-0.2Zr alloy after: a) 4 and b) 12 ECAP passes

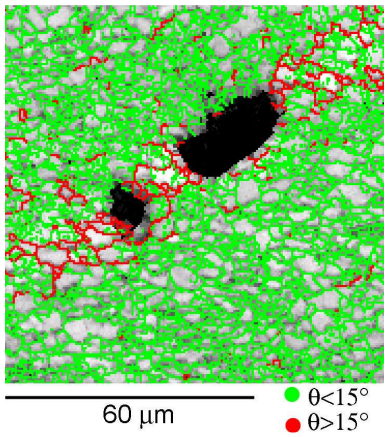


Figure 9. Microstructure on the mesoscopic shear bands

of the mesoscopic sliding and creep cavitation may be closely connected with the observed different creep behaviour of the investigated alloys especially for small numbers of ECAP passes.

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