INFLUENCE OF MICROSTRUCTURE INSTABILITY ON CREEP BEHAVIOUR OF UFG PURE MATERIALS

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Abstract
Experiments were conducted to determine an effect of creep temperature on creep behaviour of pure Cu. The ECAP pressing was performed at room temperature by route Bc. Constant load creep tests in tension were conducted at 373-573 K under different stresses. The values of the stress exponent n of the minimum creep rate for ultrafine-grained (UFG) and coarse-grained material were determined. Microstructure of samples was characterized by transmission electron microscope (TEM) and scanning electron microscope (SEM) equipped with the electron backscatter unit (EBSD). The microstructure analyses showed that microstructure of pure Cu processed by 8 ECAP passes and subsequent creep exposure contained large fraction of boundaries with coincidence sites lattice (CSL). The results showed that creep in UFG materials is influenced by additional creep mechanisms up to 0.5 Tm when the UFG microstructure is more or less stable.

Keywords:
Ultrafine-grained microstructure, EBSD, creep behaviour

1. INTRODUCTION
Methods of severe plastic deformation [1,2] enable to reduce grain size to the submicrometer or even nanometer level. The most attractive technique is equal-channel angular pressing (ECAP). The materials with ultrafine-grained structure have different mechanical properties in comparison with coarse-grained materials. The ultrafine-grained structure is not always stable in particular in the microstructure of pure metals. The grain growth and recrystallization can occur at about 0.3 Tm [3]. From this reason the UFG materials are usually tested at temperatures about 0.2 - 0.5 Tm[4-9].

Sklenicka et al. [8,9] found that the creep resistance of pure Al and Cu increase considerably after the first ECAP pass, but the creep resistance of these materials decreases with the subsequent increasing of ECAP passes. The decrease of creep resistance with increasing ECAP passes can be explained by microstructure changes and by increasing contribution of grain boundary sliding to the total creep deformation. Some creep results of UFG pure metals indicate different creep behavior because the minimum creep rates in UFG materials were found slower than in the same material in a coarse-grained material. By contrast, some alloys [10,11] showed the 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 the deterioration of the creep properties at 473 K even after one ECAP pass.

The relationship between steady state (or minimum) creep rate and time to fracture is given by the empirical Monkman-Grant relation (MGR) [12]

\[ \varepsilon_p^{\infty} \cdot t_r = \text{const.} = C \]
where m and C are the Monkman-Grant constants. The constants m and C enable to estimate the time to fracture. Monkman-Grant relationship is beneficial for verification of models of intercrystalline creep fracture. In the case that creep strain is primarily result of dislocation glide into grains the intercrystalline creep fracture is controlled by dislocation glide [13] otherwise the validity of MGR can be hardly expected. The aim of this work is to describe effect of microstructure stability on creep behaviour when the UFG is more or less stabile and simultaneously the microstructure changes occurred.

2. EXPERIMENTAL MATERIALS AND PROCEDURES

The experimental material in a received state used in this investigation was a coarse-grained copper. The billets were processed by ECAP at room temperature using a die that had an internal angle of 90° between the two parts of the channel and an outer arc of curvature of ~ 20°, where these two parts intersect. It can be shown from first principles that these angles lead to an imposed strain of ~ 1 in each passage of the sample [1]. The pressing speed was 10 mm/min. The billets were subsequently pressed by route Bc by 8 ECAP passes to give mean grain size ~ 0.4 µm. The constant load creep tests in tension were performed at 373, 473 and 573 K and under different applied stresses. The tensile samples, having gauge lengths of 10 mm and cross-sectional areas of 8 x 3.2 mm, were machined from billets parallel to the section XZ. Before creep exposure the marker lines transversal to the stress axis on the polished tensile samples were made. The creep testing was conducted in an environment of purified argon with the testing temperatures maintained to within ± 0.5 K of the desired value. All of the tests were run up to fracture. The microstructure was examined by SEM and scanning electron microscope equipped by electron back scatter diffraction (EBSD). Inhomogeneity of microstructure can be qualified by the coefficient of profile area variation CVa [14]: the higher are their values the more pronounced is the inhomogeneity. In homogeneous systems 0.55 ≤ CVa < 1, in mildly inhomogeneous systems is CVa lower then 2 and higher values are typical for systems with multimodal grain size distributions.

3. RESULTS AND DISCUSSION

3.1 Microstructural investigations

The microstructure after 8 ECAP passes and creep exposure at 373 K and 160 MPa (Fig. 1) contained mixture of small more or less equiaxed and larger elongated grains. The mean grain size was about 2.9 µm and coefficient of profile area variation CVa was determined about 2.08 which represents inhomogeneous

![Fig. 1 Microstructure of UFG Cu after creep at 373 K and 160 MPa](image1)

![Fig. 2 Microstructure of UFG Cu after creep at 473 K and 60 MPa](image2)
systems with bimodal character. The microstructure after 8 ECAP passes and creep exposure at 473 K and 60 MPa (Fig. 2) contained grains with mean size about 7.4 μm. The coefficient of profile area variation CVa was determined about 2.94 which represents systems with multimodal grain size distributions. Inhomogeneity created during creep exposure can caused non-uniformity of creep strain. Inhomogeneous structures with bimodal character can be used for optimization of strength and ductility in nanocrystalline and UFG materials at room temperature [15].

The dependence of the mean grain size of UFG copper after creep exposure on applied stress is shown in Fig. 3. It can be seen that mean grain size increases significantly with increasing creep temperature and slightly with decreasing applied stress. The samples tested at 573 K contained the grains with mean size above 10 μm. The mean grain size is significantly higher in comparison with samples tested at 473 and 373 K. The copper, with mean grain size significantly higher than 10 μm, can be considered as fully recrystallized coarse-grained material. Such mean grain size may be certain limit which markedly changes the value of the contribution of additional creep mechanisms to the total creep deformation. The instability of UFG microstructure influences the total creep strain (Fig. 4). The specimens with mean grain size lower than 10 μm exhibited the increase of the strain with increasing value of applied stress. However the opposite result was observed at 573 K when the grain size was larger than 10 μm. The increase of grain size with increasing value of applied stress (time to fracture) can be influenced not only by grain growth but also increasing volume of recrystalized grains with increasing time to fracture.

**Fig. 3** The dependence of mean grain size of UFG copper after creep exposure on applied stress.

**Fig. 4** The dependence of mean grain size of UFG copper after creep exposure on creep strain.

**Fig. 5** The dependence of CSL boundaries on time to fracture.
UFG microstructures contained relatively large fraction of CSL boundaries (Fig. 5) and some of them can be so-called as 'special' boundaries [16]. The inspection of Fig. 5 showed that the samples tested at 473 and 573 K contain high fraction of special boundaries than UFG copper tested at 373 K. The largest amount of ‘special’ boundaries was formed by boundaries with ∑3 CSL. These boundaries have twin character [16]. The fraction of CSL and ∑3 CSL boundaries increases with increasing time to fracture in the samples tested at 373 and 473 K. However, opposite tendency was observed in the microstructure of samples tested at 573 K. It is known that deformed fcc metals with low stacking fault energies containing after recrystallization higher fractions of twin boundaries than those annealed without an imposed shear stress [17]. The number of ∑3 CSL boundaries can influence creep behaviour of UFG Cu because they are resistant to crack initiation [18] and less susceptible to corrosion than high ∑CSL and ‘general’ grain boundaries [19].

### 3.2 Creep behaviour

The stress dependences of the minimum creep rate measured at 373 K for ECAP and CG copper are illustrated in Fig. 6. The results demonstrate that creep resistances of CG and ECAP copper are too different to perform the creep tests at the same stress interval. Nevertheless, the stress exponents determined at the similar creep rate interval decrease from the value of n~15 for CG material to the value of n~5 for copper processed by 8 ECAP passes. The transition from power law creep to the power law breakdown (PLB) is shifted to the higher applied stresses and higher minimum creep rate in the specimens processed by ECAP. The material after 8 ECAP passes is more resistant against the transition from power law creep to the PLB in comparison with CG and copper processed by 1 ECAP pass. The higher resistance of UFG copper is influenced by grain size and its sufficient thermal stability at 373 K.

![Fig. 6 Stress dependences of creep rate at 373, 473 and 573 K for CG and UFG copper.](image)

![Fig. 7 Monkman-Grant relation for CG and Cu after ECAP and creep exposure at 573 K](image)

The links between deformation and fracture in Cu tested at 573 K are in good agreement with well known Monkman-Grant relationship (MGR). Nevertheless copper processed by ECAP is shifted to slower values of minimum creep rate and longer time to fracture in comparison with CG state. Fig. 7 shows that MGR is valued with the same value of m for copper processed by 1-12 ECAP passes (Tab. 1) and it is independent on number of ECAP passes.

The validity of MGR for coarse-grained material with the very similar value of m (Tab. 1) is weakened neither by difference in examined materials nor in their homological temperatures. In the Fig. 8 and 9 is shown that MGR is shifted for copper processed by low number of ECAP passes. The values of m for Cu processed by
low number of ECAP passes are similar to the values for CG state. However the values of m for copper processed by higher number of ECAP passes (UFG material) decreased with decreasing temperature of creep tests. The shift and changes in values of m occur probably as a consequence of different final creep ductility.

Table 1 The values of m for coarse-grained and ECAP material

<table>
<thead>
<tr>
<th>Creep temperature [K]</th>
<th>CG</th>
<th>ECAP 1-2 passes</th>
<th>ECAP 4-12 passes</th>
</tr>
</thead>
<tbody>
<tr>
<td>373</td>
<td>-1.119</td>
<td>-1.132</td>
<td>-0.843</td>
</tr>
<tr>
<td>473</td>
<td>-1.061</td>
<td>-0.987</td>
<td>-0.937</td>
</tr>
<tr>
<td>573</td>
<td>-1.285</td>
<td></td>
<td>-1.082</td>
</tr>
</tbody>
</table>

4. CONCLUSIONS

The copper processed by 8 ECAP passes tested in region of sufficient microstructure stability is more resistant against the transition from power law creep to the PLB in comparison with CG and copper processed by 1 ECAP pass. Instability of UFG microstructure influences creep ductility and time to fracture.

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LITERATURA


