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CHARACTERIZATION OF CREEP BEHAVIOUR AND MICROSTRUCTURE CHANGES IN PURE COPPER PROCESSED BY EQUAL-CHANNEL ANGULAR PRESSING

PART I. CREEP BEHAVIOUR

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Abstract. Coarse-grained high-purity copper was processed by equal-channel angular pressing (ECAP) through 1-12 passes and then tested under creep conditions at temperature ranging from 423 to 523K. It was found that the creep resistance of the ECAP processed material was markedly improved with respect to unpressed material. The results revealed that the creep behaviour of pressed material strongly depends on the number of ECAP passes. The ECAP material exhibits the highest creep resistance after the first ECAP pass. However, successive ECAP passes lead to a noticeable decrease in the creep properties of the pressed copper. The results indicate conventional power-law creep with a stress exponent of ~5 – 6 which is consistent with an intragranular dislocation process involving the glide and climb of dislocations.

1. INTRODUCTION

Only few reports are available describing the creep behaviour of metallic materials processed by equal-channel angular pressing (ECAP) [1-3] and then tested under creep loading [4-32]. These reports have shown that there are both similarities and differences between the creep behaviour of coarse-grained metals and alloys and their ultrafine-grained counterparts. Our previous results for high-purity aluminium [7-19], which are the most extensive available to date, appear unexpected result because under same testing conditions of stress and temperature the measured minimum creep rates in the ECAP material with ultrafine grain size were slower and creep lifetimes markedly longer than in the same material in a coarse-grained unpressed state.

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The present investigation was initiated to provide additional and general information on the creep behaviour of high purity copper after processing by ECAP. The early creep experiments on ECAP copper were conducted only to a total of 4 separate passes through the ECAP die so that information is missing at the higher strains. Further, no systematic stereological measurements were performed to document microstructure evolution in high purity copper during ECAP and the creep exposure. Thus, the present work was initiated to address these deficiencies.

The two parts of the paper present and discuss the obtained results of experimental investigations. Part I reports the results of creep tests which were conducted on samples pressed through a total up to 12 passes by ECAP. These samples where then examined to provide quantitative metallographic information using orientation imaging microscopy incorporating a computer-aided facilities for electron-backscattered diffraction (EBSD) analysis. The microstructural characteristics and the results of microstructural investigations are presented and discussed in the Part II of the paper.

2. EXPERIMENTAL MATERIAL AND TECHNIQUES

The experiments were conducted using high-purity copper. This material was selected because some creep properties and behaviour associated with processing by ECAP have already been reported [4-6,18,24-29,32]. The material used was a coarse-grained (grain size ~ 1.2 mm) high purity (99.99%) copper supplied in the form of ingots. The ingots were cut into short billets having a length of 60 mm and a cross-section 10 mm x 10 mm. These 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 [33]. The pressing speed was 10 mm/min. Repetitive pressings were conducted to give totals of 1, 2, 4, 8, and 12 passes, equivalent to imposed strains from ~ 1 to ~ 12, respectively, mostly using processing route B_C in which the billets are rotated by 90° in the same sense between each pass [34]. The grain size of high purity Cu after pressing through four passes at room temperature using route B_C was measured as ~ 0.4 μm.

Creep tests were conducted in tension using samples having gauge lengths of 10 mm and cross-sectional areas of 8 x 3.2 mm. Each specimen was machined so that the longitudinal axis was oriented parallel to the pressing axis. The creep specimens were tested at temperatures 423 – 573K using various constant stress within the range from 20 to 100 MPa. The creep testing was conducted in an environment of purified argon with the testing temperatures maintained to within ± 0.5K of the desired value. All of the tests were continued until fracture. For comparison purposes, an additional creep tests were conducted on specimens of the as-received and unpressed (coarse-grained) material. The strain-time readings were continuously recorded by PC – based data acquisition system. Following ECAP and creep testing, microstructure evolution was characterized using scanning electron microscope (SEM) equipped with an electron back-scatter diffraction (EBSD) unit. The results of the EBSD techniques and microstructural data analysis are described in Part II of this paper.

3. RESULTS AND DISCUSSION

3.1. Effects of the number of ECAP passes and processing route on the creep behaviour

Representative creep curves for unpressed and pressed copper by route B_C up to 12 passes are shown in Figs. 1 and 2, respectively. Standard ε versus t creep curves in Figs. 1a and 2a can be easily replotted in the form of the instantaneous strain rate $d\varepsilon/dt$ versus t as shown in Figs. 1b and 2b. These plots were obtained at 473K (~ 0.35 T_m) and 573K (~ 0.42 T_m). The creep tests were run up to the final rupture of the creep specimens. As demonstrated by the figures, significant differences were found in the creep behaviour of the ECAP material when compared to its unpressed counterpart. First, the ECAP material exhibits markedly longer creep life than that of coarse-grained material. Second, the minimum creep rate for the ECAP material is about one to two orders of magnitude less than that of coarse-grained copper. Third, the shapes of creep curves for the ECAP material after high number of pressings differ considerably from the tests conducted at small number of the ECAP passes by the extent of individual stages of creep.

It is apparent from Figs. 1 and 2 that the times to fracture of pressed material increase enormously after the first pass but gradually decrease with increasing number of ECAP passes. Thus, the effect

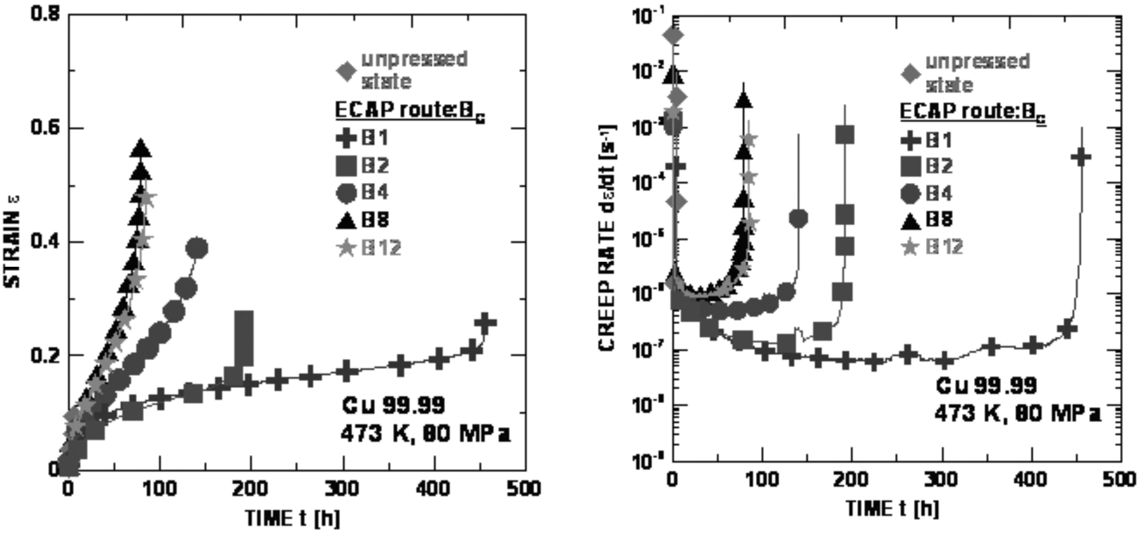

Fig. 1. Standard creep and creep rate vs. time curves at 473K and 80 MPa for unpressed state and various number of ECAP passes via route B_C.

of the number of ECAP passes on creep behaviour is generally consistent with the earlier results concerning the creep testing on pure aluminium [7-8,10-11,18,19,21,29]. These results can indicate a significant role of the microstructure in the creep in ECAP copper. The results of microstructure evolution during ECAP and subsequent creep of copper will be reported and discussed in more detail in the Part II of this paper [35]. However, previous investigations [18,19,29] of the fraction of high-angle grain boundaries after ECAP and creep in aluminium and copper showed that after the first pass, the boundaries were predominantly low-angle boundaries in character, and the population of high-angle grain boundaries had considerably increased in materials during repetitive pressings. This indicates that high-angle grain boundaries have a lower strengthening effect under creep than low-angle ones. Further, the refinement of the dislocation substructure during ECAP increases the creep resistance and leads to a decrease in the minimum creep rate [29].

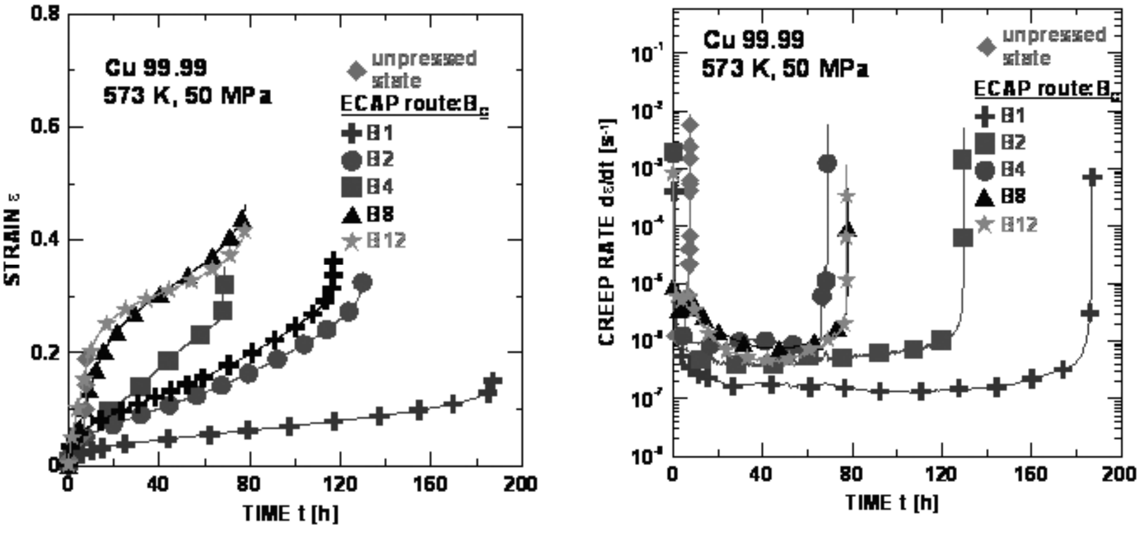

Fig. 2. Standard creep and creep rate vs. time curves at 573K and 50 MPa for unpressed state and various number of ECAP passes via route B_C.

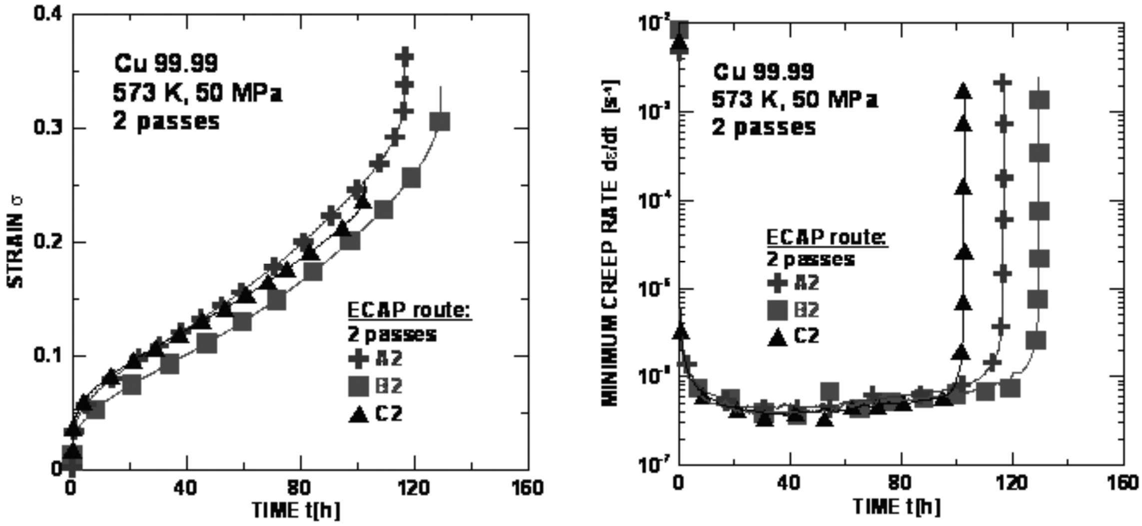

Fig. 3. Standard creep and creep rate vs time curves at 573K and 50 MPa for samples pressed by two ECAP passes with different routes ($B \equiv B_C$).

The use of repetitive pressings provides an opportunity to develop different microstructures by rotating the billet between consecutive ECAP passes. Using the notation introduced by Segal [36] and Furukawa et al [34], the billet is either not rotated (route *A*), rotated by 90° (the routes designated B_A and B_C refer to rotation in the opposite and the same sense) or rotated by 180° (route *C*).

Figs. 3 and 4 show that standard creep curves for the ECAP copper pressed by routes *A*, B_C, and *C* subjected to two and four passes, respectively. Figs. 5a,b,c summarize a complete record of three creep parameters for the ECAP copper samples after creep testing. For comparison reason, Fig. 5 includes also results of an earlier experimental investigation of the effect of different ECAP processing routes on creep behaviour of pure aluminium [8,11,19]. Each point represents the average results of two or three individual creep tests at the same number of ECAP passes and under the same creep conditions. Inspection of Figs. 3-5 shows that independently of material selected there are not very significant differences in creep behaviour of samples prepared by the various ECAP processing routes.

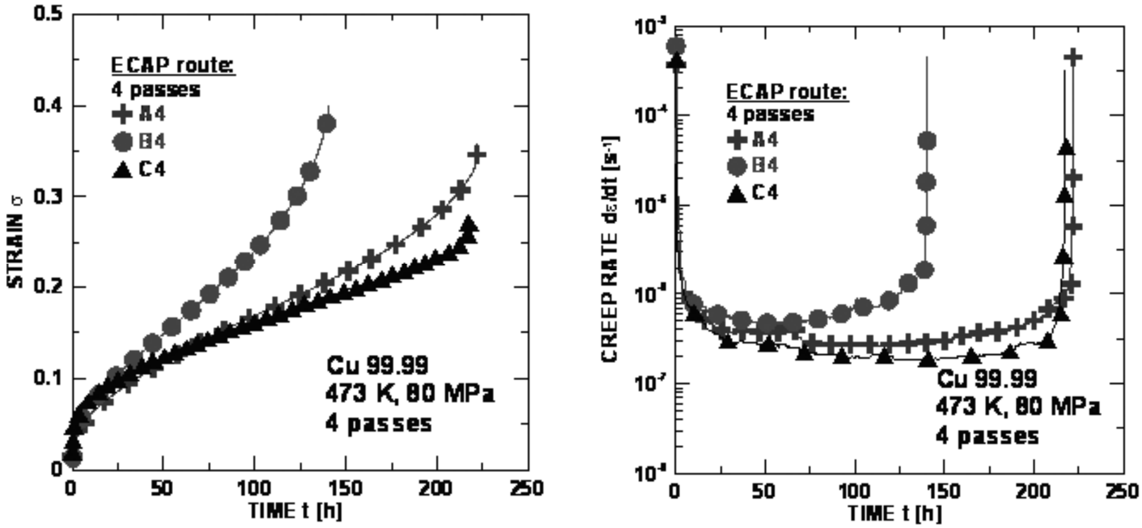

Fig. 4. Standard creep and creep rate vs time curves at 473K and 80 MPa for samples pressed by four ECAP passes with different routes ($B \equiv B_C$).

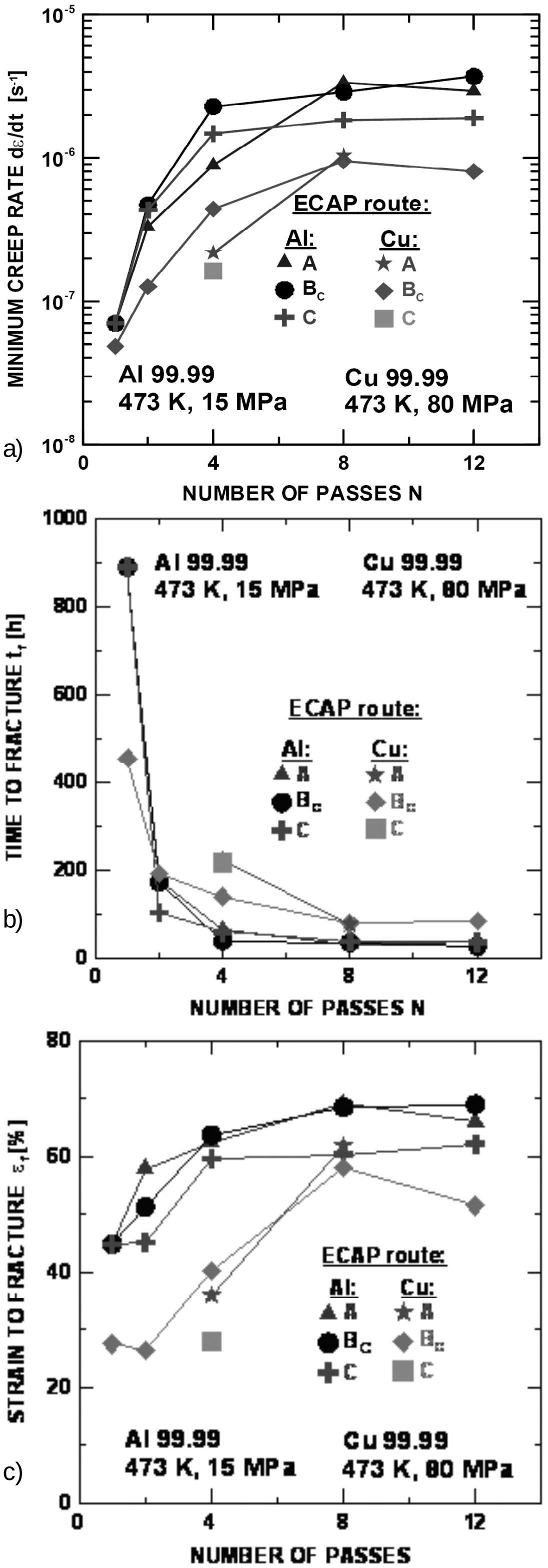

Fig. 5. Effect of various ECAP routes and different number of ECAP passes on (a) creep rate, (b) time to fracture and (c) strain to fracture. Aluminium data are from Ref. [19].

All three processing routes produce a significant increase in the minimum creep rate (Fig. 5a) and the strain to fracture (Fig. 5c) through the first four passes and a slight increase during subsequent pressing. Accordingly, the times to fracture dramatically drop through the first four passes and then there is no significant difference among the number of passes (Fig. 5b).

A conclusion following from the results shown in Fig. 5 is that there is only little apparent dependence of the creep behaviour on the ECAP route used for aluminium and copper samples crept after the same number of ECAP passes under the same loading conditions. However, in order to examine this conclusion more critically it should be noted that the ultrafine grains produced in aluminium [8,9,21] and copper [35] are not sufficiently stable at elevated temperatures and a significant grain growth takes place at the very beginning of the creep test [8]. Further, the additional difference of the creep curves may be attributed to the extraordinarily large initial grain sizes of aluminium [8,9] and coarse grain size of the present material in as-received states. Finally an another explanation in the differences of the creep behaviour after different processing routes with the same number of ECAP passes and under the same creep loading conditions may lie in the possibility that the creep experiments are probably not a sufficiently refined procedure for picking up these rather small differences in creep.

3.2. Stress and temperature dependence of creep parameters

For elevated and high temperature creep, the minimum creep rate generally varies with the applied stress through a power-law relationship $\dot{\varepsilon}_{min} \sim A\sigma^n$. To determine the value of the stress exponent *n*

$$n = \left(\frac{\partial \ln \dot{\varepsilon}_{min}}{\partial \ln \sigma} \right)_T \qquad (1)$$

for the creep rates of pure copper in the unpressed and as-pressed conditions, the minimum creep rates were determined from the standard creep curves and plotted logarithmically against the applied stress. The results are shown in Fig. 6a for specimens without ECAP (at 573K only) and after ECAP pressing at 573 and 473K for 8 passes. It can be seen that the creep rates in the pressed and unpressed samples are very similar when tests are performed at 573K and the same levels of the applied stress. The observed values of the stress exponent are ~ 6.3 (at 573K) and ~ 5 (at 473K) for pressed material

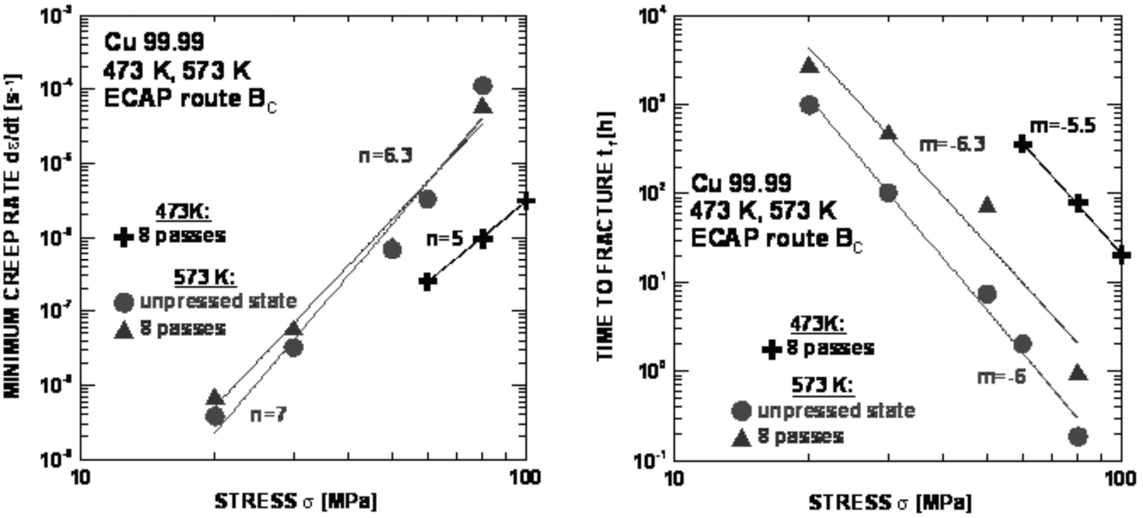

Fig. 6. Stress dependence of creep rate at 473 and 573K for samples pressed by eight ECAP passes and unpressed state (573K only).

and ~ 7 (at 573K) for the unpressed one. Very similar values of the stress exponent *m*

$$m = -\left(\frac{\partial \ln t_f}{\partial \ln \sigma}\right)_T \tag{2}$$

for the times to fracture are shown in Fig. 6b. The time to fracture t_f can be estimated as $t_f = M/\dot{\varepsilon}_{min}$ from the minimum creep rate $\dot{\varepsilon}_{min}$ if *M* is a constant (Monkman-Grant relation [37]). The observed minimum creep rates $\dot{\varepsilon}_{min}$ and the times to fracture t_f in copper processed by ECAP with different number of passes have already been published [38] and confirmed the general approximate validity of the Monkman-Grant relation. Thus higher values of the times to fracture t_f for the pressed samples may reflect also a considerable increase of creep plasticity (creep fracture strain) in the pressed samples in comparison to the unpressed ones.

As depicted from comparison of Fig. 6a and b, the slopes and therefore the values the apparent stress exponents n and m for both the pressed and unpressed samples are nearly the same indicating that deformation and fracture processes in creep are controlled by the same mechanism(s).

To determine the apparent activation energy for creep Q_C, the minimum creep rates were measured in the temperature interval from 423 to 523K and at the applied stress 80 MPa. The activation energy for creep Q_C is defined as

$$Q_C = \left[\frac{\partial \ln \dot{\varepsilon}_{min}}{\partial(-1/kT)}\right]_\sigma . \tag{3}$$

Thus, the activation energy Q_C can be derived from the slope of log *d*ε/*dt* versus 1/*T* plots shown in Fig. 7. The values of the apparent activation energy Q_C are equal to 106.1 ± 0.2 and 90.3 ± 2.1 kJ/mol for the unpressed and the pressed samples, respectively. It is generally accepted that the high fraction

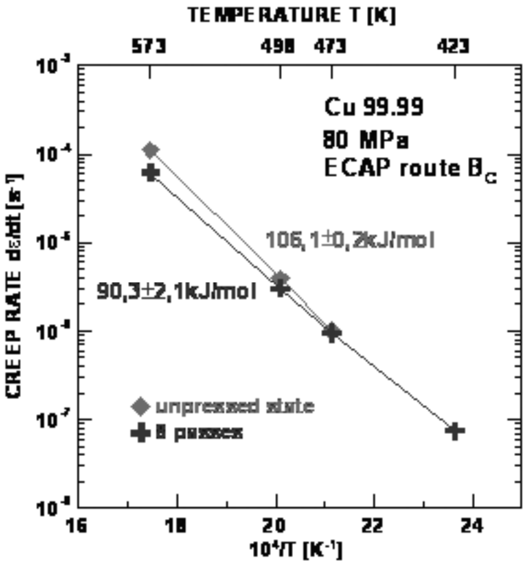

Fig. 7. Relation between creep rate and temperature for unpressed and pressed states at 80 MPa.

of non-equilibrium high-angle grain boundaries in the pressed material reduces activation energy of deformation to values in the direction of those of thermally activated grain boundary processes like the grain boundary diffusion. Valiev *et al.* [39] reported the grain boundary diffusion activation energy $Q_b \approx$ 78 kJ/mol in the pressed ultrafine-grained copper. This value is low compared to the value of $Q_b \approx 107$ kJ/mol for the grain boundary diffusion in coarse grain copper [40].

Very recent reports [10,18,21,31] have demonstrated that creep will occur in pure aluminium and its alloy after processing by ECAP by the same mechanism as in coarse-grained materials with intragranular dislocation glide and climb as the dominant rate-controlling deformation processes. Thus conventional creep mechanisms, already developed for coarse-grained materials, may be used to explain the creep characteristics and behaviour of the ECAP pressed material.

4. SUMMARY AND CONCLUSION

Coarse-grained high-purity copper was processed by equal-channel angular pressing (ECAP) through 1 – 12 passes and then tested under creep conditions at temperature ranging between 423 to 523K. The main results can be summarized as follows:

- The creep behaviour of copper pressed by ECAP depends critically upon the number of ECAP passes. The creep resistance is increased already after the first ECAP pass. However, successive ECAP pressing leads to: (i) a noticeable decrease in the time to fracture, (ii) an increase of the minimum creep rate and (iii) a continuous increase of the strain to fracture (creep plasticity).
- A little apparent dependence of the creep behaviour on the different ECAP route with the same number of ECAP passes was observed for copper specimens crept under the same creep conditions. It is believed that the effect of grain growth at creep temperature may tend to obscure the effect of processing route and the creep testing is probably not sufficiently refined procedure for picking up these rather small differences in the creep behaviour of the pressed material by different ECAP routes.
- The results indicate conventional power-law (dislocation) creep with a stress exponent of $n \approx 5 – 6$ which is consistent with an intragranular dislocation process involving the glide and climb of dislocations.

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