INFLUENCE OF ECAP TECHNIQUE ON CREEP BEHAVIOUR OF POWDER ALUMINIUM PROCESSED BY DIRECT EXTRUSION

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Abstract

Severe plastic deformation (SPD) is a perspective method for preparation of ultrafine-grained materials. The Al powder was prepared by cold isostatic pressing (CIP) at the pressure of 200 MPa. The granulometric powder fraction of a particle size less than 400 µm was compacted via conventional direct hot-extrusion (DE) at temperature 450°C by ratio 16:1. This material was then subjected to equal-channel angular pressing (ECAP) up to 4 passes by route Bc. The microstructural investigations were performed using optical microscopy (OM) and scanning electron microscopy (SEM) equipped with an electron back-scatter diffraction (EBSD) unit. Creep tests in tension were conducted at temperature of 473 K and at applied stresses in the range from 30 to 60 MPa. Some creep tests in compression were performed on material just after DE. The paper presents results of study of microstructure and creep behaviour of aluminium powder before and after ECAP. It was found that ECAP method does not lead to an additional marked refinement of grain size. However, in dependence on number of ECAP passes ECAP method markedly influences creep behaviour due to development of high-angle grain boundaries during ECAP process.

1. INTRODUCTION

The optimum consolidation process is essential to obtain a fully densified structure of powder materials. Conventional powder metallurgy route requires high temperature to remove any porosity and make it nearly dense. In this process the microstructure of the sample may coarsen which is not suitable. Usually it is desirable to employ a special consolidation process that allows lower processing temperature. It is well known that introduction of shear deformation, e.g. by direct extrusion (DE), can result in significant refinement of the microstructure of metals and alloys [1]. However, this type of working is accompanied by extreme pressing loads and high extrusion temperatures. Equal-channel angular pressing (ECAP) has become an important processing procedure for refining microstructure in metallic materials via severe plastic deformation (SPD) [2]. It is known [3] that intense shear strain in ECAP is able to disrupt the surface oxide layer and creates good bonding between particles. Furthermore, it is an effective method to consolidate the powder at relatively lower temperatures and loads than that used in conventional powders processing [4]. Recent investigations have been conducted to determine the creep properties of aluminium and aluminium-based alloys prepared by SPD techniques mostly via ECAP method. Despite considerable research effort in the methodology of equal-channel angular pressing (ECAP), only very limited reports describing the powder
materials processed by ECAP and then tested its creep behaviour under conditions of constant stress or constant load. Recently, Sklenička et al. [5] and Dvořák et al. [6] conducted extensive studies of creep behaviour and microstructure of high-purity aluminium prepared by ingot metallurgy and then processed by ECAP at room temperature. The authors used the isothermal constant load creep test technique and measured the minimum creep strain rates to determine mechanisms of creep. Further, Dvorak et al. [7] studied creep behaviour of powder Al before and after ECAP technique. It was found that material processed by 1 and 2 ECAP passes embodied higher creep resistance compared to material in DE state. In the present paper, the previously published creep data [7] are analysed with the new dates to find out, whether creep in ECAP materials occurs by the same mechanism as in the DE state of material.

2. EXPERIMENTAL MATERIAL
Commercially aluminium powder size less than about 400 µm was used as a raw material. From this powder was compacted material which was then subjected to DE and ECAP process up to 4 passes. Creep tests in tension and compression were performed at the same temperature. Simultaneously, microstructure was observed for all state of material. Full details on the fabrication of aluminium, ECAP technique and creep testing are given elsewhere [7].

3. RESULTS

3.1. Microstructural observation
Good bonding between particles was obvious with no decohesion observed. The presence of pores in the PM material was studied earlier [7]. The optical microstructure of the PM material revealed limited occurrence of pores after ECAP passes. It was shown that higher density of DE material was achieved after only one pass of ECAP. Following number of ECAP passes did not cause the change in density of pores. EBSD pictures of the samples were taken in longitudinal to the extrusion direction. For comparison, the microstructures of the DE and ECAP materials are demonstrate on the Fig. 1

Fig. 1a shows EBSD microstructures observed for the samples subjected to DE. It was found that pressing by DE leads to reduction of grain size to 6.5 µm. The grains are mostly elongated to the extrusion direction. Subsequent ECAP press (Fig.1b) leads to a substantial reduction in the grain size (~ 3.9 µm) and the more equiaxed microstructure. With further pressings, the grains subsequently evolve into a reasonably equiaxed and homogeneous microstructure. The microstructure is essentially homogeneous after four ECAP passes with an average grain size of the order of ~ 2.5 µm.

Fig. 1. EBSD images of aluminium (misorientation Δ > 15°) subjected by a) DE, b) subsequent 1 ECAP pass, and c) subsequent 4 ECAP passes.
Fig. 2a shows the dependence of number of ECAP passes on the number of high-angle grain boundaries (HAGB) with misorientation $\theta > 15^\circ$. The relative fraction of HAGBs population is about 61% for DE state only. After first pass, the boundaries were predominantly low-angle boundaries in character, and the population of high-angle boundaries had considerably increased in sample after following four ECAP passes. Further, square area of grains is illustrated on Fig. 2b. For 1 ECAP passes, average area of grain is 11.7 $\mu m^2$.

**Fig. 2a.** Dependence of number of ECAP passes on the number of boundaries with misorientation $\Delta > 15^\circ$ and grain size.

**Fig. 2b.** Frequency of square grain size for Al processed by 1 ECAP pass.

### 3.2. Creep results

The results of creep test were summarized in Fig. 3 (tensile tests) and Fig. 4 (compression tests). Representative standard creep curves are shown in Fig. 3. The creep testing was conducted on billet after direct extrusion and, for comparison purposes, on the same material additionally processed by ECAP method by route B (B=Bc). All of these plots were obtained at an absolute temperature of 473 K (~ 0.5 Tm) and at an applied tensile stress of 50 MPa. The creep tests in tension were running up to the final fracture of creep specimens. As demonstrated by Fig. 3 significant differences were found in the creep behaviour of the ECAP material when compared to its DE counterpart. First, the ECAP materials after 1 and 2 passes exhibits markedly longer creep life (Fig. 3a) than DE material. The highest creep resistance is achieved already after first ECAP pass. However, successive ECAP pressing leads to a noticeable decrease in the creep properties. The minimum creep rate for the ECAP material is less than that for DE state. Second, application of 4 ECAP passes leads to marked deterioration of creep properties. Creep life here achieves only half value as compared to DE state. There is also reflected by the minimum creep rate whose value may be up to one order of magnitude higher than that of the DE state. Third, the strain to fracture is approximately the same for both ECAP and DE state. Nevertheless, slightly higher elongation was observed on material after 4 ECAP passes.
Fig. 3. Creep curves for samples after DE and additional 2 ECAP passes: (a) standard creep curves, and (b) creep rate vs. time.

Compression creep test for DE material is illustrated on Fig. 4. The creep tests were performed at 473 K under a constant applied stress and interrupted at a true strain of about ~ 0.35. It is clear that no-one of the creep curves exhibits a well-defined steady state. In fact this stage is reduced to an inflection point of the dε/dt vs. t curve. Supposing that the instantaneous creep rate dε/dt at given stress and temperature is a certain measure of the “softness” of the microstructure, then the dε/dt-t plots reveal the time evolution of this “softness”.

Inspection of Fig. 4b shows that the samples after applied of 50 MPa exhibit creep curves with a short primary stage of creep followed by instantaneous softening. Higher stresses have different process when after first hardening the creep rate remains reasonably constant. These constant rates demonstrate that the microstructural characteristics are essentially stable at the points where the tests were terminated.

Fig. 4. Creep curves for samples after DE and additional 2 ECAP passes: (a) standard creep curves, and (b) creep rate vs. strain.
An additional difference in the minimum creep rate for the ECAP material and DE state is illustrated by Fig. 5, which shows the variation of the minimum creep rate with the applied stress for the tension (Fig. 5a) and compression test (Fig. 5b). For tensile creep test, the observed values of the stress exponent \( n = (\partial \ln \dot{\varepsilon} / \partial \ln \sigma) \), are approximately 16 for DE and 15 for ECAP material. The same value of \( n \) was found in compression test for DE material.

![Graph 1](image1.png)

**Fig. 5.** Variation of the minimum creep rate with the applied stress for the a) tension and, b) compression test

### 4. DISCUSSION

In this work microstructural investigation of all material examined indicated little differences in the grain size produced via ECAP passes. It has been demonstrated that ECAP is capable of producing refined and homogenous structures with large fractions of high-angle boundaries. The value of the relative fraction of a high-angle (\( \theta > 15^\circ \)) grain boundary population slightly decreases after first ECAP pass and than rapidly increases during subsequent ECAP pressing (Fig. 2). The highest creep resistance was achieved already after first pass. Thus we attain considerably heterogeneous microstructure with high presence of low-angle grain boundaries (LAGBs). Further pressing led to decrease in the creep properties. The coexistence of a dislocation climb and grain boundary sliding in creep of ECAP material may explain the observed decrease of the creep resistance with increasing number of ECAP passes. A progressively increasing contribution of grain boundary sliding to overall creep strain can be probably attributed to the accompanying transformation of low angle to high angle boundaries and an achievement of their more equilibrium state when the number of passes increased. The higher contribution of grain boundary sliding to the total creep elongation is higher in sample subjected to 4 ECAP passes which exhibits lower creep life and higher fracture strain in comparison to DE state.

Stress dependence of the minimum creep rate at constant temperature and at constant grain size can be described by the power-law relationship of the form: \( \dot{\varepsilon} \equiv A \sigma^n \), where \( A \) is independent of \( \sigma \), while \( n \) is the stress exponent of the creep rate. In our previous works [8] realized on pure cast state Al was found the stress exponent of \( n = 5 \) which indicates conventional power-law creep. It is consistent with an intragranular
dislocation process involving the glide and climb of dislocations. We expected the similar results on powder material. However, the observed values of n are very high. It could be cost by dispersion strengthening of powder material. In this material cannot be excluded the interaction between moving dislocations and oxides particles which are present in the matrix as a result of processing in powder metallurgy. This small oxide phases could be responsible for high value of stress exponent through the possible existence of the threshold stress for creep at very small values of the applied stress. However, lower value of the stress exponent found for ECAP aluminium indicates an enhanced role of grain boundary sliding. The detailed investigation of fracture surfaces did not revealed any differences between the mode of creep fractures. Together with creep results it seems that the similar creep mechanisms are controlling both creep behaviour and damage processes.

5. CONCLUSION

In this paper, the effect of direct extrusion and following ECAP pressing on the microstructure and mechanical properties of aluminium processed by powder metallurgy have been studied. It has been shown that DE leads to a decrease of grain size. Further, extrusion by ECAP method does not lead to an additional refinement of the grain size. The highest creep resistance is achieved already after first ECAP pass. Successive ECAP pressing leads to a noticeable decrease in the creep properties. After 4 ECAP passes deterioration of creep resistance represents up to half value of those for DE state. On the contrary, slight improvement of strain was observed. Creep in powder Al is probably controlled by the same mechanism for both DE and ECAP states.

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REFERENCES