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Microstructural influence on low-temperature superplasticity of ultrafine-grained Ti–6Al–4V alloy

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Abstract

Microstructural influence on low-temperature superplastic behavior of ultrafine-grained Ti–6Al–4V alloy fabricated by equal channel angular pressing (ECAP) was investigated. The deformed structures were analyzed with the increment of strain by transmission electron microscopy. Also, a series of tensile tests were carried out on ultrafine-grained (UFG) samples to measure elongation at temperature of 973 K and at strain rates of 10^{-4} to 10^{-2} s⁻¹. The results indicated that elongation was significantly increased with increasing ECAP straining from 4 to 8 revealing more high-angle grain boundaries. Deformation mechanisms for UFG structure were analyzed in the context of inelastic deformation theory, which consisted of dislocation glide and grain boundary sliding.

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1. Introduction

Superplasticity normally refers to capability to exhibit extremely high elongations free of noticeable necking. In order to attain the excellent superplasticity in coarse-grained Ti-6Al-4V alloy, it is essential to refine the grain size below micrometer-order by thermo-mechanical processes (TMP) such as mechanical alloying, severe plastic deformation (SePD), etc. Equal channel angular (ECA) pressing, one of the SePD processes, has been regarded as the most effective method to produce bulk ultrafine-grained (UFG) materials with intense plastic straining, resulting in no residual porosity and no dimensional changes [1,2]. From economical viewpoints, the lowtemperature superplasticity by UFG structure makes it possible to reduce cost savings in superplastic forming industries. Recently, Sergueeva et al. [3] reported that UFG structure via HPT process leads to very high elongations. Indeed, Ko et al. [4] concluded that such high elongation observed at low temperature was resulted from grain boundary sliding (GBS) accommodated mainly by grain boundary diffusion. However, the difficulty in achieving such high superplasticity, in spite of UFG structure,

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is arisen due to the presence of non-equilibrium state in grain boundaries.

Therefore, the purposes of this study are to understand and compare the microstructural changes after two ECA pressing strain levels, 4 ($\varepsilon \approx 4$) and 8 ($\varepsilon \approx 8$), and to analyze the deformation mechanisms of UFG Ti–6Al–4V alloy using load relaxation tests in relation to the inelastic deformation theory.

2. Experimental procedures

The material used in this work was a commercial Ti–6Al–4V alloy billet with a chemical composition of 6.03% Al, 3.83% V, 0.2% Fe, 0.19% O, 0.01% C, 0.007% N and balance Ti (in wt.%). The as-received microstructure was heat treated at 1223 K for 2 h and furnace cooled, resulting in coarse-grained (CG) microstructure with an average grain size of 11 μ m, and was machined (Ø 9.5 mm × *L* 80 mm) for isothermal ECA pressing. Detailed conditions on the present ECA pressing were reported elsewhere [4,5].

Load relaxation tests were used to evaluate the flow behavior of UFG structures using hourglass specimens with a gauge length of 27 mm. From the data of load relaxation test, flow stress–strain rates curves were obtained by the methods suggested by Lee and Hart [6]. Also, tensile tests were performed using dog-bone specimens (gauge length of 5 mm) lying paral-

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Fig. 2. Resulting elongations of low-temperature superplasticity of UFG Ti-6Al-4V alloy.

lel to the longitudinal axis of the ECA pressed sample to verify superplastic deformation.

3. Results and discussion

Fig. 1 represents the transmission electron microscopy (TEM) bright-field (BF) images and the corresponding selected area electron diffraction (SAED) patterns of UFG structures in the 4 and 8 ECA pressings, respectively. Regardless of the number of ECA pressings in this study, most grains were significantly refined to $0.3 \,\mu$ m in diameter without changing volume fraction of each constituent phase. Two specimens did not reveal any significant differences in their microstructures, as reported earlier [1,5]. However, it was noted that more diffused ring pattern in SAED was found in 8 ECA pressed sample than in 4 ECA pressed one, implying that the misorientation angles of each grain boundary increased with an increment of ECAP strain [7].

The elongation results of UFG samples tested at 973 K are shown as a function of strain rate in Fig. 2 together with those tested by other TMPs. It is important to note that, in spite of similar refined α -grain size, 8 ECA pressed specimens show higher elongations than 4 ECA pressed specimens at the same temperature and strain rate. Elongation rises to reach over 700% even at 973 K, confirming that low-temperature superplasticity can be achieved by grain refinement via ECA pressings. Furthermore, the strain rate showing the superplastic elongation by ECA pressings is about one order of magnitude higher than that reported by the authors [3,8]. This implies that production cost for the part made by superplastic forming can be considerably reduced by using UFG materials. From the stress–strain rate relations in region II, activation energies are estimated to be 294, 174 and 98 kJ/mol for CG, 4 and 8 ECA pressed specimens, respectively.

Fig. 3 shows stress-strain rate curves obtained from the load relaxation tests at 973 K for the CG (11 µm) and two distinct UFG (0.3 µm) samples, respectively. Comparing the stress levels of three specimens fixed strain rate, UFG structure showed lower flow stress than the CG sample. This was probably due to the decreased stress required for the GBS in UFG structure. The solid lines in Fig. 3 were obtained on the basis of the inelastic deformation theory [9], revealing a good accordance with experimental results. It was noticed that stress-strain rate curves of the UFG sample showed slight curvature changes, especially at intermediate strain rate regime $(5 \times 10^{-6} \text{ s}^{-1} \le \dot{\varepsilon} \le 10^{-3} \text{ s}^{-1})$, whereas CG sample showed only negative curvature for the most of the strain rate range. According to the inelastic deformation theory consisting of GBS and dislocation glide [9,10] this curvature change is mainly attributed to the occurrence of GBS. Therefore, GBS would be more predominant in the UFG structure than in the CG microstructure and contributed significantly to total deformation of UFG Ti-6Al-4V alloy. Also, the slope of stress-strain rate curves is expressed as the strain rate sensitivity ($m = \partial ln\sigma/\partial \ln \dot{\epsilon}$). It is clearly shown that UFG materials show much higher m values than the CG materials, approaching a maximum value of 0.45.

Quantitative analysis was made to examine the relative contribution of each deformation mechanism at 973 K using the experimental data obtained from load relaxation tests in relation with inelastic deformation theory, and the results are presented in Fig. 4. It was noted that the fractions of strain induced by the operation of GBS to the total strain approached to 40 and 55% in each UFG specimen, while that of CG specimen was less than 10%. The amount of the GBS contribution found in this study was well accorded with those suggested by Langdon [11]. In order to confirm that GBS was indeed an important deformation mechanism in UFG structures, 8 ECA pressed specimen was cut parallel to longitudinal axis and then scratched with Al_2O_3 powder of 0.05 µm. The sample was then compressed up to strain



Fig. 1. TEM micrographs and corresponding SAED patterns of UFG Ti-6Al-4V alloy after: (a) 4 ECA pressings and (b) 8 ECA pressings.

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