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Stored energy and recrystallization kinetics of ultrafine grained titanium processed by severe plastic deformation

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ABSTRACT

Equal channel angular pressing (ECAP) was conducted at 250 °C on commercial purity titanium up to 10 passes. An ultrafine-grained microstructure with a mean grain size of about 183 nm was obtained. The evolution of stored energy and recrystallization temperature was studied by differential scanning calorimetry. The activation energy involved in the recrystallization process was also determined. The results show that with increase of ECAP passes up to 6 the recrystallization temperature increases. Also, it is seen that with increasing ECAP passes up to 8 the stored energy increases, beyond which it saturates at a value of about 58 J/mol. For the recrystallization of 10 passes ECAP-ed samples, average activation energy of about 342 kJ/mol was determined.

1. Introduction

Metallic materials with ultrafine-grained (UFG) microstructure produced through severe plastic deformation (SPD) have been reported to have superior physical and mechanical properties compared with coarse-grained (CG) counterparts [1]. In addition to small grain size (average grain size less than 1 μm), ultrafine grained materials processed by the SPD methods contain a large fraction ($>50\%$) of high angle grain boundaries [2]. These materials, depending on the chemical composition and processing conditions, may also have high dislocation density of 10^{14}–10^{16} m^{-2} [1,3–6]. Moreover, a very high vacancy concentration (in order of 10^{-4}) has been reported to present in metals after processing by severe plastic deformation [4,7–9]. Thus, it is reasonable to expect that these materials have high amounts of stored energy. Besides providing a direct strengthening effect, the stored energy also provides the driving force for subsequent microstructural changes on heating by recovery and recrystallization [10]. Accordingly, it is expected that UFG materials have lower thermal stability in comparison with those processed by conventional deformation processes such as cold rolling.

Recrystallization as a solid state transformation can be studied by non-isothermal experiments conducted in a constant heating rate such as investigations by differential scanning calorimetry (DSC) [10]. Although there are some studies in which the stored energy and recrystallization kinetics of SPD-processed metals and alloys have been studied [11–14], in the case of commercial purity titanium (CP-Ti) this subject has been rarely documented. Thus, in the present study differential scanning calorimetry (DSC) is used to measure the stored energy and activation energy for recrystallization of CP-Ti that has been subjected to 10 passes ECAP at 250 °C.

2. Experimental procedure

The chemical composition (wt%) of the CP-Ti investigated was 0.18% O, 0.03% N, 0.16% Fe, 0.17% Pd, 0.18% Cr and balance Ti. At first, the material was annealed at 800 °C for 1 h in argon atmosphere and then air cooled. With this procedure, an equiaxed microstructure having an average grain size of about 20 μm was obtained (Fig. 1). ECAP samples with a length of 70 mm and diameter of 14.5 mm were machined from the annealed material. These samples were subjected to ECAP in a die with the channel angle $\Phi=105^\circ$ and the corner angle $\Psi=20^\circ$ which leads to an imposed strain of about 0.8 for each pass. ECAP was performed up to 10 passes with route B_C at 250 °C.

The microstructure of ECAP-ed specimens was investigated by transmission electron microscopy (TEM) with a Philips CM 200 electron microscope operated at 200 kV. Specimens for TEM were cut from middle sections of the ECAP-ed billets perpendicular to the pressing direction (cross section). Thin foils were first mechanically polished and finally electropolished in a Tenupol 5 double jet polishing unit in a solution of 5 vol% $HClO_4$ in methanol at −30 °C.

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The stored energy and recrystallization temperature of the material were determined by DSC using a Netzsch DSC 200 F3 under a constant heating rate of 20 °C min^{-1} in high purity Ar atmosphere. Samples in disc form of approximate 80 mg were cut from ECAP-ed billets. These samples were ground to 1200 grit, cleaned in soap solution and rinsed ultrasonically in ethanol. The DSC runs were initiated from ambient temperature to 850 °C and at least two samples were measured. For each sample, two DSC runs were obtained and the curve from the second run was used as a baseline. The final DSC curve was the difference between the second run and the first run. The activation energy was determined based on DSC measurements at heating rates of 10, 20, and 30 °C min^{-1}. The activation energy for the transformation can be calculated from the experimental results obtained by DSC by the use of isoconversion methods. In these methods, the dependence of the peak temperature on the heating rate is used to calculate the activation energy for recrystallization [15]. The most popular isoconversion methods used for calculation of activation energy developed by Kissinger [16], Boswell [17], Ozawa [18] and Starink [19] are given by Eqs. (1)–(4), respectively. In these equations, β is the linear heating rate, E_a is the activation energy, T_P is the maximum temperature of peak, R is the universal constant of gases, and C is a particular constant of each method

$$\ln\left(\frac{\beta}{T_P^2}\right) = C - \frac{E_a}{RT_P} \quad (1)$$

$$\ln\left(\frac{\beta}{T_P}\right) = C - \frac{E_a}{RT_P} \quad (2)$$

$$\ln\beta = C - 1.0518\frac{E_a}{RT_P} \quad (3)$$

$$\ln\left(\frac{\beta}{T_P^{1.92}}\right) = C - 1.0008\frac{E_a}{RT_P} \quad (4)$$

According to Eqs. (1)–(4), the plots of $\ln(\beta/T_P^2)$, $\ln(\beta/T_P)$, $\ln\beta$, and $\ln(\beta/T_P^{1.92})$ versus $(1/T_P)$ should yield a straight line each and from each slope the activation energy can be determined. By isochronal DSC analysis, the recrystallized fraction X at temperature T is given by $X = A_T/A$ where A is the total area of the exothermal peak and A_T is the area under the curve between the temperature of beginning of peak (T_0) and the temperature T_X. Using this method, sigmoidal curves are obtained and the recrystallization temperature can be calculated [20].

Vickers microhardness (Hv) was measured on a plane perpendicular to the pressing direction (cross section) by imposing a load of 200g for 15 s and taking 15 separate measurements at different randomly selected positions.

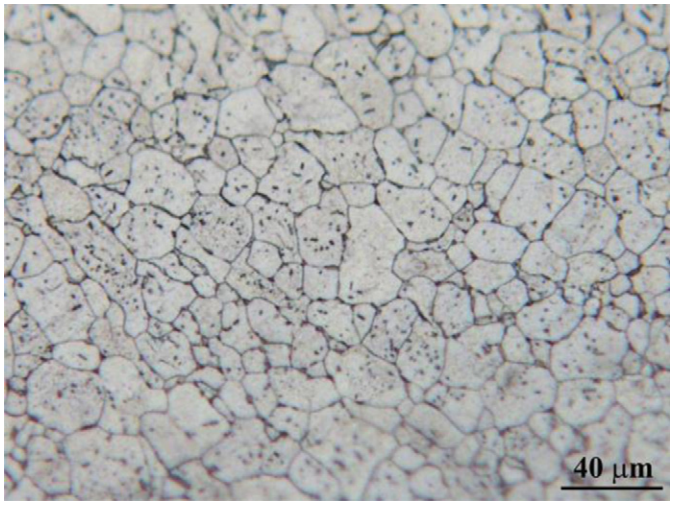

Fig. 1. Micrograph of CP-Ti in the as-annealed condition.

3. Results

3.1. Microstructure

Fig. 2(a) shows a typical microstructure of CP-Ti after 10 passes ECAP taken from the cross section of processed billet together with the associated SAD pattern recorded from a region of 1 μm diameter. The microstructure consists of equiaxed grains with average grain size of 183 nm which were evaluated from a number of TEM micrographs. Three types of grains can be distinguished in this micrograph. There are grains with sharp boundaries so that their interiors are nearly free of dislocations, e.g. the grains marked as A. Grains containing sub-boundaries and dislocation tangles are, labeled B and the grains containing a high

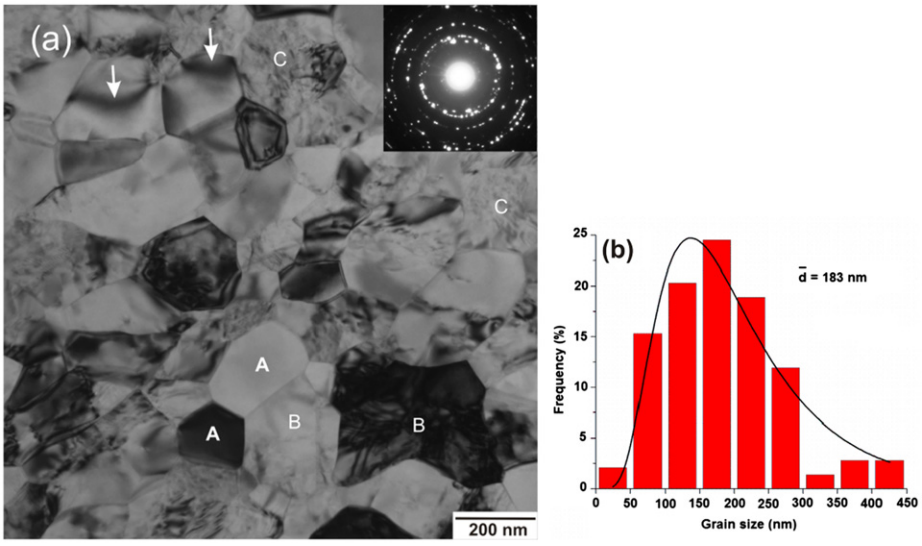

Fig. 2. (a) Bright field TEM micrograph and the corresponding SAD pattern from the cross section of CP-Ti billet, and (b) the grain size distribution in CP-Ti processed by 10 passes ECAP at 250 °C