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Characteristics of the allotropic phase transformation in titanium processed by high-pressure torsion using different rotation speeds

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ABSTRACT

An investigation was initiated to examine the effect of rotation speed on the allotropic α to ω-phase transformation in titanium. A grade 2 commercial purity titanium was processed by high-pressure torsion (HPT) at room temperature up to a maximum of 10 turns using a pressure of 5.0 GPa and different rotation speeds from 0.5 to 2 rpm. It is shown that the allotropic phase transformation occurs during HPT at different rotation speeds but the volume fraction of the ω-phase decreases when the rotation speed is increased. The results indicate that the hardness and strength are decreased by processing the specimens at higher rotation speeds. Additional annealing for 15 min at 423 and 473 K after HPT processing is also effective in changing the volume fractions of the ω-phase and producing an ω to α reverse phase transformation. The results are interpreted in terms of the temperature rise during HPT processing.

1. Introduction

It is well known that titanium (Ti) displays three phases depending on the pressure and temperature. The hexagonal close-packed α-phase is stable at ambient temperature and pressure but this phase transforms to the body-centered cubic β-phase at temperatures above 1155 K under ambient pressure [1]. The existence of the β-phase was also predicted theoretically at pressures higher than ~36 GPa but this transformation was never observed experimentally [2,3]. An α to ω martensitic phase transformation occurs under high pressures at ambient temperature [1] where the crystal structure of the ω-phase is composed of a regular hexagonal close-packed lattice but with an open structure having only 3 atoms per unit cell at (0,0,0), (1/2, 2/3, $c/2a$), and (2/3, 1/3, $c/2a$) where a and c are the lattice parameters in the basal plane and perpendicular to the basal plane, respectively, and the axial ratio is $c/a \approx 0.62$ [4].

The high-pressure ω-phase exhibits a reverse phase transformation upon heating or when the pressure is reduced [5]. It is now well established that straining using high-pressure torsion (HPT) may produce a stabilization of the high-pressure ω-phase under ambient conditions [6–11]. Furthermore, the ω-phase is harder than the α-phase so that the α to ω-phase transformation during HPT processing produces extra hardening and strengthening of the pure Ti. In practice, it appears that HPT may be the only technique that is easily available for introducing a high volume fraction of the ω-phase in pure Ti [12].

Processing by HPT is now a well-established procedure for obtaining nanometer and submicrometer microstructures in bulk solids [13,14]. In processing by HPT, a disk-shaped specimen is deformed by simple shear between two anvils where it is constrained under a high pressure and subjected to concurrent torsional straining [15]. The applied pressure is an important factor in initiating the α to ω-phase transformation since it is necessary to apply a pressure greater than a critical value of ~2 GPa in order to produce this transformation: thus, an earlier report demonstrated that the fraction of the ω-phase increased both with increasing pressure from 3 to 6 GPa and with increasing shear strain [6]. In practice, it appears that this straining facilitates, but does not initiate, the phase transformation [9] so that a large shear strain is insufficient to produce a phase transformation in the absence of a high hydrostatic pressure [16].

The effects on the phase transformation of the processing temperature and initial grain size were investigated recently [10]. Initially, it was expected that decreasing the processing temperature may promote the formation of the ω-phase but, on the contrary, experiments showed that the fraction of the ω-phase decreased when the processing temperature was decreased from room temperature to cryogenic temperatures and this was related to the formation of smaller nanometer grain sizes when processing by HPT at cryogenic temperatures [10]. Thus, the ω-phase

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volume fraction decreased with decreasing average grain size and the ω-phase was absent when the average grain size was ~20 nm [10]. Another important parameter in the α to ω transformation is the role of any impurities in Ti. There is evidence that the presence of oxygen suppresses the α to ω-phase transformation because oxygen increases both the ω-phase energy relative to the α-phase and the energy barrier for the transformation [17]. As a consequence, the ω-phase was not detected in commercial purity Ti containing a relatively high oxygen content even after processing at 6.0 GPa [18].

Although numerous reports document the effects of impurities and processing parameters, such as pressure, numbers of rotations and processing temperature, on the occurrence of the α to ω-phase transformation in HPT, only very limited information is at present available on the effect of strain rate, and thus anvil rotation speed, on the nature of the phase transformation. Furthermore, most reports have focused on samples deformed at a conventional rotation speed of 1 rpm [8,10,16,18]. There is only a single report showing that the strain rate has little significance on the α to ω-phase transformation when using rotation speeds of 0.2 and 0.5 rpm [9]. Accordingly, the present investigation was initiated to address this deficiency by examining the effect of different HPT rotation speeds on the α to ω-phase transformation, the microstructural evolution and the mechanical properties of the processed material. As will be demonstrated, the results show that the rotation speed has a critical effect on the magnitude of the transformation and especially on the total volume fraction of the ω-phase.

2. Experimental material and procedures

The experiments were conducted on commercial purity (CP) grade 2 titanium (99.2% purity) that was annealed for 2 h at 972 K under an Ar-controlled atmosphere to give an α-phase microstructure with an average grain size of ~65 μm. Titanium disks with thicknesses of ~0.8 mm and diameters of 10 mm were processed by HPT at room temperature under an applied pressure of $P=5.0$ GPa using rotation speeds of 0.5, 1 and 2 rpm and rotations through totals of 1, 5 and 10 revolutions under quasi-constrained conditions [19,20]. A K-type (chromel–alumel) thermocouple was inserted in a hole at the back of the upper anvil and this was used to record the temperature of the upper anvil at a position ~10 mm from the HPT sample by making a small vertical hole in the centre of the upper anvil as described in an earlier report [21].

After HPT, some samples processed for 10 turns at 0.5 rpm rotation speed were annealed at temperatures of 423 or 473 K for 15 min under an Ar-controlled atmosphere. Each HPT disk, both after HPT processing and after HPT processing and annealing, was polished to a mirror-like quality and hardness measurements were taken using a Vickers microhardness tester with a load of 500 gf and dwell times of 10 s The average microhardness values, Hv, were measured along randomly selected diameters on each disk with the measurements taken at intervals of ~0.5 mm and at every point the local value of Hv was obtained from an average of four separate hardness measurements. The transformation temperatures were measured by differential scanning calorimetry (DSC) using a Mettler-Toledo instrument with the analysis performed using non-isothermal (scanning) experiments upon heating at a scanning rate of ~10 K min^{-1} and covering a temperature range of 298–773 K. The phase constituents were determined using X-ray diffraction (XRD) (Rigaku SmartLab) employing Cu Kα radiation (wavelength $\lambda=0.154$ nm) at 45 kV and a tube current of 200 mA. The XRD measurements were performed over a 2θ range from 30° to 90° using a scanning step of 0.01° and a scanning speed of 2° min^{-1}. The analyses using DSC and XRD were conducted using sample areas with diameters of 3 mm located near the edges of the disks. A foil for transmission electron microscopy (TEM) was prepared using a focused ion beam (FIB) (Zeiss Nvision 40 FIB) method at 3 mm from the disk centre in the normal section of the disk so that the normal of the image lay in the shear direction. The TEM micrographs were obtained using a JEOL JEM-3010 microscope operating under an accelerating voltage of 300 kV.

Miniature tensile specimens were cut from near the edges of the HPT disks with gauge dimensions of $1.1\times1.0\times0.6$ mm^3 and the stress-strain curves were recorded under conditions of constant rate of cross-head displacement at room temperature using an initial strain rate of 1.0×10^{-3} s^{-1} with a Zwick universal testing machine. The stress-strain curves were plotted for each specimen to give the yield stress and ultimate tensile strength. Two samples were tested for each condition. The elongations were estimated by carefully measuring the gauge lengths before and after tensile testing using an optical microscope.

3. Experimental results

3.1. Microstructure and hardness after HPT processing

Fig. 1(a) shows the X-ray diffraction patterns at the edges of the disks of the CP-Ti after HPT processing for different rotation speeds, w, at $N=10$. Earlier results in a fully-annealed condition without HPT processing showed that, as anticipated, the initial microstructure consisted only of the α-phase Ti with a main peak of $(10\text{–}10)_\alpha$. Inspection of the X-ray patterns demonstrates that ω-phase peaks are clearly visible in Fig. 1(a) with the main peak position corresponding to the $(11\text{–}20)_\omega$ planes at $2\theta\approx39°$ thereby confirming the occurrence of the α to ω-phase transformation. The results indicate that the intensities of the ω-phase peaks decrease with increasing rotation speed such that the intensities of these peaks are essentially negligible when using the fastest rotation rate of 2 rpm. The volume fractions of the ω-phase after HPT processing were calculated using standard procedures [22] and the results are shown in the last column of Table 1. Estimates of the ω-phase volume fractions based on the XRD results are summarized in Table 1 and show changes from ~65% for 0.5 rpm to ~27% and ~12% at rotation speeds of 1 and 2 rpm, respectively.

The results for the corresponding Vickers microhardness measurements are shown in Fig. 1(b) for the same number of 10 rotations after processing through different rotation speeds with the average values of Hv plotted along each disk diameter and with the lower dashed line at $Hv\approx170$ corresponding to the initial hardness in the fully-annealed condition. The microhardness results in Fig. 1(b) show that the hardness decreases significantly with increasing rotation speed. Thus, the hardness values at the edges of the disks after 10 turns decrease from $Hv\approx370$ to ~304 and ~294 when the rotation rate increases from 0.5 to 1 and 2 rpm, respectively. These hardness measurements are consistent with the measured equilibrium volume fractions of the ω-phase since this phase is harder than the α-phase [1].

The microstructure and an appropriate selected area electron diffraction (SAED) pattern are shown in Fig. 2 at a region ~3 mm from disk centre after HPT through 10 turns at 0.5 rpm. This microstructure consists of an array of ultrafine equiaxed grains having an average size of ~70 nm. This is typical of materials prepared using severe plastic deformation (SPD) techniques and it is consistent with the presence of a large volume of high-energy non-equilibrium boundaries [23,24]. Strain contrast is visible in many of these small grains which is associated with the presence of dislocations. In addition, the arrangement of the diffraction spots in semi-continuous circles in the SAED pattern confirms that