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Using heat treatments, high-pressure torsion and post-deformation annealing to optimize the properties of Ti-6Al-4V alloys

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ABSTRACT

Experiments were conducted to investigate the processing parameters that may be used to optimize the properties of Ti-6Al-4V alloys. The alloy was initially subjected to two different heat treatments leading to the formation of martensitic α' and lamellar $\alpha+\beta$ microstructures and then both materials were processed by high-pressure torsion (HPT) for 10 turns at room temperature. This gave significant grain refinement to the nanometer range in both conditions and the occurrence of an allotropic *hcp* to *fcc* phase transformation in the martensitic alloy. These nanostructured alloys were subjected to post-deformation annealing (PDA) at temperatures in the range of 473–1023 K. The results show the hardness increases slightly to 773 K due to $\alpha'+fcc \rightarrow \alpha+\beta+fcc$ and $\alpha \rightarrow \alpha+\beta$ phase transformations in the martensitic α' and lamellar $\alpha+\beta$ alloys and then decreases up to 1023 K due to recrystallization and grain growth. An optimum property of a very high yield strength (~1120 MPa) and ultimate tensile strength (~1200 MPa), together with excellent ductility (elongation to failure of ~26%), was achieved in the Ti-6Al-4V martensitic alloy processed by a combination of HPT followed by PDA at 873 K for 60 min.

1. Introduction

Ti-6Al-4V is the titanium alloy used most frequently in commercial and industrial applications. This alloy possesses low density, high strength, good toughness, a general corrosion resistance, excellent bio-compatibility and very good high temperature properties and formability. Therefore, it is used widely for many applications in aerospace and chemical engineering, for power generation and as an implant material in medicine [1–4]. In equilibrium, the alloy consists mainly of an α-phase (*hcp*) with some β-phase (*bcc*) at room temperature. The existence of the α/β transformation means that it is possible to achieve a variety of microstructures and property combinations in the alloy through thermomechanical processing thereby permitting the development of properties for specific applications [5].

Depending upon the cooling rate and the prior heat treatment, the microstructure of the alloy may be divided into several types. For very slow cooling rates from high within the α+β region or above the β-transus temperature (1263 ± 20 K), the β-phase primarily transforms into a globular type of α. Increasing the cooling rate accelerates the α nucleation rate in the β grain boundaries thereby enhancing the formation and growth of α platelets into prior β grains. The β-phase fully or partly transforms into a martensitic type during high cooling rates and this martensite exists in two different forms, α' (*hcp*) and α'' (orthorhombic) [6–8]. The type and amount of α' and α'' formed on quenching depends upon the chemical composition (vanadium enrichment) of the β phase that exists at the temperature prior to quenching [9]. Thus, by controlling the phase transformations occurring during thermal processing, particularly during cooling from elevated temperatures, an optimal mechanical performance may be achieved in the α+β Ti alloys.

It is now well established that grain refining is a very effective procedure for improving the mechanical properties of materials. Furthermore, processing through the application of severe plastic deformation (SPD) provides an opportunity for reducing the grain size to the submicrometer or even the nanometer level [10,11]. In practice, high-pressure torsion (HPT) is an excellent processing method because it produces materials with exceptionally small grain sizes and with a large fraction of grain boundaries having high angles of misorientation [12–14]. In 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]. Generally, nanostructured metals and

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alloys processed by HPT exhibit high strength but their ductility is limited because they have both a low rate of strain hardening and a low strain rate sensitivity [16–18]. Accordingly, post-deformation annealing (PDA) is often an important tool for improving the ductility both for titanium [19] and for other materials [20,21]. Nevertheless, annealing at excessively high temperatures may lead to an acceleration in recovery and a reduction in hardness so that it is important to select an appropriate annealing condition to produce both good ductility and high strength [22].

Several reports are now available describing the processing of the Ti-6Al-4V alloy by HPT [23–29] but no systematic investigations are available describing the effect of the initial microstructure on the structural evolution and on the mechanical behavior of the alloy after PDA. Furthermore, there has been no attempt to date to optimize the properties of Ti-6Al-4V alloys using selected combinations of HPT and PDA. Accordingly, the present investigation was initiated to address these deficiencies by subjecting a Ti-6Al-4V alloy to two different heat treatments in order to produce significantly different initial microstructures and then evaluating the significance of these initial conditions on the subsequent microstructural evolution and the mechanical properties attained within the alloy after HPT and PDA.

2. Experimental material and procedures

The experiments were conducted using a Ti-6Al-4V alloy where the composition is given in wt%. Prior to processing by HPT, the as-recieved material was divided into two separate batches and these batches were treated using two different heat treatments. The first batch was subjected to a solution annealing at 1273 K for 30 min followed by water quenching to obtain a martensitic microstructure. The second batch was solution annealed at 1223 K for 45 min followed by air cooling to room temperature and then a stress relief anneal at 873 K for 3 h followed by furnace cooling to obtain a lamellar ($\alpha+\beta$) microstructure. Henceforth, the materials processed by these two procedures are denoted as α' (martensitic) and $\alpha+\beta$ (lamellar) alloys, respectively. Following the initial heat treatments, disks with thicknesses of ~0.8 mm and diameters of 10 mm were processed by HPT at room temperature under an applied pressure of $P = 6.0$ GPa using a rotation speed of 1 rpm and rotations through totals of 10 revolutions under quasi-constrained conditions [30]. These disks were then processed by PDA at temperatures from 473 to 1023 K for 60 min.

Each processed disk was polished to a mirror-like quality and hardness measurements were taken using a Vickers microhardness tester with a load of 500 gf and a dwell time of 10 s. The average microhardness values, Hv, were measured at 3 mm from the disk centres and at every point the local value of Hv was obtained from an average of five separate hardness measurements. The phase constituents were determined using X-ray diffraction (XRD) employing Cu Kα radiation (wavelength $\lambda = 0.154$ nm) at 45 kV with 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 analysis using XRD was conducted over sample areas with diameters of 3 mm located near the edges of the disks. Microstructural characterizations were carried out using optical microscopy (OM), scanning electron microscopy (SEM) and transmission electron microscopy (TEM). Foils for TEM were prepared after HPT processing using a focused ion beam (FIB) Zeiss Nvision 40 FIB facility at 3 mm from the disk centres in the normal sections of the disks so that the normals of the images lay in the shear direction. The TEM micrographs were obtained using a JEOL JEM-3010 microscope operating under an accelerating voltage of 300 kV.

Two miniature tensile specimens with gauge dimensions of $1.1 \times 1.0 \times 0.6$ mm^3 were cut from symmetric off-centre positions near the edges of each disk using electro-discharge machining. The mechanical properties were examined in the martensitic and lamellar alloys after HPT followed by PDA at 773–1023 K for 60 min. Stress-strain curves were recorded using an initial strain rate of 1.0×10^{-3} s^{-1} with a Zwick universal testing machine. Two samples were tested for each condition. The stress-strain curves were plotted for each specimen and the ultimate tensile strengths were derived directly from the curves. The elongations were also estimated by carefully measuring the gauge lengths before and after tensile testing using an optical microscope.

3. Experimental results

3.1. Microstructures of the Ti-6Al-4V alloy before and after HPT processing

The microstructure of the alloy after solution annealing at 1273 K for 30 min followed by water quenching is shown in Fig. 1(a). The coarse prior β grains, with an average size of ~500 μm, were fully transformed to martensite (α') and the microstructure shows martensitic laths distributed throughout the microstructure having different orientations and with an average lath thickness of ~0.8 μm. Fig. 1(b) shows the microstructure after solution annealing at 1223 K for 45 min followed by air cooling to room temperature and then stress relief annealing at 873 K for 3 h followed by furnace cooling. The microstructure reveals a lamellar $\alpha+\beta$ structure having different orientations. In the lamellar $\alpha+\beta$ the retained β-phase lies between the α platelets so that the resulting microstructure consists of average prior-β grains of ~500 μm, colony size parallel-oriented α-phase lamellae of ~200 μm and an average α lamellae lath width of ~1.5 μm.

TEM micrographs and selected area electron diffraction (SAED) patterns of the HPT processed α' and $\alpha+\beta$ alloys are shown in Fig. 2(a) and (b), respectively, taken at regions of ~3 mm from the disk centres. The microstructures of both conditions are highly strained from the HPT processing with complex non-uniform contrasts because of the presence of high densities of lattice defects. These images show that many grains have an irregular shape with sharp corners while many of the grain boundaries are wavy and ill-defined. Some equiaxed grains appear to form from the fragmentation of elongated grains and these and other similar images suggest that the average sizes of the separate fragments of structures are ~30 and ~40 nm for the processed α' and $\alpha+\beta$ alloys, respectively. The diffraction patterns show numerous spots arranged along circles indicating the presence of crystallites separated by high-angle grain boundaries (HAGBs). The appearance of significant streaking of the diffraction spots denotes the presence of high internal stresses and elastic distortions of the crystal lattice. All of these characteristics are typical of materials prepared using SPD techniques and they are consistent with the presence of a large volume of high-energy non-equilibrium grain boundaries [31–34]. The observed diffraction patterns correspond to the α/α'-phase and the *fcc* phase for the processed α' samples but only to the α-phase for the processed $\alpha+\beta$ samples. Thus, in the latter material there is no evidence for the presence of a β-phase and this demonstrates the dissolution of the β-phase during the SPD-processing.

Further observations of the microstructure of the HPT-processed α' alloy by TEM and the relevant SAED pattern are given in Fig. 3(a). The dark-field image in Fig. 3(b) is based on the spot corresponding to the *fcc* phase marked by a circle in the diffraction pattern in Fig. 3(a) and it shows the distribution of this phase within the *hcp*-phase matrix and suggests the occurrence of twinning in *fcc*.

3.2. Mechanical properties after PDA

Figure 4 shows the measured values of the microhardness for