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Mechanical behavior and microstructure properties of titanium powder consolidated by high-pressure torsion

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ABSTRACT

Research was conducted to investigate the potential for consolidating titanium powder using high-pressure torsion (HPT) at room temperature. The nanostructured samples processed by HPT were characterized by X-ray diffraction (XRD) and transmission electron microscopy (TEM). The results show there is a significant refinement of the Ti powder and it consolidates into bulk nanostructured titanium with a mean grain size estimated by TEM as ~200–300 nm and a mean crystallite size measured by XRD as ~20–30 nm. Microhardness measurements and tensile testing show high strength and low ductility after consolidation under a pressure of 6.0 GPa for 5 revolutions. Additional short annealing at a temperature of 300 °C for 10 min leads to a significant enhancement in ductility while maintaining the high strength.

1. Introduction

Processing bulk nanostructured materials [1] through the application of severe plastic deformation (SPD) [2], using techniques such as equal-channel angular pressing (ECAP) [3] and high-pressure torsion (HPT) [4], is now recognized as an important tool for achieving grain refinement. However, less attention has been paid to the very powerful ability of HPT to consolidate metallic powders [5–9], as well as composites [10–14], amorphous compounds [15–19], machining chips [20–22] and even ceramic powders [23].

Titanium and titanium alloys are excellent metals for structural and bioengineering applications due to their high corrosion resistance and biocompatibility [24–28]. Since pure Ti has low strength, alloying with other elements is generally used to increase its mechanical strength but this may lead to degradation in biocompatibility. To solve this problem, processing by SPD may be used to improve the mechanical properties of pure titanium. One way deserving special attention is to produce bulk nanostructured titanium through the cold consolidation of titanium powder by means of HPT. In order to significantly increase the strength, there is a report of ball-milled (BM) powder of Ti which was consolidated by HPT to form bulk nanocrystalline disks [29]. A relative high density (99.9%) and a high tensile strength were achieved after cold consolidation from ball-milled titanium powder and an additional sample was also consolidated from a non-BM Ti powder. Although the published information is limited, it appears from the report that this specimen exhibited not only a high strength but also a reasonable level of ductility. Based on these results, research was initiated specifically to examine the mechanical properties and the microstructural evolution of HPT-consolidated titanium powder.

2. Experimental materials and procedures

Experiments were conducted on commercial purity (CP) titanium powder (99.5 wt%) having a mesh size of ~150 μm obtained from Goodfellow Ltd., Cambridge, UK. The chemical composition of the Ti powder was (in ppm) C < 100, chlorides 1700, Fe 200, N < 100, O

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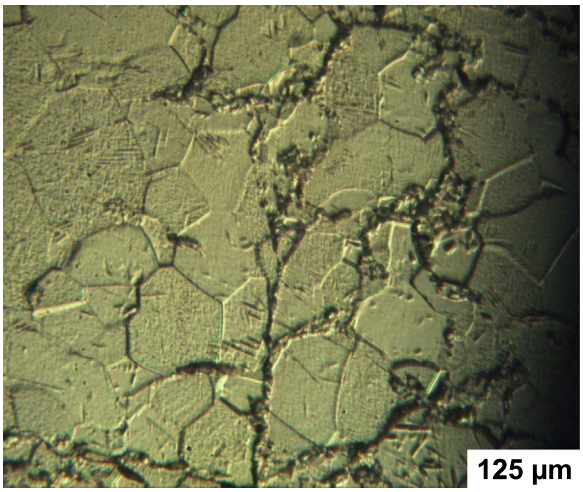

Fig. 1. Optical image of HPT consolidated titanium powder (P=6.0 GPa, N=0, loading time=1 min+annealing at 700 °C for 40 min) (d =85.1 ± 29.5 μm).

1000. The powder was pre-compacted into disk-shaped tablets with diameters of 10 mm and thicknesses of approximately 1.5 mm. These tablets were further consolidated to thicknesses of ~0.85 mm by HPT under an applied pressure of 6.0 GPa for 1 min without any anvil rotation so that no torsional deformation was applied. These consolidated powder Ti samples are henceforth designated N0 samples. Some N0 samples were selected for annealing in an argon atmosphere at 700 °C for 40 min and they were used as a reference to show the initial coarse-grained material of powder Ti before HPT processing. Fig. 1 shows an optical image of the microstructure of the Ti powder specimen processed by simple compression and then annealed at 700 °C for 40 min. The mean grain size was measured by the intercept method using ImageJ™ [30] and this gave 85.1 ± 29.5 μm. The remaining N0 samples were then further processed by HPT at room temperature (RT) under an applied pressure, P, of 6.0 GPa, using a rotation speed of 1 rpm and torsional straining through numbers of revolutions, N, of 5 turns and then annealed for 10 min in air at consecutive temperatures of 250, 300, 450, 650 and 750 °C.

All disks were polished to a mirror-like quality and hardness measurements were taken using a Vickers micro-hardness tester with a load of 500 gf and a dwell time of 10 s. The average microhardness values, Hv, were measured along randomly selected diameters on each disk. These measurements were taken at intervals of 0.5 mm and at every point the local value of Hv was obtained from the average of four separate hardness values.

The analyses by X-ray diffraction (XRD) were undertaken using a Bruker D2 Phaser instrument with Cu radiation ($K_{\alpha 1}$=1.54060 Å) and a Ni monochromator with a 1D LYNXEYE detector. The scan step was 0.02° and the delay time was 2.5 s. A Rietveld analysis using MAUD software [31] was performed in order to monitor the phase composition, the lattice parameters, a, c, the microcrystallite size, d, and the microstrain $< \varepsilon^2 >^{1/2}$.

Transmission electron microscopy (TEM) (JEOL) was employed to characterize the fine microstructure of nanostructured titanium after processing by HPT. Foils for TEM studies were cut out by electro-discharge machining (EDM). After mechanical thinning to about 100 μm, they were subjected to electrolytic polishing using a "Tenupol-5" set. Electropolishing was conducted using chemical solution consisting of 5% perchloric acid, 35% butanol and 60% methanol in the temperature range of −20 to −35 °C. The grain size distribution was obtained by examining at least 5 dark field images for each sample. Since many low-angle boundaries were not well-defined in this analysis and hence these boundaries were discounted, the grains used for detailed analysis are primarily those separated by high-angle misorientations. The mean grain size was defined as the diameter of a circle with the same area as a grain in the dark field image, so that $d=(4 \cdot S/\pi)^{1/2}$. The area of the individual grains was measured using the ImageJ software.

For mechanical testing, two miniature tensile specimens were cut from symmetric off-centre positions in each disk near the edges using EDM. This specimen configuration was described earlier and the gauge dimensions were 1.1×1.0×0.6 mm^3 [32].

The mechanical properties were examined at room temperature and at elevated temperatures of 250 and 300 °C. All specimens were heated rapidly to the testing temperature, typically in a time of ~5–10 min, and then held for 10 min to reach a uniform temperature prior to testing. Stress-strain curves were recorded using an initial strain rate of 1.0×10^{-3} s^{-1}. The stress-strain curves were plotted for each specimen and the yield stress (YS) and the ultimate tensile strength (UTS) were then measured from each curve. At least two samples were tested for each condition. All elongations were carefully calculated by measuring the gauge lengths before and after tensile testing using an optical microscope.

3. Experimental results

3.1. Microhardness

Fig. 2 shows results for the microhardness measurements. The microhardness distribution along the diameter of the HPT-processed Ti powder compressed to a disk is given in Fig. 2a. It is apparent that after pure compression without torsional straining (P=6.0 GPa, N=0 and loading time of 1 min) and annealing at 700 °C for 40 min the microhardness distribution is highly inhomogeneous along the disk diameter. This inhomogeneity is so large that there is a difference in Hv of up to more than 100%. By contrast, after HPT processing for 5 whole revolutions the microhardness is essentially fully homogeneous and equal to Hv≈300. This latter value is consistent with earlier results [27] where the microhardness for consolidated BM titanium powder was Hv≈350. Fig. 2b shows the evolution in the disk consolidated for 5 whole revolutions and annealed for 10 min at consecutive temperatures of 250, 300, 450, 650 and 700 °C. This plot shows that the HPT-consolidated Ti powder is thermostable in short annealing but there is a drop of about 50 Hv in the microhardness value between 450 and 650 °C.

3.2. XRD results

The crystallite size and microstrain were measured by XRD using Rietveld refinement in the MAUD software. Fig. 3 shows a typical example of the analysis for an HPT-consolidated disk at P=6.0 GPa and N=5 turns. Thus, the material is single alpha-phase titanium and neither the omega phase nor titanium oxides were detected or if these phases exist then they are below the detectable level of the XRD analysis. This is not consistent with the earlier result using BM Ti powder [29] where an omega phase was detected in a sample consolidated at P=6.0 GPa through 4 whole revolutions. As noted earlier [33], the alpha-omega phase transformation represents the accommodation process during HPT processing of metals of Group VI of the periodic table. Obviously in the nanocrystalline BM Ti powder used earlier [29] the grain size was already in the nanometer range so that any dislocation movement was difficult. By contrast, in the present investigation using Ti powder with large particles (~150 μm mesh), dislocation slip was an active deformation process.

The XRD results on the evolution of the crystallite size and the microstrain of the samples of HPT-consolidated Ti powder are shown in Fig. 4a, where HPT_0 is assigned for the sample compressed without torsion (HPT, P=6.0 GPa. N =0, loading for 1 min) and annealed at 700 °C for 40 min and HPT_5 corresponds to the disk of Ti powder consolidated at room temperature for 5 whole revolutions at a load of 6.0 GPa. The microstrain was obtained from the MAUD software which