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Thermal stability and mechanical properties of HPT-processed CP-Ti

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Abstract. A grade 2 commercial pure titanium was processed by high-pressure torsion (HPT) at room temperature to 20 turns. X-ray analysis showed that an ω phase formed during HPT processing but disappeared immediately during 10 minutes post-HPT short-term annealing even at a very low temperature of 473 K. The thermal stability of HPT-processed microstructural evolution was studied by electron backscatter diffraction (EBSD). After short-term annealing at lower temperatures (473 and 673 K), the disc centre had relative higher hardness value than the edge area, and it was found that the centre retained the feature of deformed microstructure whereas the edge showed recovery / recrystallization of the microstructure. Short-term annealing at higher temperatures (873 and 973 K) led to almost uniformly distributed hardness and microstructures in the disc centre and edge areas. Grain structures and hardness measurements indicate complete recrystallization occurred at 873 K.

Keywords: Electron backscatter diffraction; Grain size; Hardness; High-pressure torsion; Titanium; x-ray.

1. Introduction

Commercial purity titanium (CP-Ti) is widely used for medical implants in trauma surgery, orthopedic and oral medicine due to its good biocompatibility [1]. Material strength is an important consideration for practical bio-implantation. However, the mechanical strength of CP-Ti is relatively low compared to other metals such as Ti-6Al-4V used in medical device. It is now well established that equal-channel angular pressing (ECAP) and high-pressure torsion (HPT) are effective techniques in generating ultrafine-grained structure / nanocrystalline structure, and can improve the mechanical strength of CP-Ti [2-12]. CP-Ti normally needs to be heated to high temperatures between 473 and 773 K for ECAP processing [8], whereas HPT can easily process CP-Ti at room temperature [9-12]. In practice, HPT is more effective in refining the grain structure and enhancing the strength.

To benefit from the high strength and the nanocrystalline microstructure developed during HPT processing for use in practical environments, it is essential the materials have the ability to maintain

this nanocrystalline microstructure over a certain time. Therefore, the current research on the thermal stability and mechanical properties was initiated to investigate the microstructure and hardness evolution of CP-Ti during short-term annealing after HPT processing.

2. Experimental material and procedures

The experimental material was grade 2 CP-Ti in the form of a round bar with diameter of 12.7 mm, supplied by Titanium Industries UK Ltd (Birmingham, UK). The as-received material was first annealed at 973 K for 2 h in a vacuum furnace to obtain a microstructure with average grain size of ~65 μm, and this is henceforth designated the as-annealed state (N0). The rod was then machined to a diameter of 9.9 mm, sliced into discs with thicknesses of ~1.1 mm and ground with abrasive papers to final thicknesses of ~0.8 mm.

All discs were processed by HPT at room temperature through total numbers of turns, N, of 1, 5, 10 and 20 using an imposed pressure of 6.0 GPa and a rotational speed of 1 rpm. The HPT was conducted under quasi-constrained conditions where there is a small outflow of material around the periphery of the disc during the processing operation. To investigate the thermo-stability of the HPT-processed discs, samples processed through 10 turns were selected for 10 minutes short-term annealing at temperatures of 473, 673, 873 and 973 K.

The microhardness was measured on HPT-processed and post-HPT annealing samples using a FM300 hardness tester equipped with a Vickers indenter with a load of 500 gf and a dwell time of 15 s. A JEOL1200 transmission electron microscope (TEM) was used to characterize the deformation microstructure after 10 turns of HPT processing at higher magnifications. The centre of the TEM foil was 3 mm away from the centre of the HPT sample. The grain structures near the edge of disc after post-HPT annealing were examined by electron backscattered diffraction (EBSD) using a JSM6500F thermal field emission scanning electron microscope (SEM). The EBSD patterns were collected using a step size of 0.15, 0.21, 0.52, 0.7 μm for 473, 673, 873 and 973 K for post-HPT annealing samples respectively.

The microstructures of the HPT-processed and post-HPT annealing samples were also evaluated using a Bruker D2 Phaser X-ray diffractometer equipped with a Cu target using Cu Kα (wavelength λ = 0.15406 nm) radiation and a Ni monochromator with a 1D LYNXEYE detector. The whole disc surface was analysed by X-ray diffraction (XRD) and θ - 2θ scans were conducted from 2θ = 25° to 100° with scan steps of 0.02° to record the XRD patterns.

3. Experimental results

Figure 1 shows the variation of the microhardness along the diameter of the discs processed to 5, 10 and 20 turns. The as-annealed material had a microhardness of ~157 Hv. After 5 turns, the disc centre had a lower hardness than at the edge area, which is consistent with a rigid body assumption that strain imposed on the disc has a linear dependence on the distance from the disc centre and the strain is a maximum value at the outer edge of the disc [13]. After 10 turns, the hardness along the disc diameter tended to distribute more homogenously although there were some jumps in the local hardness values. The highest hardness values were achieved in the 20 turns sample, with reasonable homogeneity along the disc diameter.

The microstructure of CP-Ti after 10 turns HPT processing is shown in the TEM image of Fig. 2. At the half-radius of the disc, the microstructure consists of equiaxed grains with average grain size of ~70 nm. Therefore, HPT produced significant grain refinement in CP-Ti with an average grain size reduced from ~65 μm for as-annealed to ~70 nm after 10 turns of HPT processing. The ring-like selective area diffraction pattern (SADP) demonstrates that the microstructures have boundaries mainly with high angles of misorientation. The strengthening effect after HPT processing is obvious and significant.

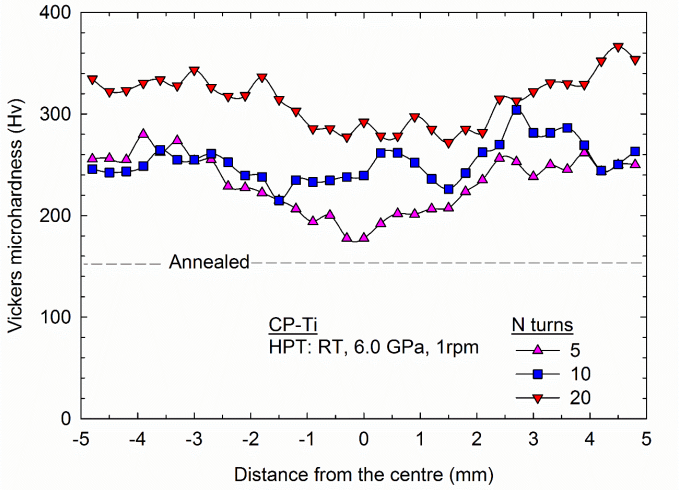

Fig. 1. Distribution of Vickers microhardness, Hv, along the diameter of disc processed by HPT through 5, 10, 20 turns.

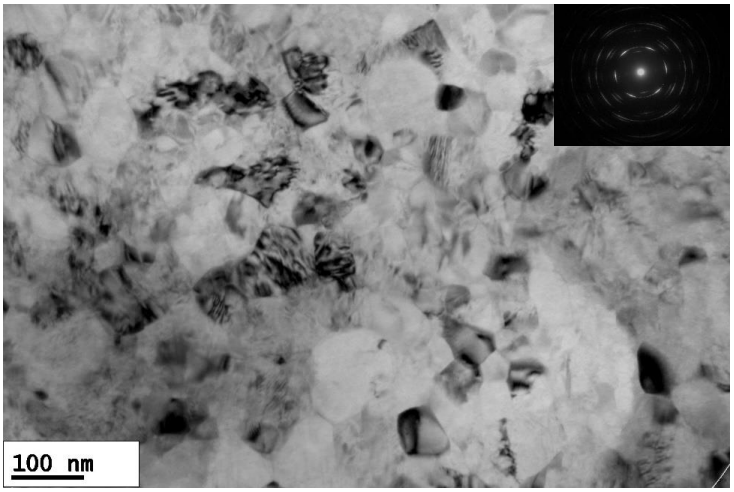

Fig. 2. Microstructure in CP-Ti after 10 turns in disc half-radius area.

Figure 3 shows the x-ray patterns of CP-Ti in the as-annealed (N0) condition and after HPT processing to different number of rotations. In the as-annealed condition, there are only α peaks and no ω phase was detected. After HPT processing to 1 turn, ω phase peaks become visible with main peak $(11\text{-}20)_\omega$ at $2\theta \approx 39°$ to replace the α peak $(0002)_\alpha$ at $2\theta \approx 38.42°$. As deformation proceeded to 5, 10 and 20 turns, three more ω peaks at $2\theta \approx 56.5°$, 84° and 97° appeared. This result confirms an α to ω phase transformation due to HPT deformation and indicates the intensity of the ω phase increases with increasing numbers of rotations.

The hardness variations along the disc diameter during 10 minutes post-HPT short-term annealing on 10 turns samples are shown in Fig. 4. After short-term annealing at lower temperatures of 473 and 673 K, the disc centre had relative higher hardness values than the edge area, and this indicates that the disc edge area had more rapid recovery / recrystallization than the centre area. Short-term annealing at higher temperatures of 873 and 973 K gave almost uniformly distributed hardness in disc centre and edge areas, and also the hardness at 873 and 973 K were almost at the same level. Overall, the hardness values in the disc edge area decreased with increasing temperature from 473 to 873 K. This indicates that HPT-processed CP-Ti is very sensitive to the applied temperatures.

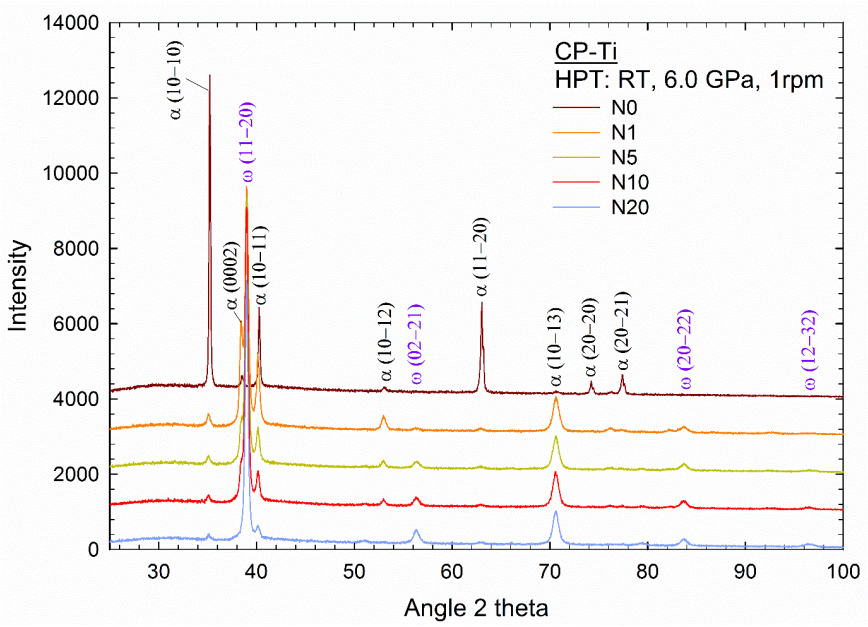

Fig. 3. X-ray patterns of CP-Ti in both the as-annealed (N0) and after HPT processing through different numbers of rotations.

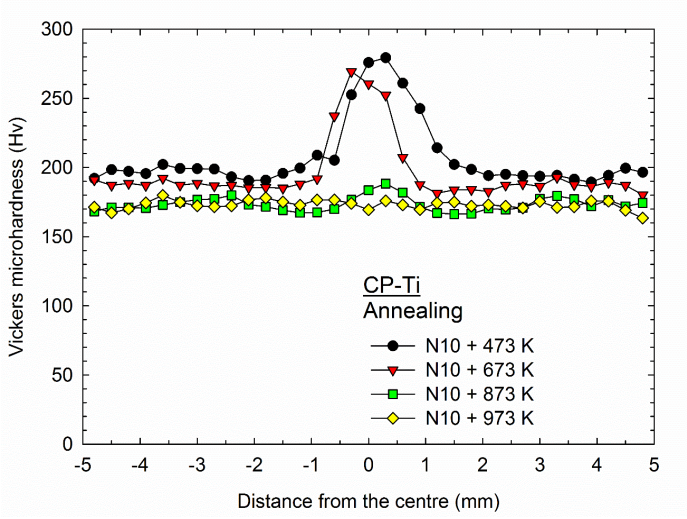

Fig. 4. Distribution of Vickers microhardness, Hv, along the diameter of disc during post-HPT short-term annealing at different temperatures.

Figure 5 shows the microstructure development in disc edge area during post-HPT short-term annealing at 473, 673, 873 and 973 K. At the lowest temperature of 473 K, the microstructure should have a high dislocation density and it is difficult to obtain a clear EBSD pattern, whereas at 673, 873 and 973 K the average grain sizes were 3.1, 3.9 and 9.7 μm, respectively. The grain structures coarsen rapidly from ~70 nm after 10 turns to the microns level after 10 minutes short-term annealing.

Figure 6 shows the x-ray patterns of CP-Ti in 10 turns samples during post-HPT short-term annealing. Upon heating to 473 K for 10 minutes, the ω phase peak $(11\text{-}20)_\omega$ is replaced by α phase peak $(0002)_\alpha$ at $2\theta \approx 38.42°$. Meanwhile the other three ω phase peaks disappeared at $2\theta \approx 56.5°$, 84° and 97°. At higher temperatures of 673, 873 and 973 K, no ω peak was detected. This confirms the reverse ω to α phase transformation due to heating.

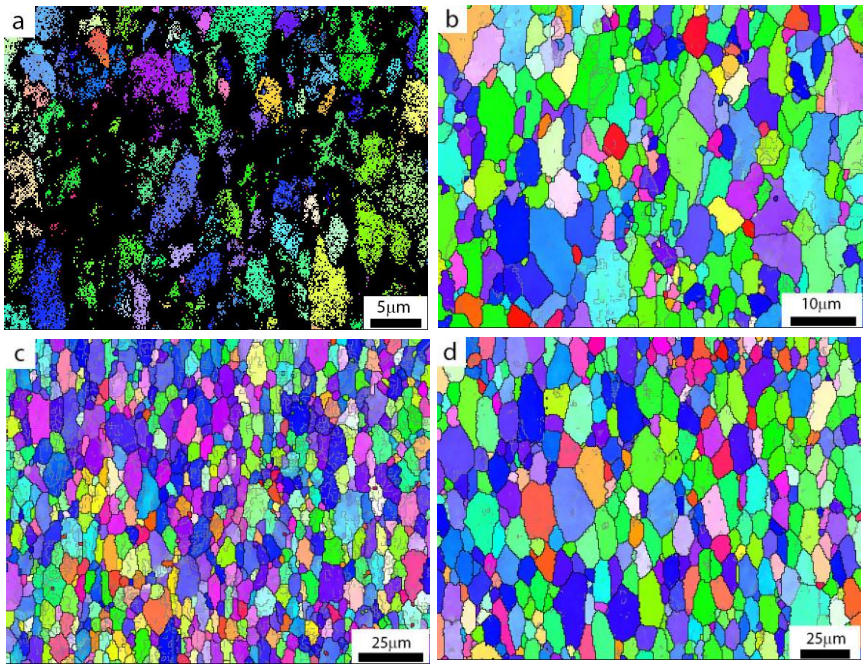

Fig. 5. Microstructure development during post-HPT short-term annealing at (a) 473, (b) 673, (c) 873, and (d) 973 K.

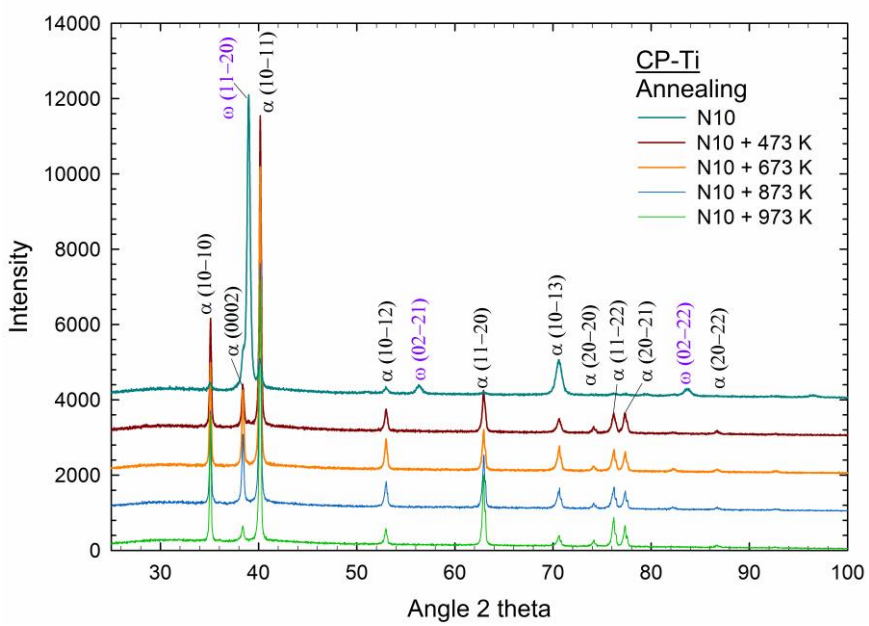

Fig. 6. X-ray patterns of CP-Ti in 10 turns samples during post-HPT short-term annealing.

4. Discussion

There are many reports on grain refinement and strength enhancement in HPT processed grade 2 CP-Ti [9-12]. Grain size and hardness measurement in the CP-Ti processed in this research confirmed the ability of HPT to achieve a nanostructure and to improve the material strength. HPT processing gave nanostructured grains with an average grain size of ~70 nm. However, after 10 minutes short-term annealing, the grain size changed to 3.1, 3.9 and 9.7 μm at 673, 873 and 973 K, respectively. The post-HPT short-term annealing behaviour indicates HPT-processed CP-Ti is temperature sensitive, with

significant grain growth in short holding times. This may be related to the high dislocation density introduced by heavy shear deformation which places the material in a high stress and nonequilibrium state with therefore a high tendency for recovery and recrystallization.

Not all grade 2 CP-Ti processed by HPT showed the ω phase [11], but there are other reports of the ω phase in HPT samples [10, 12]. This may be due to the fact that different materials are from different suppliers with different composition control and this affects the α to ω phase transformation during HPT. It was found that the pressure and rotation speed during HPT processing affects the amount of ω phase transformation [12]. In this research, HPT was carried out at a constant pressure 6.0 GPa and using a constant rotation speed of 1 rpm, and Fig. 3 shows the amount of ω phase transformation increased with increasing numbers of rotations. This confirms that the α to ω phase transformation during HPT is deformation-induced. There is a report that the high-pressure ω phase exhibits a reverse phase transformation upon heating [14]. The x-ray patterns from short term annealing samples in Fig. 6 shows the reverse ω to α phase transformation at a very low temperature of 473 K for 10 minutes. This reverse ω to α phase transformation means that the short-term annealed material loses the strengthening effect from the ω phase.

5. Summary and conclusions

1. Significant grain refinement and strength enhancement in CP-Ti were achieved through HPT processing.

2. HPT processed CP-Ti showed temperature sensitivity during short-term annealing. The material loses the nanostructure feature derived from 10 turns HPT processing and turns into a micron coarse grain structure during the short-term annealing.

3. A deformation induced α to ω phase transformation and heating-induced reverse ω to α phase transformation occurred in CP-Ti during HPT processing and post-HPT short-term annealing.

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