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Development of an ω-Phase in Grade 2 Titanium Processed by HPT at High Hydrostatic Pressure

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High-pressure torsion was used to process grade 2 titanium to 10 turns using different pressures up to a maximum of 8 GPa. X ray diffraction showed that the as-received material and the material processed at pressures of 2 GPa and 4 GPa were single phase but the material processed at 8 GPa contained both the h.c.p. and the simple hexagonal ω-phase. Nanoindentation was used to determine the mechanical behavior and the results show a pronounced increase in hardness due both to the severe plastic deformation processing and to the phase transformation at the highest pressure. A maximum hardness of ~4.7 GPa was attained.

Keywords: *commercial purity titanium, high-pressure torsion, phase transformation, severe plastic deformation*

1. Introduction

Titanium and its alloys have great potential for applications in the transportation industries because of their high strength to density ratio which allows the fabrication of structural components with low weight. Also, titanium can be used as a biomaterial due to its high resistance against corrosion and its biocompatibility. In both applications, transportation and medical, it is important to improve the strength of the material in order to satisfy any structural demand. Grain refinement and strain hardening are widely used as methods for improving the strength of metallic materials. These methods are particularly important for biomaterial applications because hardening by alloying may compromise the biocompatibility of the material.

Severe plastic deformation (SPD)[1] techniques are now recognized as effective processing operations to introduce grain refinement and strain hardening into metallic materials. These techniques have been used to process commercially pure titanium and significant improvements in strength were reported. For example, Equal-Channel Angular Pressing (ECAP)[2] has been used to process commercial purity (CP) Ti and the results show that ultrafine-grained structures are developed and the strength is significantly improved[3-13]. In addition, it was shown that SPD processing by ECAP improves the fatigue life of dental implants fabricated from CP-Ti[13].

However, even finer grain structures and improved strength may be developed in CP-Ti when processing by high-pressure torsion (HPT)[14]. The large hydrostatic stress developed during this processing operation prevents cracking and permits processing at room temperature. Thus grain sizes in the range of a few hundreds of nanometers have been reported in CP-Ti processed by HPT[15-21]. Also, titanium exhibits a phase transformation at high pressures into an hexagonal ω-phase[22,23]. It was reported that the ω-phase is denser despite the less-packed structure and this was attributed to the smaller atomic size[22]. Recent research shows that the ω-phase is formed in CP-Ti processed by HPT[16,17,20,21,24]. The high pressures developed in this process lead to a phase transformation and the imposed torsional strain stabilizes the hexagonal phase upon pressure release. It is known that the α to ω phase transition is favored with decreasing oxygen content in the alloy[16], with increasing pressure[17], with increasing the numbers of turns in HPT[16,17,20,21] and with decreasing the rotation rate[21]. However, although the phase transformation has been reported at pressures as low as 5 GPa in Ti-0.05% O [16] and in grade 2 Ti [21], the ω-phase was not detected in CP-Ti of similar grade even after processing by HPT using a pressure of 6 GPa for up to 10 turns[19]. This was attributed to the presence of different amounts of impurities compared to other investigations. It has been reported that larger pressures are needed for transformation in samples with higher impurity contents[23].

To date, most reports describe the processing of titanium up to a maximum pressure of 6 GPa in HPT. Thus, the objective of the present study was to evaluate the formation of the ω-phase in grade 2 CP-Ti processed by HPT at a higher pressure of 8 GPa. In addition, nanoindentation was used to evaluate the distribution of hardness and the strain rate sensitivity of the processed material.

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2. Experimental Material and Procedures

The material used in the present experiments was a CP-Ti of grade 2 (99.5% purity) provided by Global Metal Trading (UK) Ltd. (Birmingham, U.K.). The material was received as rods with 10 mm diameter. Discs with 0.9 mm thickness were cut from the as-received rods using spark erosion and these discs were ground using paper grids to a final thickness of ~0.8 mm.

The material was processed by HPT using a quasi-constrained facility[25,26] in which each anvil contains a shallow circular depression of ~0.25 mm depth and ~10 mm diameter. The discs were placed inside the depression in the bottom anvil for processing. The compression load in the HPT equipment was varied in order to provide different levels of nominal stresses during the processing operation. Thus, samples were processed by 10 turns of HPT using the different pressures of 2, 4 and 8 GPa. The samples were processed at 1 rpm (~0.1 rad/s) and, by considering a flow stress of 1000 MPa (= hardness/3), it was anticipated that the temperature rise on the sample during the HPT processing is ~50 ºC [27,28].

Samples of the as-received and the HPT-processed material were embedded in resin, ground and polished to a mirror-like finish on the upper circular surface. The phase constituents of the samples were determined by X-ray diffraction analysis with Cu (Kα) radiation using a scanning rate of 0.02 rad/s and $10^o < 2\theta < 120^o$. Nanoindentation measurements were carried out using a NanoTest Platform 2 nanoindenter (Micro Materials Ltd., Wrexham, UK). A maximum load of 200 mN and dwell time of 30 s were used in the tests. The loading rate was varied between 1 mN/s and 5 mN/s to estimate the material strain rate sensitivity. Clusters of 10 indentations were used to determine the average hardness at selected positions on the processed samples.

3. Experimental Results and Discussion

The X-ray diffraction patterns of the material before and after processing by HPT at different pressures are shown in Figure 1. The peaks observed in the unprocessed material correspond to the h.c.p. α-phase and also in the material processed by HPT under pressures of 2 and 4 GPa. The α-phase is also present in the material processed under a pressure of 8 GPa but additional peaks are present in the diffraction pattern and these correspond to the hexagonal ω-phase. Thus, the material processed under the largest pressure exhibits both crystalline structures. This result agrees with earlier reports as ω-phase formation and stabilization was reported after HPT processing at pressures of 5 GPa and larger and after multiple turns[16,17,20,21,24]. On the other hand, no phase transformation was observed in a similar material after HPT processing up to 10 turns at 6 GPa of pressure which suggested higher impurity content in the material[19]. Therefore, the present results show that a higher HPT pressure of 8 GPa favors a phase transformation in this material.

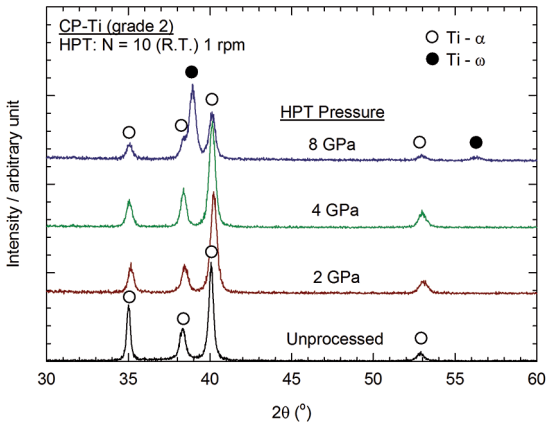

Figure 1: X ray diffraction patterns for the material before and after processing by HPT.

Figure 2 shows representative load vs. depth curves obtained from nanoindentation in the material before and after HPT processing. The indentations were carried out at the mid-radius position of the discs processed under different levels of pressure. There is a clear tendency of decreasing the depth at peak load in the material processed by HPT and this effect is more pronounced in the material processed at 8 GPa. It is also noted that the variation in depth during the 30 s period at peak load is larger in the unprocessed material which suggests a higher strain rate sensitivity in this condition.

The hardness was determined at different locations of the processed discs and the average values are plotted as a function of the distance from the center in Figure 3: the hardness of the unprocessed material is also shown as a dashed line for comparison. The data show that HPT processing under pressures of 2 and 4 GPa increase the hardness to values between 3.55 - 3.87 GPa and the distribution is fairly homogeneous throughout each disc. Processing under a larger hydrostatic pressure of 8 GPa leads to an increased hardness in the range of 4.10 - 4.71 GPa but the distribution is slightly less homogeneous. Specifically, lower hardness values are observed near the center and at the edge of the disc while the mid-radius is harder. The significant increase in hardness in the material processed under the maximum pressure is attributed to the formation of the ω-phase and this is in agreement with other reports. For example, a saturation hardness of ~2.6 GPa was observed in Ti processed at 2 GPa without a phase transformation while a saturation hardness of ~3.4 GPa was observed in the same material processed at 6 GPa which exhibited ω-phase formation[17].

The hardnesses near the center and at the edge of the disc of the material processed at 8 GPa are lower than at the mid-radius position but nevertheless they are higher than observed in the material processed at lower pressures. This suggests these areas may have experienced lower amounts of phase transformation. The area near the center of the disc is subjected to a lower shear strain and it is known that the ω-phase requires high plastic deformation to prevent

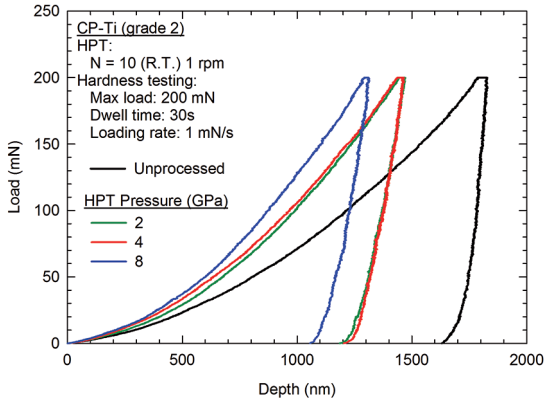

Figure 2: Load vs depth curves for the material before and after processing by HPT using different pressures.

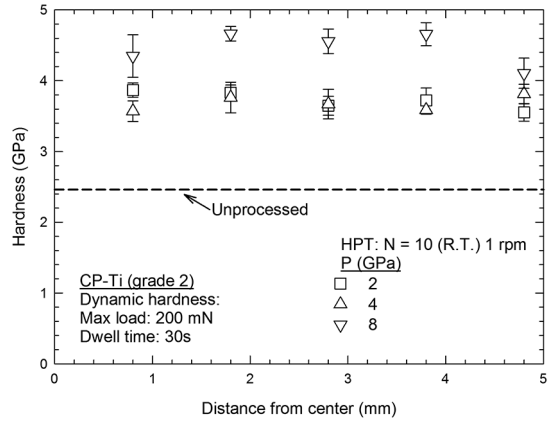

Figure 3: Hardness distribution on discs processed by HPT using different pressures.

any reverse transformation during unloading. This is in agreement with experimental evidence of an increasing volume fraction of ω-phase with increasing imposed strain during HPT[16,17,20,24] and a lower hardness distribution near the center of the disc even after 10 turns of HPT[16,17,20]. By contrast, the edge of the disc undergoes maximum plastic deformation and the reduced volume of ω-phase in this case is attributed to a lower hydrostatic stress. Thus, although the disc is subjected to a compressive load through the massive anvils in the HPT facility, finite element modeling has shown that the distribution of hydrostatic stress is not homogeneous and is larger near the center and lower near the edge[25]. Thus, the edge of the disc may be processed under a lower pressure and this effectively reduces the amount of the phase transformation. This is in agreement with the reduced volume fraction of the ω-phase detected by synchrotron characterization at the edges of discs[24].

Nanoindentation tests were also carried out using different loading rates. An early report showed that the strain rate, $\dot{\varepsilon}$, during hardness testing is given by $\dot{\varepsilon} = 1/h$ (dh/dt) where h is the displacement of the indenter and t is the time, and it is possible to estimate the strain rate sensitivity of the material by comparing its response at different loading rates[29]. Figure 4 shows representative curves for tests in the unprocessed material and the material processed at 8 GPa. Figure 4(a) shows the evolution of displacement as a function of time and it is possible to estimate the strain rate during the test. The strain rate variation was found to be proportional to the loading rate change. Figure 4(b) shows the load vs displacement curves and it confirms that the unprocessed material exhibits a different response to the different loading rates while the material processed by HPT at 8 GPa is essentially rate-insensitive. Calculations showed that the strain rate sensitivity in the unprocessed material is ~0.03 whereas it is ~0.0 in the material processed by HPT under all testing conditions. The strain rate sensitivity of the unprocessed material agrees with reports for pure titanium[30,31] although a slight lower value of 0.020 was also reported[32]. On the other hand, the lower values determined for the material processed by HPT differs from the trend of increasing strain rate sensitivity with refining grain structure which was reported elsewhere[31].

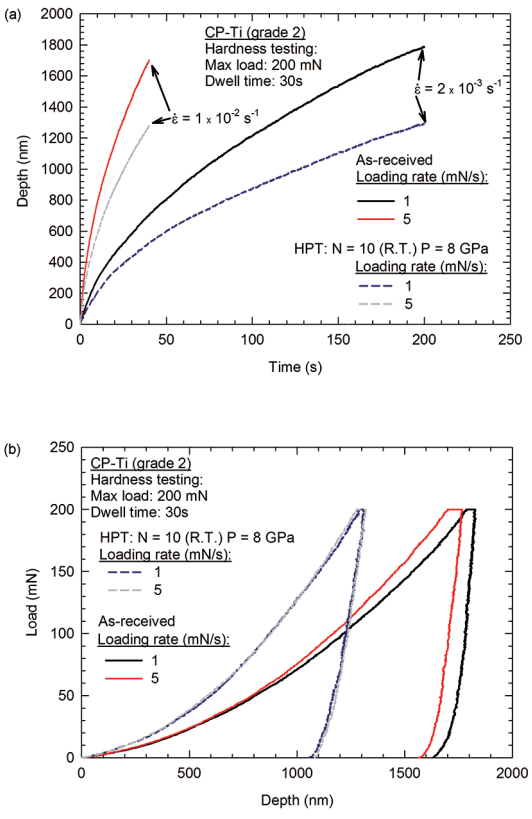

Figure 4: (a) Depth vs Time curves and (b) Load vs depth curves at different loading rates for the unprocessed material and the material processed by HPT at 8 GPa.

Table 1: Summary of data on hardness and development of ω phase in pure titanium processed by HPT[16,17,19-21,24].

Oxygen content (%)	HPT Pressure (GPa)	Number of turns of HPT	Rotation rate (rpm)	ω phase	Maximum hardness (GPa)	Reference
0.05	1.5	5	0.2	no	~2.5	Todaka et al. 2008
0.05	5	5	0.2	yes	~3.7	Todaka et al. 2008
0.2	2	10	0.5	no	~2.6	Edalati et al. 2009
0.2	6	10	0.5	yes	~3.4	Edalati et al. 2009
0.2	40	5	0.5	yes	~4.0	Edalati et al. 2009
<0.25	6	10	1	no	~3.0	Wang et al. 2014
<0.25	6	1/4	1	yes	~2.9	Shirooyeh et al. 2014
<0.25	6	10	1	yes	~3.9	Shirooyeh et al. 2014
<0.25	6	5	1	yes	~4.1	Bolmaro et al. 2014
<0.25	5	10	0.5	yes	~3.6	Shahmir and Langdon 2016
<0.25	5	10	1	yes	~3.0	Shahmir and Langdon 2016
<0.25	4	10	1	no	~3.7	Present work
<0.25	8	10	1	yes	~4.7	Present work

The development of the ω-phase and increase in hardness during HPT processing at large pressures for pure titanium has been reported in several investigations and Table 1 shows a summary of the available data. It is apparent that a phase transformation is readily observed after HPT processing of high purity Ti and at pressures larger than 4 GPa. Thus, the present results agree with the available reports since the phase transformation was observed only at the highest pressure of 8 GPa.

Comparing similar materials processed at the same rotation rate and the same number of turns of HPT, the hardness increases significantly in materials exhibiting an ω-phase due to processing at higher pressures. This confirms the effectiveness of enhancing the hardness of titanium through a phase transformation. There is also a trend of increasing the hardness of the processed material with increasing processing pressure even after reaching the threshold pressure for ω-phase formation. For example, ω-phase formation was reported in samples processed at 6 GPa but the hardness of the processed material increased with increasing processing pressure up to 40 GPa[17]. Thus, the present results show a higher hardness of ~4.7 GPa in titanium in Figure 3 and this higher value is attributed to increased impurities and to the higher processing pressure.

4. Summary and Conclusions

1. Commercial purity titanium of grade 2 was processed by 10 turns of HPT at pressures of 2, 4 and 8 GPa. X ray diffraction revealed the presence of an ω-phase in the material processed at the highest pressure.

2. Nanoindentation revealed a pronounced increase in hardness due to HPT processing and this effect was even more pronounced in the material processed at 8 GPa of pressure due to the phase transformation which gave a maximum hardness of ~4.7 GPa.

3. The distribution of hardness in the material processed at 8 GPa was heterogeneous with higher values at the mid-radius position and lower values near the center and at the edge of the disc. These lower values were attributed to the reduced ω-phase content due to the low shear strain near the center of the disc and the reduced pressure at the edge.

4. Nanoindentation tests with different loading rates showed that HPT processing reduced the strain rate sensitivity of titanium essentially to 0.

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