Microstructures and mechanical properties of Ti5553 alloy processed by high-pressure torsion

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Abstract. In the present research, the effects of high-pressure torsion (HPT) processing on the microstructure and mechanical properties of Ti-5Al-5Mo-5V-3Cr (Ti5553) alloy were studied. HPT processing produced a white etching layer (WEL) in the middle section of the cross-section and numerous shear bands in the surface region of the cross-section. And the thickness of the WEL increased with increasing the HPT revolutions. TEM observation of the WEL revealed an ultrafine-grained structure with high degree of lattice distortions. The mechanical properties measurements showed that the hardness and ultimate tensile strength increased by HPT processing, accompanied with a decrease in the elongation to failure. It is considered that the mechanical properties of HPT processed Ti5553 alloy are mostly dominated by the shear banded region and the WEL where have the finest grain size and high density of dislocations.

1. Introduction
Metastable β-Ti alloys have great potentials as structural materials due to their good formability at room temperature, high strength-to-density ratio, excellent age hardenability and good corrosion resistance [1]. In recent years, the metastable β-Ti alloys are increasingly used in aerospace industry. For example, Ti-5Al-5Mo-5V-3Cr (Ti5553, all elemental concentrations in mass%) alloy, which is a newly developed metastable β-Ti alloy, is being used for landing gear components on Boeing 787 [2]. The required high-level of mechanical properties of Ti alloys depend on several strengthening mechanisms such as solid solution strengthening, precipitation strengthening (such as precipitation of ω phase and α phase), grain refinement strengthening and dislocation strengthening [3].

In recent years, severe plastic deformation (SPD) by high-pressure torsion (HPT) has attracted much attention. The advantage of HPT is that the large shear strain can generate a combination of exceptional grain refinement (< 100 nm) and high density of dislocations [4]. HPT processing was applied to commercially pure titanium (CP Ti) and Ti-6Al-4V alloy. Valiev et al. reported that the application of HPT processing at room temperature resulted in the grain refinement of CP Ti with a mean grain size of around 120 nm, the ultimate tensile strength of 980 MPa and elongation to fracture of 12% [5]. Islamgaliev et al. observed that as a result of isothermal HPT processing at 573 K the ultimate tensile strength of CP Ti was up to 1600 MPa while the ductility was around 5% [6]. Ti-6Al-4V alloy processed by HPT exhibited ultrafine grains of 100-200 nm, outstanding room temperature tensile strength of 1.5 GPa and superplastic elongation of up to 500 % [7]. Meanwhile, there are few reports on the HPT processing of β-Ti alloys.

The motivation of the present study is to investigate the effects of HPT processing on the microstructure and mechanical properties of Ti5553 alloy in a single β phase condition.

2. Experimental
The Ti5553 alloy was prepared by cold crucible levitation melting (CCLM). The weight of an ingot was around 1.2 kg. The ingot was homogenized at 1473 K for 1 h, hot forged at 1473 K into a 40 mm
square block and then hot rolled into a 17 mm square bar, followed by air cooling. Then the bar was wrapped by a Mo foil and treated at 1473 K for 3 h, followed by water quenching. All the heat treatments were carried out in air. After grinding the oxide layer on the surface, the bar was cold swaged to the diameter of 10 mm. The Ti5553 alloy was solution treated (ST) above the β transus temperature (≈ 1123 K) at 1273 K for 1 h in Ar, followed by water quenching. The disks for HPT were sliced from the bar with a thickness of 0.85 mm. HPT processing was carried out under a compressive pressure of 5 GPa and rotation speed of 0.2 rpm for 1/2, 1 and 10 revolutions.

Planar samples were mechanically ground down to roughly the median plane and polished to mirror surface, and X-ray diffraction (XRD) characterization was performed on a RIGAKU RINT-TTR3 diffractometer with Cu-Kα radiation (40 kV, 150 mA). The microstructural characterization was carried out using optical microscopy (OM), scanning electron microscopy (SEM, JEOL JSM-7001F, 20 kV) and transmission electron microscopy (TEM, JEOL JEM-2100F, 200 kV). OM and SEM observations were carried out on the cross-sections of the samples mounted in resin. Disk samples for TEM analysis were cut from the median plane and perforated by twin-jet electropolishing at a temperature of 228 K in an electrolyte of 6%HClO₄, 30%C₄H₁₀O and 64%CH₃OH in volume.

Vickers microhardness measurements were carried out with an applied load of 0.98 N for 15 seconds on the cross-section of the disk. The measurements were made along the lines parallel to the radial directions at the positions 1/4 and 1/2 of the thickness. The miniature tensile specimens with a gauge section of 4 mm x 1 mm x 0.7 mm were cut by electro discharge machine (EDM) from the disks and the center of the gauge section was 1.5 mm away from the disk center. The tensile specimens were electrochemical polished to remove the damage layer from EDM cutting and to obtain a smooth surface. The tensile tests were carried out at room temperature with a strain rate of 2 x 10⁻³ s⁻¹ and the strain was monitored by video-extensometer with 3 µm resolution. The fracture surface was observed by scanning electron microscopy (SEM, TM-1000, 15 kV).

3. Results and discussion

![XRD patterns](image)

Figure 1. X-ray diffraction patterns of Ti5553 alloy before and after HPT processing.

XRD profiles of the Ti5553 samples before and after HPT processing with different revolutions are
shown in Figure 1. Only the peaks of β phase are detected in every sample. HPT processing led to a marked decrease in the peak intensity. The main peak corresponds to the one for β{110} after HPT processing. In addition, peak broadening can be seen in HPT processed samples, indicating significant grain refinement and an increase in the density of defects such as dislocations. The peak broadening is the most significant in the sample subjected to HPT of 10 revolutions.

The OM observations on the etched cross-section of the Ti5553 samples before and after HPT processing are shown in Figure 2. Coarse β phase grains can be seen in the sample subjected to ST. After HPT processing, the microstructure on the cross-section exhibits different structures in the middle region and near the both sides of the surface. The microstructure in the middle region consists of a white band structure aligned into radial direction, which is called as white etching layer (WEL). The formation of the WEL is due to their high resistance to etching. The OM observations also indicate that the WEL starts to form on the head region of the cross-section (as shown by the microstructure of the Ti5553 sample subjected to HPT 1/2 revolution) and then expands to the center region of the cross-section with increasing the HPT revolutions. In addition, the thickness of the WEL increases with increasing the HPT revolutions.

Details of the microstructures were further studied by backscatter electron SEM observation on the cross-section of the sample subjected to HPT processing of 10 revolutions, as shown in Figure 3. Figure 3(a) shows the SEM image contains the microstructure outside (upper region) and inside (lower region) the WEL. The microstructure outside the WEL shows numerous shear bands passing through the β grains. The shear bands show a bright contrast and the β grains show a dark contrast. The microstructure inside the WEL is uniform and featureless, showing a bright contrast. The enlarged views on the microstructure outside and inside the WEL are shown in Figure 3(b) and (c), respectively. It is clearly that the deformation inside the WEL is more intense and homogeneous. And it is considered that the coalescing and the accumulation of the shear bands during HPT processing results in the formation of WEL.

Figure 2. Optical micrographs on the cross-section of Ti5553 alloy before and after HPT processing.
Figure 3. (a) Backscatter electron SEM micrographs on the cross-section of Ti5553 alloy subjected to HPT processing of 10 revolutions. (b) Enlarged view outside the WEL. (c) Enlarged view inside the WEL.

Figure 4. TEM microstructure inside the white etching layer of the sample subjected to HPT processing of 1 revolution. (a) Bright field image and corresponding selected area electron diffraction (SAED) pattern. (b) Dark field image.
The microstructure inside the WEL was further studied by TEM on the sample subjected to HPT of 1 revolution, as shown in Figure 4. The BF image ((a)) and DF image ((b)) show complex strain contrast possibly originated from a high density of dislocations uniformly distributed, and it is difficult to find grain boundaries, suggesting the low mobility of dislocations and thus sluggish dynamic recovery. Distortion of diffraction spots along the hoop direction in the selected area electron diffraction (SAED) pattern also suggests a high degree of lattice distortions associated with a large internal stress. It is rather difficult to determine the true grain size in such severely deformed microstructure. Some bright areas observed in the dark filed image may not be a single grains but only a part of grains [8]. Similar microstructure was seen in Ti-Mo alloys after HPT processing [9].

Results of Vickers microhardness of Ti5553 samples after HPT processing are summarized in Figure 5. The level of hardness in solution treated sample is indicated by the broken line with a value of 288.1 ± 5.3 Hv. The hardness values after the HPT of 1/2, 1 and 10 revolutions are plotted as a function of distance from the center of the disk. It is obviously that HPT processing causes a marked increase in the hardness. The average hardness values are 329.8 ± 17.3 Hv, 341.3 ± 15.5 Hv and 366.6 ± 18.4 Hv for the samples with HPT of 1/2, 1 and 10 revolutions, respectively. It also shows that the hardness is lower in the center region of the disk at the early processing stage (1/2 revolution), but becomes uniform across the disk when HPT processing is continued to the higher number of revolutions. The large scatters in the hardness in N=1 and N=10 samples imply that the deformed structure is heterogeneous, as mentioned before by the optical micrographs. The hardness is much higher inside the shear bands or in the WEL than outside the WEL. For example, for the sample subjected to HPT of 10 revolutions, the average hardness inside the WEL is 389.6 ± 8.3 Hv and the hardness outside the WEL is 350.6 ± 14.4 Hv.

Figure 6 shows the tensile stress-strain curves of Ti5553 alloy before and after HPT processing. For the solution treated sample, the ultimate tensile strength and elongation-to-failure are 924 MPa and 14.6%, respectively. The first 1 revolution of HPT processing changed the stress-strain curve drastically. By the HPT processing of 1/2 revolution, the tensile strength increased a little but the ductility was lost significantly. Following the yield and rapid work hardening, the flow stress decreased to about 4% strain, which is a typical behavior in ultrafine grained materials. After applying the HPT processing of 1 and 10 revolutions, the tensile strength increased markedly but the ductility was lost. Figure 7 shows the SEM images of the fracture surface. Figure 7(a) shows the fracture surface. A big and deep pit is observed in the central region, and it is considered to be the crack source. For the HPT processed samples, the fracture surface is flat and covered with small dimples, as shown in Figure 7(b) to (d). In the HPT processed Ti5553 alloy, the WEL and shear banded regions should
have the finest grains and the highest density of dislocations because of the localized intense deformation. So it is considered that the mechanical properties of HPT processed Ti5553 alloy are mostly dominated by the shear banded region and the WEL.

4. Summary
Effects of high-pressure torsion processing on the microstructure and mechanical properties of Ti-5Al-5Mo-5V-3Cr alloy (all elemental concentrations in mass%) were studied. HPT processing
produced a white etching layer (WEL) in the middle region of the cross-section and numerous shear bands in the surface region of the cross-section. TEM observation of the WEL revealed an ultrafine-grained structure with high degree of lattice distortions. It is considered that the WEL and shear bands contribute to the high strength and the low ductility of the HPT processed samples.

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References
[1] Ankem S and Greene C A 1999 Mater. Sci. Eng. A 263 127-131
[2] Boyer R R and Briggs R D 2005 J. Mater. Eng. Perform. 14 681-685
[3] Clement N, Lenain A and Jacques P J 2007 JOM 59 50-53
[4] Zhilyaev A P and Langdon T G, 2008 Prog. Mater. Sci. 53 893-979
[5] Valiev R Z, Sergueeva A V and Mukherjee A K 2003 Scripta Mater. 49 669-674.
[6] Islamgaliev R K, Kazyhanov V U, Shestakova L O, Sharafutdinov and Valiev R Z 2008 Mater. Sci. Eng. A 493 190-194.
[7] Sergueeva A V, Stolyarov V V, Valiev R Z and Mukherjee A K 2002 Mater. Sci. Eng. A 323 318-325
[8] Popov A A, Pyshmintsev I Y, Demakov S L, Illarionov A G, Lowe T C, Sergeyeva A V and Valiev R Z 1997 Scripta Mater. 37 1089-1094
[9] Frajami S, Tsuchiya K, Todaka Y and Umemoto M 2009 Processing and Fabrication of Advanced Materials - XVIII 1053-1060