High Strength and Ductility in Ball-Milled Titanium Powders Consolidated by High-Pressure Torsion

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High Strength and Ductility in Ball-Milled Titanium Powders Consolidated by High-Pressure Torsion

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Abstract. Pure Ti powders were subjected to ball milling and subsequently high-pressure torsion (HPT) for consolidation. It is found that a fully dense (99.9%) disc with ultrafine grained structure (~50-300 nm) was produced. The strength and ductility were well comparable to those of ball-milled Ti-6%Al-4%V powders after hot roll sintering.

Introduction

It is well established that processing through the application of ball milling results in a significant grain refinement and a marked increase in hardness [1]. In a ball milling process, a powder mixture is loaded into a container together with grinding balls and is subjected to milling for a period of time. The ball-milled powders are then consolidated into bulk forms using hot processing methods. An alternative process is now gaining a considerable interest for consolidation of powders, which utilizes a process of high-pressure torsion (HPT) [2]. Because the powders are pressed by high pressures and intense strain is introduced through torsion, the powders are not only compacted without heating but also significant grain refinement is achieved. The HPT process was applied successfully as a cold consolidation technique without sintering for ball-milled powders including powders of pure Ni [3], pure Co [4], an Al-Mg alloy [5], Co-based metal-ceramic composites [6] and amorphous alloy [7]. In spite of various publications regarding the ball milling of Ti [8], little attention has been paid to the consolidation of ball-milled Ti powders through the application of HPT.

In this study, thus, cold consolidation of ball-milled Ti powders are attempted using HPT and the evolutions of mechanical properties and microstructure are investigated.

Experimental Procedures

High purity Ti (99.5%) powders were subjected to ball milling using a planetary device with a tungsten carbide vial for 50 hours with a rotation speed of 200 rpm under pure argon atmosphere. The appearance of powders before and after processing with HPT is shown in Fig. 1. Ball to powder weight ratio was 1.8:1 and the material of balls was a high-carbon chromium steel. HPT was conducted at room temperature under a pressure of 2 GPa, which is smaller than the critical pressure for ω phase formation [9]. The ball-milled powders were consolidated to discs with 10mm diameter and 0.8mm thickness while they were strained through either 1, 4, 10 or 40 revolutions with a rotation speed of 1 rpm. The details of HPT facility and operating procedures were reported elsewhere [10,11].

The HPT-processed discs were evaluated using density measurement, Vickers microhardness, bending test, tensile test, scanning electron microscopy (SEM) and transmission electron microscopy. First of all, both sides of the disc samples were polished to a mirror-like surface and the density was determined by Archimedes' principle. Second, Vickers microhardness was measured with an applied load of 200 g for 15 s along the radii from the center to edge. Third, for bending test, miniature rods with a 0.5×0.5mm^2 square cross section and 9mm length were cut from the 10mm diameter discs at 1 mm away from the disc center. Three-point bending tests were carried out at a cross-head speed of 0.5 mm/s to measure the bending stress and displacement in the radial direction and the pressing direction as described elsewhere [12]. Fourth, miniature tensile specimens having 1.5mm gauge length, 0.7mm width and 0.5mm thickness were cut from the 10mm discs at the position 2 mm away from the center. Each tensile specimen was mounted horizontally on grips and pulled to failure using a tensile testing machine with an initial strain rate of 2×10^{-3} s^{-1}. Fifth, SEM was performed at 20 kV for observation of fractographs of tensile specimens after failure. Sixth, for TEM, 3mm discs were punched out from the 10mm discs at 3.5 mm away from the center and further thinned with a twin-jet electro-chemical polisher. TEM was performed at 200 kV for microstructural observation and for recording selected-area electron diffraction (SAED) patterns.

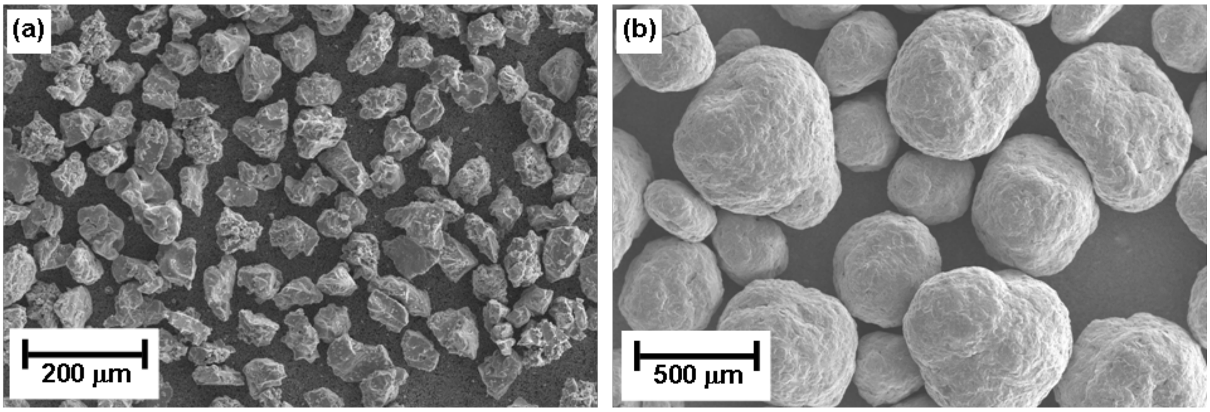

Fig. 1 Appearance of titanium powders before (left) and after (right) ball milling.

Results and Discussion

The measured values of microhardness are plotted against the equivalent strain in Fig. 2. Here, the equivalent strain, ε, was calculated by the equation as $\varepsilon = (r\theta/t)/\sqrt{3}$, where r is the distance from the center of disc, θ is the rotation angle and t is the thickness of disc [2]. It is apparent from Fig. 2 that the hardness increases with increasing equivalent strain at an early stage of straining but appears to saturate to a level of 330 Hv at higher strains. This hardness variation with strain is similar to that of bulk Ti processed by HPT [9].

From the bending tests, the stress-displacement curves are delineated in Fig. 3 for the samples processed for 10 revolutions. The tests were carried out in the radial direction and pressing direction as in the inset. The bending strength in the radial direction is higher than that in the pressing direction but the total displacement to failure shows an opposite trend.

Figure 4 shows the results of the tensile testing after ball milling followed by HPT of Ti powders. For comparison, Fig. 4 includes the published data after processing of Ti [8,9,13-15] and of Ti-6%Al-4%V [8]. Solid line represents a relation delineated through the points obtained by the annealed sample and conventional cold-rolled and hot-rolled samples for bulk Ti. It is noted that HPT processing provides two important advantages. First, the strength of pure Ti is significantly enhanced by the HPT so that it becomes comparable to the strength and even better ductility of ball-milled

Ti-6%Al-4%V after hot roll sintering, and second the ball-milled Ti powders processed by ball-milling shows the highest strength after HPT when compared to other processing methods as shown in Fig. 4.

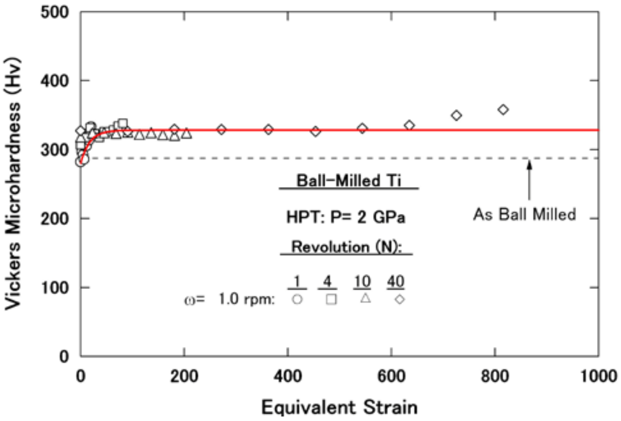

Fig. 2 Vickers microhardness plotted against equivalent strain for samples processed after various revolutions.

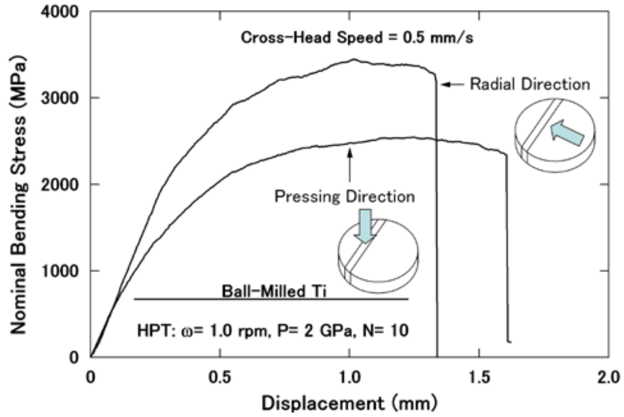

Fig. 3 Bending stress versus displacement curves obtained from discs processed for 10 revolutions in radial direction and pressing direction.

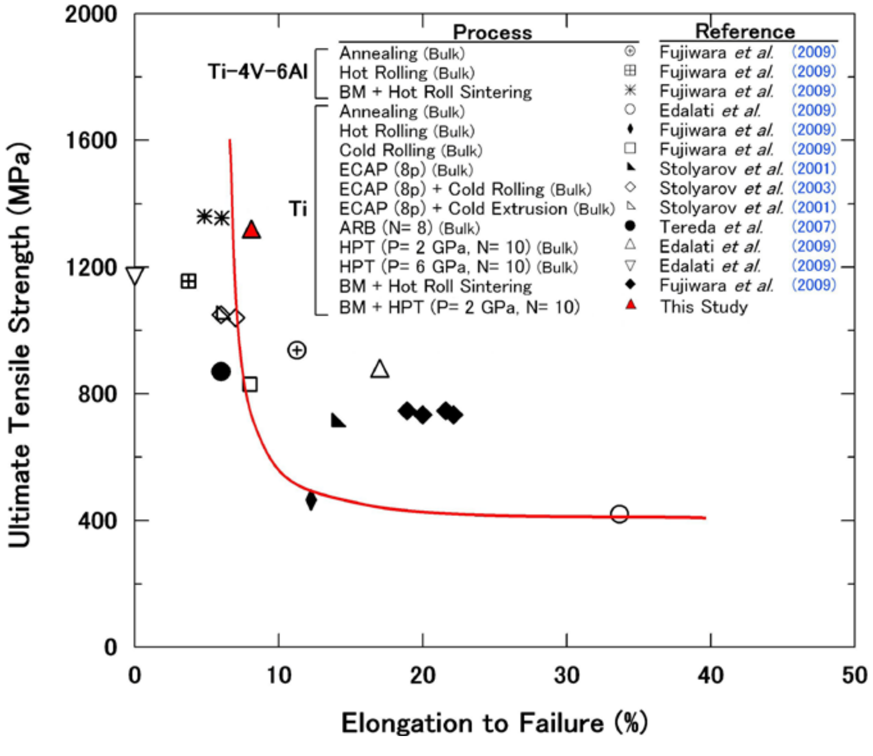

Fig. 4 Plots of ultimate tensile strength versus elongation to failure including published data for samples of pure Ti and Ti-6%Al-4%V after processing with different methods [8,9,13-15].

Figure 5 shows (a) SEM fractographs after tensile testing, (b) TEM micrographs and (c) SAED patterns of samples processed for 10 revolutions. It is apparent from Fig. 5(a) that the fracture surface has a vein-like pattern. This observation indicates that plastic deformation occurred in consistence with the results of tensile testing shown in Fig. 4. TEM microstructure and the corresponding SAED patterns show that the microstructures consist of ultarfined grains with grain sizes in the range of 50 to 300 nm. Close examination of the microstructures reveals that there are many dislocations within the grains and most of the grains are surrounded by ill-defined grain boundaries with high misorientation angle. Density measurement in this study revealed that a relative density as high as 99.9% is achieved after cold consolidation by HPT for 10 revolutions.

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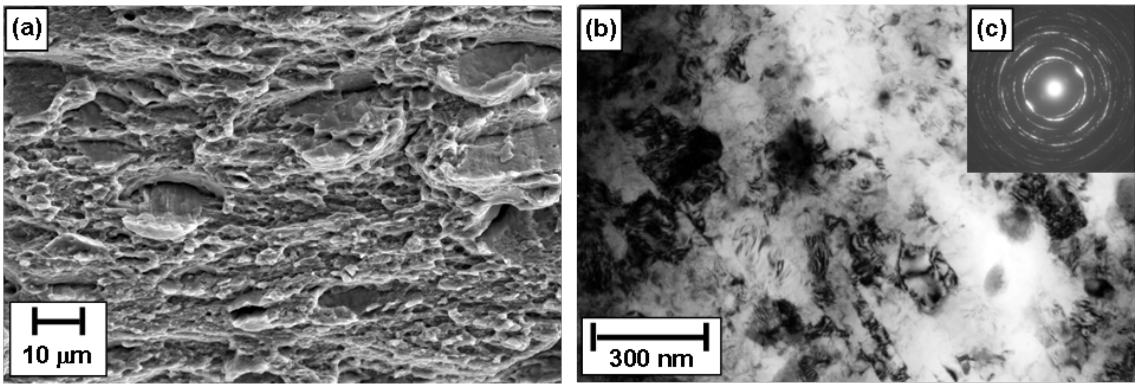

Fig. 5 (a) SEM fractograph, (b) TEM micrograph and (c) SAED pattern of samples processed by HPT for 10 revolutions.

Conclusions

Ball-milled Ti powders were consolidated to fully dense discs with ultrafine grained structure by high-pressure torsion (HPT). It was found that the strength and ductility are improved by application of ball milling followed by HPT when compared with those of other deformation methods, and they become well comparable to those of ball-milled Ti-6%Al-4%V after hot roll sintering.

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