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Application of high-pressure torsion for consolidation of ceramic powders

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Abstract

Alumina (α-Al_2O_3) powders were successfully consolidated by application of high-pressure torsion (HPT) at ambient temperature and by a subsequent annealing process. Introduction of strain by HPT was confirmed by peak broadening in X-ray diffraction analysis. It was shown that consolidation was greater, giving rise to increased hardness with increasing imposed strain, annealing temperature and annealing time. Scanning electron microscopy showed that consolidation occurred at lower temperatures due to the presence of strain.

Keywords: Alumina; Ceramics; High-pressure torsion; Severe plastic deformation.

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Processing by severe plastic deformation (SPD) has become an attractive research issue in recent years. The grain size is significantly refined through SPD and, consequently, mechanical properties such as hardness and strength are enhanced. Several techniques are available for SPD processing, but high-pressure torsion (HPT) is especially effective in introducing extremely large strains under high hydrostatic pressures and thus it is applicable to hard and less ductile materials such as tungsten [1], magnesium alloys [2] and intermetallics [3].

In the HPT method a thin disc specimen is placed between two massive anvils under high pressure and intense strain is introduced by rotating the two anvils with respect to each other [4]. In addition to grain refinement, HPT is also applicable as a processing tool for consolidation of powders [5–23] such as metallic materials [5–12], metal–ceramic composites [11–17] and amorphous materials [17–23]. The consolidation of metal–ceramic composites was successfully achieved with fractions of ceramics up to as large as 50% [15,16]. However, to the best of the authors' knowledge there appears to have been no application of HPT for the consolidation or pre-compaction of pure ceramic powders, including an examination of the influence of strain on consolidation.

In this study, and for the first time, commercially pure α-Al_2O_3 powders were subjected to HPT and subsequently to annealing. The evolution of microhardness and microstructures were investigated, with attention paid to the consolidation process during HPT and post-HPT annealing.

Materials used in this study were commercially pure α-Al_2O_3 powders of ~1 μm particle size. Approximately 0.5 g of the powders was placed in a flat-bottomed shallow hole of 10 mm diameter and 0.25 mm depth located in the center of the lower anvil of a HPT facility. The powders were first pressed by lifting the lower anvil against the upper anvil and both anvils were rotated with respect to each other while pressing. The HPT operation was conducted at room temperature at a rotation speed of ω = 0.5 r.p.m. for either N = 2, 4 or 10 revolutions under a pressure of P = 6 GPa. Details concerning the HPT facility have been reported elsewhere [24,25]. The disc samples after processing by HPT were annealed at temperatures of T = 600, 1000, 1200 and 1300 ^{o}C for periods of t = 2 and 16 h. In this annealing an argon atmosphere was used to eliminate atmosphere-induced structural evolution [26]. The appearance of the powders before and after processing by HPT is shown in Figure 1.

The disc samples were evaluated through Vickers microhardness measurements, density measurements, scanning electron microscopy (SEM) and X-ray diffraction (XRD) analysis. First, after processing by HPT the discs were polished and the Vickers microhardness was measured at the center (within 1 mm of the center, r < 1 mm) at 8 different points and edge (>4 mm from the center, r > 4 mm). Second, the sample density was determined by Archimedes' principle using an electronic balance with an accuracy of 0.1 mg. Third, SEM at 20 keV was used for microstructural observation of the powders and disc samples. Fourth, structural analyses with XRD were performed using CuKα radiation at 40 kV and 40 mA with a scanning step of 0.02^{o} and a scanning speed of 0.5^{o} min^{-1}.

It should be noted that the dimensions of HPT processed discs were too small to carry out an evaluation of fracture toughness.

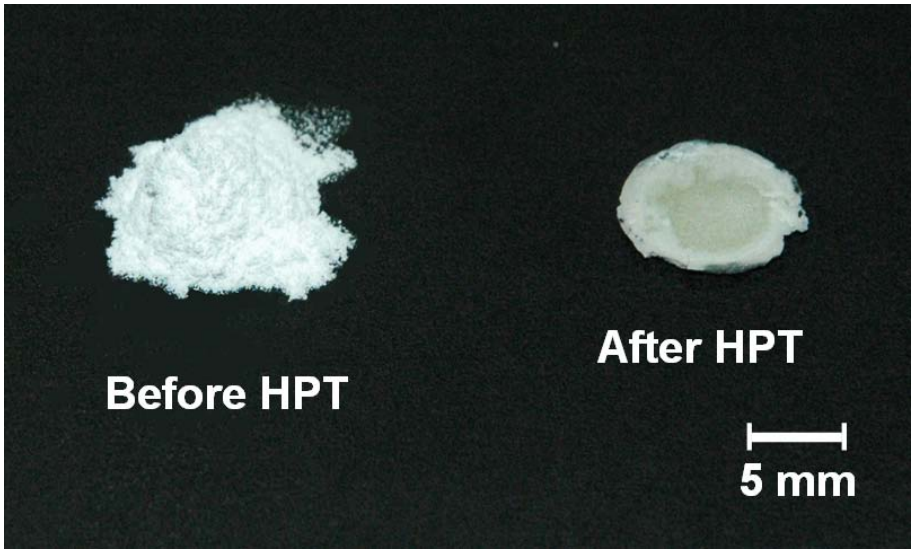

Figure 1. Appearance of alumina powders before (left) and after (right) HPT for 10 revolutions.

Figure 2a plots the microhardness against the number of revolutions at the center and edge of the discs subjected to HPT and subsequent annealing at 1300 ^{o}C for 16 h. The hardness after HPT but without annealing is also plotted in Figure 2a for the edge. For both parts of the discs the hardness increased monotonically with increasing number of revolutions, but the hardness values at the edge were invariably higher than those in the center. This trend can be attributed to the difference in strain, because the magnitude of strain (e) created through HPT varies number of revolutions and distance from the disc center, as given by the following equation [27]:

$$\varepsilon = \frac{2\pi rN}{\sqrt{3}\,t} \qquad (1)$$

where r is the distance from the disc center, N is the number of revolutions and t is the thickness of the disc. Thus, the center of the disc corresponds to a shear strain theoretically equal to zero and undergoes nearly pure compression. Figure 2a suggests that consolidation improves as the strain increases with an increase in the number of revolutions and/or with distance from the disc center. It should be noted that compaction of powders was not feasible by mere application of a pressure, even at 6 Gpa, without rotation to induce strain. These observations are consistent with our earlier report concerning the consolidation of amorphous chips using HPT [23].

The effect of imposed strain is more clearly demonstrated in Figure 2b, plotting all hardness values in Figure 2a as a function of the equivalent strain. It is apparent that the hardness after annealing increased with increasing equivalent strain. It should be noted that calculation of the equivalent strain did not take into account the effect of slippage and thickness reduction during HPT processing, because this estimation is difficult for powders [28,29]. Inspection of Figure 2a and b reveals that the increase in hardness with the increase in equivalent strain was ~1.8 times. However, annealing gave rise to a 10 times increase in hardness when compared with the sample without annealing. Thus, post-HPT annealing plays a crucial role in densification of ceramic powders.

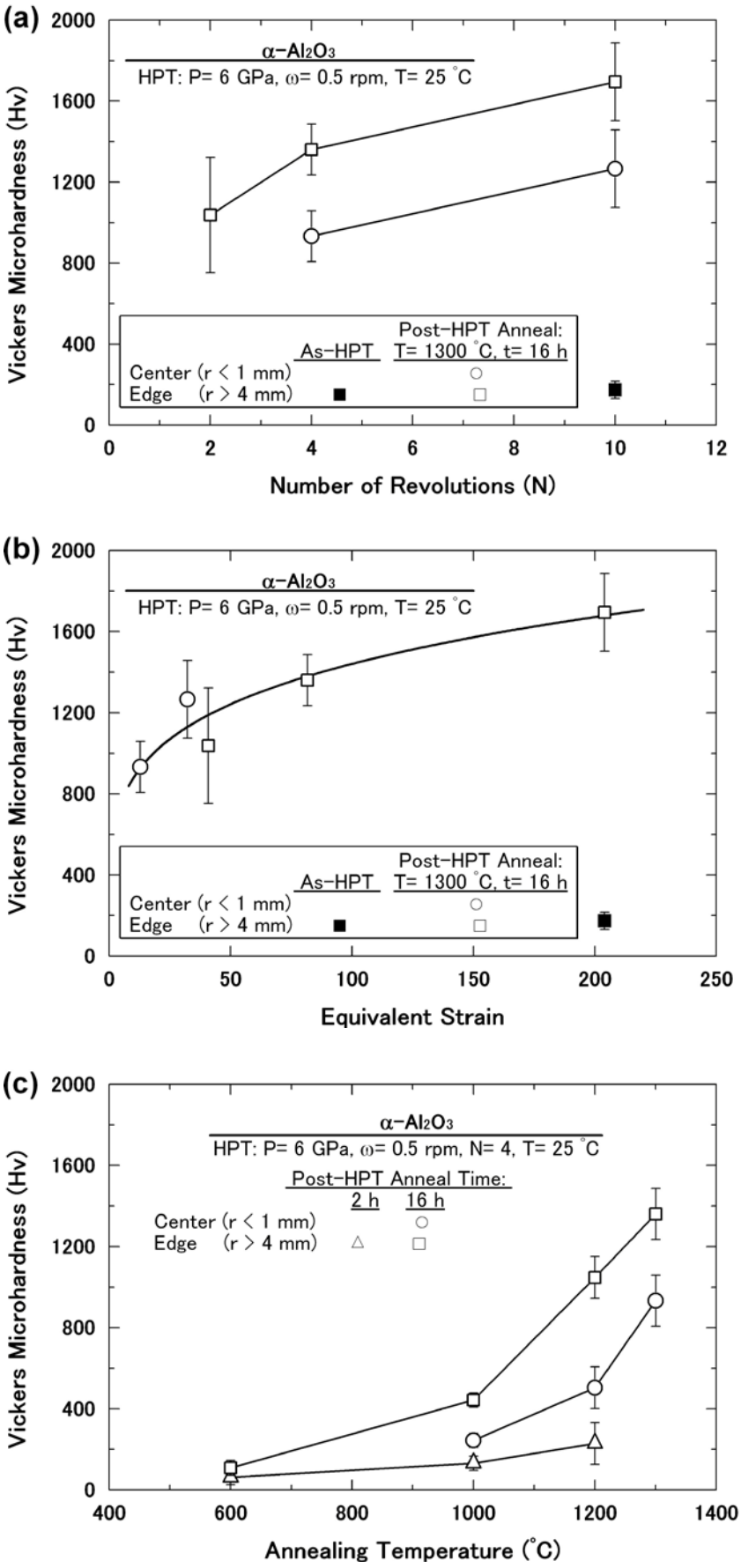

Figure 2. Vickers microhardness plotted against (a) number of revolutions, (b) equivalent strain and (c) annealing temperature at the center and edge of discs.

Figure 2c plots microhardness as a function of annealing temperature for the HPT processed discs. The plots include hardness values at the center and edge of the discs after annealing for 2 and 16 h. The microhardness increased with increasing annealing temperature and with the longer annealing time. Moreover, hardness values at the edge were higher than those at the center. Thus, provided that the hardness level reflects the degree of consolidation, all these trends suggest that a higher temperature and longer period of annealing, as well as a greater strain, are important in enhancing the consolidation of alumina powders. It is considered that better consolidation occurs because the connectivity of individual powders improves with an increase in annealing time and temperature, as reported earlier [30,31].

The microhardness values reported in the literature for bulk alumina vary over a wide range, 1200–3000 Hv [30–35]. This variation could be due to differences in either the conditions for measuring hardness [32–34], the level of purity of the powders [32], the microstructure [34], the processing conditions [30,31] or a combination of some or all of these. It should be noted that the maximum hardness attained in this study, ~1700 Hv, is comparable with those reported in the literature [31–34]. Nevertheless, the consolidation temperature used in this study was low and it is suggested that sintering should be accelerated due to the significant strain introduced by HPT prior to heating. Table 1 documents the measured density and relative density for the powders subjected to HPT for 10 revolutions before and after post-HPT annealing at 1300 ^{o}C for a period of 16 h, including the theoretical value. It appears that annealing of the HPT processed sample resulted in increased density and consolidation to a relative density level as high as 98.6%. This relative density is comparable with those reported in the literature [26,30–32].

Figure 3 shows SEM micrographs of the powders (Fig. 3a), the edge of a disc after HPT processing for 10 revolutions in plan view (Fig. 3b) and the edge part in a plan view of a disc after HPT processing for 10 revolutions followed by annealing at 1300 ^{o}C for 16 h (Fig. 4c). The particle size appears to be ~1 μm, with a spherical morphology for the as received powders in Figure 3a. The particles were deformed after processing by HPT, as shown in Figure 3b. Although the overall consolidation and connectivity appeared to be weak, consolidation partially occurred even on HPT processing at room temperature, as indicated by the arrow in Figure 3b. Consolidation improved on annealing after HPT, as shown in Figure 3c, but the connectivity of individual particles was still incomplete, so that the gaps were visible in the sample, as indicated by the arrows in Figure 3c.

XRD profiles are shown in Figure 4 for the powders, for the disc subjected to HPT for 10 revolutions and for the disc subjected to HPT for 10 revolutions and annealed at 1300 ^{o}C for 16 h. Neither phase transformation nor impurities were detected in any of the samples within the sensitivity limit of the present XRD analysis. Close examination of Figure 4 indicates that peak broadening occurred appreciably after HPT, but the peak broadening disappeared on annealing after HPT. The appearance of the peak broadening indicates that the HPT process was effective in introducing many of the lattice defects which facilitate the consolidation of alumina powders.

In summary, it is emphasized that HPT processing is promising in pre-compaction and subsequent consolidation of alumina ceramics. Although full consolidation at room temperature cannot be attained in ceramics, unlike metallic materials [5–23], the hardness measurements suggest

that introducing intense strain under high pressure should be effective for complete consolidation at sintering temperatures as low as 1300 ^{o}C or for shorter sintering time without using binders. It should be noted that the present procedure using HPT contrasts with conventional hot pressing processes where the sintering temperature for a dense alumina sample should be about 1700 ^{o}C [30,31].

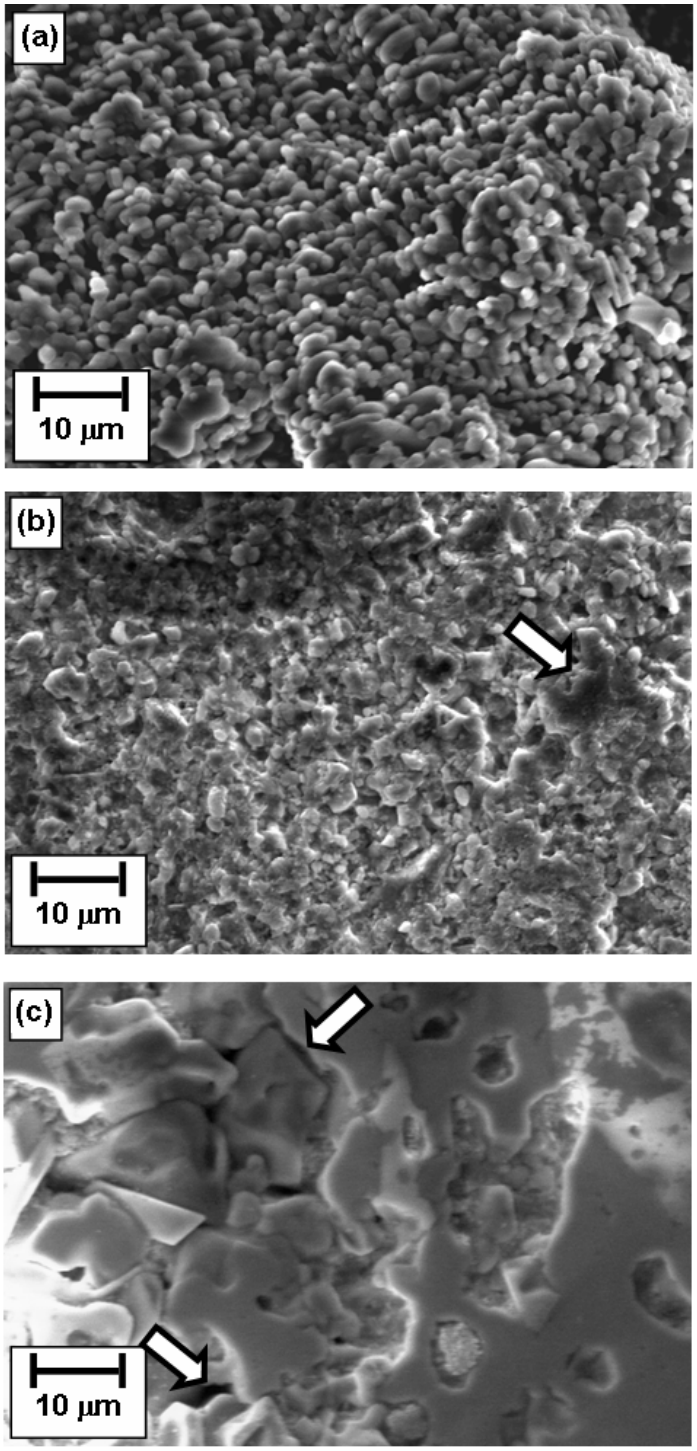

Figure 3. SEM micrographs of (a) powders, (b) edge of disc in plan view after processing by HPT for 10 revolutions and (c) edge of disc in plan view after processing by HPT for 10 revolutions and annealing at 1300 ^{o}C for 16 h.

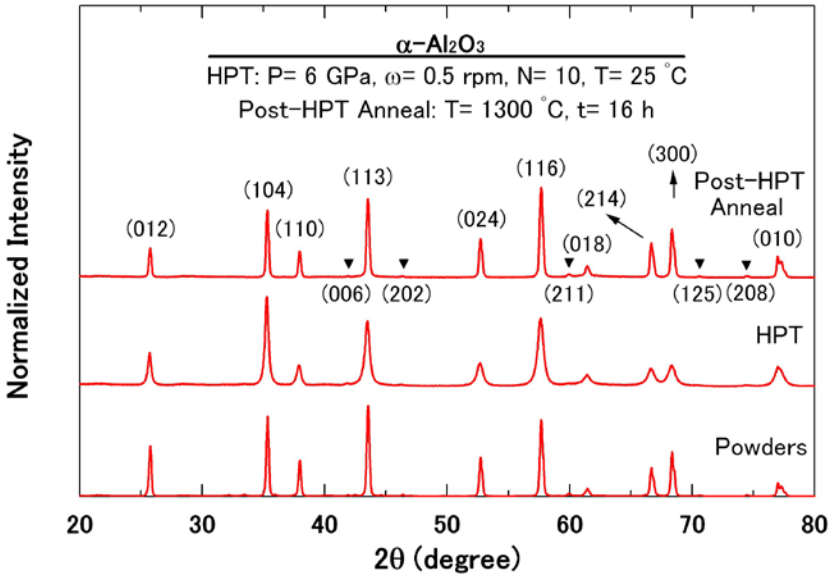

Figure 4. XRD profiles of as received powders, after HPT processing for 10 revolutions and after HPT processing for 10 revolutions and post-HPT annealing at 1300 ^{o}C for 16 h.

Alumina (α-Al_2O_3) powders were processed by HPT followed by annealing and the following conclusions were made.

1. The powders are consolidated satisfactorily using HPT followed by annealing.
2. XRD analysis reveals that HPT processing is effective in introducing lattice strain in alumina ceramics.
3. Hardness increases and consolidation improves with increasing strain imposed by HPT.

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