

High-pressure torsion of pure magnesium: Evolution of mechanical properties, microstructures and hydrogen storage capacity with equivalent strain

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Pure Mg (99.9%) is processed by high-pressure torsion (HPT) at room temperature. The hardness behavior with imposed strain is similar to pure Al (99.99%), having a hardness maximum followed by a steady state. HPT processing increases the hardness and tensile strength. A bimodal microstructure with an average grain size of $\sim 1\ \mu\text{m}$ is developed by HPT with some grains free of dislocations. Hydrogen absorption is improved by HPT after 10 revolutions, and a total hydrogen absorption of 6.9 wt.% is achieved. © 2011 Acta Materialia Inc. Published by Elsevier Ltd. All rights reserved.

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High-pressure torsion (HPT) is a severe plastic deformation (SPD) technique where a thin disc or ring is placed between two massive anvils under a high pressure and intense shear strain is introduced by rotating the two anvils with respect to each other [1,2]. It has been shown that the hardness after HPT processing is represented by a unique function of equivalent strain for various metals such as Al [2–7], Cu [8], Fe [9], Ti [10], Hf [11], V [12], and Mo [12]. However, the hardness behavior of pure Al is different from that of other pure metals. In most metals, a steady-state level is reached directly following an initial increase with straining [8–12]. However, in pure Al with a purity level of 99.99%, the hardness initially increases with increasing strain and, after reaching a maximum, decreases to a constant level [2–7]. Hardness–strain behavior similar to that of Al may be observed in pure Mg because both Al and Mg are similar in terms of melting temperature (T_m) and stacking fault energy (SFE) ($T_m = 933\ \text{K}$ for Al and $922\ \text{K}$ for Mg, and $\text{SFE} = 166\ \text{mJ m}^{-2}$ for Al and $125\ \text{mJ m}^{-2}$ for Mg [13]), and these parameters are the most important in terms of the

deformation behavior. Thus, the first part of this study is to investigate the hardness behavior of pure Mg with respect to strain imposed by HPT.

In addition, the second part of this study examines hydrogen absorption behavior with HPT straining. It is well known that Mg produces a hydride in an atmosphere of hydrogen and Mg is considered a good candidate for a hydrogen storage material [14]. The main drawbacks of Mg in this respect are that its absorption and desorption temperatures are very high and the hydrogenation reaction is too slow. It is therefore important to look at how the absorption speed can be increased at low temperatures. The influence of SPD on the hydrogen storage performance of Mg alloys has been investigated in several papers and improvements in this respect have been reported after processing by equal-channel angular pressing (ECAP) [15–18] and HPT [19–23]. Moreover, it was shown that HPT is effective in formation of strain-induced hydride in pure Hf as well as in decreasing the dehydrogenation temperature [11]. However, little is understood to date regarding the hydrogen absorption behavior in high-purity Mg after processing by SPD. Since the alloying elements and their interaction with plastic strain may affect both thermodynamic and kinetic of hydrogen absorption, investigation of HPT-processed pure Mg is important to understand the effect of SPD, grain refinement and dislocation density on hydrogen storage capacity.

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In this study, pure Mg is severely deformed by HPT and the evolution of mechanical properties and microstructures together with hydrogen storage performance are examined as a function of imposed strain.

Cylindrical rods of pure Mg (99.9%), 10 mm in diameter and 60 mm long, were cut from a $60 \times 80 \times 180 \text{ mm}^3$ ingot. The rods were sliced to discs 0.8 mm thick using a wire-cutting electric discharge machine and further annealed for 1 h at 773 K under an argon atmosphere. HPT was conducted at room temperature using the annealed discs under a pressure of $P = 6 \text{ GPa}$ and subsequently shear strain was imposed through either $N = 0.25, 0.5, 1, 3$, or 10 revolutions with a rotation speed of $\omega = 0.5 \text{ rpm}$. The HPT-processed discs were evaluated using Vickers microhardness measurement, tensile test, optical microscopy (OM), transmission electron microscopy (TEM) and hydrogen storage analysis.

First, after processing by HPT, the 10 mm discs were polished to a mirror-like surface and the Vickers microhardness was measured with an applied load of 50 g for 15 s along the radii from the center to edge at eight different radial directions at 0.5 mm increments.

Second, miniature tensile specimens with a gauge length of 1.5 mm, a width of 0.7 mm and a thickness of 0.5 mm were cut from the discs at a 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 $2 \times 10^{-3} \text{ s}^{-1}$. It should be noted that the dimensions of the present tensile specimens are fairly small, and therefore care is required when compared with other sizes of tensile specimens.

Third, for OM observations, the annealed discs were polished to mirror-like surfaces and etched in a solution of 5% HNO_3 and 95% $\text{C}_2\text{H}_5\text{OH}$.

Fourth, for TEM, 3 mm diameter discs were punched from the HPT discs at 3.5 mm away from the center. The 3 mm diameter discs were ground mechanically to a thickness of 0.15 mm and further thinned with a twin-jet electrochemical polisher using a solution of 2% HClO_4 , 28% $\text{C}_3\text{H}_5(\text{OH})_3$, and 70% CH_3OH at 263 K under an applied voltage of 30 V. TEM was performed at 200 kV for microstructural observation and for recording selected-area electron diffraction (SAED) patterns.

Fifth, for hydrogen storage analysis, two discs were polished on both sides to a thickness of 0.55 mm and the hydrogen absorption rate was measured using a Sieverts-type commercial gas adsorption apparatus (Bel Japan Inc., Bel Sorp HG) at 423 K. An approximate weight of 130 mg of the samples was exposed under a hydrogen pressure of 3 MPa for 100 ks and the content of hydrogen absorbed in the sample was recorded every $\sim 5 \text{ ks}$.

Figure 1a shows the variation in hardness with the distance from the centers of disc samples after 0.25–10 revolutions. The hardness variation is irregular and strongly depends on the extent of revolution. The hardness increases with respect to the distance from the center for 0.25 revolutions. The hardness increases, reaches a maximum and decreases as the distance from the center increases for 0.5 revolutions. However, the hardness exhibits a decrease with an increase in the distance from the center for 1, 3, and 10 revolutions.

To demonstrate the hardness behavior with respect to equivalent strain, all hardness values in Figure 1a are

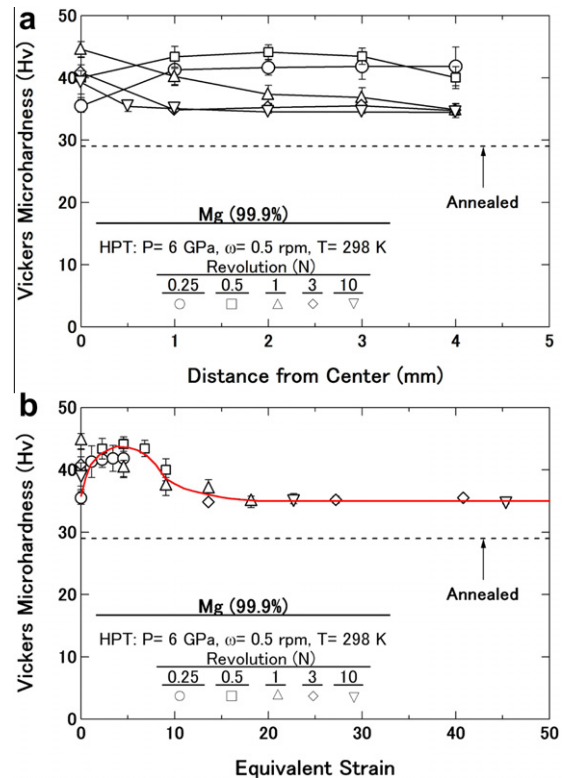


Figure 1. Vickers microhardness plotted against (a) distance from center and (b) equivalent strain for disc samples processed after various numbers of revolutions.

plotted against the equivalent strain in Figure 2b as attempted in earlier papers [2–12]. Here, the values of slip-page fraction and thickness reduction during HPT were measured as described earlier [9,24]. Since both slippage and thickness reductions during HPT were negligible irrespective of the number of revolutions, the equivalent strain was calculated as:

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

where r is the distance from the disc center, N is the number of revolutions and t is the thickness of the disc. It is apparent that all data points now lie on a unique curve, reaching a maximum at an equivalent strain of 4, thereafter leveling off at an equivalent strain of ~ 20 . This is then followed by the onset of a steady state where

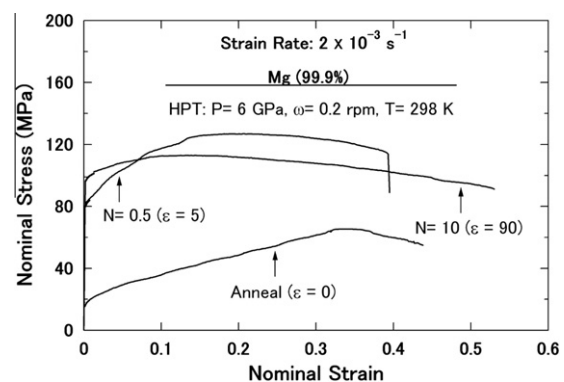


Figure 2. Nominal tensile stress vs. nominal strain curves for annealed sample and samples processed by HPT for 0.5 and 10 revolutions.

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