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Micro-Mechanical Responses of Ultrafine-Grained Materials Processed Through High-Pressure Torsion

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Abstract. The processing of metals through the application of high-pressure torsion (HPT) provides the potential for achieving exceptional grain refinement in bulk metal solids. These ultrafine grains in the bulk metals usually show superior mechanical and physical properties. Especially, the development of micro-mechanical behavior is observed after significant changes in microstructure through processing and it is of great importance for obtaining practical future applications of these ultrafine-grained metals. Accordingly, this presentation demonstrates the evolution of small-scale deformation behavior through nanoindentation experiments after HPT on various metallic alloys including a ZK60 magnesium alloy, a Zn-22% Al eutectoid alloy and a high entropy alloy. Special emphasis is placed on demonstrating the essential microstructural changes of these materials with increased straining by HPT and the evolution of the micro-mechanical responses in these materials by measuring the strain rate sensitivity.

Introduction

The processing of metals through the application of severe plastic deformation (SPD) provides the potential for achieving exceptional grain refinement in bulk solids. Among the reported SPD techniques, one of the most attractive methods refers to the processing by high-pressure torsion (HPT) where this type of processing leads to exceptional grain refinement that is not generally achieved using other procedures [1]. The fundamental principles of HPT processing were described in detail in an earlier review [2]. Specifically, the sample in a disk shape is applied to receive both very high compressive straining and torsion straining concurrently. Numerous reports are now available demonstrating that bulk materials after HPT having ultrafine and nanometer grain sizes generally show superior mechanical properties which include high strength dictated through the Hall-Petch relationship at low temperatures and a superplastic forming capability at high temperatures due to possible fast diffusion.

Nevertheless, there is no comprehensive report to date demonstrating the interdependency between the evolution in microstructure through HPT processing and the improvement in plastic responses at room temperature (RT) after processing. Accordingly, this study was initiated to evaluate the correlation between the microstructural changes and evolution in hardness and in the strain rate sensitivity, *m*, calculated through nanoindentation testing on a series of alloys after HPT with increasing numbers of HPT turns: a ZK60 magnesium alloy, a Zn-22% Al eutectoid alloy and a high entropy alloy (HEA). The report suggests the recognition of a consistent trend of enhanced micro-mechanical behavior in a wide range of metals and alloys after HPT.

ZK60 Magnesium alloy

An extruded ZK60 magnesium alloy in a billet shape with a diameter of 10 mm was cut into disks with thicknesses of ~1.5 mm and these disks were polished on both sides to have final thicknesses of ~0.83 mm. The HPT processing was conducted at RT under quasi-constrained conditions [3] under a pressure, *P*, of 6.0 GPa and a rotational speed of 1 rpm and through numbers of revolutions, *N*, up to 5 turns.

The microstructure at the edges of the disks was examined using an optical microscope. The representative microstructures are shown in Fig. 1 for (a) an as-extruded condition and for the samples after HPT for (b) 1/4, (c) 1/2 and (d) 2 turns [4]. The initial microstructure of the as-extruded material before HPT consists of a bi-modal grain distribution with a fraction of >50% of coarse grains having ~25 µm surrounded by several finer grains having ~4-5 µm as shown in Fig. 1(a). A noticeable change is visible in the microstructure after HPT through 1/4 turn where the fraction and size of the coarse grains were reduced to ~35% and ~20 µm, respectively, and the fine grain size was reduced to ~2-3 µm with an increased area fraction of ~65% as shown in Fig. 1(b). After 1/2 turn in Fig. 1(c), the coarse grain sizes remained the same at ~20 µm whereas the fine grains having an average size of ~1.0-1.5 µm exist with a high area fraction of ~75%. Thereafter, the microstructure remained constant with increasing torsional rotation though 2 turns as shown in Fig. 1(d). Thus, the HPT processing introduced a reasonable level of microstructural saturation up to at least 2 turns in the ZK60 alloy.

The bi-modal microstructure observed after HPT for 2 turns is in excellent agreement with a ZK60 alloy after ECAP at 473 K [5] and after HPT at RT under 2.0 GPa for 5 turns [6]. The unique microstructural evolution in magnesium alloys is explained by the necklace-like dynamic recrystallization (DRX) where the concentration of deformation occurs across the new fine grains [7]. Thus, the evolution in grain structure depends on the initial grain size, the size of the new grains and the amount of imposed strain within magnesium alloys.

Two different nanoindentation tests were conducted on the as-extruded sample and on the disk edges of the ZK60 alloy after HPT through 2 turns at four different indentation strain rates from 0.0125 to 0.1 s^{-1} using two different indentation procedures; constant strain rate (CSR) testing and strain-rate jump (SRJ) testing [4]. These two tests were performed with a three-sided pyramidal Berkovich indenter in order to estimate the strain rate sensitivity, *m*, where the *m* values are determined by following the earlier procedures and applying the measured hardness, *H*, through nanoindentation [8]. The estimated *m* values were plotted with increasing *N* as shown in Fig. 2. It should be noted that the essential merit of the SRJ test is that the estimation is conducted from a single indentation whereas multiple indentations are required in the CSR tests.

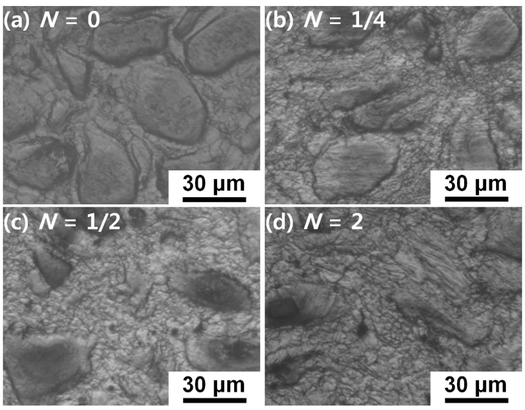

Fig.1 Representative optical micrographs taken at the edges of the ZK60 disks (a) in the as-extruded condition and after HPT under 6.0 GPa for (b) 1/4, (c) 1/2 and (d) 2 turns [4].

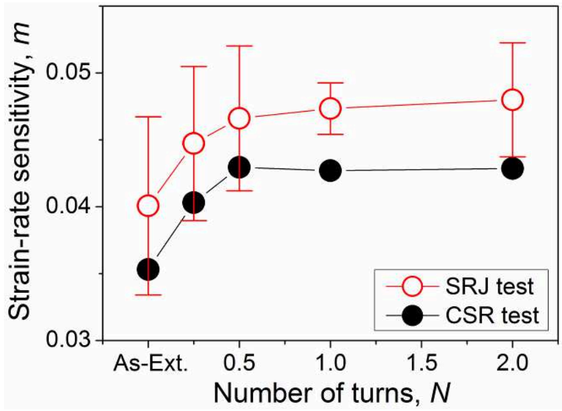

Fig. 2 Variations of the strain-rate sensitivity with different levels of torsion straining: data were obtained through CSR and SRJ testing. [4].

Slight differences are observed in the *m* values for these two procedures, such as ~0.048 from SRJ and ~0.043 from CSR at $N = 2$ turns. However, it is apparent that the overall trend is consistent for these two testing methods through the nanoindentation analysis. Thus, the *m* value of ~0.035-0.040 in the as-extruded condition prior to HPT was enhanced significantly to $m \approx 0.043$-0.047 after HPT through 1/2 turn and thereafter the values remain reasonably constant through 2 turns. The result is in good agreement with a recent report on a pure Mg after extrusion showing $m = 0.03$ which was also measured by the CSR method through nanoindentation analysis [9].

It should be noted that the ZK60 alloy demonstrated the hardness behavior of strain hardening without any apparent dynamic recovery [10] whereas, as documented in a recent report [11], most commercial purity metals and alloys exhibit this type of hardness behavior. There are two additional very different hardness behaviors. The first behavior is for the high-purity metals which shows strain hardening followed by softening due to the microstructural recovery. The other behavior is for metals and alloys having low melting temperatures where these materials show strain softening with weakening and a representative metal for this behavior is a Zn-22% Al eutectoid alloy [12].

Zn-22% Al eutectoid alloy

The experiments were conducted using a commercial Zn-22% Al eutectoid alloy consisting of a binary microstructure with an Al-rich α phase and a Zn-rich β phase. Following the conventional procedure as described in the earlier section, the processing by HPT was conducted at RT on the Zn-Al alloy under a fixed compressive pressure of 6.0 GPa for total numbers of torsional revolutions of $N = 0$, 1, 2 and 4 turns at 1 rpm where $N = 0$ turn denotes the sample in an as-annealed condition prior to processing.

Following processing, the microstructure at the disk edges was observed by a scanning electron microscopy (SEM) and the micrographs are shown in Fig. 3 for the samples with *N* of (a) 0 turn, (b) 1 turn, (c) 2 turns and (d) 4 turns where the grains appearing white represent the Zn-rich phase and the grains appearing black represent the Al-rich phase [13].

As shown in Fig. 3(a), the initial material contains both essentially equiaxed grains with an average size of ~1.4 μm and a lamellar structure with thicknesses of ~100 nm. However, this binary microstructure of equiaxed and lamellar structures disappeared at the disk edges after HPT and instead there were refined microstructures with only equiaxed ultrafine grains as seen in Fig. 3(b)-(d). Moreover, a banded microstructure by an agglomeration of each phase was observed at the edges of the disks in an early stage of HPT though 2 turns whereas the equiaxed grains with homogeneous phase distributions were observed in the sample after HPT for 4 turns. Significant grain refinement to ~400 nm and ~350 nm was observed through HPT after 2 and 4 turns, respectively [12,13].

Nanoindentation measurements were conducted on the as-annealed sample and on the disk edges of the Zn-Al alloy after HPT through 4 turns at four different indentation strain rates from 0.0125 to 0.1 s^{-1} [13]. The value of *m* was calculated by measuring the slope of the line for each sample in a logarithmic plot of H/3 versus constant displacement rate as shown in Fig. 4 where the strain rate is empirically equivalent to two orders of magnitude slower than the indentation strain rate. It is apparent that the estimated *m* value of 0.125 for $N = 0$ was enhanced significantly to ~0.226 after HPT for 1 turn, thereby demonstrating the improvement in plasticity of the alloy through HPT. A maximum value of $m \approx 0.265$ was calculated after 2 turns and it decreased slightly after 4 turns.

The change in *m* is related to the capability of grain boundary sliding influenced by the homogeneous distribution of the two phases with increasing torsional straining by HPT [14]. Nevertheless, the results revealed superior plasticity at RT in the Zn-Al alloy after HPT. The result is in good agreement with a recent report for a Zn-22% Al alloy with very high purity demonstrating a similar value of $m \approx 0.24$ measured by tensile testing at RT and the alloy demonstrated excellent RT superplasticity with an elongation to failure of 500% at 10^{-3} s^{-1} [15].

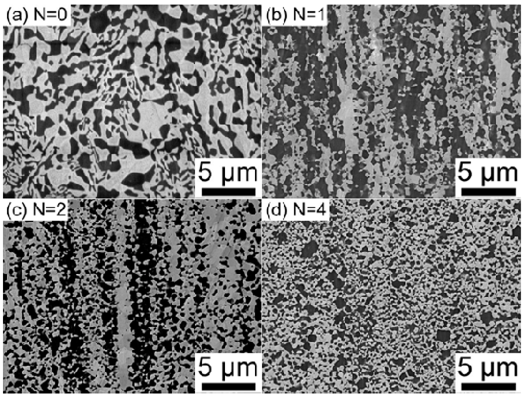

Fig.3 SEM photos taken at the edges of the disks for (a) N = 0, (b) N = 1, (c) N = 2 and (d) N = 4. [13].

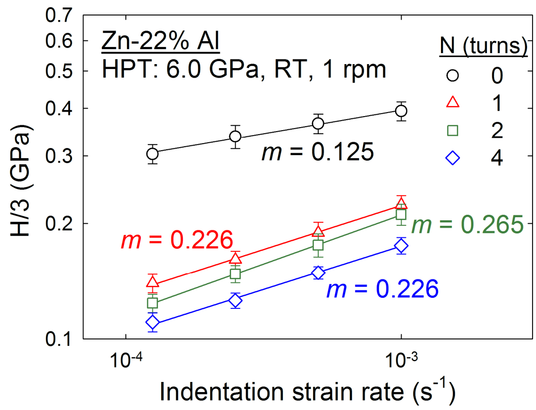

Fig. 4 Variation in strain-rate sensitivity for the Zn-Al alloy before and after HPT for increasing numbers of turns [13].

High entropy alloy

The high entropy alloy (HEA) system having a composition of $Co_{20}Cr_{20}Fe_{20}Mn_{20}Ni_{20}$ (in at.%) was prepared by casting. A detailed examination showed the as-cast HEA has an f.c.c. single phase with the average grain size of ~40 μm before HPT processing [16]. The alloy was processed by HPT in a conventional manner at RT under 6.0 GPa for totals of 1/4, 1/2, 1 and 2 turns.

The microstructure after HPT was observed by transmission electron microscopy (TEM) and the micrographs with SAD patterns are shown in Fig. 5(a) and (b) for the disk edges of the HEA after N = 1/4 and 2 turns, respectively [16]. The direct measurement from the micrographs showed the alloy contains equiaxed fine grains with average sizes of ~60 and ~40 nm at the disk edges after HPT for 1/4 and 2 turns, respectively. The SAD patterns demonstrating clear ring patterns indicates the successful achievement of nanoscale grains without preferred crystallographic orientation for both samples. Thus, the HEA developed a nanocrystalline structure in the very early stages of HPT processing while HEA has a stable microstructure during plasticity attributed to the requirement of a high activation energy leading to sluggish diffusion. It should be noted that there was no evidence of a phase transformation during the processing.

An examination by nanoindentation was conducted on the HEA before and after HPT up to 2 turns at the same indentation strain rates used for the other alloys in the earlier sections. As noted earlier, the values of m were estimated by plotting H/3 versus strain rate for each sample condition as shown in the inset of Fig. 6 and the evolution in the value of m with increasing numbers of HPT turns is shown in the main plot of Fig. 6 where N = 0 denotes the as-cast sample without HPT processing [16].

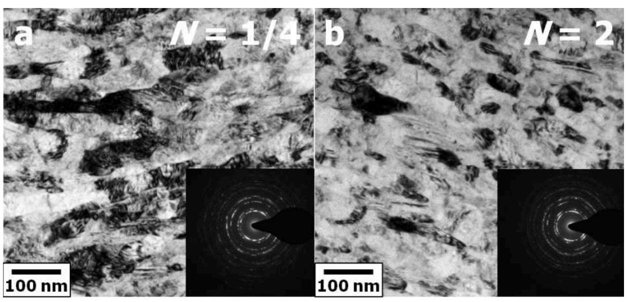

Fig. 5 Representative TEM images and SAD patterns (inset) taken at the edges of the HEA disks after HPT for (a) 1/4 and (b) 2 turns [16].

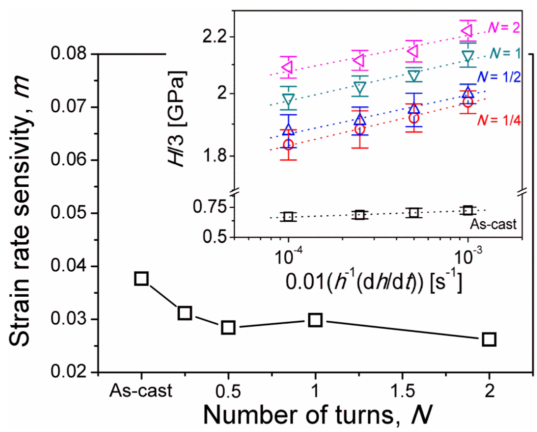

Fig. 6 Variation in m for the HEA before and after HPT for increasing N. Inset showing H/3 vs. strain rate [16].

The *m* value for the as-cast HEA sample was ~0.038 which is much higher than general coarse-grained f.c.c. metals, thereby demonstrating the excellent initial plasticity of the initial HEA. There is a negligible reduction in the value of *m* to ~0.031 after HPT for 1/4 turn and thereafter the value remained reasonably constant with increasing *N* to 2 turns. By contrast, the inset shows the HEA exhibited significant increase in hardness after HPT with increasing numbers of turns.

The results demonstrate that the HPT processing provides an excellent potential for achieving superior strength while maintaining excellent plasticity at RT in the nanostructured HEA. There is very limited research on HEA processed by HPT [17,18] and additional study is indispensable to better understand the significant change towards the nano-scale microstructure through HPT processing at ambient temperature.

Summary

The present report shows successful HPT processing leading to significant grain refinement in a ZK60 magnesium alloy, a Zn-22% Al eutectoid alloy and a high entropy alloy. The examination through nanoindentation shows that these alloys exhibited excellent micro-mechanical responses at room temperature after successful microstructural refinement by HPT. The results demonstrate the feasibility of HPT processing to a wide range of metals and alloys.

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