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Micro-Scale Mechanical Behavior of Ultrafine-Grained Materials Processed by High-Pressure Torsion

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Abstract. Bulk ultrafine-grained (UFG) materials usually show superior mechanical and physical properties. The development of micro-mechanical behavior is observed after significant changes in microstructure through high-pressure torsion (HPT) processing. This report summarizes recent results on the evolution of small-scale mechanical response examined by the nanoindentation technique on two UFG materials including a high-entropy alloy and an Al-Mg metal matrix nanocomposite processed by HPT. Special emphasis is placed on demonstrating the interrelationship of essential microstructural changes with increasing torsional strain and applying a post-deformation annealing treatment and the evolution of the micro-mechanical behavior in these UFG materials by estimating the strain rate sensitivity.

Introduction

The processing of metals through the application of severe plastic deformation (SPD) was well demonstrated for achieving exceptional grain refinement in bulk solids in the last two decades. Among the various SPD techniques, one of the most effective procedures for grain refinement refers to the processing by high-pressure torsion (HPT). The exceptional microstructural refinement through HPT is not generally achieved using other SPD procedures [1]. The fundamental principles of HPT processing were well described in an earlier review [2]. Specifically, a metal disk is applied to receive severe torsional straining under very high compressive pressure. Numerous reports are now available showing there is an excellent potential for achieving superior mechanical properties in the HPT-processed metals having ultrafine grains.

For characterizing mechanical properties of the HPT-processed materials, it is important to note that the properties taken at the overall disk surfaces are often very different from those measured at the local regions at the disk center or peripheries. This is attributed to the principles of HPT processing where accumulated torsional strain increases from the disk center towards the periphery [3]. Thus, the mechanical properties and texture of the materials after HPT may vary depending on the measurement location [4] which suggests a new strategy for the application of a nanoindentation technique for characterizing the ultrafine-grained (UFG) materials processed by HPT [5].

This study was initiated to summarize very recent reports evaluating the correlation between the microstructural changes and evolution in hardness and plasticity by calculating the strain rate sensitivity through nanoindentation testing on a high entropy alloy (HEA) and an Al-Mg alloy system produced by HPT and additional annealing. The report suggests the recognition of a consistent trend of enhanced micro-mechanical response in the UFG metals and alloys processed by HPT.

High Entropy Alloy Processed by HPT

An HEA system having a composition of $Co_{20}Cr_{20}Fe_{20}Mn_{20}Ni_{20}$ (in at.%) was prepared by casting. The as-cast HEA before HPT has an f.c.c. single phase with the average grain size of ~40 μm [6]. After machining the material into disks with a 10 mm diameter and ~0.83 mm thickness, the disks were processed by HPT at RT under a pressure of 6.0 GPa for totals of 1/4, 1/2, 1 and 2 turns. An XRD analysis demonstrated that there was no phase transformation during HPT processing.

The microstructure observed by transmission electron microscopy (TEM) with an SAD pattern is shown in Fig. 1 for the edge regions of the HEA after HPT for (a) 1/4 turn and (b) 2 turns [6]. The micrographs show reasonably equaxed grains with sizes of ~60 and ~20 nm at the disk edges after HPT for 1/4 and 2 turns, respectively. The significant microstructural refinement to nanoscale grains is also suggested by the SAD patterns demonstrating clear ring patterns without significant preferred crystallographic orientation for both samples. Thus, the HEA developed a true nanocrystalline structure in the very early stages of HPT while HEAs are known to have a stable microstructure during plasticity attributed to the requirement of a high activation energy leading to sluggish diffusion.

Nanoindentation measurements were conducted on the as-cast HEA and on the disk edges of the HEA after HPT up to 2 turns at four different indentation strain rates from 0.0125 to 0.1 s^{-1} [6]. The values of *m* were calculated by measuring the slope of the datum line for each sample in a logarithmic plot of H/3 versus constant displacement rate as shown in the inset of Fig. 2 where H denotes nanoindentation hardness. The inset shows the HEA exhibited significant increase in hardness after HPT and with increasing numbers of HPT turns.

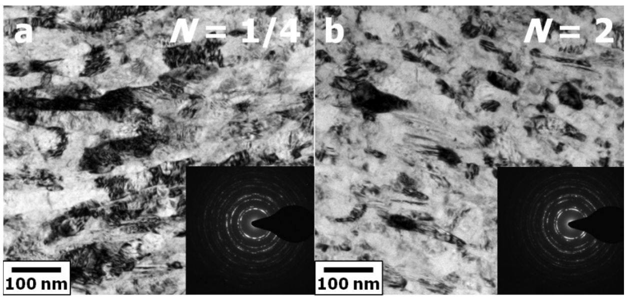

Fig. 1 Representative TEM images and SAD patterns (inset) taken at the edges of the HEA disks after HPT for (a) 1/4 and (b) 2 turns [6].

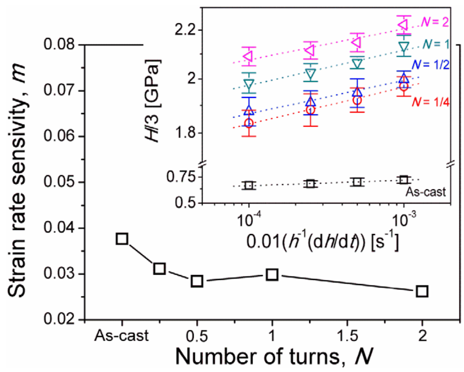

Fig. 2 Variation in *m* for the HEA before and after HPT for increasing HPT turns. Inset showing *H*/3 vs. strain rate [6].

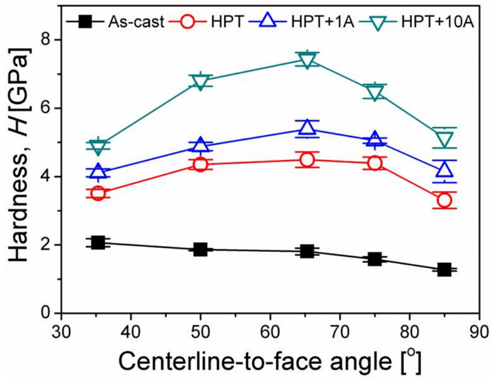

Fig. 3 Variations in nanoindentation hardness as a function of indenter angle for the HEA after HPT and annealing [8].

The *m* value of the HEA in the as-cast HEA sample was ~0.038 which is much higher than general coarse-grained f.c.c. metals where it is generally $m \approx 0.01$ at grain sizes larger than 1 μm [7], thereby demonstrating high plasticity of the initial HEA. HPT processing resulted in a negligible reduction in the value of *m* to ~0.031 after HPT for 1/4 turn and thereafter the value remained reasonably constant through 2 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.

A very resent nanoindentation study demonstrated annealing-induced hardening in the HPT-processed HEA for 2 turns followed by annealing at 450 ºC for 1 and 10 hours (designated as HPT+1A and HPT+10A, respectively) due to the formation of a nanostructured multiphase microstructure [8]. The results with several different indenter angles are shown in Fig. 3 [8].

Micro-Mechanical Properties of the Al-Mg Hybrid System Processed by HPT

The Al-Mg intermetallic-based metal matrix nanocomposite (MMNC) was synthesized by HPT up to 20 turns and after HPT for 20 turns followed by post-deformation annealing (PDA). Specifically, the HPT-processed material was prepared from separate conventional metals of Al (Al-1050 alloy) and Mg (ZK60 alloy) through diffusion bonding at RT by applying conventional HPT processing [9-11]. The separate Al and Mg disks were well bonded without any segregation even after HPT for 5 turns due to diffusion bonding [10]. Thereafter, the disk edge where the highest torsional straining is available showed no presence of Mg-rich phase and all Mg dissolved into the Al matrix, and formed an Al-Mg intermetallic phase after 10 turns [9]. Thus, the disk edge of the Al-Mg alloy is transformed into an intermetallic-based MMNC through HPT.

Figure 4 shows the microstructure at the disk edges after (a) HPT for 20 turns and (b) HPT followed by PDA at 523 K for 1 hour [11]. A reasonably equiaxed grain structure was observed with an average grain size of $d \approx 60$ nm in the HPT-processed Al-Mg system and after PDA the material demonstrated a homogeneous equiaxed microstructure with an average grain size of $d \approx 380$ nm.

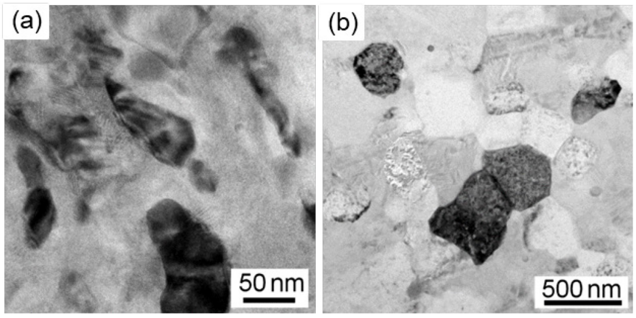

Fig. 4 TEM images taken at the disk edges after (a) HPT for 20 turns and (b) HPT followed by PDA in the Al-Mg system [11].

The micro-mechanical behavior of the Al-Mg metal system was examined by applying the nanoindentation technique. All measurements were conducted under a predetermined peak applied load of P_{max} = 50 mN at constant indentation strain rates from 1.25×10^{-4} to 1.0×10^{-3} s^{-1}. Figure 5 shows representative load, P, versus displacement, h, curves for the Al-Mg disk edges after (a) HPT for 20 turns and (b) HPT followed by PDA when measuring at four equivalent strain rates [11].

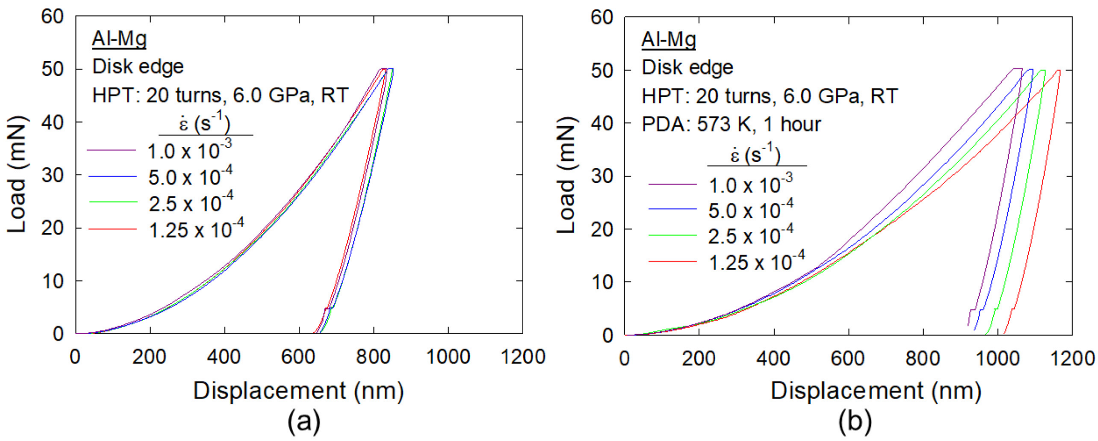

Fig. 5 Representative load-displacement curves for the Al-Mg disk edges after (a) HPT for 20 turns and (b) HPT and PDA when measuring at four strain rates of 1.0×10^{-3} - 1.25×10^{-4} s^{-1} under a predetermined maximum peak load of 50 mN [11].

It is apparent by comparing these P-h curves between the two sample conditions that the Al-Mg alloy system after HPT without PDA showed much lower displacements than the material after HPT followed by PDA at all strain rates, thereby indicating high hardness of the Al-Mg disk edge immediately after HPT for 20 turns. Closer inspection showed the disk edge immediately after HPT having true nano-scale grains demonstrated all separate *P-h* curves in reasonably consistent locations at all four strain rates as shown in Fig. 5(a). This demonstrates no strain rate dependency of plasticity in the testing conditions and implies a typical character of UFG metals demonstrating hard and brittle responses. On the contrary, the microstructure after HPT was recovered and showed grain growth through PDA so that the *P-h* curves for this material exhibited an apparent positive strain rate dependency where there is increasing displacements at slower strain rates of nanoindentation as shown in Fig. 5(b). Thus, a significant improvement in plasticity was attained by PDA in the HTP-processed Al-Mg alloy system.

The micro-mechanical behavior of the Al-Mg system after HPT for 20 turns was evaluated by calculating the strain rate sensitivity, *m*, from the slopes of the lines in a logarithmic plot of H/3 against strain rate as shown in Fig. 6 [11]. It should be noted that the error bar on each datum point represents the standard deviation of the total numbers of measurements of 20 tests but the error ranges are too small to recognize in the plot.

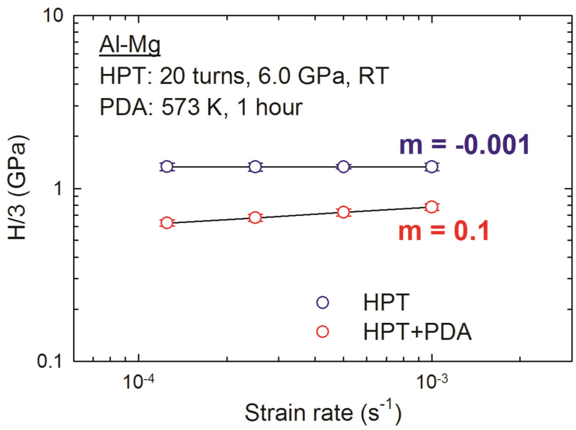

Fig. 6 Variations in the strain rate sensitivity with increasing strain rate for the disk edges of the Al-Mg system after HPT for 20 turns and after HPT and PDA [11].

The analysis estimated *m* values of −0.001 and 0.1 for the Al-Mg alloy system after HPT for 20 turns and HPT followed by PDA, respectively. Thus, the strain rate sensitivity was very low after HPT and the PDA treatment providing a significant enhancement in the *m* value in the Al-Mg system, thereby demonstrating the significance of PDA for improving plasticity in the UFG materials processed by HPT.

It is important to note that the improved *m* value of 0.1 for the PDA-treated Al-Mg system processed by HPT is even higher than the reported *m* values of ~0.07 for a commercial purity Al after ECAP for 6-12 passes at RT [12-15] and after accumulative roll bonding (ARB) for 8 cycles at RT [13] and ~0.035-0.050 for a ZK60 alloy after HPT for 2 turns at RT [16] where all these reference data were also acquired through nanoindentation testing. Thus, the UFG alloy system synthesized by HPT followed by PDA demonstrates a significantly higher value of *m* in comparison with any initial material and any similar alloys processed by SPD.

The nanoindentation analysis showed feasibility of a PDA treatment for enhancing the plasticity of the Al-Mg system after HPT while the microstructure was maintained reasonably in the nano-scale as shown in Fig. 4(b). The significance of PDA was demonstrated earlier for improving the overall ductility of nanostructured Ti after HPT [17]. Specifically, PDA enables the production of an ordering of the defect structures within the grain boundaries leading to an equilibrium state without any significant grain growth [18]. Moreover, short-term annealing reduces the dislocation density in the grain interior of the UFG materials so that the dislocation storage capacity may increase and thus the strain hardening capability is enhanced. This leads to a high potential for achieving excellent ductility in the UFG material after SPD.

Significance of a Nanoindentation Technique for UFG Metals Processed by SPD

Recent developments in characterization techniques provide a better understanding of the enhanced mechanical properties of UFG materials processed by SPD. In particular, the novel technique of nanoindentation has become a common tool for the simultaneous measurements of sets of essential mechanical properties on the material surfaces and interfaces at the submicron scale. [19]. There have been several studies to date demonstrating the use of the nanoindentation technique for examining the mechanical properties and parameters of SPD-processed metals. A very recent review examined and summarized the available experimental results showing the enhancement in strength and ductility in terms of the micro-mechanical response at room temperature in a range of metals and alloys processed by several different SPD processing procedures [5].

The main benefit of applying the nanoindentation technique is because of the requirement of limited volume for each measurement. It is especially favorable for relatively small HPT-processed materials prepared in the current laboratory-scale studies. Moreover, the microstructure after HPT processing often includes a gradient-type nanostructure in terms of grain size, phase and composition [20] and thus the nanoindentation tests allows an examination at arbitrarily selected local points within a sample. A recent report successfully examined the presence of a newly formed phase of an intermetallic compound having thicknesses less than 30 µm at an interface of the two-phase Al-Mg alloy system after HPT followed by annealing [21]. Thus, in addition to conventional tensile testing, the nanoindentation technique is promising for examining mechanical response of UFG materials processed by HPT where the materials may have smaller overall dimensions and include gradient-type microstructures. Further investigations are needed to fully develop this approach.

Summary

The present report shows successful HPT processing leading to significant grain refinement in a high entropy alloy and an Al-Mg alloy system produced from two separate metals. An examination through nanoindentation shows that these alloys exhibit excellent micro-mechanical responses at room temperature after HPT and after an additional PDA treatment. The novel technique of nanoindentation provides a wide range of information including mechanical properties and the local microstructure. The capability of the measurements will have an excellent potential for examining the UFG materials processed by HPT.

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