Materials Science Forum
ISSN: 1662-9752, Vol. 1016, pp 3-8
doi:10.4028/www.scientific.net/MSF.1016.3

Submitted: 2020-01-15
Revised: 2020-01-21
Accepted: 2020-05-13
Online: 2021-01-05

Recent Developments in the Processing of Advanced Materials Using Severe Plastic Deformation

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Keywords: Hardness, High-pressure torsion, Nano-composites, Severe plastic deformation, Ultrafine grains

Abstract. The processing of bulk metals through the application of severe plastic deformation (SPD), using procedures such as equal-channel angular pressing (ECAP) and high-pressure torsion (HPT), is now well established for the fabrication of materials with exceptionally small grain sizes, usually in the submicrometer range and often having grain sizes at the nanometer level. These grain sizes cannot be achieved using thermo-mechanical processing or any conventional processing techniques. Recently, these procedures have been further developed to process alternative advanced materials. For example, by stacking separate disks within the HPT facility for the synthesis of bulk nanocrystalline metastable alloys where it is possible to achieve exceptionally high hardness, or by pressing powders or metallic particles in order to obtain new and novel nano-composites exhibiting unusual properties.

Introduction

Conventional thermo-mechanical processing (TMP) entails the use of mechanical deformation processes, such as compression, forging or rolling, together with heat treatments in order to produce materials having improved properties such as high strength and/or grain refinement. However, the minimum grain size attainable by TMP is generally of the order of a few micrometers and it is not generally feasible to use this approach for the production of metals having grain sizes within the submicrometer or the nanometer range. In practice, it is attractive to produce metals having grain sizes in these very small ranges because it follows from the Hall-Petch relationship [1,2] that these small grains will lead to a high strength and they may provide, for example, a potential for achieving a superplastic forming capability. The strength-grain size relationship is now well documented for a very wide range of metals [3] and it has become of increasing importance in recent studies designed to develop new and advanced functional materials.

Over the last two decades it has become well established that submicrometer or nanometer grains may be achieved by processing metals through the application of severe plastic deformation (SPD) where these processing methods refer to procedures in which a sample is subjected to a very high strain without incurring any significant change in the overall dimensions of the sample [4]. The two main examples of these procedures are equal-channel angular pressing (ECAP) where a sample is pressed through a die constrained within a channel [5] and high-pressure torsion (HPT) where a disk is subjected to an applied pressure and concurrent torsional straining [6] but HPT is especially attractive because it produces smaller grain sizes than ECAP [7,8] and also a higher fraction of grain boundaries having high angles of misorientation [9]. Recently, there have been new developments in processing by HPT in order to produce new advanced materials. The principles of these developments are reviewed in this report.

The Principles of Conventional Processing by HPT

In conventional HPT, a single disk is placed between two massive anvils in an HPT facility and subjected to an applied pressure, *P*, and then to torsional straining through rotation of one of the anvils. Typically, the processing is conducted under quasi-constrained conditions where there is a small outflow of material around the periphery of the disk during the processing operation [10].

An example is shown in Fig. 1 for disks of an Al-7075 alloy having diameters of 10 mm and processed by HPT at room temperature (RT) under a pressure of 6.0 GPa [11]. This plot shows the measured Vickers microhardness across diameters of the disks after processing through numbers of turns, *N*, from 1/8 to 10, where the lower broken line is the hardness value in the unprocessed and as-annealed condition and the experimental points were recorded at regular intervals across each disk. The results show that initially the hardness increases at the disk edges but with only a small increase in the central region. This result is reasonable because the imposed strain varies with the position on the disk with a maximum strain occurring at the edge and with no strain imposed in the center of the disk. But after increasing numbers of turns the hardness increases in the center to the extent that there is a reasonably homogeneous distribution of hardness values across the diameter after a total of 10 turns.

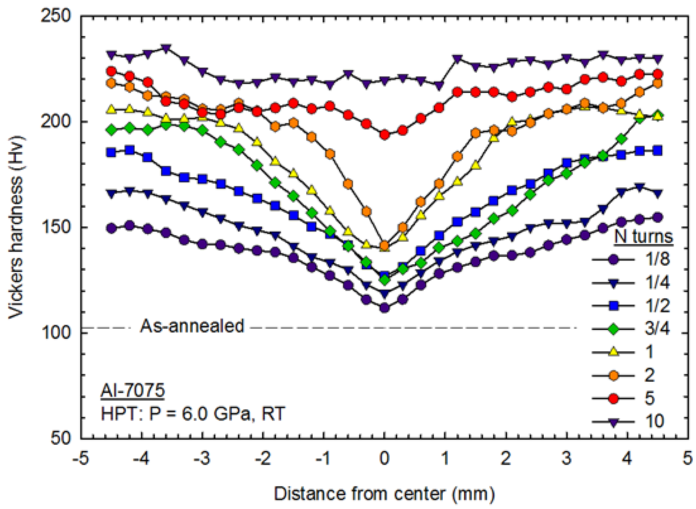

Fig. 1 Vickers microhardness across diameters of disks for various numbers of turns after processing Al-7075 by HPT [11].

This type of behavior is termed *without recovery* and it is typical of a large number of metals where the hardness increases with equivalent strain and then essentially saturates [12]. However, in some materials, such as pure Al [13], the material may initially harden and then soften to a saturation value in the behavior termed *with recovery* and in other materials, such as the Bi-Sn eutectic alloy [14], the material softens to a lower saturation level in the behavior termed *with weakening* [12]. There are various microstructural effects that account for these different types of behavior [15].

Bulk-State Reactions to Synthesize Metal-Matrix Nanocomposites and Bulk Metastable Alloys

Numerous recent studies demonstrated a new feasibility of HPT processing for the synthesis of intermetallic-based bulk nanocomposites and ultimately nanocrystalline bulk metastable alloys. A comprehensive summary of the new strategy was reported recently [16] reviewing several unique sample set-ups of dissimilar conventional metals and alloys in HPT processing to let the materials show bulk-state reactions at room temperature and the enhanced functionalities of the mechanically-bonded nanocrystalline hybrid alloy systems.

One of the most reported sample set-ups for the bulk-state reactions in HPT processing is shown in Fig. 2 where separate metal disks of Al-1050 (Al) and ZK60 magnesium (Mg) alloys are stacked in the order of Al/Mg/Al and the conventional HPT procedure involving severe compressive pressure and concurrent torsional straining is applied at room temperature without any special sample surface treatment [17].

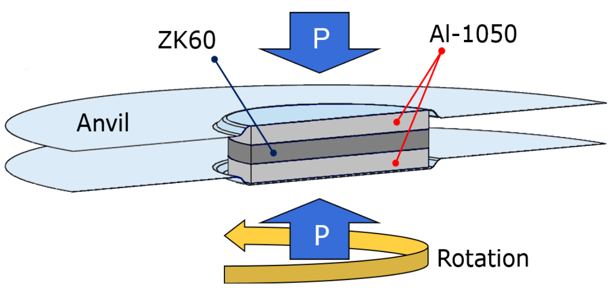

Fig. 2 Schematic illustration of the sample set-up for the bulk-state reaction in the HPT facility [17].

Fig. 3(a) describes a series of micrographs taken at the vertical cross-sectional planes of the hybrid Al-Mg alloy system after HPT for, from top, 1, 5, 10, 20 and 100 turns under 6.0 GPa at room temperature [16-18]: the dark phase represents the Mg-rich and the bright phase represents the Al-rich in the microstructure. When dissimilar metal disks are mechanically bonded by HPT, the separate disks tend to diffusion bond without any visible segregations. The continuous interfaces of the separate phases are visible throughout the disk diameter and form a multi-layered structure when the stacked metals are compressed and torsionally strained in the very early stage of HPT for 1 turn.

Increasing the numbers of HPT turns to 5 demonstrates a severe mixture of the dissimilar metal phases at the disk edge and this complicated microstructure extends towards the disk centers through 10 and 20 turns, while the multi-layered microstructure remains at the disk centers after 20 turns. A series of detailed structural examinations concluded that the formation of nanostructured Al-Mg intermetallic compounds of Al_3Mg_2 and $Al_{12}Mg_{17}$ was observed in the severe phase mixtures at the Al-Mg disk edges leading to the synthesis of metal-matrix nanocomposites by HPT. By contrast, further processing for 100 turns under 6.0 GPa demonstrates a homogeneous nanostructure with complete dissolution of Mg and the earlier formed intermetallic phases within the Al matrix and ultimately this introduces a bulk nanostructured metastable Al alloy in a supersaturated solid solution state [18].

The hardness variations along the disk diameter for the HPT-induced Al-Mg system after 5, 10, 20 and 100 turns are shown in Fig. 3(b) [18] where the dashed horizontal lines are the saturation hardness values observed for the Al-1050 and ZK60 alloys after HPT for 5 turns for comparison purposes. The low hardness values at the disk centers are initiated by the presence of the multi-layered structure at the disk centers up to 20 HPT turns. At the disk edges, the severe phase mixture by HPT demonstrates the achievement of extreme hardness of Hv ≈ 260 and 330 after 10 and 20 turns, respectively. An increase in the upper limit of hardness at the disk edges was attributed mainly to the significant grain refinement with a minor but essential contribution of the hard Al-Mg intermetallic compounds through the activated fast diffusion. Moreover, the extreme severe plastic deformation for 100 HPT turns leads to a homogeneous microstructure yielding a uniform distribution of Vickers microhardness of ~350 across the disk diameter.

These series of studies on the bulk-state reaction and mechanical bonding of separate dissimilar metals by HPT demonstrates significant opportunities for the production of advanced metallic alloys and composites as well as for excellent contributions to current manufacturing techniques in diffusion bonding, welding and mechanical joining.

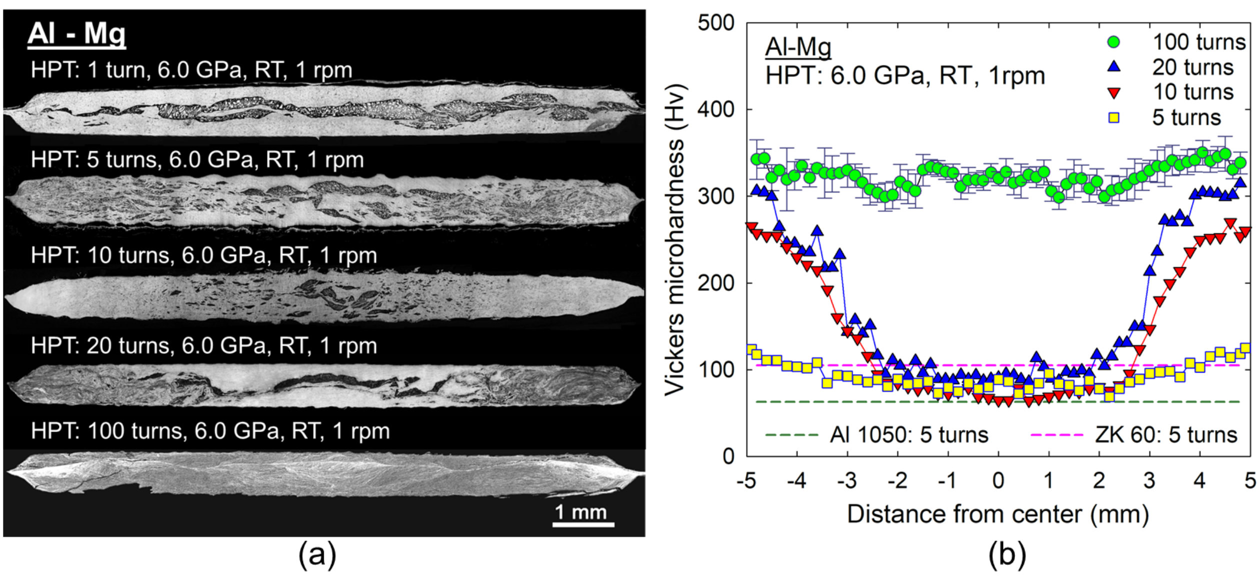

Fig. 3 (a) Cross-sectional microstructure of the Al-Mg alloy system after 1, 5, 10, 20 and 100 HPT turns and (b) the hardness distributions across the disk diameter of the HPT-induced Al-Mg alloy disks after 5, 10, 20 and 100 HPT turns [16-18].

The Incorporation of Hard Particles to Synthesize Metal-Matrix Nanocomposites

HPT also provides the opportunity to consolidate mixtures of particles. As a consequence, particles of a hard phase can be introduced in a softer nanostructured metallic matrix to produce a metal matrix nanocomposite. Recent reports demonstrated the successful incorporation of Al_2O_3 [19,20], AlCuFe quasicrystals [21], hydroxyapatite and bioactive glass [22] particles into a magnesium matrix. Magnesium was used as the matrix material due to its low density and biocompatibility which makes this material attractive for multiple applications.

High-pressure torsion processing promotes deformation and bonding of the metallic particles producing a solid disk. It was observed that agglomerations of the second phase particles may take place at the early stage of processing [19] but an increase in the number of turns promotes homogenization. The hard particles can increase the deformation imposed to the matrix during processing promoting enhanced grain refinement [20] and may increase the strength. Figure 4 shows the flow stress of pure Mg and an Mg-Al_2O_3 composite plotted as a function of the strain-rate and it is observed that the hard ceramic particles increase the overall strength of the composite. Tensile tests also revealed an increase in strength of a Mg-Quasicrystal composite compared to the unreinforced metal although the ductility was compromised [21].

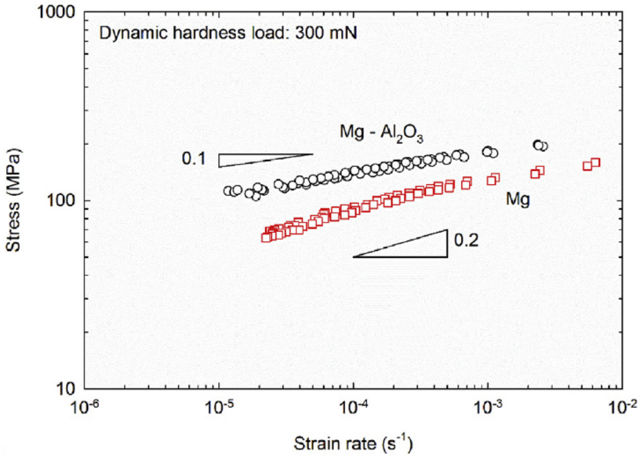

Fig. 4 Flow stress plotted as a function of the strain-rate for pure Mg and an Mg-Al_2O_3 composite [19].

In addition to improvements in mechanical properties, the particles can contribute to biological response of the material. It is known that magnesium is a biocompatible and biodegradable material. Thus, it can be used to produce temporary implants which gradually degrade. A recent report [22] showed it is possible to introduce bioactive particles such as hydroxyapatite (HA) and bioactive glass (BG) in a magnesium matrix to produce bioactive and biodegradable composites. Figure 5 shows the microstructure of an Mg-HA composite with elemental distributions of Mg, Ca, P and O and it is observed a homogeneous distribution of HA in a magnesium matrix. It is known that hydroxyapatite displays good interaction and can improve growth of cells. These results showed good strength, ductility and biocompatibility of this composite. This provides the opportunity to develop metallic biodegradable implants with the ability to gradually deliver the particles incorporated into the composite.

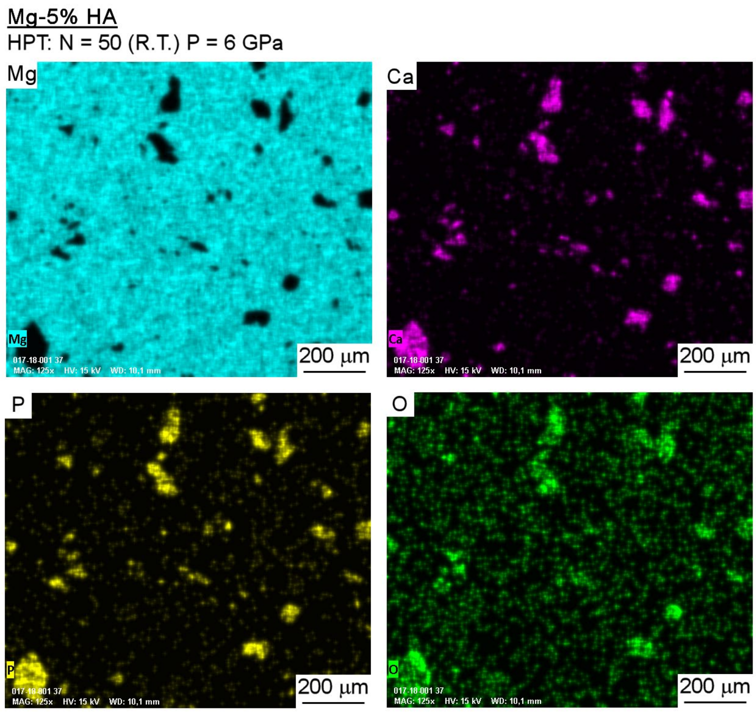

Fig. 5 Microstructure of an Mg-HA composite produced by HPT [22].

Fabrication of Composites Reinforced with Graphene Nanoplates

The fabrication of metal matrix composites generally involves the use of continuous fibre or particle reinforcements. An alternative is to use graphene nanosheets consisting of multiple layers of graphene that may be obtained more easily than single layer graphene [23]. These nanosheets have been used extensively for the production of graphene-reinforced aluminum matrix composites but the processing techniques involve procedures such as hot isostatic pressing, sintering or friction stir processing and these are high-temperature methods which may cause oxidation or the production of Al_4C_3. Processing by HPT has the advantage that it can be conducted at relatively low temperatures.

Experiments were conducted in which an Al powder similar to Al-1050, with a mean particle size of 125 μm, was mixed with 5% of graphene nanoplates (GNPs), subjected to pressing under a pressure of 40 MPa for 1 minute to provide good compaction and then processed by HPT with P = 6.0 GPa through various numbers of turns up to a maximum of 20 [24]. Miniature tensile specimens were cut from the pressed disks and then pulled to failure at room temperature (298 K) at an initial strain rate of 1.0×10^{-3} s^{-1}. Figure 6 shows examples of the engineering stress-strain curves for the samples processed by HPT through 20 turns at temperatures of 298, 373 and 473 K. These curves show that the strength of the nanocomposite decreases with increasing processing temperature and there is a maximum strength of ~350 MPa after processing at 298 K. For comparison, Al-1050 sheet of commercial purity and in a work hardened state generally exhibits tensile strengths within the range of ~105-145 MPa thereby demonstrating the potential for achieving exceptional strengthening through the addition of only 5% of GNPs. It should be noted that the measured grain size in the nanocomposite processed by 20 turns at 298 K was ~70 nm thereby showing that grain refinement makes a significant contribution to the overall strengthening of the nano-composite.

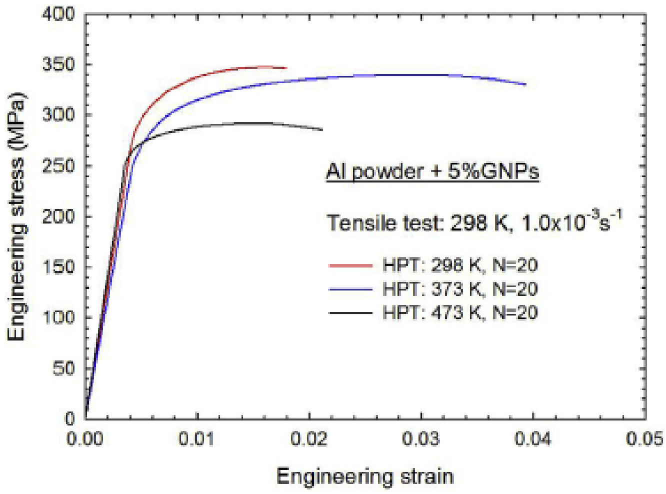

Fig. 6 Engineering stress-strain curves for samples of Al with 5% GNP processed for 20 turns at three different temperatures and tested at 298 K [24].

Acknowledgements

This work was supported in part by the National Science Foundation of the United States under Grant No. DMR-1810343 (MK), in part by the Serrapilheira Institute under Grant #Serra-1709-17750 (RBF) and in part by the European Research Council under ERC Grant Agreement No. 267464-SPDMETALS (TGL).

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