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Developing magnesium-based composites through high-pressure torsion

M. M. Castro[1], P. H. R. Pereira[1], R. B. Figueiredo[1], T. G. Langdon[†,2]

[†]langdon@soton.ac.uk

[1]Department of Metallurgical and Materials Engineering, Universidade Federal de Minas Gerais, Belo Horizonte, 31270-901, Brazil
[2]Materials Research Group, Department of Mechanical Engineering, University of Southampton, Southampton, SO171BJ, U. K.

Magnesium and its alloys display interesting properties such as low density and biocompatibility but the lack of ductility and low strength compromise their performance in many applications. Fabrication of metal matrix composites may alleviate these challenges and improve overall performance. Magnesium matrix composites can be produced by cold consolidation of particles through high-pressure torsion and this paper summarizes recent findings in processing routes for the fabrication of composites, the microstructure developed, mechanical properties obtained and potential applications. It is shown that ductile materials can be mixed with magnesium by processing half samples placed side by side and hard and brittle materials can be incorporated by processing mixed particles. The distribution of phases may be controlled by the amount of rotation imposed during processing. Well-dispersed second phase particles within a continuous magnesium matrix can be obtained. Thus, it is possible to incorporate bioactive materials within a biodegradable magnesium matrix. Detailed characterization using transmission electron microscopy reveals the processing also refines the grain structure of the metallic matrix. The composites may display good ductility, improved strength and an ability to precipitate intermetallics through thermal treatment. The composites produced using high-pressure torsion have different potential applications including the development of bioactive and biodegradable biological implants.

Keywords: biodegradable material, composites, high-pressure torsion, magnesium, mechanical properties.

1. Introduction

Magnesium and its alloys exhibit very low density and they are biocompatible and biodegradable. These characteristics make these materials attractive to produce low-weight components and temporary biological implants. However, it is of great interest to improve other properties that may compromise their performance, such as mechanical strength, ductility and corrosion resistance. Thus, research has focused on evaluating the effect of alloying elements or thermo-mechanical processing on the properties of magnesium. Another route to improve the aforementioned properties is through the incorporation of different materials to produce magnesium-based composites.

The fabrication of metal-based composites is usually carried out through dispersion of the different material in molten metal followed by solidification or through the sintering of a mix of particles. Both routes require heating which can trigger reactions between the different materials. Moreover, magnesium is a highly reactive metal which undergoes significant oxidation at high temperatures. Another route to produce metal-based composites is through the cold consolidation of particles using high-pressure torsion (HPT). The high compressive stresses and severe shear straining, which are typical of this process, promote bonding of the metallic particles and the formation of a solid disc. The consolidation of copper [1,2] and aluminum [3–7] particles have been reported as well as other metallic materials [8–10].

High-pressure torsion refines the grain structure of pure magnesium and its alloys and may improve strength, ductility and introduce superplastic properties [11]. Also, this processing technique does not compromise biocompatibility of these materials [12,13] and may enhance corrosion resistance [12,14–19]. Recent papers have reported the use of this technique to produce different magnesium-based composites. The present paper provides a summary of the major findings in this area. It is divided into sections on the processing of the composites, the microstructures obtained, the mechanical properties and potential applications.

2. Processing

Different routes have been tested for mixing magnesium with other materials including the stacking of discs, placing half discs side by side and mixing particles followed by HPT processing. The effectiveness of the route depends on the nature of the materials being mixed. It has been shown that pure magnesium machining chips can be fully consolidated after 5 turns of HPT while the boundaries between the original chips of the magnesium alloy AZ91 are still visible after the same number of turns [20].

The incorporation of hard and brittle particles, such as ceramics, is carried out by mixing particles. In this case, the amount of each phase and the size ratio between the particles play a role in the bonding and the dispersion of the phases. Fig. 1 shows X-ray tomography images of discs produced by compaction of pure magnesium machining chips mixed with alumina particles. As the size of the alumina particles is significantly smaller than the chips, a high heterogeneity of distribution of phases takes place in the early stages of processing and a thick agglomeration of alumina was observed after 1 turn (shown at centre). A high number of turns is required in order to produce a solid disc with a well-dispersed reinforcement phase [21]. Accordingly, homogeneous distributions of particles of bioactive glass and hydroxyapatite in magnesium matrices were also obtained after 10 and 50 turns of HPT, respectively [22].

In the case of incorporation of a ductile phase, such as metallic aluminum, it is possible to stack discs [23–25] or to place half discs side by side [26]. It has been shown that the bonding between the phases takes place after a reduced number of turns. However, the thickness of the phases decreases with increasing numbers of turns [26]. Fig. 2 shows cross-sectional images, after different numbers of turns, at the centre and at the edge of discs of magnesium-aluminium composites which were produced by initially placing half discs side by side [26]. Good bonding between the phases is observed after only 1 turn and the phases become gradually thinner by increasing the number of turns.

3. Microstructure

The number of phases and their distribution in the microstructure depends upon the number of materials used in the mixing, the volume fraction of each material and their original size. Thus, the processing of particles of pure magnesium, without any additional phase, leads to a single phase disc after sufficient number of turns when the material is observed using optical microscopy [20]. It is, however, expected that the oxide layer of the original particles will be dispersed within the metallic matrix after consolidation. Also, the amount of magnesium oxide within the material increases when decreasing the size of the original particles.

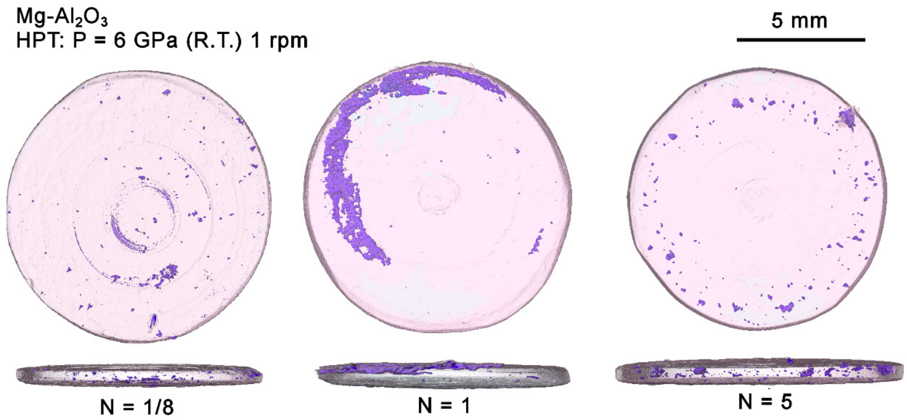

Fig. 1. (Color online) X-ray tomography images of discs of Mg-Al_2O_3 composite at different stages of processing by HPT where *N* denotes the number of turns. Reproduced with permission [21] Copyright 2019, Elsevier.

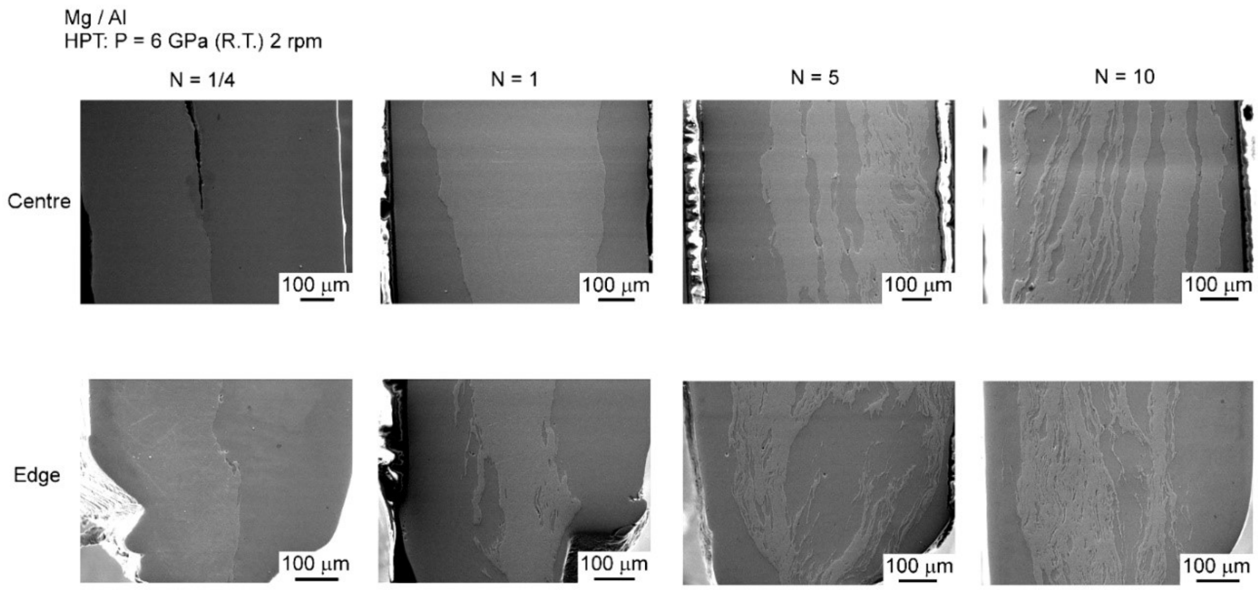

Fig. 2. BSE images of discs of Mg/Al composite at different stages of processing by HPT. Reproduced with permission [26] Copyright 2019, Elsevier.

In fact, transmission electron microscopy images revealed a significant amount of MgO particles after processing nanoparticles of pure magnesium [27,28].

Multiple phases are observed when other materials are mixed with magnesium. Fig. 3 shows a representative image of the microstructure of a composite with 5 wt.%. of hydroxyapatite (HA) particles which were mixed with magnesium particles and processed to 50 turns of HPT [22]. A continuous magnesium matrix is developed and the ceramic phase is apparently well-dispersed. Similarly, a continuous magnesium matrix incorporating ceramic particles is observed in a Mg-10% Al_2O_3 composite [21]. However, as the size of the alumina particles is smaller, the average distance between the particles is also reduced. It is important to note that breakup of the reinforcement phase may take place during HPT processing which leads to a decrease in the size of this phase and in the average distance between phases. This effect has been observed when processing a Mg-bioactive glass (BG) composite in which the original BG particles had dimensions of hundreds of microns and particles of tens of microns were observed after 10 turns of HPT [22].

As the processing is carried out at room temperature and the magnesium matrix is subjected to severe plastic deformation, its grain structure is significantly refined. Fig. 4 shows images of a TEM lamella extracted from a Mg/Al composite [26]. Fig. 4b shows a scanning transmission electron microscopy (STEM) image, which enhances the compositional contrast between the phases. The thickness of the phases is ~1 μm and the grains within each phase are ultrafine, as shown in Fig. 4c.

4. Mechanical properties

The ability to incorporate a second phase and to control the amount and distance between phases provides the opportunity to tailor the mechanical properties of the composite. Furthermore, the pronounced grain refinement induced by the severe plastic deformation affects significantly the mechanical behaviour. Hence, it is expected that the incorporation of a hard phase will increase the overall strength of magnesium. Fig. 5 shows the relationship between the flow stress and the strain rate, determined using hardness creep testing at room temperature, for pure magnesium and a Mg-Al_2O_3 composite processed by HPT [21]. It is known that the grain refinement induced by HPT triggers creep-assisted plastic deformation at room temperature in magnesium [29,30]. As a consequence, this material exhibits moderate strength and a high strain rate

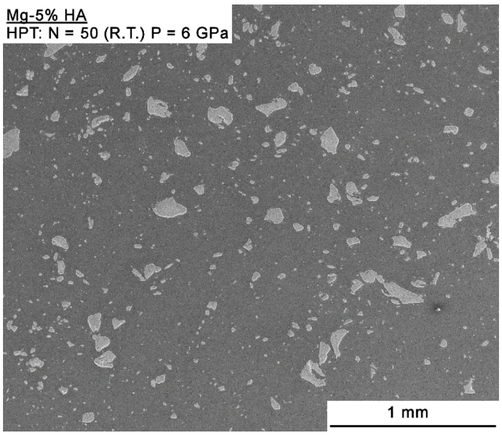

Fig. 3. SEM backscattered electron image of the mid-radius area of the Mg-HA composite [22].

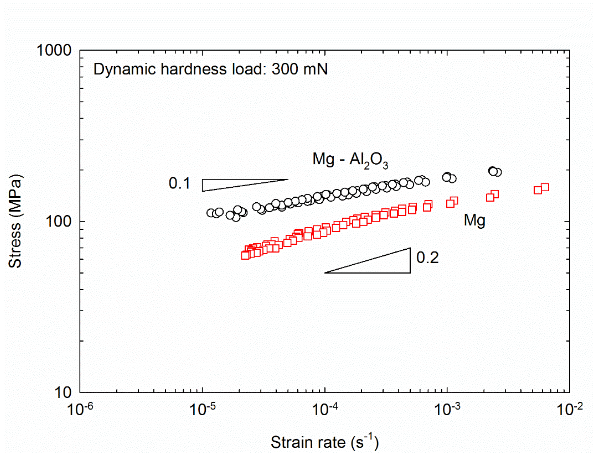

Fig. 5. Stress vs strain rate curves for pure magnesium and a magnesium-alumina composite processed by HPT. Reproduced with permission [21] Copyright 2019, Elsevier.

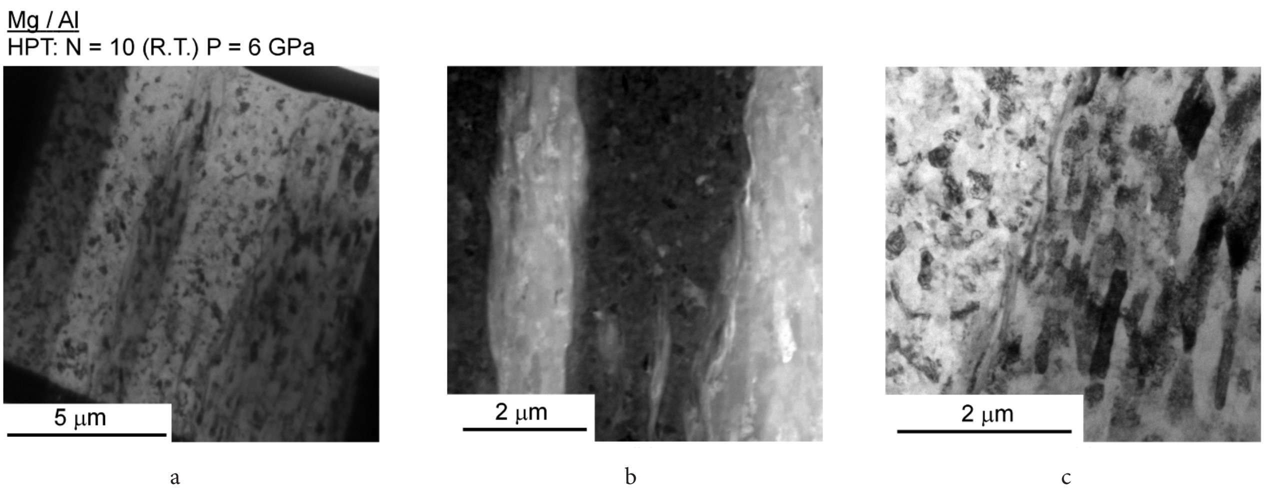

Fig. 4. TEM bright field image of the whole lamella (a), STEM image of the different phases (b) and higher magnification at the interface between Mg and Al phases (c). Reproduced with permission [26] Copyright 2019, Elsevier.

sensitivity of ~0.2. The addition of the hard ceramic particles in the composite increased the overall strength and reduced the strain-rate sensitivity. This means an improvement in room temperature creep resistance. An increase in hardness, compared to the unreinforced metal, was also reported for Mg-HA and Mg-BG composites [22].

It is now known that fine-grained pure magnesium displays extraordinary ductility at room temperature [29,31,32]. The high density of grain boundaries in this material induces a significant contribution of grain boundary sliding deformation even at room temperature. Thus, provided the composite contains a significant proportion of fine-grained magnesium, it is expected it will also display good ductility and this has been observed in a Mg/Al composite. Fig. 6 shows a stress vs strain curve, determined by tensile testing, of such a composite where a significant elongation was achieved [26]. Moreover, the presence of the aluminum phase in close contact with pure magnesium provides an opportunity to induce the precipitation of intermetallic phases by controlled thermal treatment. As a consequence, an increase in hardness is attained after thermal treatment [26].

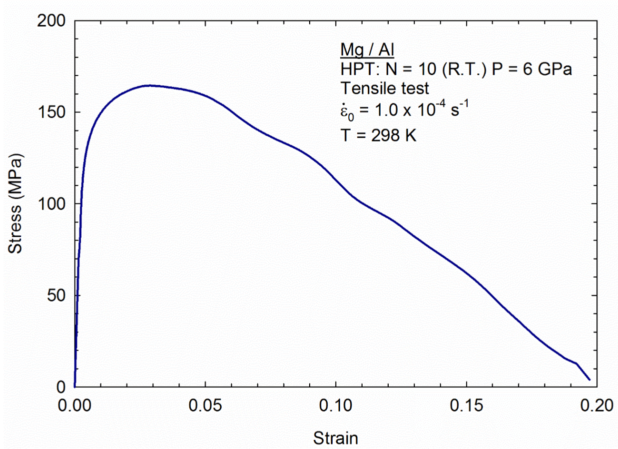

Fig. 6. Stress vs strain curves for a Mg/Al composite processed by HPT. Reproduced with permission [26] Copyright 2019, Elsevier.

5. Applications

The results obtained so far show it is possible to consolidate particles of magnesium and to incorporate other materials using HPT. The processed composites display well-dispersed phases within a matrix of ultrafine grained magnesium and the changes in properties open up the possibility of multiple applications. For example, it is possible to mix magnesium with other metallic materials without significant reactions between them. Since pure magnesium exhibits a unique ability to develop extraordinary ductility with grain refinement, the composite may also exhibit good ductility. Also, tailoring the metallic system provides the possibility of strengthening the composite by precipitation of hard intermetallics using posterior thermal treatment. Therefore, it is possible to produce ductile composites which can be shaped using forming operations and which may be hardened by thermal treatment after the final shape is attained.

Also, it is known that magnesium is biocompatible and biodegradable. This places this material as a strong candidate for the fabrication of temporary biological implants which play a structural role and gradually degrade and are absorbed within the body as the surrounding tissue regenerates. The possibility to incorporate other materials within a magnesium-based composite provides the opportunity to add a bioactive function to this material. For example, a magnesium-bioactive glass composite showed the ability to develop calcium phosphate after a short immersion in body fluid solution [22]. Fig. 7 shows an SEM image of the surface of a Mg-5% BG composite processed by HPT after 2 hours of immersion in Hank's solution. The composition of the area

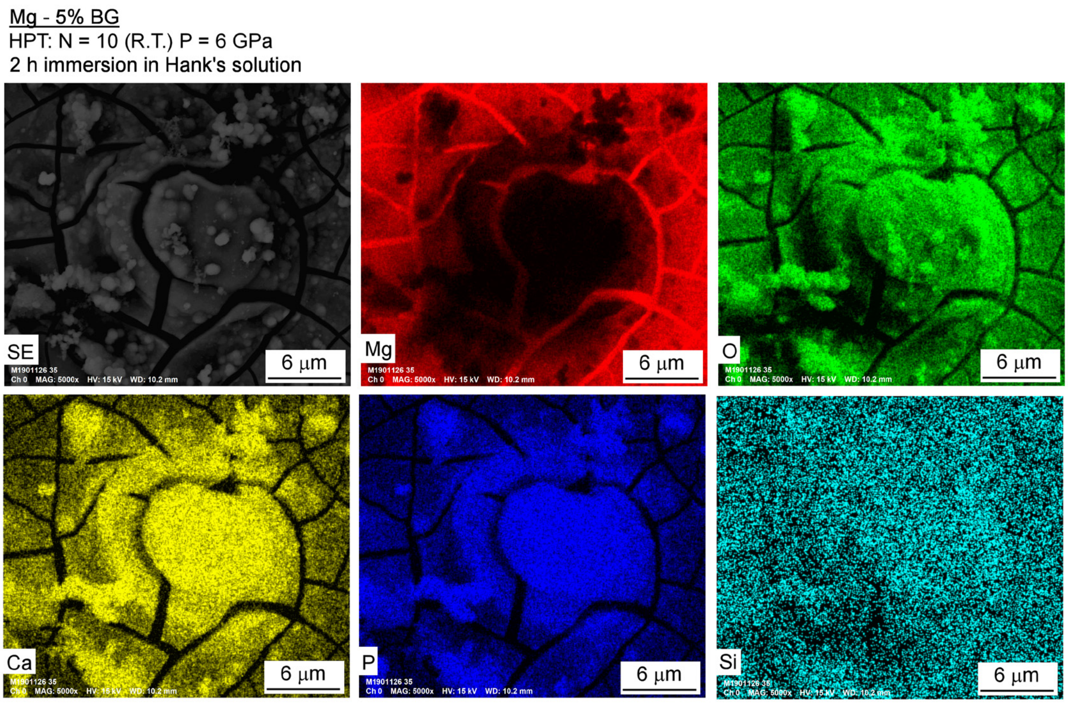

Fig. 7. (Color online) SEM image and compositional maps of surface products of a Mg-BG composite processed by HPT and immersed in Hank's solution for 2 hours [22].

is also revealed by EDS mapping and it is observed that the surface product is rich in calcium, phosphorous and oxygen. This surface product is expected to form better bonding with the bone tissue. This observation confirms it is possible to produce magnesium composites with drug delivery ability.

6. Conclusions

• It is possible to consolidate magnesium particles into a bulk solid disc using high-pressure torsion. It is also possible to incorporate different materials to produce a magnesium matrix composite.

• The route for composite fabrication depends on the material to be incorporated. Ductile metallic materials can be incorporated by placing half discs side by side or by stacking discs while hard and brittle materials can be incorporated by mixing with particles of magnesium.

• The size and distribution of phases can be controlled by the fabrication route and by the number of turns of HPT.

• Ultrafine grains are produced within the magnesium matrix and this provides the opportunity to improve the ductility of the composite. The incorporation of hard particles or the precipitation of intermetallics strengthen the composite.

• The fabrication of magnesium-based composites through HPT provides the opportunity to develop ductile materials which can be hardened by thermal treatment after the final shape of a component is attained. It is also possible to incorporate bioactive materials to produce bioactive and biodegradable composites.

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