Superior ductility in magnesium alloy-based nanocomposites: the crucial role of texture induced by nanoparticles

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ABSTRACT

In an expansive field of metals, magnesium has been trending of late in automobile, aerospace, defense, sports, electronic and biomedical sectors as it offers an advantage in lightweighting. In the realm of Mg-based materials, Mg nanocomposites have a good combination of specific strength, thermal and damping properties, but lack a high ductility and do not typically undergo a large amount of uniform elongation. The current work bridges this gap by reporting a magnesium nanocomposite (Mg–1.8Y/1.5Y₂O₃) that exhibits a significantly high tensile ductility of 36%. Microstructural characterization of the nanocomposite revealed that the striking presence of micron-Mg–Y phases and nano-Y₂O₃ particles in matrix led to a bimodal particle distribution which affected the dynamic recrystallization mechanism. It was also observed that the addition of Y₂O₃ nanoparticles weakened the texture of nanocomposite. The dominating influence of texture weakening over other mechanisms (grain refinement and alleviated micro-strain) on the plastic deformation/ductility of the nanocomposite is highlighted, and the contribution of nanoparticles toward the enhancement of ductility is ascertained. In contrast to the previous studies where Mg-based nanocomposites are known to have improved strengths, this approach can be used to develop magnesium nanocomposites that are exceptional.

Introduction

Magnesium is garnering a lot of attention in different engineering sectors, particularly in the transportation sector, to enable lightweighting, fuel efficiency and

reduction in the greenhouse gas emissions [1]. One major constraint for magnesium is its restricted ductility due to the availability of limited deformation modes originating from its HCP crystal structure [2]. At room temperature, basal slip is readily

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activated because of its low CRSS value; hence, the von Mises criterion for homogeneous deformation is not met. However, in the last decade, this limitation was overcome by employing different mechanisms that impart ductility, and these mechanisms are listed below, briefly.

- (a) *Grain size reduction* (i.e., an increased grain boundary area) allows homogeneous distribution of stress concentration at boundaries/triple junctions, thereby activating non-basal slip and imparting ductility at lower temperatures [3].
- (b) *Bimodal grain characteristic* In an ultrafine-grained material, the dislocation storage efficiency inside the tiny grains is quite low especially in the presence of dynamic recovery, making the material prone to plastic instability (early necking). This severely limits the desirable uniform elongation unless larger grains of appropriate sizes and volume fractions are present. These larger grains produce pronounced strain hardening to sustain the useful uniform deformation to large strains [4]. Thus, the bimodal grains are useful to impart high strength and uniform elongation in a material [5].
- (c) *Alleviated micro-strain* indicates a decreased dislocation density, one of the root causes for homogeneous deformation in a material, imparting it ductility.
- (d) *Promoting activity of tension twins* (for example, in ZX11 alloys) helps deformation along the thickness direction after twinning which is an important criterion for high stretch formability of Mg alloys [6].
- (e) *Tailored initial orientation* with maximized Schmid factor for basal slip and reduced Schmid factor for non-basal slips prolonging the strain hardening stage II and giving the material ductility [3].
- (f) *Anisotropy in SFE* of the basal (low SFE) and non-basal planes (high SFE) leads to a differential in the extent of strain hardening exhibited by them. This increases the CRSS for basal slip with respect to the non-basal slips rendering magnesium plastically deformable [3].
- (g) *Modified texture* (preferred crystallographic orientation of grains) is one of the most effective ways to ductilize a Mg-based material [7, 8]. So far, there have been two ways to control

texture: (1) placing most of the grains in the ideal orientation with basal plane tilting 45° from tensile axis, i.e., a strong non-basal texture and (2) randomizing the orientation as much as possible to get quite a weak texture (samples processed by torsion extrusion, alloyed by rare earth elements, Ca, etc. [9]). In this work, a third and a novel approach of weakening texture is proposed, i.e., nanoparticles-induced texture weakening which mediates ductility in Mg matrix.

Magnesium matrix nanocomposites (MMNCs) are generally defined as materials that demonstrate extraordinarily increased strengths without compromising ductility due to the presence of nanoparticles that promote dislocation looping and resist immediate failure [10, 11]. However, the effect of nanoparticles on improvement in ductility is still unclear, and there has been very limited reported work that discusses a significant improvement in ductility. This work presents a highly ductile alloy nanocomposite whose ductility is achieved through nanoparticle and alloying element addition and discusses the mechanism with aid of micro- and macro-texture analysis. In contrast to the previous studies, this work highlights the possibility of developing highly ductile magnesium-based nanocomposites using conventional processing techniques.

Materials and methods

Mg–1.8Y/1.5Y₂O₃ (wt%) nanocomposite was synthesized by melting and casting commercially pure Mg turnings (99.9% purity; supplied by Acros Organics, USA), Mg–30% (wt%) Y master alloy (99% purity; supplied by Sunrelief Metal Co., Limited, China) and Y₂O₃ nanoparticles (Nanostructured and Amorphous Materials, Inc., USA; 32–36 nm average size) using DMD (disintegrated melt deposition) method [12]. The ingot obtained was turned to billets (length: 45 mm; diameter: 36 mm), homogenized at 400 °C for 1 h and extruded at 350 °C to obtain rods of 8 mm diameter. Samples were taken from the rods for further analysis. Phase analysis was performed using 200 kV FEI Tecnai G2 T20 super twin (ST) Transmission Electron Microscope (TEM) on samples (50–90 nm thick) prepared using a FIB system attached to FEI Helios Dual-beam Nanolab 650.

Macro-textures were measured using a Bruker D8-Discover texture goniometer using Co-K α radiation. During X-ray texture measurements, all the samples were irradiated perpendicular to the extrusion direction along the radial plane. Six pole figures, viz. $\{1\ 0\ \bar{1}\ 0\}$, $\{0\ 0\ 0\ 2\}$, $\{1\ 0\ \bar{1}\ 1\}$, $\{1\ 0\ \bar{1}\ 2\}$, $\{1\ 1\ \bar{2}\ 0\}$ and $\{1\ 0\ \bar{1}\ 3\}$ were measured, and orientation distribution functions (ODFs) were calculated using ADC algorithm using a Labotex Software. Inverse pole figures (IPF) were calculated from the ODFs. Microtexture of the extruded rods was obtained using the electron backscatter diffraction (EBSD) technique in a field emission gun (FEG) scanning electron microscope (SEM) by FEI Company at an accelerating voltage of 25 kV. The EBSD scans were recorded on the radial plane parallel to the extrusion direction (ED), and it was ensured that the confidence index (CI) was greater than 0.1 and the step size was maintained to be 0.1. The software that was used for dealing the EBSD data was TSL OIM version 7.2. Kernel average misorientation (KAM) of each EBSD spot with all its neighboring spots were calculated with the provision that misorientations exceeding 5° were excluded from the average calculation. A fully automated servo-hydraulic Model MTS 810 mechanical testing machine, with a clip-on Instron 2630e100 extensometer, was used for tensile testing, conforming to ASTM test method E8/E8M-13a. Specimens with a diameter of 5 mm and gauge length of 25 mm were used for the tensile tests and were tested at a strain rate: $1.6 \times 10^{-4}\ \text{s}^{-1}$. Five tests were performed to ensure consistency, and the representative result is presented in this work. Fracture surfaces' analysis was performed using SEM (JEOL JSM-6010 coupled with EDS). Grain size measurement from the SEM images and optical microscopic images was done using ImageJ and Origin softwares where about 200 grains were measured and plotted as histograms and the grain size distribution was ascertained. This was later fitted to a 'LogNormal' function, and the average value was determined to be the average grain size.

Results and discussion

The addition of Y₂O₃ nanoparticles to a Y containing Mg matrix resulted in Mg–Y secondary phases and Y₂O₃ nanoparticles dispersed ex situ in the matrix as shown in a high-magnification SEM image in Fig. 1a.

The secondary phases were found to be distributed throughout and their presence was not confined to grain boundaries, while the nanoparticles were particularly seen to segregate toward the grain boundaries as shown in Fig. 1b.

Figure 2 represents the EBSD-generated IPF maps of Mg–1.8Y/1.5Y₂O₃ nanocomposite along with its monolithic alloy and pure Mg. From the images, the following can be deduced:

- (a) *Recrystallized structure* The IPF maps indicate completely recrystallized microstructures, i.e., the samples (Mg, Mg–1.8Y and Mg–1.8Y/1.5Y₂O₃) underwent dynamic recrystallization during hot extrusion processing [13]. There was no evidence of the presence of deformed grains in any of the samples.
- (b) *Grain size* The aforementioned recrystallization phenomenon was different in pure Mg, Mg–1.8Y alloy and Mg–1.8Y/1.53ZnO nanocomposite due to the variation in the microstructural constituents. The average grain sizes of the alloy and nanocomposite were much lesser than those of Mg as seen from Fig. 2a–c. This is attributed to the presence of Y that forms secondary phases that act as hard restrictive obstacles against grain growth, restricting the migration of grain boundaries [14]. However, it is evident that the average grain size of the nanocomposite is marginally higher than that of Mg–1.8Y alloy and the grain size distribution is quite high with grain sizes varying from 2 to 10 μm as seen in Fig. 2c. It is generally known that the nanoparticles contribute to the grain refinement of Mg. This is because of the accelerated nucleation kinetics originating from the large driving force due to the geometrically necessary dislocations and the large number of nucleation sites. However, when nanocomposites were compared to alloys in terms of the grain sizes (keeping the processing parameters the same), it is seen that the nanocomposites did not achieve as effective grain refinement as alloys, i.e., grain refinement was not possible to less than 5 μm in case of most Mg nanocomposites [11]. Such results are intriguing as the alloy nanocomposites reported previously also exhibited a similar grain coarsening compared to the alloys [15]. The reason behind this has not been discussed so far, particularly for Mg–

Figure 1 **a** Representative micrograph of the nanocomposite with Mg–Y phase and Y₂O₃ nanoparticles; **b** TEM image of the nanocomposite showing the presence of the nanoparticles,

particularly at the boundaries; **c** EDS spectrum for the Mg–Y phase shown in (a) and **d** EDS spectrum of Y₂O₃ nanoparticle shown in (a).

Figure 2 EBSD generated microstructures presented as IPF maps for **a** pure Mg [19] **b** Mg–1.8Y alloy [19, 20] and **c** Mg–1.8Y/1.5Y₂O₃ nanocomposite. Average grain sizes are given in the inset in the IPF maps. Inverse pole figures obtained from the X-ray

texture analysis for the **d** pure Mg; **e** Mg–1.8Y alloy and **f** Mg–1.8Y/1.5Y₂O₃ nanocomposite. The projected direction is the radial direction (RD).

based nanocomposites, to the authors' best knowledge. This coarsening of the grains is because of the variation in the size of the secondary phases (coarser) and particles (finer). This leads to a bimodal-type distribution of particles and phases in the matrix, and hence, the mechanism can be interpreted similar to the mechanism of recrystallization of alloys containing bimodal particle distribution. Both particle stimulated nucleation (PSN) (by particles $> 1 \mu\text{m}$ that act as sites for PSN) and Zener pinning (by small particles spaced close enough to pin the grain boundary migration) play a role in controlling the recrystallization kinetics [16]. The accelerated recrystallization kinetics by PSN and the retarded kinetics by pinning lead to a recrystallized grain size larger than predicted. Thus, the nanocomposite has a coarser grain size in comparison with the alloy.

- (c) *Texture* Figure 2d–f shows the inverse pole figures of the materials. The extruded pure Mg has the strongest texture as can be seen from the maximum intensities, with the *c*-axis of the grains aligned perpendicular to the extrusion direction. With addition of Y, it is known that the texture weakens to a large extent and this is also reported in several other works previously [14, 17]. The addition of Y_2O_3 nanoparticles to Mg–1.8Y alloy led to the further weakening of texture of the nanocomposite as can be observed from the maximum intensity demonstrated by the nanocomposite in Fig. 2f. Further, no evidence of twinning was detected in any of the as-extruded materials. The completely weakened texture with the addition of nanoparticles and its effect is studied in this work.

Engineering tensile stress–strain curves of the nanocomposite subjected to tensile test along extrusion direction are shown in Fig. 3a. From the curves, it is noticeable that the nanocomposite stands out in its properties as compared to the alloy and pure Mg. It exhibits highest tensile yield and ultimate strengths coupled with highest ductility of 36% (higher than the Mg–1.8Y alloy by 33% and Mg by 500%).

The increased strength of the nanocomposite in comparison with Mg–1.8Y alloy, despite having a weaker texture, is due to the obvious presence of nanoparticles. The nanoparticles contribute to the

Orowan strengthening and dislocation/forest strengthening. It must be noted that there is no effect of Hall–Petch strengthening in the case of this Mg–1.8Y/ Y_2O_3 nanocomposite as the grains coarsened with addition of nanoparticles as compared to Mg–1.8Y alloy. It can also be noted that the yield strength of the nanocomposite is very similar to that of Mg due to the variation in textures of the two. Pure Mg exhibits a stronger texture, thereby demonstrating high yield strength despite the lack of a smaller grain size and other strengthening mechanisms, whereas the nanocomposite exhibits a weak texture. Hence, the nanocomposite's weak texture was offset by the presence of nanoparticles, dislocations and refined grains to match the yield strength of pure Mg.

The ductility of the nanocomposite matches/exceeds that of commercial Al alloys and mild steels. These results are interesting as nanocomposites of Mg base, reported thus far, had average ductilities ranging from 3 to 18% only [11] as given in Fig. 3b, and it is seen that Mg–1.8Y/1.5 Y_2O_3 nanocomposite has surpassed the ductilities of all the previously reported nanocomposites.

Out of the major factors that contribute to the increased ductility of the nanocomposite, the grain refinement effect can be neglected due to a coarser grain size of the nanocomposite as compared to that of the alloy. Further, from Fig. 3b, it can be established that grain refinement does not play a major role in ductilizing the Mg nanocomposites, as there is no general trend followed with respect to grain size and ductility. The other possible factors that contribute to the ductilization of Mg nanocomposites are (1) micro-strain and (2) texture. The micro-strain in a material is estimated through KAM. It is often known that a presence of lower dislocation density leads to a lower micro-strain (lower KAM value) resulting in a high ductility, as in the case of Mg–1.8Y alloy (Fig. 3c). However, the nanocomposite exhibited a much higher micro-strain (nanoparticles lead to generation of dislocations due to a mismatch in CTE) and still exhibited higher ductility. This indicates the dominance of weak texture over other factors like grain size and micro-strain on the ductility of magnesium-based materials. From Fig. 2, the nanocomposite exhibited the weakest texture and hence the nanoparticles-induced texture mediated in the highest ductility of 36% in the nanocomposite. Since, the nanocomposite exhibited a weaker texture and higher ductility than the Mg–1.8Y alloy, yttrium is

Figure 3 **a** Representative tensile engineering stress versus engineering strain curves; **b** grain size versus ductility plots for several magnesium-based nanocomposites reported so far [15, 21–24]. It is to be noted that the average grain size value is considered for plotting the graphs and all the reported materials are synthesized using similar casting and processing techniques and **c** Kernel average misorientation distribution of the materials.

not the sole reason for the ductile behavior of the nanocomposite. The weakened texture in the nanocomposite as compared to Mg is due to the combined addition of alloying element as well as nanoparticles. In comparison with the alloy, the further texture weakening is primarily due to the nanoparticles. The nanoparticles altered the deformation activity due to their interaction with dislocations and could also assist in improving the hardening behavior, as can be seen from the tensile stress–strain curves. This, in turn, results in an increase in activity of other slip systems and hence improved deformation behavior and enhanced ductility of the nanocomposite [18].

To understand the failure mechanism of the Mg–1.8Y/1.5Y₂O₃ nanocomposite further, the fracture surfaces after tensile testing were investigated. Figure 4 represents the fractograph of the Mg–1.8Y/1.5Y₂O₃ nanocomposite with the insets displaying the EDS analysis of the Mg–Y secondary phases (Fig. 4a) and Y₂O₃ nanoparticle (Fig. 4b). Despite having a very high ductility, the surface of the nanocomposite still assumes a few features of cleavage fracture adjacent to dimples. It is also characterized by secondary cracks, phases and nanoparticles. Interestingly, it was noted that these secondary cracks were present in Mg–Y phase in the nanocomposite as indicated with arrows in Fig. 4a, a clear indication of movement of dislocations through the Mg–Y phases. Further, a visible pullout was observed in the vicinity of Y₂O₃ nanoparticles (Fig. 4b), which could be the

resultant of the dislocation looping around the nanoparticles (Orowan dislocation looping mechanism [10]) before failure leading to pullouts. This dislocation looping mechanism is thought to resist immediate failure and impart ductility to the nanocomposite.

Conclusions

In summary, while most Mg nanocomposites, reported thus far, have had ductility in the range of 3–18% under wrought conditions, in this study, a Mg-based nanocomposite is reported to exhibit a highest ductility of 36% matching/exceeding that of ductile Al alloys and mild steels. The experimental results indicated the effect of the presence of Y₂O₃ nanoparticles in the matrix in controlling the recrystallization phenomenon and altering texture. Although, the nanoparticles did not aid in grain refinement and led to the generation of dislocations (because of mismatch in CTE resulting in higher micro-strain), the highest ductility was achieved due to the very weak texture exhibited by the nanocomposite. Thus, it is concluded that texture dominates the other factors (grain size, micro-strain) in ductilizing magnesium-based nanocomposites. Further, this work reinforces the role of nanoparticles in not just strengthening of the magnesium matrix, but also imparting it a very high ductility.

Figure 4 Tensile fractographs of the nanocomposite indicating **a** cracks in the Mg–Y secondary phase and **b** occurrence of pullout around the Y₂O₃ nanoparticles.

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Conflict of interest The authors declare no conflict of interest.

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