



Compressive deformation behavior of Mg and Mg/(Y₂O₃ + Ni) nanocomposites

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ABSTRACT

Uniaxial compressive properties of Mg and Mg/(Y₂O₃ + Ni) hybrid nanocomposite are studied in the present paper. A remarkable improvement in both 0.2% CYS and UCS is exhibited by hybrid nanocomposites when compared to pure Mg. Subsequently, plastic deformation due to twinning activity and basal plane orientation detected by X-ray diffraction at different strain levels are investigated. Both Mg and hybrid composites yield by twinning deformation and extensive twinning supports reorientation of basal (0002) planes after yielding. Upon twin saturation, basal planes get orientated perpendicular to the compression axis in composite sample, causing a steep increment in work hardening with limited failure strain. However, in the case of pure Mg, complete basal reorientation did not take place. As a result, continuous twin deformation provided gradual work hardening leading to appreciable failure strain in the case of Mg when compared to its reinforced counterparts.

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1. Introduction

The demand of light weight materials for energy saving in structural applications especially in automobile and aerospace industries has driven the development of magnesium based materials [1,2]. Magnesium matrix composites have recently captured the global attention primarily due to their reasonably superior specific mechanical properties arising due to their low density which is of prime importance in material selection for weight critical applications [3–11]. Although their tensile properties are reported extensively in open literature, little information is available about compressive properties irrespective of many engineering applications of Mg materials in which compressive loads are dominant [12–19]. Since magnesium is a strongly textured metal, not only reinforcement addition but also texture evolution (crystal orientation) has strong effects on variation in strength or ductility of magnesium [15–19]. The anisotropic mechanical properties of magnesium and its alloys depend on their crystallographic texture, particularly at low temperatures. Extruded or rolled Mg and Mg alloys generally exhibit strong texture with basal (0002) planes parallel to the compressive loading direction (extrusion texture) [20]. Under this basal orientation, {10–12} extension twinning is a common deformation mode in magnesium. For the materials with basal (0002) planes perpendicular to the compressive loading direction (i.e. *c*-axis parallel to the loading direction), some slip systems are activated besides twinning. Obara et al. [21] reported the

activation of basal and {11–22} {–1–123} slip system from room temperature to 500 °C in magnesium single crystal under *c*-axis compression. Other studies [22–25] also reported the possibility of plastic deformation due to basal slip and/or pyramidal slip system in Mg alloys. It is therefore obvious that the initial orientation of basal planes or position of *c*-axis with respect to the loading axis influence the deformation behavior of magnesium. Previous studies [26–28] have also been investigated the orientation dependence of mechanical properties of different Mg alloy systems by controlling the initial crystal orientation of the materials. Although basal planes align parallel to the loading axis in extruded materials, {10–12} twinning during compressive loading causes 90° reorientation of basal planes. Brown et al. [23] showed the relation between changes in peak intensities from diffraction pattern and the twinning activity in AZ31B Mg alloy by using neutron diffraction-based internal strain measurements. Another study [25] also reported sharp texture evolution with a marked increase in basal peak intensity in XRD pattern of compressively failed AZ31B Mg alloy as a signal of twinning reorientation. However, the correlation between microstructure evolution in terms of twinning deformation and the flow behavior of material is not conducted in detail. From the open literature, a systematic investigation on the plastic deformation behavior of Mg nanocomposites at different stages of deformation/flow curve under compressive loading has not yet been reported.

Accordingly, the present study discusses the effect of second phases on the significant increment in compressive strength of Mg nanocomposites over pure magnesium. An investigation is also made on the evolution of microstructure with twin formation at different levels of total strain to gain insight into the plas-

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tic deformation behavior of pure Mg and Mg/(Y₂O₃ + Ni) hybrid nanocomposites during compression. Twin formation is also correlated with the changes in basal (0002) peaks from XRD analyses at different strains during uniaxial compressive loading. The materials in the current study were synthesized using powder metallurgy route incorporating hybrid microwave sintering followed by hot extrusion.

2. Experimental procedures

2.1. Materials

In this study, magnesium powder of 98.5% purity and with a size range of 60–300 μm (Merck, Germany) was used as the matrix material. Yttria (Y₂O₃) with a particulate size range of 30–50 nm (Inframat Advanced Materials, USA) and nickel (Ni) with an average particulate size of 20 nm (Nanostructured & Amorphous Materials Inc., USA) were used as the particulate reinforcements.

2.2. Processing

Monolithic magnesium and hybrid nanocomposites containing 0.7 vol.% yttria [7] and nickel (0.3, 0.6 and 1.0 vol.%) were synthesized using the powder metallurgy technique. The synthesis procedure involved blending pure magnesium powder with nano-sized powder in a mechanical alloying machine [Model: RETSCH PM-400] at 200 rpm for 1 h (60 min). The blended powder mixtures were subsequently cold compacted at a pressure of 97 bar (510 MPa) using a 100-ton press to form billets that measured 35-mm in diameter and 40-mm in height. Monolithic magnesium was compacted using the same parameters but without blending of the powder. The compacted billets were immediately sintered using an innovative hybrid microwave sintering technique [29] for 13 min to reach a temperature (643 °C) near the melting point of magnesium using a 900 W, 2.45 GHz SHARP microwave oven. The sintered billets were subsequently hot extruded at a temperature of 350 °C at an extrusion ratio of 25:1.

2.3. Density measurements

Density of the extruded magnesium and magnesium nanocomposites in the as-polished condition was measured using Archimedes' principle. Three samples were randomly selected from the extruded rods and were carefully weighed both in air and when fully immersed in distilled water. An electronic balance [Model: A&D ER-182A] with an accuracy of 0.0001 g was used for recording the weights. The theoretical density of each of the hybrid nanocomposite formulation was calculated using rule-of-mixtures. For this purpose, the density of pure Mg, Y₂O₃ and Ni were taken to be 1.74 g/cc (provided by Merck, Germany), 5.03 g/cc (provided by Inframat Advanced Materials, USA) and 8.908 g/cc (provided by Nanostructured & Amorphous Materials Inc., USA), respectively.

2.4. Microstructural characterization

Microstructural characterization studies were conducted for determining grain size and formation of twinned grains. A metallographic optical microscope [Model: OLYMPUS] and a Scion Image Analyzer were used for this purpose.

2.5. X-ray diffraction studies

X-ray diffraction analysis was carried out on the as-extruded and compressively deformed Mg and Mg nanocomposite samples using automated Shimadzu LAB-X XRD-6000 diffractometer. The phases formed in the samples were identified and reorientation of crystal planes before and after compression as well as at specific stages during compressive loading was subsequently analyzed.

2.6. Compression test

Room temperature compressive tests were performed on cylindrical monolithic and composite samples according to ASTM E9-89a using an automated servo hydraulic testing machine (MTS 810). Extruded rod of 7 mm diameter was cut into 7 mm length samples for compression tests to provide the aspect ratio (l/d) of unity. Samples were tested at a strain rate of $5 \times 10^{-3} \text{ min}^{-1}$ and the compression load was applied parallel to the extrusion direction.

Fracture surface characterization studies were carried out on the compressive fracture surface of Mg and Mg composites with the objective of establishing the failure mechanisms. Fractography was accomplished using a Field Emission Scanning Electron Microscope (FESEM) [Model: HITACHI S-4300].

3. Results

3.1. Density and grain size measurements

The density results from Table 1 show that there was no significant difference in theoretical and experimental densities for both monolithic and composite samples. This indicates the feasibility of current processing route, PM method involving hybrid microwave sintering route, to synthesize nearly dense materials [7,29,30]. The densification response was also not affected markedly due to increasing presence of second phases in Mg. The average grain size was reduced significantly in composite samples when compared to Mg. However, no significant change in grain size was observed with increasing presence of nickel in nanocomposites considering the standard deviation.

3.2. Analysis on twinning

Figs. 1 and 2 show the microstructures of Mg and Mg/(0.7Y₂O₃ + 1.0Ni) hybrid nanocomposite in as-extruded condition and following compression at different total strain levels (elastic + plastic) up to failure. In case of Mg, less amount of twinning was observed in the sample compressed to a total strain of 2.5% and 21%, and to fracture point (Fig. 1b, e and f).

Table 1
Results of density and grain morphology determinations.

Material	Reinforcement (vol.%)		Theoretical density (g/cm ³)	Experimental density (g/cm ³)	Grain size (μm)
	Y ₂ O ₃	Ni			
Mg	–	–	1.740	1.738 \pm 0.007	20 \pm 3
Mg/(Y ₂ O ₃ + Ni)	0.7	0.3	1.785	1.778 \pm 0.002	9 \pm 3
Mg/(Y ₂ O ₃ + Ni)	0.7	0.6	1.806	1.802 \pm 0.002	6 \pm 2
Mg/(Y ₂ O ₃ + Ni)	0.7	1.0	1.835	1.829 \pm 0.002	5 \pm 2

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