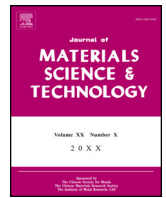




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## Nanoscale deformation of multiaxially forged ultrafine-grained Mg-2Zn-2Gd alloy with high strength-high ductility combination and comparison with the coarse-grained counterpart

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### ABSTRACT

Cold processing of magnesium (Mg) alloys is a challenge because Mg has a hexagonal close-packed (HCP) lattice with limited slip systems, which makes it difficult to plastically deform at low temperature. To address this challenge, a combination of annealing of as-cast alloy and multi-axial forging was adopted to obtain isotropic ultrafine-grained (UFG) structure in a lean Mg-2Zn-2Gd alloy with high strength (yield strength: ~227 MPa)-high ductility (% elongation: ~30%) combination. This combination of strength and ductility is excellent for the lean alloy, enabling an understanding of deformation processes in a formable high strength Mg-rare earth alloy. The nanoscale deformation behavior was studied via nanoindentation and electron microscopy, and the behavior was compared with its low strength (yield strength: ~46 MPa) – low ductility (% elongation: ~7%) coarse-grained (CG) counterpart. In the UFG alloy, extensive dislocation slip was an active deformation mechanism, while in the CG alloy, mechanical twinning occurred. The differences in the deformation mechanisms of UFG and CG alloys were reflected in the discrete burst in the load-displacement plots. The deformation of Mg-2Zn-2Gd alloys was significantly influenced by the grain structure, such that there was change in the deformation mechanism from dislocation slip (non-basal slip) to nanoscale twins in the CG structure. The high plasticity of UFG Mg alloy involved high dislocation activity and change in activation volume.

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### 1. Introduction

Magnesium is abundant in nature and its alloys exhibit a number of attractive properties including high specific strength and specific stiffness, good damping and shock absorbing capacity, high thermal conductivity, and strong electromagnetic shielding ability [1,2]. While these properties encourage the use of Mg alloys in automobile, aerospace, and packaging industries, there are two aspects that inhibit their widespread usage. First, magnesium alloys typically have lower yield strength than their aluminum counterpart. Second, cold processing of Mg alloys is a challenge because Mg has a hexagonal close-packed (HCP) lattice with limited slip systems, which makes it difficult to be plastically deformed at low temperature.

In regard to the strength, Mg-rare earth (RE) alloys, either binary or with additional alloying elements, are of particular interest

because of the high yield strength that can be achieved through precipitation hardening. In recent years, a wide range of RE elements (La, Ce, Nd, Gd, Y) have been explored to increase strength and improve formability. Their addition has been promising in terms of weakening the texture and improving their formability and increasing the strength through precipitation hardening-induced by phases such as Mg<sub>12</sub>RE, Mg<sub>3</sub>RE or Mg<sub>2</sub>RE phases, besides solid solution strengthening [3].

Grain refinement is an important approach that increases the strength of engineering materials. The superior properties of ultrafine-grained alloys include a combination of high strength with uniform elongation. Recently, we have processed as-cast Mg-2Zn-2Gd alloy via a combination of annealing and multiaxially forging (MAF) to obtain an ultrafine-grained (UFG) structure with high strength-high ductility combination [4]. In this regard, we considered a relatively dilute and model ternary alloy system, with minimum targeted strength of ~200 MPa and elongation exceeding ~20% in the multiaxially forged (MAF) alloy. Prior to MAF, the as-cast alloy was annealed at 500 °C for 2 h to relieve the inter-

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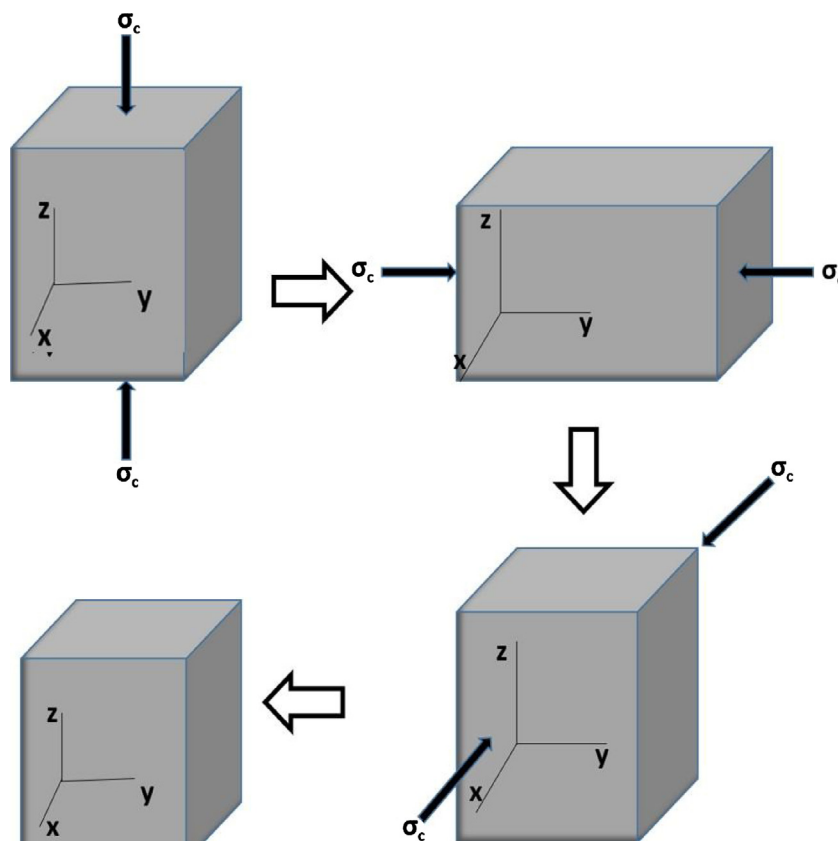


Fig. 1. Schematic of the MAF process.

nal residual stresses. The details of processing of the multiaxially forged alloy are described elsewhere [4].

In designing the alloy, the following aspects were considered after a critical review of the phase diagrams of Mg-RE alloys and fundamental knowledge available in the literature. (a) Gadolinium (Gd) has a high solubility of 23.49 wt% at eutectic temperature [5] in Mg and significantly contributes to solid solution strengthening when alloyed with Mg [6]. (b) 1–2 wt% of Gd weakens recrystallization texture effects and improves ductility. (c) Alloying Mg with 2 wt% Gd exhibits maximum tensile strength as compared to any other rare earth element for similar percentage of alloying element. Beyond 2 wt% Gd, there is no significant effect on strength and may reduce the strength because of solute segregation. (d) Gd and Zn together form FCC w-phase with  $Mg_3Zn_3Gd$  that is suitable for grain refinement [7]. Based on the aforementioned aspects, the nominal chemical composition of the alloy in wt% was Mg-2Zn-2Gd, ensuring the formation of precipitation strengthening w-phase and simultaneously obtaining solid solution strengthening, besides strengthening from grain refinement. The objective of the study described here is to elucidate the interplay between grain structure and nanoscale deformation behavior in a lean and model Mg-2Zn-2Gd alloy with high strength-high elongation, not previously obtained.

## 2. Experimental procedure

The alloy was melted in a resistance furnace under a protective gas mixture of Ar + 2%  $SF_6$ . Pure Mg, Zn, and Gd were used to prepare the alloy. Initially, Mg and Zn were melted in a boron nitride (BN)-coated mild steel crucible, and Gd was added into the melt at  $\sim 770^\circ C$ . After the addition, the melt was mechanically stirred at 200 rpm for 30 min, for complete dissolution of Gd and to obtain uniform composition in the melt. The melt was poured into a pre-

heated ( $350^\circ C$ ) metallic round mold with an inner diameter of 100 mm and a length of 400 mm attached with a sprue at the top.

Prior to MAF, the as-cast alloy was annealed at  $500^\circ C$  for 2 h to relieve internal residual stresses. MAF of the as-cast Mg-2Zn-2Gd alloy was carried out at a pressing speed of 12–15 mm/s, using a press of a 2000 kN load limit. Rectangular samples were subjected to MAF, as schematically illustrated in Fig. 1 [4]. The sample was placed in MAF die and heated to  $450^\circ C$  in a muffle furnace. The temperature of the MAF die was monitored using a K-type thermocouple. Once the MAF die attained the desired temperature, a period of 30 min was allowed to elapse prior to MAF. This time was adequate for the die to obtain a steady-state temperature. The sample was reheated to the deformation temperature of  $450^\circ C$  ( $450^\circ C$  was the experimentally observed lowest and optimal temperature for MAF).

The MAF device consisted of a punch for external loading and a die, and the punch moved vertically inside the chamber comprising of punch and die. The dimensional ratio of samples was maintained constant throughout the MAF processing, while the loading direction was changed  $90^\circ$  from pass to pass. The samples were MAF processed (2 passes), and in each pass the true strain induced was 2.1, such that the total strain after the second pass was 4.2.

The as-cast alloy + annealed alloy and multiaxially forged (MAF) alloy samples were cut into  $5\text{ mm} \times 5\text{ mm} \times 5\text{ mm}$  dimensions and metallographically polished to mirror finish. The samples were etched for  $\sim 2$  min in a solution containing 8 g picric acid, 5 ml acetic acid, 10 ml distilled water, and 100 ml ethanol for microstructural examination. Transmission electron microscopy (using a Hitachi H-9500 TEM operating at 300 kV accelerating potential) was carried out using 3 mm disks that were electropolished in a solution containing 3% perchloric acid in ethanol.

Given that the nanoindenters were to be subsequently examined by transmission electron microscopy (TEM) for microstructural

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