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## Materials Chemistry and Physics

journal homepage: www.elsevier.com/locate/matchemphys



# High mechanical efficiency, microstructure evaluation and texture of rheocasted and extruded AZ80-Ca alloy reinforced with processed Al<sub>2</sub>O<sub>3</sub>/GNPs hybrid reinforcement

GRAPHICAL ABSTRACT

A: AZ80-1.5CNT[13]

B: AZ61-3GNP [14]



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#### HIGHLIGHTS

- AZ80/Ca alloy reinforced with Al<sub>2</sub>O<sub>3</sub>/ GNPs was successfully fabricated by rheo casting.
- · Single distribution of GNPs and fairly uniform dispersion of Al2O3 was observed.
- · Hybrid reinforcement affected grain size and precipitations morphology of base alloy.
- AZ80-Ca/Al2O3+GNP composite exhibited superior strengthening and failure strain efficiency.

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# CYS UCS CFS C: AZ31-1.5GNP [15] D: AZ31-3GNP [15] 35 Efficiency(%) 15 C

TYS UTS TES

#### ARTICLE INFO

Keywords: Mg based nanocomposite Hybrid reinforcement Microstructure Texture Mechanical properties

#### ABSTRACT

In this study, Mg based nanocomposite reinforced with nano alumina and graphene nano-platelets as hybrid reinforcement was fabricated by rheo casting followed by hot extrusion at 450 °C. Microstructure investigations showed that in comparison with the base alloy, addition of hybrid reinforcement has significant effects on grain refining (up to  $\sim$  44%), decrease of area percent of eutectic phase (up to  $\sim$  38%), evaluation of dynamic precipitations morphology and decrease of basal texture (up to ~ 23%). Mechanical properties results showed that fabricated nanocomposite exhibits simultaneously enhancement of micro-hardness (~ 33% increase), tensile and compressive yield strengths (~ 27% and 12% increase, respectively), ultimate tensile and compressive strengths (~ 33% and 10% increase, respectively), and tensile and compressive fracture strains (~ 42% and 13% increase, respectively). Simultaneous enhancement of strength and ductility in the nanocomposite introduced this hybrid reinforcement as the new class of reinforcements with higher performance.

#### 1. Introduction

Magnesium (Mg) is the lightest of all structural metals, hence is a proper choice for the application fields of ultra-light weight materials which have high specific mechanical properties such as aircraft and other transport concerns [1,2]. Nevertheless, the usage of magnesium is restricted due to its low strength and ductility [3]. In order to decrease this shortage, some strategies such as (i) alloying [4], (ii) incorporation of reinforcement [5-7], (iii) grain refinement [4,6,8] and (iv) texture modification [9,10] were used by researchers. It is expected that

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simultaneously use of these strategies provides desirable properties.

AZ series alloys of magnesium are generally used as composite matrixes due to ease of production, low cost and high strength and ductility [6]. Further, positive effect of Ca addition as an alloy element on mechanical properties of AZ series alloys was reported [6,11].

In other hand, adding reinforcement particles such as Al<sub>2</sub>O<sub>3</sub>, SiC and CNTs is one of the possible methods to further improvement of the mechanical properties of Mg alloys [5]. Often, strengthening effect of reinforcement is frequently concomitant with a decrease in failure strain. Moreover, the reports on the simultaneous increase of compressive and tensile properties are very low. Tensile strength and ductility of AZ series alloys are increased simultaneously by addition of Al<sub>2</sub>O<sub>3</sub> in nano scale [11,12]. It has been reported that addition of 1.5 vol% Al<sub>2</sub>O<sub>3</sub> in AZ80 + 0.5Ca alloy increases tensile and compressive mechanical properties [6]. Paramsothy et al. [13] showed that AZ81-CNT nanocomposite presents higher tension failure strain compared to base alloy while compressive failure strain and tension and compressive strengths are decreased. Rashad et al. [8] found that tensile and compressive strengths of AZ31 alloy are increased with CNT addition while tensile and compressive failure strains are decreased. It is indicated that reduction of failure strain and slightly increase in strength of Mg matrix nanocomposites reinforced with CNTs can be attributed to structural in-homogeneities in these composites due to non-uniform distribution of CNTs and low load transfer capability from Mg alloy matrix to the CNT due to weak interfacial bonding strength [14]. In recent years, GNP as reinforcement has received more consideration by researchers. The mechanical properties of GNPs with two dimensional structure and high specific surface are comparable to CNTs, while are dispersed easer into metal matrix. Moreover, GNPs are inexpensive and produced easily [8]. Therefore, GNPs have highly efficient in reinforcing metal matrix composites. Rashad et al. [14] have prepared AZ61-3GNP composite by disintegrated melt deposition (DMD) method followed by hot extrusion. Experimental results revealed lower both tension and compressive failure strains compared to base alloy while tensile and compressive yield strengths and ultimate tensile and compressive strengths are increased. In another research, Rashad et al. [5] found that addition of GNPs increases tensile strength, compressive strength and tensile failure strain while decreases compressive failure strain compared to Mg-6Zn alloy. Rashad et al. [15] have fabricated AZ31-3GNPs using stir casting method followed by hot extrusion. It was reported that addition of GNPs reinforcement into AZ31 matrix alloy increases mechanical properties except the compressive failure strain. Also, it was reported that using of GNPs as a constituent of hybrid reinforcement has positive effect on mechanical behavior of Mg based composite [16,17]. Rashad et al. [17] reported that addition of hybrid reinforcement improves both tensile and compressive mechanical properties compared to Mg. Although usually overall strength and failure strain of Mg-GNPs composites are better than Mg-CNTs composites [18], however mechanical properties of Mg-GNPs composites are lower than expected which can be attributed to poor wettability of GNPs by Mg alloy matrix [14,16,19]. It has been indicated that semi-solid casting can improve wettability and reinforcement distribution and reduces casting defects [7].

Microstructure and texture evolutions of matrix during secondary process affect mechanical properties of Mg based materials. Extrusion as a secondary process can improve reinforcement distribution and reduces grain size, casting defects and caused basal texture [20]. In our previous study [7,21], it was shown that modification of morphology of  $\rm Mg_{17}Al_{12}$  eutectic phase, dynamic precipitates and recrystallization during extrusion process resulting development of a fine grain structure, significantly enhance mechanical properties of extruded AZ80 + Ca based materials compared to as-cast ones.

Literature search results indicated that limited attempts have been made to select AZ80  $\,+\,$  Ca as a composite matrix and use of rheo casting method in order to fabricate Mg based composites. Also, the combination of GNPs with excellent mechanical properties and nano sized

alumina with better wettability is expected to enhance remarkably the mechanical properties of AZ80  $\,+\,$  Ca Alloy which has not been reported so far. Therefore, the purpose of this work is to investigate effect of hybrid reinforcement on microstructural evaluation, texture and mechanical properties of the AZ80  $\,+\,$  Ca alloy. So, the AZ80  $\,+\,$  Ca alloy reinforced with hybrid reinforcement containing 1.5 vol% nanoalumina and 0.5 vol% GNPs was produced by rheo casting route followed by hot extrusion. Mechanical properties and microstructure examination were used to evaluate fabricated nanocomposite.

#### 2. Experimental procedure

#### 2.1. Materials

The AZ80 magnesium alloy (7.81% Al, 0.46% Zn, 0.1% Mn and balance Mg, in wt.%) and 0.5 wt% Ca granular (Merck Co. Germany) were selected as the matrix alloy. The selected hybrid reinforcement was nano sized alumina with the average diameter of 50 nm (Merck Co. Germany) with the volume fraction of 1.5% along with 0.5 vol% GNPs which had entangled morphology with the average thickness of 2–18 nm and average diameter of 4–12  $\mu m$  (US research Co. USA). Based on the data sheet, GNPs possess fewer layers than 30 graphene layers.

#### 2.2. Fabrication

The fabrication of AZ80 + Ca/(Al $_2$ O $_3$ +GNPs) hybrid nanocomposite was included the pre-synthesizing the GNP/Al $_2$ O $_3$  hybrid reinforcement and the rheo casting process. Firstly, GNPs and Al $_2$ O $_3$  were dispersed by ball milling for 4 h and speed of 300 rpm with a ball to powder ratio of 5:1, by a ball milling machine. In this process, 1 wt% stearic acid was added to inhibit serious cold welding. Main goal was to remove the layers of gas on the surface of the hybrid reinforcement caused by surface abrasion of the particles what leads to achieve better distribution of hybrid reinforcement.

In the second step, about 350 g AZ80 alloy, Ca granular and hybrid reinforcement were set in the stainless steel crucible and melted at 700 °C under argon protective atmosphere using an electrical resistance furnace. Then molten slurry was cooled to 600 °C where the matrix alloy was in the semi-solid region. At this time, composite slurry was stirred for about 12 min at 450 rpm using dual Chemineer HE-3 impeller [22], while temperature was reduced to 585 °C. After stirring was finished, slurry was kept for 3 min and then poured into a preheated steel die (at 450 °C). To identify the effects of hybrid reinforcement on the AZ80 + Ca alloy, the monolithic alloy was also fabricated by the same experimental parameters.

#### 2.3. Hot extrusion of materials

The cast billets were machined into diameter of 34 mm and height of 70 mm and subsequently homogenized for 24 h at 415 °C. After homogenizing, the billets were hot extruded at 450 °C with an extrusion ratio of 12:1. A graphite powder was used for lubrication purposes during process.

#### 2.4. Microstructure and mechanical characterizations

In order to characterize graphitic structure of as received GNPs, Raman spectra was obtained by a Raman spectrometer (Takram P50C0R10, laser wavelength = 532 nm). Microstructural examinations of as casted and extruded specimens were performed by Optical Microscopy (Olympus-GX51 model) and Field Emission Scanning Electron Microscopy (FESEM) equipped with EDS (FESEM model: Mira3XMU). The Microstructural examinations were applied to determine the morphological characteristics of  $\alpha\text{-Mg}$  grains and  $\text{Mg}_{17}\text{Al}_{12}$  phase, presence and distribution of reinforcements in the composite

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