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Enhancement of thermal, mechanical, ignition and damping response of magnesium using nano-ceria particles

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ABSTRACT

Magnesium (Mg)-based nanocomposites owing to their low density and biocompatibility are being targeted for transportation and biomedical sectors. In order to support a sustainable environment, the prime aim of this study was to develop non-toxic magnesium-based nanocomposites for a wide spectrum of applications. To support this objective, cerium oxide nanoparticles (0.5 vol%, 1 vol%, and 1.5 vol%) reinforced Mg composites are developed in this study using blend-press-sinter powder metallurgy technique. The microstructural studies exhibited limited amounts of porosity in Mg and Mg-CeO_2 samples (< 1%). Increasing presence of CeO_2 nanoparticles (up to 1.5 vol%) led to a progressive increase in microhardness, dimensional stability, damping capacity and ignition resistance of magnesium. The compressive strengths increased with the increasing addition of the nanoparticles with a significant enhancement in the fracture strain (up to ~48%). Superior energy absorption was observed for all the composite samples prior to compressive fracture. Further, enhancement in thermal, mechanical and damping characteristics of pure Mg is correlated with microstructural changes due to the presence of the CeO_2 nanoparticles.

1. Introduction

Magnesium (Mg) based materials are the lightest (density ~1.74 g/cc) of all structural metals including iron, titanium and aluminium based alloys [1]. In addition to properties like low density and high specific mechanical properties, Mg-based materials also exhibit an excellent combination of specific strength, castability, machinability, recyclability, thermal stability, damping behavior and electromagnetic radiation resistance [2]. These advantages make Mg-based materials an excellent choice for aerospace, automotive, consumer electronics and sports sectors [3]. Worsening climate and the global move to cut the carbon dioxide emissions by 2 billion tons by 2025 to keep the temperature rise within 2 °C from the pre-industrial levels (Paris agreement 2017) is expected to be catalytic in increasing the usage of Mg-based materials in very near future [4]. Further, the on-going demand for biodegradable materials that possess modulus properties close to that of natural bone alongside excellent biocompatibility is also making Mg-based materials promising candidates in orthopaedic applications [5]. Mg as a biodegradable, bioresorbable and biocompatible metal enhances cell adhesion and osteoblastic activity and is also responsible for improved bone regeneration and healing [6]. Further, biomaterials should possess adequate strength to withstand adequate mechanical loads besides exhibiting good biocompatibility [7]. However, there are factors such as limited ductility, limited strength, lower creep resistance, high corrosion rate and perceived easy susceptibility to ignition that limit the adaptability of Mg in structural and biomedical applications [8].

An effective way to overcome these limitations is by developing novel composites with the addition of inexpensive low volume fraction of nanoparticulates (NPs) into Mg matrix [9]. The introduction of NPs into Mg matrix at lower concentrations assists in achieving superior specific strength and ductility properties by means of dispersion strengthening without adversely affecting the density of the material [10,11]. For example, Tun et al. [10] developed Mg-ZnO nanocomposites using microwave sintering assisted powder metallurgy (P/M) technique and reported that Mg-1.5 vol% ZnO nanocomposite achieved proof stress of 125 MPa with an elongation of ~17% which is much higher than commercial Mg-alloys like AZ31, AZ91, WE43, and ZK21. In addition to ZnO, other metal oxides such as Al_2O_3, TiO_2, ZrO_2 and SiO_2 have been used as potential reinforcements for Mg due to their superior mechanical properties and thermal stability at elevated temperatures [12]. The metal oxide NPs are chemically stable and do not

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undergo further oxidation which plays a vital role in the ease of their handling and further processing [13]. Apart from enhanced physical and mechanical properties, a high degree of biocompatibility and non-toxicity of these metal oxides is also a crucial factor for targeting these materials for biomedical applications [14].

Incorporation of rare earth metal oxides (REOs) including those of the lanthanum series as well as yttrium and scandium into metallic matrices can induce 'reactive element effect' (REE), due to their strong rare-earth-oxygen interactions [15]. Along with enhancing the protective characteristics of oxides on many metals and alloys, the addition of such REOs also helps in realizing a refined microstructure and good strength-ductility combination in the composites [15,16]. Further, REOs are thermally stable reinforcements and hence can be used in extremely high-temperature applications [17]. Also, the use of nano REOs in lower amounts (typically ~2 vol%) helps in minimizing the possible usage of RE elements which have found to cause latent toxicity and several other harmful effects on the human body [12]. However, compared to other MO reinforcements, REOs offer unique advantages in enhancing the performance of Mg composites which may not be realized otherwise. Only one recent study so far characterized the ignition, compressive and damping properties of Mg reinforced with Samarium (III) oxide nanoparticles (SNPs) [16]. Simultaneous and significant improvements in the compressive strengths and fracture strain values were realized with increased addition of SNPs, with $Mg1.5Sm_2O_3$ nanocomposite showing a compressive strength of ~395 MPa (~58% higher than pure Mg) and $Mg1.0Sm_2O_3$ nanocomposite showing ~15% enhancement in the ductility as compared to monolithic Mg. Incorporation of 1.5 vol% SNPs also enhanced the damping capacity of pure Mg significantly by ~189%. As a result of reactive element effect induced by SNPs, $Mg1.5Sm_2O_3$ nanocomposite exhibited the onset of ignition at ~650 °C which surpassed the ignition temperature of most of the commercial aerospace Mg alloys like AZ61, ZK40A, ZK60A, AM50, and AZ91A and Federal Aviation Administration (FAA) approved alloy like WE43 whose ignition temperature is in the range 600–640 °C [12]. Promising results obtained for such REOs reinforced Mg nanocomposites is a great encouragement to develop new REO containing Mg-based composites with superior combination of overall properties.

Ceria (CeO_2) is one such REOs with unique properties such as high thermal stability [18], high temperature oxidation resistance [19], high hardness [20], superior wear and corrosion resistance [21,22], specific chemical reactivity [20], high refractive index and UV absorbing ability [23]. As a result, recently ceria nanoparticles (CNPs) have gained a lot of interest as a coating material for corrosion protection of metals and alloys [22], high temperature oxidation safe guards [20], catalytic convertors for removing toxic gases [24], electrochemical cells [22], UV-absorbers [25], as an oxygen ion conductor in solid oxide fuel cells [24], additives in ceramics [26], thermal barriers [27], solar cells [28], electrochromic thin-film applications [24] and as glass abrasives [29], to name a few. Further, CNPs are used for applications in nanobiology and regenerative medicine due to their unique regenerative properties owing to their low reduction potential and the coexistence of both Ce^{3+}/Ce^{4+} on their surfaces. Also, CNPs, when incorporated in scaffolds, can act as a stimulus and oxidant, enabling faster growth or healing in soft or hard tissue regeneration [30].

Owing to the several advantages of Mg and CNPs in the field of structural and biomedical applications, the present study was aimed to develop high-performance Mg-based nanocomposites using CNPs as a reinforcement through the powder metallurgy technique, using microwave sintering of the powder compacts followed by hot extrusion. The effect of presence of CNPs on microstructural, mechanical, damping and thermal properties was investigated. Also, the structure-property correlations have been drawn and analyzed. The properties of Mg-CeO_2 nanocomposites with varying amount of reinforcement (0.5, 1 and 1.5 vol%) are reported and the test results are benchmarked against monolithic Mg.

2. Materials and methods

2.1. Materials

Pure magnesium (Mg) powder with a size range of 60–300 μm and a purity of ≥ 98.5% supplied by Merck (Darmstadt, Germany) was used as the matrix material and CeO_2 powder with an average size of 20–30 nm was procured from US Research Nanomaterials, Inc., USA.

2.2. Synthesis

The synthesis process for Mg-CeO_2 nanocomposites involved blending pure Mg powder with CNPs using the RETSCH PM-400 mechanical alloying machine (Haan, Germany) without balls at 200 rpm for 1 h. The blended powder mixtures were then cold compacted using a 100-ton press to form billets measuring 35-mm diameter and 45-mm height. Monolithic Mg was also compacted using the same parameters. The compacted billets were then sintered using hybrid microwave sintering technique for 16 min to reach a temperature of 640 °C using a 900 W, 2.45 GHz SHARP microwave oven. The sintered billets were homogenized at 450 °C for 2 h prior to hot extrusion at 400 °C at an extrusion ratio of 20.25:1. Detailed information on processing methodology can be found in Refs. [31–34].

2.3. Materials characterization

2.3.1. Microstructural characterization

Microstructural characterization studies were conducted on the extruded polished samples to determine the presence, distribution and interfacial integrity of CNPs using JEOL JSM-6010 Scanning Electron Microscope (JEOL Ltd., Tokyo, Japan).

2.3.2. Density measurement

The experimental densities of the extruded pure Mg and Mg-CeO_2 nanocomposites were determined using Archimedes' principle. The samples were first weighed in air and then in distilled water using an A &D ER-182A electronic balance maintaining an accuracy of ± 0.0001 g. The theoretical densities of the synthesized materials were calculated using rule of mixture.

2.3.3. Microhardness

Microhardness measurements were performed on the polished samples using Shimadzu-HMV automatic digital microhardness tester with a Vickers indenter. An indentation load of 245.5 mN and a dwell time of 15 s was used in accordance with the ASTM standard E384-08.

2.3.4. Coefficient of thermal expansion (CTE)

The CTE values of the extruded materials were determined utilizing a heating rate of 5 °C min^{-1} using a LINSEIS TMA PT 1000LT thermomechanical analyzer (New Jersey, USA). Argon flow rate was set at 0.1 L min^{-1}. The displacement of the samples was measured using an alumina probe as a function of temperature (30–350 °C).

2.3.5. Phase transformation determination

DSC studies using Shimadzu DSC-60 instrument was carried out at a heating rate of 5 °C min^{-1} from room temperature to 600 °C in flowing argon atmosphere to determine the phase transformations in the nanocomposites. The deflection in the curve (exothermic or endothermic peak) indicate the occurrence of reactions due to phase transformations.

2.3.6. Ignition testing

The ignition temperatures of the synthesized materials were determined using a Thermo Gravimetric Analyzer (TGA). Samples of dimensions 2 × 2 × 2 mm^3 were heated from 30 to 750 °C at a heating rate of 10 °C min^{-1} in the purified air with a flow rate of 50 mL min^{-1}.