MICROWAVE SINTERED MAGNESIUM NANOCOMPOSITE: HYBRID (Y_2O_3+Ni) NANO-SIZE REINFORCEMENT AND TENSILE PROPERTIES

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ABSTRACT

In the present study, results revealed that strength of the microwave sintered commercially pure magnesium was significantly enhanced till 100°C due to the incorporation of hybrid (0.7 % yttria + 0.6 % nickel) nano-particle reinforcement, which diminishes gradually with a further increase in test temperature. Presence of the hybrid reinforcement particles assisted in complete recrystallization of magnesium matrix at 150°C. The hybrid reinforcement also assisted in the deformation process of magnesium and achieved large ductility at a relatively low temperature.

Keywords: Metal-matrix composites; Powder processing; High-temperature properties; Tensile Test

1. INTRODUCTION

Low density magnesium based materials are the most obvious substitute in consideration to replace the existing aluminum and/or iron based materials in automobiles. With virtually unlimited supply (eighth most common element in earth and third most common element in dissolved seawater minerals [1]), successful application of magnesium will significantly improve fuel efficiency and sequential reduction in carbon emission. An estimated twenty kilogram of carbon dioxide emission reduction calculated for every kilogram of automobile weight loss [2]. It is assumed that automobile could be at least 35% lighter once magnesium replaces traditional steel in their space frame application [3]. However, execution of this ambitious plan requires application of magnesium in extruded form. Till now cast magnesium are in automobile application and hexagonal closed packed crystal structure induced relatively poor ductility limits magnesium application in extruded and sheet form. Recent development of high performance reinforced magnesium [4-6] brought hope to the anticipated application of magnesium in extruded form. In the development process of these materials it was found that nano-size thermally stable reinforcement simultaneously improves the strength and ductility of magnesium. Induction of non-basal slip system at room temperature deformation process [6] was attributed to the extraordinary improvement in the ductility of magnesium at ambient temperature and concurrently clear the ways of extrusion of magnesium into intricate near net shaped objects at a temperature significantly below the most commonly used temperature of above 225°C [1, 7]. Exploiting the understanding of effect of the nano-size ceramic powder [4-6] in ductility improvement process and metallic powder [8, 9] in strength improvement process of magnesium led to the recent development of a high performance hybrid nanocomposite [10]. Nano-size yttria and nickel particles were combined as hybrid reinforcement in commercially pure magnesium matrix, which led to the significant improvement in the room temperature tensile strength and ductility of the matrix. A comprehensive investigation on the developed material is needed to understand the effect of the hybrid nanoparticles reinforcement on the tensile properties of the magnesium matrix at higher temperature to ensure its possible industrial applications. Accordingly, this work was focused to investigate the effect of hybrid (i.e., yttria and nickel) nanoparticles reinforcement on the tensile properties of commercially pure magnesium matrix when subjected to higher test temperatures.

2. EXPERIMENTAL PROCEDURES

2.1 Processing

Magnesium powders (Merck, Germany) matrix (98.5% purity, 60–300 μm size) was reinforced with yttria (Inframat Advanced Materials, USA, 0.7 volume percentage, 30–50 nm size) and nickel (Nanostructured and Amorphous Materials Inc., USA, 0.6 volume percentage, 20 nm) particles using blend-press-microwave sintering powder metallurgy technique. Magnesium and hybrid reinforcement powders were blended together in a mechanical alloying machine (RETSCH PM-400) for 60 minutes at a 200 revolution per minute followed by cold compaction on a 100 ton uniaxial hydraulic press using 510MPa pressure. Balls and process control agent were not used during the blending process. Compacted billets (35 mm in diameter and 40 mm in height) were sintered for 13 minutes at a near melting temperature (i.e., 643°C) using a hybrid microwave sintering technique [11] followed by hot extrusion (350°C) to cylindrical rods with an extrusion ratio of 19.14:1.

2.2 Microstructural characterization

Microstructural characterization of the extruded hybrid nanocomposite was done to study the nanoparticles reinforcement distribution pattern and their effect on the grain morphology of commercially pure magnesium matrix. A Hitachi S4300 Field-Emission Scanning Electron Microscope (FESEM) with energy dispersive X-ray spectroscopy (EDS) and a Olympus metallographic microscope were used in these purpose. Grain characteristics were analyzed using image analysis software Scion.

2.3 Tensile testing

Tensile characteristics of the extruded hybrid nanocomposite samples were investigated at different test temperatures (i.e., 25°C, 100°, 150°C and 200°C). Tensile tests samples (diameter of 5 mm and gauge length of 25 mm) were tested in Instron 5500 machine coupled with air circulated resistance heating chamber using a strain rate of $0.01s^{-1}$. Room temperature elongation-to-fracture tests were conducted in accordance to ASTM E8M-05. In the high temperature elongation-to-fracture test, the samples were soaked at testing temperature for ~5 minutes prior to the test. Upon fracture of each of the tensile test, one half of the fractured sample left for furnace cooling and the other half of the fractured sample quenched into cold water to freeze the grain morphology existed during the fracture process. Olympus metallographic microscope was used to investigate the quenched grain morphology of the fractured magnesium matrix Fractography on the furnace cooled magnesium fracture surface conducted using JEOL JSM-5800 LV Scanning Electron Microscope (SEM).

3. RESULT AND DISCUSSION

3.1 Macrostructural Characteristics

No visible oxidation was found on the surfaces of extruded rods and high-temperature tensile tested samples. Porosity in the magnesium matrix increased marginally due to the incorporation of the hybrid nano-reinforcement, which remain low enough (i.e., 0.21 volume percentage) to have dense hybrid nanocomposite [10].

3.2 Microstructural Characteristics

Added reinforcement with the intermetallic (see Fig. 1) were found to be present predominantly at the grain boundary. Mg2Ni intermetallic was formed due to the reaction occurred at high sintering temperature between magnesium matrix and nickel reinforcement [12]. Presence of relatively thermally stable reinforcement and intermetallic at the grain boundary facilitated the grain boundary pinning in magnesium matrix and significantly refined the microstructure (see Table 1) [10].

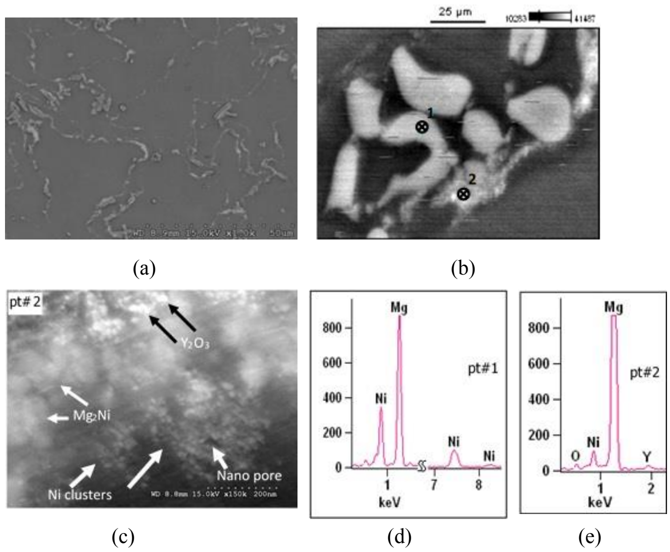


Fig. 1: Reinforcement and intermetallic phase distribution in progressively higher magnification FESEM micrographs (a), (b) and (c), and their identification using EDS analyses (d) and (e), respectively, in hybrid nanocomposites [10].

3.3 Tensile Characteristics

Result of room temperature tensile test revealed that commercially pure magnesium matrix experienced significant improvement in 0.2% yield strength as well tensile strength due to the presence of hybrid nano-reinforcement as shown in Table 2. The strengthening of magnesium matrix was attributed to the cumulative effect of: (a) presence of increasing dislocation density in magnesium matrix, due to the misfit in elastic modulus (44.7GPa for magnesium [13], 171.5 GPa for Y_2O_3 [14] and 207 GPa for nickel [13]) and coefficient of thermal expansion (CTE) (28.9 x 10^{-6} K^{-1} for magnesium [13], 7.6 x 10-6 K^{-1} for Y_2O_3 [14] and 13.9 x 10-6 K^{-1} for nickel [13]) between the matrix and hybrid reinforcement, (b) Orowan strengthening due to the presence of harder reinforcement (including Mg-Ni intermetallic), and (c) Hall-Patch strengthening effect due to significant grain refinement. Details of the room temperature strengthening effect of the hybrid nano reinforcement in magnesium matrix is available in reference [10].

Results of increasing temperature tensile tests further revealed that strengthening effect of hybrid nano reinforcement on commercially pure magnesium gradually decreased with increasing temperature as shown in Fig. 2 and Table 2. Thermally activated deformation process, e.g., activation of non-basal slip system, managed to gradually decrease the strength of the magnesium under increas-

Table 1: Physical and microstructural characteristics of hybrid nanocomposite [10].

Material	Density (g/cm^3)		Porosity (vol%)	Grain Characteristics	
	Theoretical	Experimental		Size (μm)	Aspect Ratio
Mg	1.740	1.738 ± 0.007	0.13	20	1.4 ± 0.1
$Mg/(0.7Y_2O_3+0.6Ni)$	1.806	1.802 ± 0.002	0.21	6	1.4 ± 0.3

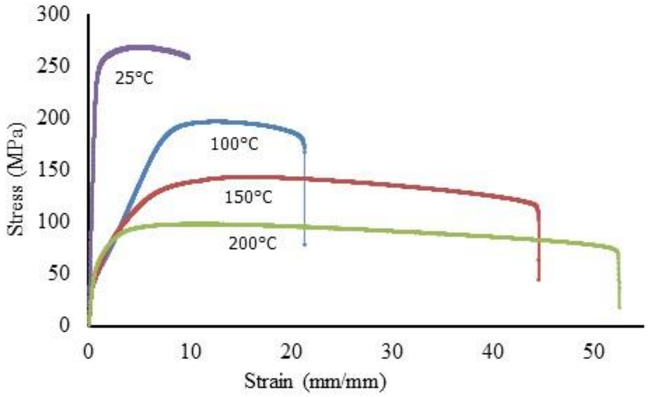


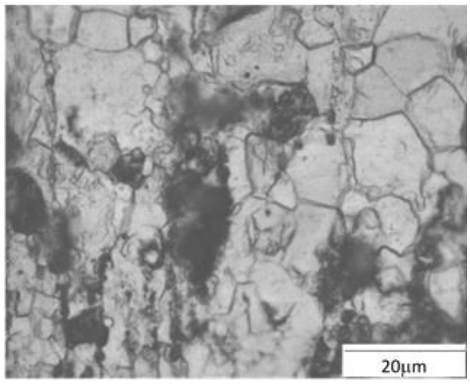
Fig. 2: Elongation-to-tensile behavior of hybrid nano particles reinforced magnesium at different testing temperature

Table 2: Tensile behaviour of hybrid nanocomposite.

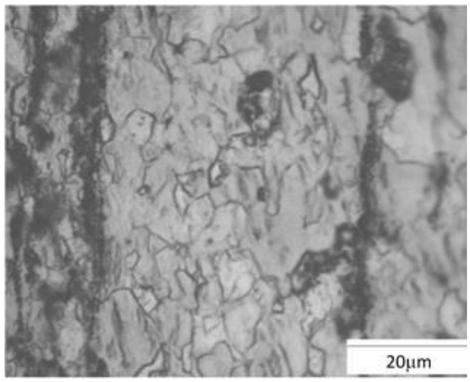
Materials	Temperature (°C)	0.2%YS (MPa)	UTS (MPa)	Failure Strain (%)
Mg	25	134	193	6.9
Mg/(0.7Y_2O_3+0.6Ni)		232	272	9.5
	100	192	197	21.3
	150	127	143	44.6
	200	78	98	52.5

ing testing temperature. Thermally activated deformation process (e.g., weakening of bond, increasing vacancy, activation of non-basal slip system) led to an easy dislocation motion in the matrix, which reduced the required applied stress for matrix deformation. It should be noted that the nano yttria particles are eligible in activation of non-basal slip system in magnesium to a certain extent at room temperature [4-6], which otherwise activated above 225°C [7]. However, the thermally stable hybrid nano reinforcement (i.e, melting temperature for yttria is ~2690°C, nickel is 1455°C) maintained their strengthening effect on the commercially pure magnesium matrix till 100°C (as compared to the room temperature strength of un-reinforced matrix). The diminishing strengthening effect of the hybrid nano-reinforcement (with increasing temperature) was due to the cumulative effect of: (a) a significant reduction in dislocation density (by complete recrystallization and grain growth, see Fig. 3), and (b) dislocation bypass of reinforcement particles without strong interaction.

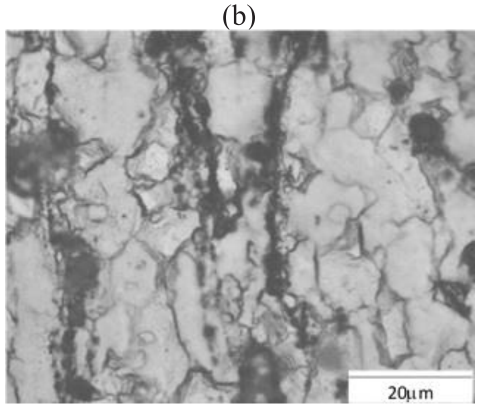
Room temperature tensile test also revealed a significant increase in ductility of magnesium matrix (see Table 1) due to presence of hybrid nano reinforcement due to the cumulative effect of: (a) grain refinement [15], (b) presence of extremely fine nano-size reinforcement particles [16], and (c) nano-particle induced non-basal slip system [6]. The fine reinforcement particles in hexagonal closed packed magnesium matrix: (i) provide sites to open cleavage crack to an advancing crack front to dissipate stress concentration, and (ii) alter the local effective stress from plane strain to plane stress state in the neighborhood of the crack tip.



(a)



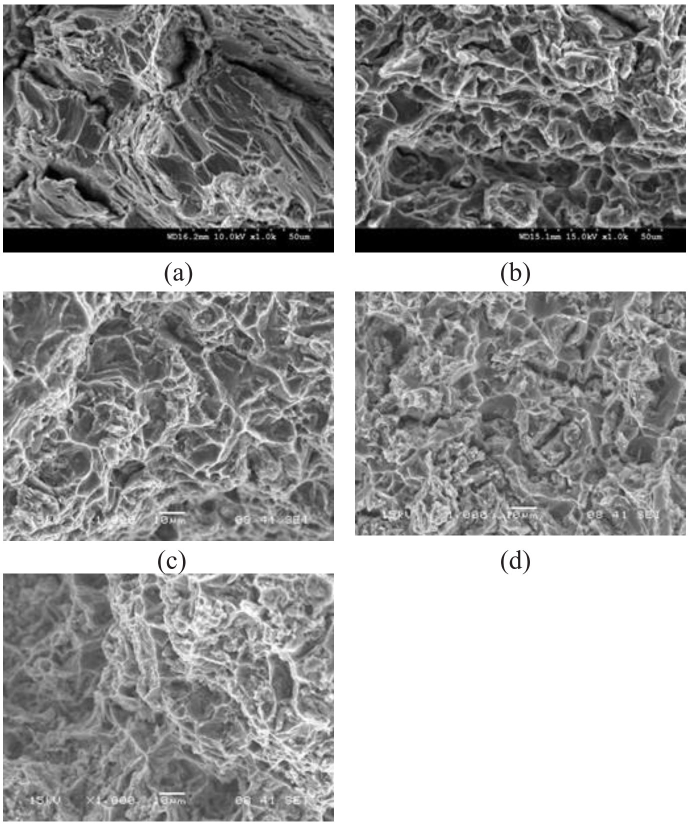
(b)



(c)

Fig. 3: Optical micrographs showing grain morphology in magnesium matrix with hybrid nano reinforcement near the fracture tip at: (a) 100°C, (b) 150°C, and (c) 200°C, respectively.

Matrix ductility increment continues with increasing test temperature (see Fig. 2 and Table 2) used in this study. Presence of hybrid nano reinforcement induced high ductility in the commercially pure magnesium at a much lower deformation temperature when compared to the temperature used in common hot work process for magnesium based materials [1, 7]. Lattice parameters of the magnesium crystal structure was remained unchanged due to the extremely negligible diffusivity of nickel atoms (e.g., 0.04 at% at 500°C) [12]) and, hence, crystal structure induced intrinsic deformability also remained unchanged. However, the remarkable large increment in ductility of the magnesium could be attributed to the cumulative effect of: (a) reduction in dislocation density, (b) thermally activated easy dislocation motion, (c) complete recrystallization by dynamic recrystallization (see Fig. 3). Presence of face



(e)

Fig. 4: Representative fractographs showing: brittle failure in unreinforced magnesium (a), ductile failure in hybrid nano reinforcement incorporated magnesium at (b) 25°C, (c) 100°C, (d) 150°C, and (e) 200°C, respectively.

centered cubic structure ductile nickel particles at grain boundaries might have acted as sink for dislocation and/ or stress concentration triple point and assisted in ductility enhancement by avoiding triple point microvoid formation. In essence the current study indicated the potential of near-net-shape fabrication process of magnesium into intricate shapes, when incorporated with small volume percentage (i.e., ~1%) of thermally stable nano size ceramic and metallic hybrid particles, at a much lower processing temperature than the currently used 350°C and above.

3.4 Fracture Characteristics

Tensile fracture surface study revealed the microstructural effect on the ductility and related fracture characteristics of hybrid nano reinforcement incorporated magnesium. Brittle failure characteristic microscopically rough small steps (see Fig. 4a) in magnesium transformed into ductile failure characteristic fine dimples of varying size (see Fig. 4b-e) with overall microscopically fine fracture surfaces. Presence of extremely finer particle, as low as 5 nanometer, favors the formation of dimples through void nucleation in the matrix-reinforcement interface [17] and was evident in reinforced magnesium matrix. The ductile dimples formation on the tensile fracture surfaces of reinforced magnesium matrix continued to be evident as failure characteristics with increasing testing temperature.

4. CONCLUSIONS

In this study, thermally stable hybrid nanoparticle reinforcement significantly improved the room temperature tensile strength and ductility of the commercially pure magnesium matrix. The hybrid nanoparticles reinforcement was able to retain their strengthening effect on the magnesium matrix until 100ºC, which gradually diminished with further increase in the test temperature. The hybrid nano-reinforcement particles assisted dynamic recrystallization and thermally activated deformation induced large ductility in reinforced magnesium at relatively low temperature.

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