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S. FIDA HASSAN*#, KHIN SANDAR TUN**, F. PATEL*, NASSER AL-AQEELI*, M. GUPTA***

NICKELIZATION EFFECT ON THE HIGH TEMPERATURE TENSILE PROPERTIES OF Mg-0.7Y_2O_3 NANOCOMPOSITE

In this study, high performance magnesium-yttria nanocomposite's room temperature, strength and ductility were significantly enhanced by the dispersion of nano-sized nickel particles using powder blending and a microwave sintering process. The strengthening effect of the dispersed nano-sized nickel particles was consistent up to 100°C and then it gradually diminished with further increases in the test temperature. The ductility of the magnesium-yttria nanocomposite remained unaffected by the dispersed nano-sized nickel particles up to 100°C. Impressively, it was enhanced at 150°C and above, leading to the possibility of the near net shape fabrication of the nanocomposite at a significantly low temperature.

Keywords: Magnesium; Nanocomposite; High-Temperature; Tensile Strength; Deformation

1. Introduction

Stiff and strong particle dispersion can effectively improve the structural properties of metals beyond the limit achievable by alloy addition. The compatibility of particle incorporation methods with matrix, type, size, morphology and volume fraction, govern the resultant properties of the reinforced metal. Magnesium is considered to be the strongest candidate to replace the existing structural materials in the automobile and aerospace industries due to its light weight and abundance [1]. Thermally stable nano-sized yttria particles were found to be an effective reinforcement in improving the structural properties of magnesium [2-7] and their strengthening effect was maximized when added at a level of 0.7 volume percentage [5-7], as shown in Table 1. Anomalous to the general trend of reinforced metals, nano-sized yttria particles significantly increased the ductility of intrinsically brittle magnesium. The strength of nano-sized yttria reinforced magnesium was reportedly enhanced further by the addition of 0.6 volume percentage of nano-sized nickel particles [8]. Nickel, with its large atomic size and different crystal structure, has an extremely negligible solubility in magnesium (e.g. 0.04% at 500°C) [9]) and is reported to have a significant strengthening effect on magnesium when present as a reinforcement [10,11]. Limited reactivity between nickel and magnesium induced good reinforcement-matrix interfacial integrity and stronger Mg-Ni intermetallic in the matrix. An extensive investigation is necessary to analyze and understand the effect of nickelization on the strength and ductility of nano-sized yttria reinforced magnesium when heated to a high temperature in order to develop its potential for industrial fabrication, as well as other applications. Hence, the focus of this study was to investigate the effect of nano-sized nickel on the strength and ductility of nano-sized yttria reinforced magnesium at an increasing temperature.

TABLE 1

Mechanical Properties of Mg/0.7Y_2O_3 nanocomposite processed by different primary processing techniques

Processing	Microhardness (HR15T)	0.2%YS (MPa)	UTS (MPa)	Ductility (%)	Work of Fracture (MJ/m^3)
IM [5]	58 (57%↑)	312 (222%↑)	318 (84%↑)	6.9 (7%↓)	18.2 (64%↑)
PM (TS) [6]	46 (7%↑)	151 (14%↑)	202 (5%↑)	12.0 (186%↑)	20.9 (194%↑)
PM (MS) [7]	45 (22%↑)	157 (17%↑)	244 (26%↑)	8.6 (15%↑)	21.8 (69%↑)

IM = Ingot Metallurgy, PM = Powder Metallurgy, TS = Traditional Sintering, MS = Microwave Sintering
Values within parentheses () showed changes in properties of magnesium due to incorporation of nano Y_2O_3 particles.

* KING FAHD UNIVERSITY OF PETROLEUM & MINERALS, DEPARTMENT OF MECHANICAL ENGINEERING, P.O. BOX 1061, DHAHRAN 31261, KINGDOM OF SAUDI ARABIA
** TECHNOLOGY DEVELOPMENT ENGINEER, ITE COLLEGE CENTRAL, H1-01, 2 ANG MO KIO DRIVE, SINGAPORE 567720
*** NATIONAL UNIVERSITY OF SINGAPORE, DEPARTMENT OF MECHANICAL ENGINEERING, 9 ENGINEERING DRIVE 1, SINGAPORE 117576

Corresponding author: sfhassan@kfupm.edu.sa, itsforfida@gmail.com

2. Experimental procedures

2.1. Processing

Magnesium matrix powder (98.5% purity, 60-300 μm size, by Merck, Germany) was reinforced with yttria (30-50 nm size, 0.7 volume percentage, by Inframat Advanced Materials, USA) and nickel (20 nm, 0.6 volume percentage, by Nanostructured and Amorphous Materials Inc., USA). Nano-sized nickel particles were dispersed in magnesium-yttria nanocomposite powder by blending magnesium powder with nano yttria and nickel reinforcement powders in a mechanical alloying machine (RETSCH PM-400) at 200 revolutions per minute for 60 minutes followed by cold compaction in a 100-ton press at a pressure of 510 MPa. No balls and process control agents were used during the blending step. The compacted billet size was found to be 35 mm in diameter and 40 mm in height. Billets were sintered using the hybrid microwave sintering technique for 13 minutes, which heated the billet close to the melting temperature of the matrix (i.e. 643°C), followed by hot extrusion at a temperature of 350°C to produce cylindrical rods with an extrusion ratio of 19.14:1. Magnesium-yttria nanocomposite was prepared using a similar procedure to the one discussed above except for the addition of nickel particles. Details of this materials processing is available elsewhere [7,8].

2.2. Microstructural characterization

The microstructural characterization of extruded magnesium-yttria nanocomposite with and without nano-sized nickel particles was done to study the distribution pattern of the nickel particles and their effect on the grain morphology of the yttria reinforced magnesium matrix. A Hitachi S4300 Field-Emission Scanning Electron Microscope (FESEM) equipped with energy dispersive X-ray spectroscopy (EDS) and an Olympus metallographic microscope were used in this study. Scion software was used for metallographic image analysis to find the grain's characteristics.

2.3. Tensile testing

The tensile characteristics of the extruded magnesium-yttria nanocomposite samples, with and without nano-sized nickel particles, were studied at 25°C, 100°, 150°C, and 200°C. A room temperature elongation-to-fracture tensile test was performed in accordance with the ASTM E8M-05. The samples used were 5 mm in diameter and 25 mm in length. High temperature tests were conducted using an Instron 5500 machine coupled with an air circulated resistance heating closed chamber with a strain

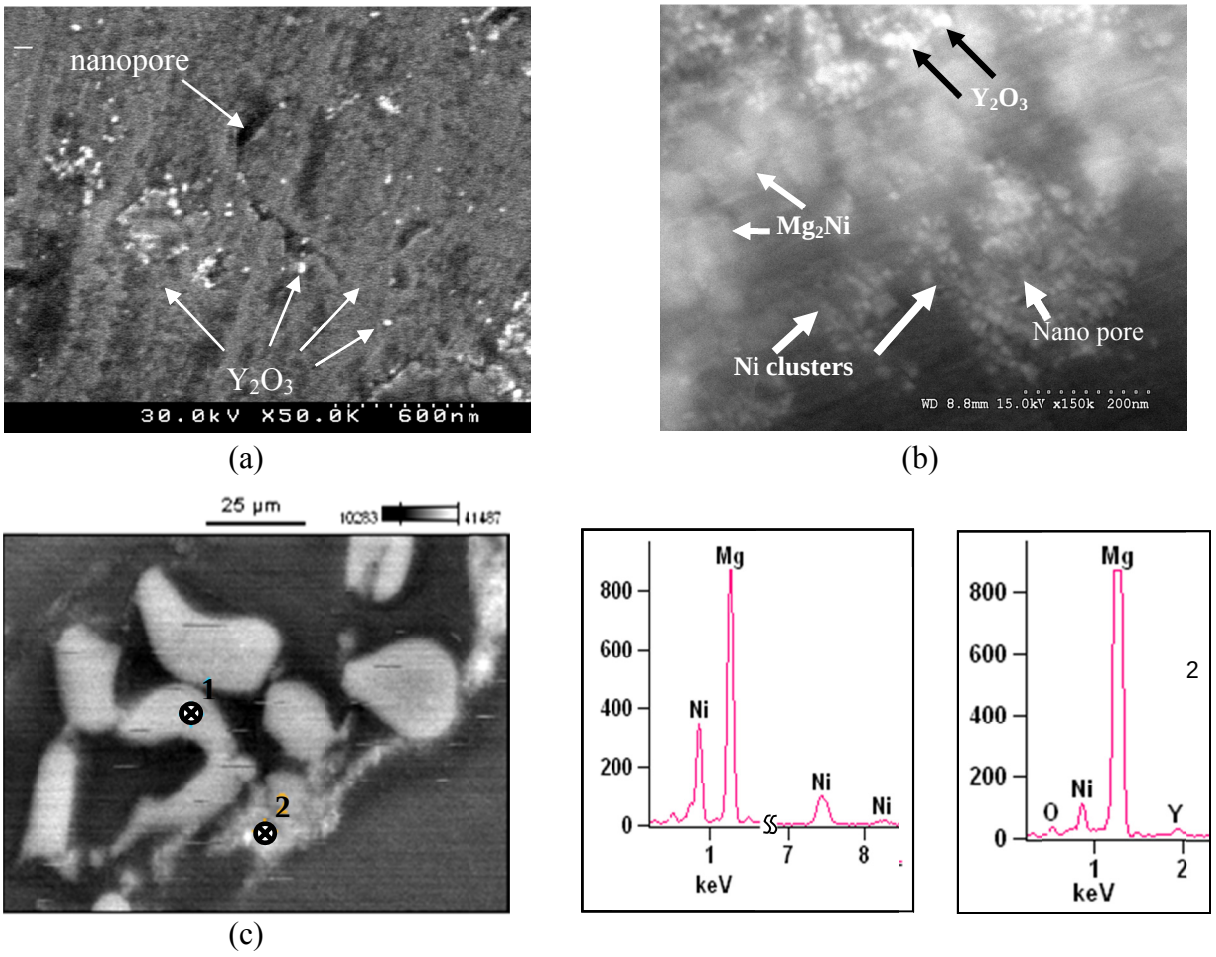

Fig. 1. Reinforcements and intermetallic phase distribution in FESEM micrographs: (a) magnesium-yttria nanocomposite, (b) nano-sized nickel incorporated magnesium-yttria nanocomposite, and (c) intermetallic identification using EDS analyses, respectively [8]

rate of 0.01 s^{-1}. Tensile samples were kept soaked at the testing temperature for ~5 minutes prior to the test. Stress-strain curves at all test temperatures were obtained from the tensile test data and the fracture values were also computed using a simple mathematical conversion of these data. Fractography on the fracture surfaces was conducted using a JEOL JSM-5800 LV Scanning Electron Microscope (SEM) to understand the effect of nano-sized nickel particles on the fracture behavior of the magnesium-yttria nanocomposite.

3. Results and discussion

3.1. Macrostructural characteristics

There were no recognizable macroscopic defects observed on the surface of the sintered billet. Careful macroscopic observation also did not reveal any visible oxidation on the surfaces of the high temperature tensile test samples. The incorporation of the nano nickel marginally increased the porosity volume percentage but maintained the dense nature of the magnesium-yttria nanocomposite.

3.2. Microstructural characteristics

Microstructural characterization revealed the presence of Mg-Ni intermetallic in the yttria reinforced magnesium matrix [8]. Mg-Ni intermetallic is the reaction product of the matrix and the nano-sized nickel particles [9] due to the high sintering temperature used in this study. The presence of nano-sized nickel and the Mg-Ni induced significant grain refinement in the magnesium matrix (see Figure 2 and Table 2) of the nano-sized yttria reinforced magnesium.

3.3. Tensile characteristics

The room temperature elongation-to-failure tensile test revealed (see Table 3) that the addition of nano-sized nickel particles notably improved the strength (both 0.2% yield strength and tensile strength) of the magnesium-yttria nanocomposite. The nano-sized nickel particle strengthening effect could be attributed to the cumulative effect of: (a) increased dislocation density in the nanocomposite due to the considerable difference in elastic modulus (44.7GPa for magnesium and 207 GPa for nickel) and the coefficient of thermal expansion (28.9×10^{-6} K^{-1} for magnesium and 13.9×10^{-6} K^{-1} for nickel) mismatch [12] between the matrix and the nano-sized nickel particles, (b) Orowan strengthening due to the presence of stiffer nano-sized nickel particles and their reaction derivative, and c) Hall-Patch strengthening due to the significant grain refinement. Details on the strengthening mechanism are available in reference [8].

The high temperature elongation-to-failure tensile test revealed that the presence of nano-sized nickel particles and its

TABLE 2

Physical and microstructural characteristics of nano-sized nickel incorporated $Mg/0.7Y_2O_3$ nanocomposite [7,8]

Material	Density (g/cm^3)		Porosity (vol%)	Grain Characteristics	
	Theoretical	Experimental		Size (μm)	Aspect ratio
$Mg/0.7Y_2O_3$	1.763	1.757 ± 0.006	0.35	18	1.4 ± 0.2
$Mg/0.7Y_2O_3$/0.6Ni	1.806	1.802 ± 0.002	0.21	6 (67%↓)	1.4 ± 0.3

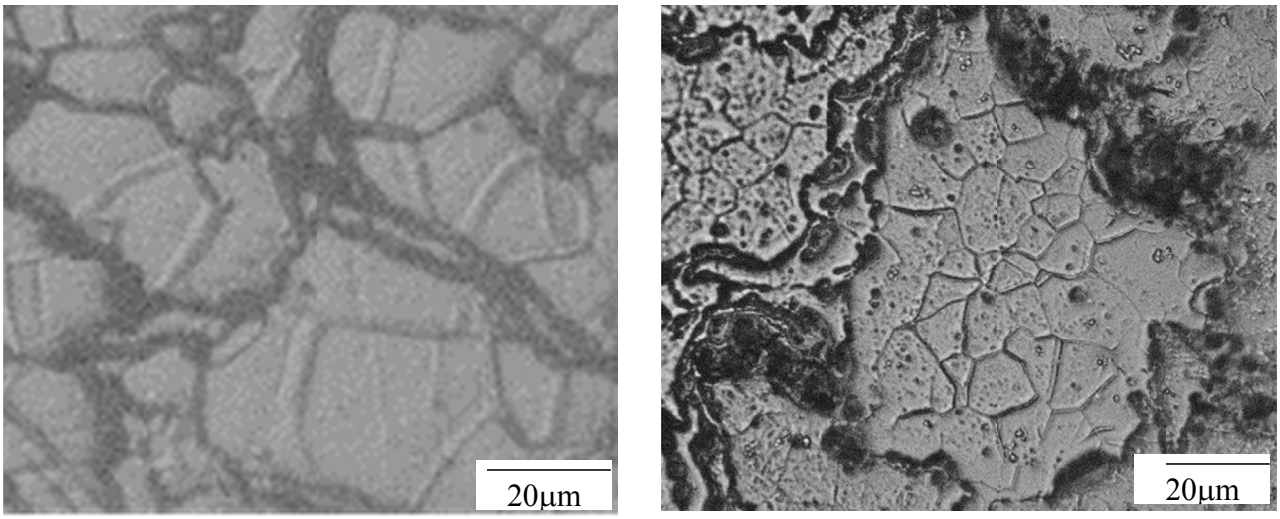

Fig. 2. Optical micrographs showing grain morphology in (a) magnesium-yttria nanocomposite, (b) nano-nickel incorporated magnesium-yttria nanocomposite, respectively

TABLE 3

Tensile behavior of nano-sized nickel incorporated Mg/0.7Y_2O_3 nanocomposite

Material	Mg/0.7Y_2O_3				Mg/0.7Y_2O_3/0.6Ni			
	25°C	100°C	150°C	200°C	25°C	100°C	150°C	200°C
0.2%YS (MPa)	157	94	88	69	232	192	127	78
UTS (MPa)	244	150	128	91	272	197	143	98
Elongation (%)	9.1	27.7	32.1	34.4	9.5	21.3	44.6	52.5
WoF (MJ/m^3)	21.8	33.1	32.2	25.1	21.8	34.8	57.2	46.7

reaction derivative effectively resisted the initiation of yield in the magnesium-yttria nanocomposite matrix with the increasing temperature used in this study (see Figure 3b and Table 3). The strengthening effect of nano-sized nickel particles (i.e. melting temperature 1455°C) and their reaction derivative weakens as the temperature increases. Thermally activated deformation processes (e.g. weakening of bond, increasing vacancy, activation of a non-basal slip system) assisted in lowering the strength level of the nickel particle incorporated magnesium-yttria nanocomposite. High temperature stress-strain flow curves were nearly flat (see Figure 3a), proof of perfect elastic-plastic behavior without the strain hardening effect, which leads to the marginal difference between the yield strength and tensile strength of the nickel particle incorporated magnesium-yttria nanocomposite. The elastic-plastic behavior of the nickelized nanocomposite could be attributed to the easy dislocating motion coupled with decreasing dislocation density and weakened nickel particles (and intermetallic) to pin and/or resist the moving dislocation at a gradually increasing temperature. The incorporated nano-sized nickel particles effectively exerted resistance to the yielding of magnesium-yttria nanocomposite up to 100°C. However, the overall thermal stability of the magnesium-yttria nanocomposite was improved due to the incorporation of nano-sized nickel particles.

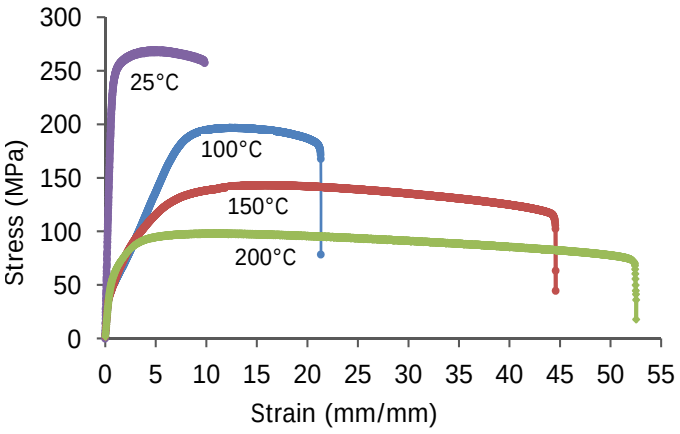

Fig. 3. Temperature dependent elongation-to-fracture tensile behavior of nano size-sized nickel particles incorporated magnesium-yttria nanocomposite

The room temperature elongation-to-failure tensile test revealed no noticeable effect on the ductility of magnesium-yttria nanocomposite (see Figure 3d and Table 3) due to the presence of nano-sized nickel particles. The effect of grain refinement in the ductility enhancement of hexagonal close pack structured magnesium [13] might have been suppressed by the presence of intermetallic in the matrix. The ductility of the nickel particle incorporated magnesium-yttria nanocomposite increased impressively when subjected to a temperature of 150°C and above. The ductility enhancement could be accorded to the easy dislocation motion coupled with their reduced presence (absence of strain hardening) in the matrix. The presence of incorporated nano-sized nickel particles could not have contributed to any modification of the intrinsic ductility of magnesium through changes in the lattice parameters and the axial ratio (i.e., *c/a*) [14] as it has an extremely negligible diffusivity (e.g., 0.04 at% at 500°C) [9] in the matrix of the magnesium-yttria nanocomposite. The results also revealed a potential for the near net shape fabrication of the magnesium-yttria nanocomposite into an intricate shape at a much lower temperature compared to the most commonly used 350°C for common magnesium based materials. The formation of ductile dimples on the fracture surfaces of the magnesium-yttria nanocomposite matrix continued to be evident in the nano-sized nickel incorporated samples (see Figure 4) as characteristics of failure with increasing testing temperature.

4. Conclusions

Nano-sized nickel particles improved the room temperature strength and ductility of the magnesium-yttria nanocomposite significantly when dispersed using powder blending and the microwave sintering process. The strengthening effect of the added nickel particles persisted in the nanocomposite up to 100°C and then gradually diminished with further increase in temperature. The ductility of the nano-sized nickel particle dispersed magnesium-yttria nanocomposite remained unaffected up to 100°C. The impressive enhancement in ductility at 150°C and above led to the possible near net shape fabrication of the nanocomposite at a significantly low temperature.

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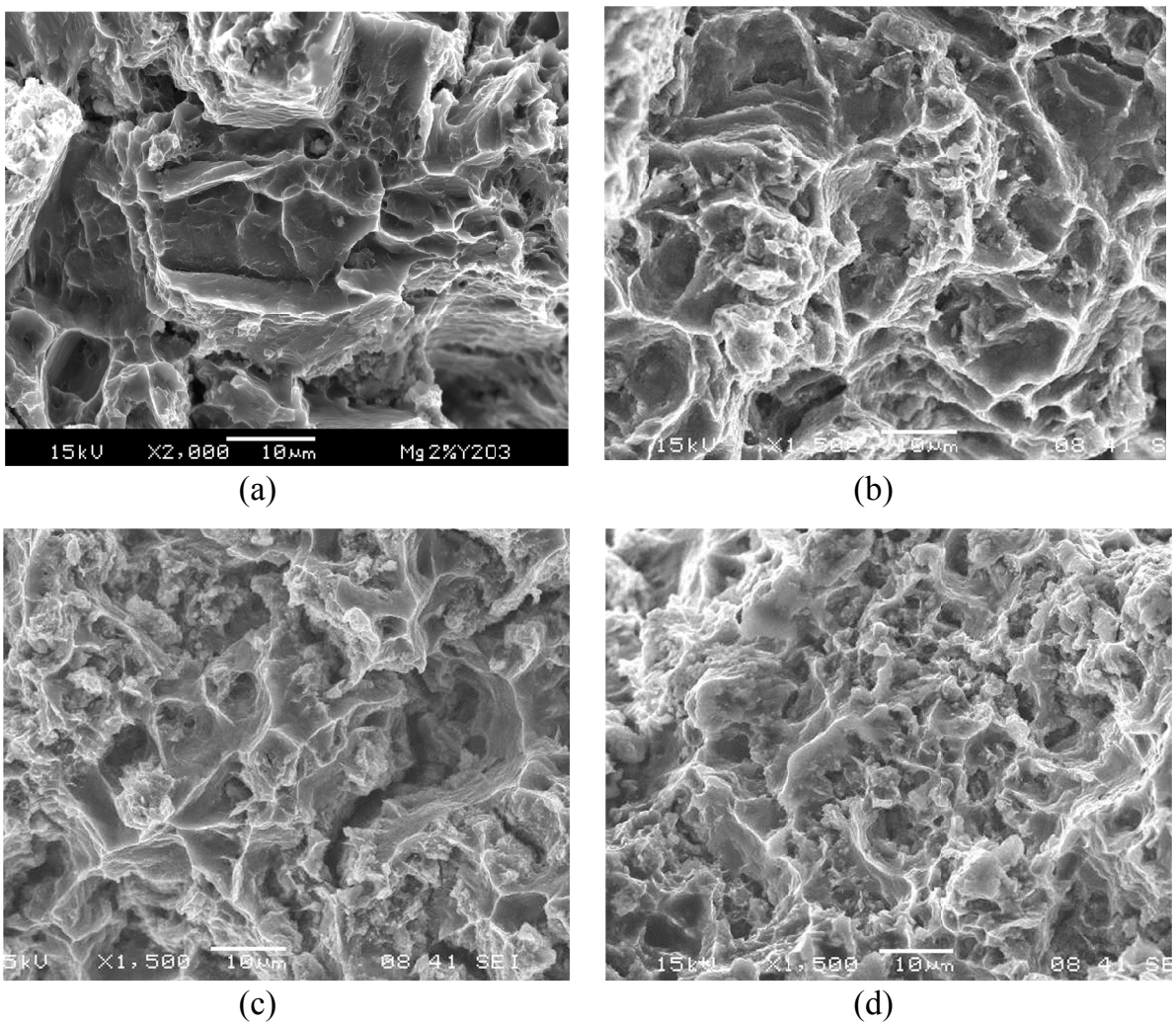

Fig. 4. Representative fractographs showing ductile dimples in: (a) magnesium-yttria nanocomposite at ambient temperature, and, (b) (c) and (d) nano-sized nickel incorporated magnesium-yttria nanocomposite at 100°C, 150°C, and 200°C, respectively

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