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CHARACTERIZATION OF HOT EXTRUDED Mg/SiC NANOCOMPOSITES FABRICATED BY CASTING

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Abstract

Mg-1%SiC nanocomposites were fabricated using an ultrasonic-cavitation based casting method, resulting in the dispersion of the reinforcing SiC nanoparticles to form Mg-metal matrix nanocomposite (Mg-MMNC) billets. The MMNC billets were then processed using hot extrusion at 350°C. Micrographic observations illustrate a significant grain size reduction and the presence of micro-bands that align the SiC nanoparticles parallel to the direction of extrusion for Mg-MMNCs. Observations from the cross-section at 90^0 of the extrusion direction show uniform nanoparticles dispersion contrasting previous observations. Results from the extruded Mg-MMNCs tensile testing at different temperatures (25^0C, 125^0C and 177^0C) reveal an increase of the yield strength, ultimate tensile strength, and ductility values as compared to the un-reinforced and extruded Mg-alloy; such increase was also observed from the microhardness testing results where an increase from 19 to 34% was measured.

Introduction

Magnesium alloys are becoming ever more prevalent in electronics, automotive and aerospace industries as energy conservation and performance demands increase. Mg alloys are one-third lighter than the equal volume of aluminum alloys, and this fact is one of the contributing factors that makes these alloys so desirable [1, 2]. Production with Mg alloys has been mostly in the field of pressure die casting because of its high productivity and dimensional accuracy [3], and extrusion is very useful for its technical and economic advantages in the production of structure components. Magnesium alloys offer high specific strength, superior damping capability, excellent machinability and good electromagnetic shielding characteristics [3]. However, Mg alloys exhibit poor mechanical performance at elevated temperatures [1, 2], their applications are usually limited to temperatures below 120 °C; in consequence, the improvement in the high-temperature mechanical properties of magnesium alloys will expand their industrial applications [4].

In general, the use of thermally stable ceramic reinforcements to create Mg based metal matrix composites (MMC) facilitates the retention of enhanced mechanical properties at elevated temperatures [4]. The desirable characteristics of MMCs include enhancements in stiffness, strength, creep resistance, and wear resistance [5]; however, MMCs normally provide lower ductility than the original matrix [4]. Cao et al. [6, 7] have been reported that nanocomposites can increase the ductility of as-cast Magnesium based metal matrix nanocomposites (MMNCs). Furthermore, by using silicon carbide (SiC) nanoparticles, Cao et al [6-13] (among others) have reported enhancement in yield strength and tensile strength without the loss of ductility by reinforcing the matrix with small volumes (<2%) of ceramic nanoparticles. However, the production of magnesium-based metal matrix nanocomposites (MMNCs) is extremely challenging for conventional casting and mechanical stirring methods. Such methods have not been successful in fabricating MMNCs due (among others) to the high specific surface area of nanoparticles and poor wettability between nanoparticles and molten metal [8]. On the other hand, the use of a technique that combined solidification processes with ultrasonic cavitation based dispersion of nanoparticles in metal melts achieved promising results for a uniform dispersion and distribution of reinforcing nanoparticles in Mg MMNCs [6-13]. During this process, ultrasonic cavitations generate micro "hot spots" with temperatures of approximately 5000°C, pressures exceeding 1000 atm, and heating rates exceeding 10^{10} K/s [14]. During MMNCs processing once nanoparticles are added to the melt, any air bubbles trapped around nanoparticle agglomerates serve as nucleation sites for cavitation generating the desired nanoparticle dispersion [9].

This paper shows results in implementing the ultrasonic cavitation based casting process to fabricate pure-Mg and Mg-1%SiC nanocomposite billets for hot extrusion. After extrusion, the mechanical properties of the MMNC rods were tensile tested at 25°C, 125°C, and 177°C. Finally, to observe the dispersion of nanoparticles and grain sizes in extruded MMNCs, optical micrographs and SEM images were obtained using samples from the tensile specimens.

Methodology

Figure 1 illustrates the experimental setup schematic of ultrasonic cavitation based solidification processing of SiC nanoparticles (SiCnp) in an Mg matrix. The experimental system was comprised of resistance heating furnace for melting the magnesium alloy, nanoparticle feeding mechanism, protective gas system, and an ultrasonic processing unit. The crucible used for melting and ultrasonic processing was made of mild steel with an inside diameter of 114 mm and a height of 127 mm. A Permendur power ultrasonic probe made of niobium C-103 alloy was used to generate a 17.5 kHz and maximum 4.0kW power output (Advanced Sonics, LLC, Oxford, CT) for melt processing. The niobium C-103 probe is 31.12 mm in diameter and 223.5 mm in length. The ultrasonic probe was dipped into the melt about 13mm. A thin walled niobium cage (31.8 mm upper diameter, 88.9 mm base diameter; 76.2 mm high, 254μm wall thickness) in

a shape of truncated cone was used to hold nanoparticles inside the melt pool during the ultrasonic processing. The niobium cage has a total of 55 holes 7.94 mm in diameter to allow the circulation of the melt and nanoparticles.

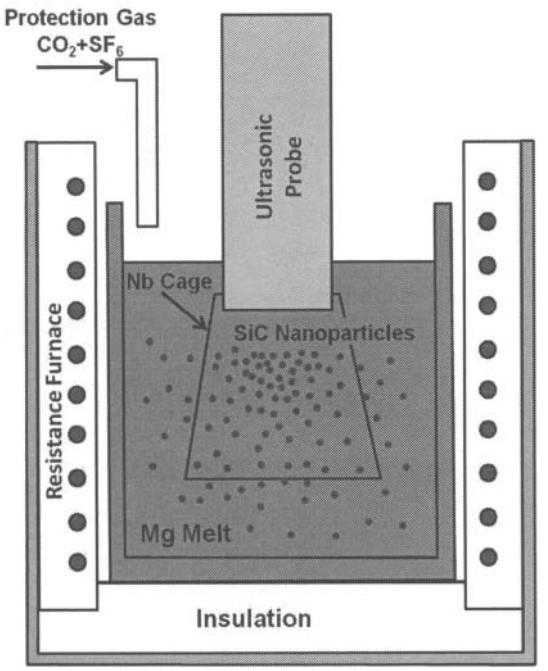

Figure 1 Schematic of Experimental setup for fabricating nanocomposites

800g of Mg was melted in the steel crucible while being protected by CO_2 and 0.75% SF_6. Once the melt temperature of 700°C was attained the niobium cage containing 1 wt. % β-SiC nanoparticles was submerged in the melt beneath the ultrasonic probe. The average size of the SiC nanoparticles used was 50nm. The melt was then ultrasonically processed at 3.5kW power level for 15 minutes for the samples with and without SiC nanoparticles. The ultrasonic probe and niobium cage were then removed from the melt and the melt was elevated to a pouring temperature of 725°C. The melt was cast into a steel permanent mold purged with CO_2 + 0.75% SF_6 and preheated to 350°C, which was designed and fabricated to produce a cast billet of 63.5 mm in diameter and 102mm in length. The casting was allowed to cool for 30 minutes before the mold was opened and the billet was removed. A graphite pouring cup was used to guide the melt into the mold, and also mounts a Pyrotech SIVEX ceramic foam filter (55mm x 55mm x 12mm, 20 pores/in).

The cast billets were preheated for two hours prior to extrusion, and were extruded at 350°C to obtain rods of 12.7 mm in diameter. The rods were extruded at a 25:1 ratio, with a ram speed of 10 mm/s and a load of 500 tons. The extruded rods of 12.7 mm (0.5 inches) in diameter were then machined into tensile bars of approximately 105 mm long with a 35 mm gauge length and 6.0 mm diameter. Tensile specimens were tested at 25°C, 125°C, and 177°C. The tensile specimens were tested in an Instru-Met TTC 90 in (228.6 cm) 4,535.92 Kg load frame using an Epsilon 3542-0100-050-HT2 extensometer with a 25.4 mm gage length clamped to each specimen. The furnace and tensile grips were preheated to the test temperature. The test specimens were then loaded into the tensile grips and the furnace was re-heated to testing temperature. The specimens were held at the testing temperature for 10 minutes before testing to ensure a uniform temperature distribution. The cross head velocity was set to 1.27 mm/min and the test was run until the specimen failure. The specimens were removed from the furnace and test fixture within 30 seconds and allowed to air cool to room temperature. The microstructures of the tested samples were studied by optical microscopy and scanning electron microscopy (SEM). Average grain size was measured using the linear intercept method from optical micrographs of the samples taken at room temperature. Samples were cut, mounted, and manually ground and polished from the grip sections of the tensile bars after tensile testing and samples were etched with 5% acetic acid in 95% water for 30 seconds. SEM was conducted in a LEO 1530 machine. Microhardness tests were conducted with a Buehler Micromet 2003 microhardness tester (load 500 gf, load time 30s). All microhardness tests were conducted at room temperature from specimens that had undergone tensile testing.

Results

Mechanical properties

The average yield strength, tensile strength, and ductility for the Mg and Mg-1%SiCnp tensile specimens conducted at various temperatures are shown in Table I. At room temperature and with the addition of 1%SiCnp to the Mg, there are significant enhancements in yield strength by 49% (from 93 to 133 MPa), tensile strength increases from 198 to 224 MPa (+13%), and ductility increase ~37% (from 5.9 to 8.1%). Below recrystallization temperature (150°C [15, 16]) for Mg and at 125°C there are moderate enhancements in yield strength (by 18%, 65MPa for pure Mg and 77 MPa for Mg-1%SiCnp) and tensile strength (by 15%), however a significant increase in ductility (by 85% measuring 13.8 for pure Mg and 25.4% for the Mg-1%SiC composite) with the addition of 1%SiC. Also shown in Table I, at 177°C (after the recrystallization temperature of Mg, 150°C) there is almost no enhancement in yield strength (by only 3%, 55MPa for pure Mg and 57 MPa for Mg-1%SiCnp), a moderate enhancement in tensile strength and 11% only (from 88 to 97 MPa), also a significant increase in ductility was averaged (91%) with the addition of 1%SiC (20.9% and 39.9% respectively).

Table I. Tested properties of hot-extruded pure Mg and Mg-1%SiC at 25°C, 125°C, and 177°C.

Testing Temperature		Yield Strength, MPa	Tensile Strength, MPa	Ductility, %	Microhardness, HV	Avg. Grain Size, μm
Room temp. (25°C)	Pure Mg	93±2.9	198±2.4	5.9±1.3	29.2 ± 1.1	33 ± 11
	Mg-1%SiCnp	133±0.7	224±1.3	8.1±1.1	39.0 ± 2.1	22 ± 4
	Variation	*49%*	*13%*	*37%*	*34%*	---
125°C	Pure Mg	65±2.9	118±1.4	13.8±1.0	30.8 ± 1.4	36 ± 27
	Mg-1%SiCnp	77±0.7	136±1.3	25.4±1.1	39.3 ± 0.8	25 ± 12
	Variation	*18%*	*15%*	*85%*	*28%*	---
177°C	Pure Mg	55±2.4	88±2.0	20.9±0.7	31.6 ± 1.5	30 ± 7
	Mg-1%SiCnp	57±3.2	97±1.8	40±1.8	37.7 ± 0.4	28 ± 7
	Variation	*3%*	*11%*	*91%*	*19%*	---

Table I shows the average microhardness of the specimens tested at room temperature (25°C), 125°C, and 177°C). For comparison, microhardness of 125°C and 177°C specimens were taken after

mechanical testing and conducted at room temperature. Samples from room temperature testing show an average microhardness of Mg and Mg-1%SiC was averaged 29.2 and 39.0 HV, respectively. The microhardness enhancements of extruded Mg-1%SiCnp compared to the tested Mg at 25°C, 125°C, and 177°C are 34%, 28%, and 19%, respectively. The performance increases in microhardness at all temperatures are largely attributed to the reinforcement that SiC nanoparticles to the Mg matrix.

Characterization

Last columns in Table I show average grain size longitudinal to the direction of extrusion in the specimens tested at 25°C, 125°C, and 177°C. The average grain size longitudinal to the direction of extrusion for extruded Mg and extruded Mg-1%SiCnp at all temperatures is 33 μm and 25 μm, respectively. Beside the calculated averages, a large variation was found (see Table I). There is not significant difference in grain refinement between the Mg and Mg-1%SiCnp due to dynamic recrystallization (DRX) during hot extrusion at 350°C. As described by Buschow et al. [17] and reported by Liu et al. [18] grains begin to recrystallize at initial grain boundaries in hot extrusion growing in layers until the old grains are consumed. DRX is thought to initiate at original (prior to extrusion) grain boundaries due to an accumulation of dislocations at the grain boundaries during the hot extrusion [19]. Optical micrographs in the longitudinal (extruded) direction of extruded Mg and extruded Mg-1%SiCnp tested at 25°C, 125°C, and 177°C, are shown in Figure 2. Elongated nanoceramic agglomerations are shown in Figures 2b, 2d, and 2f where the SiC nanoparticles are distributed in the extruded Mg matrix.

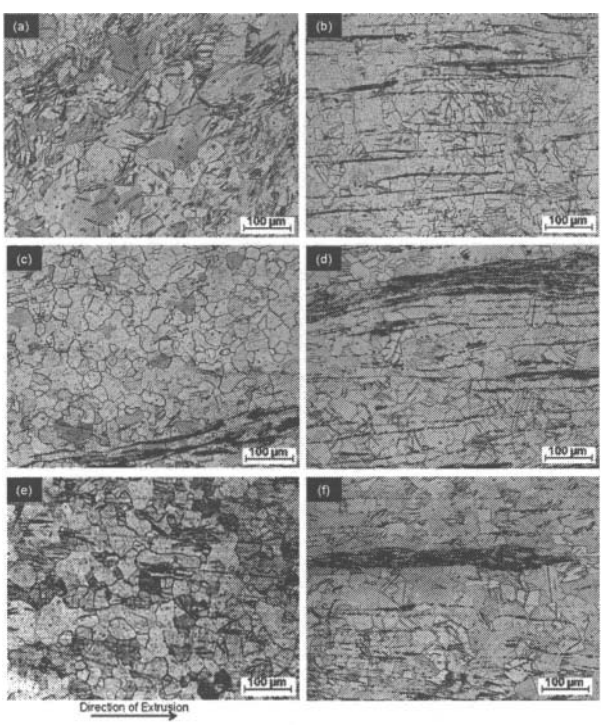

Figure 2. Optical micrographs in longitudinal (extruded) direction of extruded specimens: a) Mg tested at 25°C, b) Mg-1%SiC tested at 25°C, c) Mg tested at 125°C, d) Mg-1%SiC tested at 125°C, e) Mg tested at 177°C, f) Mg-1%SiC tested at 177°C.

The SiC nanoparticle-bands in Figures 2b, 2d, and 2f are not the only regions that SiCnp are located in; however, larger concentrations and clusters of SiC nanoparticles form these bands. The most distinguishing characteristic between the extruded Mg samples (Fig. 2a, c, e) and the extruded Mg-1%SiC (Fig.2b, d, f) are the dark SiC bands running parallel to the axis of extrusion. Such observation are similar to the described by Wang et al. [19] that observed similar phenomena in pure Mg with 40 μm sized SiC particles that were extruded at a ratio of 13:1. Streaks of SiC micro particles were observed parallel to the direction of extrusion; however, there were no clusters of SiC micro particles and the particles were bonded well to the matrix [8].

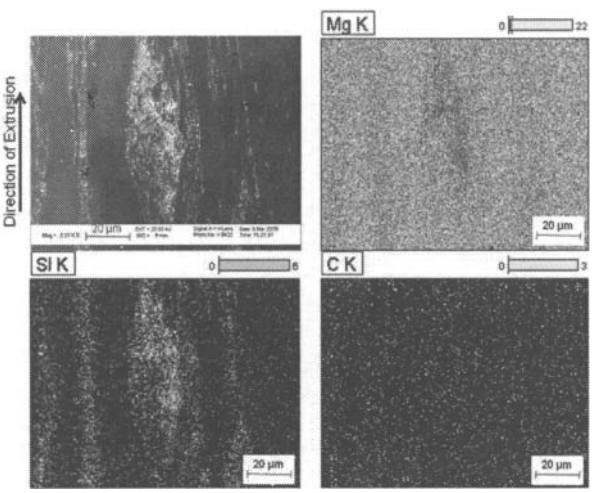

Figure 3. a) SEM image of a Mg-1%SiCnp nanocomposite of a sample after testing at 177°C, illustrating SiC band formation parallel to the axis of extrusion and corresponding EDS images b) Mg K c) Si K and d) C K.

Similar bands have also been observed in extruded Mg alloys with intermetallics that have been destroyed and dispersed into bands during the extrusion process [19]. The SEM image in Figure 3 is from a Mg-1%SiCnp nanocomposite sample tested at 177°C. Figure 3a exemplifies a SiCnp micro-band formed parallel to the axis of extrusion. Energy Dispersive X-ray Spectroscopy (EDS) images confirm that the micro bands observed in Figures 2b, 2d, and 2f are in fact elongated SiC nanoparticle agglomerations. EDS spectral imaging in Figure 3c shows the SiC particle bands in an Mg matrix in the longitudinal direction, while in contrast Mg spectral image (Figure 3b) shows its complement spectra.

Besides of the recrystallization process, the addition of 1%SiC nanoparticles enhanced yield strength, tensile strength, and ductility. At the tested temperatures below and higher recrystallization temperature the Mg-1%SiCnp nanocomposite preserves its uniform distribution, as seen in the SEM image in Figure 4 also showing the SiC nanoparticle agglomerations contouring the extrusion flow. The observed nanoparticle dispersion and mechanical properties' variation indicates a thermally stable bond between the reinforcing nanoparticles throughout the matrix. The observed increase in ductility with the addition of 1%SiC nanoparticles is in contrast to the described results shown in Cao et al, where the ductility decreases with the addition of micron-sized particles and fibers to Mg matrix [7]. Mg/SiCnp nanocomposite results are in concordance to the

characterization reported by Cao et al. Cao et al [7] also described the characterization of the Mg/SiC interface behavior where is shown no intermediate phases at the interface of Mg and SiC, and found that SiC nanoparticles were well bonded to the matrix. Same authors also report that nanoparticles aid in maintaining the grain structure in the extruded Mg-1%SiC by pinning grain boundaries at elevated temperatures thus resulting in higher ductility which explain in part the effect of the SiC nanoparticles in the Mg matrix. Explaining why (partially at least) even though clusters of nanoparticles exist in the Mg-1%SiCnp samples and why the tensile tests revealed an increase in ductility at all tested temperatures.

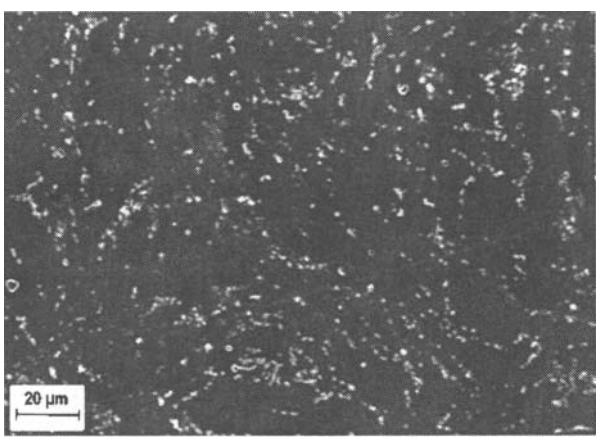

Figure 4. SEM of the SiC nanoparticle dispersion in Mg-1%SiCnp after tested at 177°C, illustrating the achieved dispersion of SiC agglomerations in transverse (extruded) direction

Conclusions

The incorporation of SiC nanoparticles by using ultrasonic cavitation based dispersion processing help to achieve a uniform distribution of reinforcing SiC nanoparticles in the Mg matrices. As an effect of the extrusion process, MMNC billets contained micro bands of SiC nanoparticles oriented parallel to the direction of extrusion evidencing the SiC nanoparticles dispersion. Along the cross section or transverse to the direction of extrusion the achieved dispersion of SiC agglomerations shows to be homogeneous. Micrographs show no significant grain refinement between the Mg and Mg-1%SiC MMNC samples after hot extrusion. In contrast to previous reports using SiC micron-sized particles or fibers, samples from Mg-1%SiCnp consistently exhibit larger ductility and enhancement of other mechanical properties. Yield strength, tensile strength, ductility and microhardness were improved also as compared to the extruded Mg samples at various testing temperatures.

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