MICROSTRUCTURE AND PROPERTIES OF AZ91D/CNF METAL MATRIX COMPOSITE

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Abstract. *Magnesium alloys are potential candidates to replace heavier materials in automobile and airplane parts due to their high strength-weight ratio. However, further utilization of those alloys is hinded by insufficient mechanical properties in such requiring applications. In the present work, carbon nano-fibers (CNF) surface-coated by silicon carbide were used to reinforce AZ91D magnesium alloy in order to enhance its mechanical and tribological properties. The AZ91D/CNF composites were produced by mechanical stirring compocasting followed by ultrasonic agitation to include and disperse reinforcement into the matrix. Composites obtained were also post-processed by high pressure thixodie-casting (HPTD), thixoinjection and hot extrusion. Microstructures of the composites were observed by optical and scanning electron microscopy, while their properties were evaluated by hardness and tensile tests. Refined primary phase and adequate dispersion of CNF was obtained due to intense turbulence and energy input promoted by ultrasonic agitation, although primary phase coarsening could be observed after HPTD. Vickers tests indicated significant improved hardness (20%) and ultimate tensile strength (40%) for composites containing 3 volume percentage of silicon coated CNF in comparison to unreinforced AZ91D alloy after HPTD. Extruded composites did not present significant enhancement in comparison to the unreinforced alloy, although values for ultimate tensile strength were higher than those obtained in high pressure thixodie-cast and thixoinjected samples.*

Keywords*: AZ91D magnesium alloy, metal matrix composite, carbon nano-fiber, thixoforming, extrusion.*

1. INTRODUCTION

Magnesium alloys have recently attracted significant interest of researchers due to their high strength-weight ratio, which makes them potential options for replacing heavier materials in some automobile parts (Mordike and Ebert, 2001) and in structural parts in electronic devices, such mobile phone external parts (Kojima and Kamado, 2005). It is also expected that magnesium will play an important role in lightweight construction in the near future (Jaschik et al., 2000), due not only to its reduced weight, but also because it can be found in abundance in Earth's crust. However, further utilization of Mg alloys is limited by its mechanical properties, which have high specific values but are insufficient at more requiring applications.

Among different new materials based on Mg alloys, metal matrix composites (MMC) reinforced with ceramic materials have appeared as a good options, since addition of ceramic short-fibers or particulates to magnesium alloys are reported in literature to have improved significantly their mechanical and tribological properties (Fritze, 2003; Moll and Kainer, 2003; Lan et al., 2004). Considering such properties to be strongly dependent on various parameters, such as quantity, size and properties of the reinforcement, utilization of carbon nano-tubes (CNT) has great potential, since this recently discovered allotrope of carbon which are predicted to have Young modulus values in the order of 0.8TPa (Salvetat et al., 1999), with tensile strength around 63GPa (Yu et al., 2000), several times higher than graphite fibers (Callister, 1997), although there is no reported macro application of nano-fibers with such properties. However, further utilization of carbon nano-fibers in metal matrix composites is hindered by the difficulties faced when forcing them to disperse into liquid metals. Aside from agglomerating due to poor wettability with some metals, even when properly distributed, CNF must be properly wetted by matrix to prevent poor cohesion with the composite.

Although data concerning wettability between carbon nano fibers and AZ91D is scarce, some values of contact angles between graphite, which has similar structure to CNF, could be found. According to Shi (2000), the contact angle between pure magnesium and porous graphite is about 74^{o}, indicating as expected good wettability of carbon materials with magnesium. However, existence of aluminum and zinc in the composition of AZ91D alloy can increase significantly the contact angle, considering that the wettability between graphite and aluminum is about 155^{o} at 700^{o}C (Ejiofor and Reddy, 1997). A method to improve the wettability between insoluble particle-liquid mixtures that has been used in previous works was the application of ultrasonic vibration to induce cavitation in the liquid metal containing particulate reinforcement in suspension. The cavitation phenomenon consists of forcing microscopic gas bubbles dispersed in a liquid to oscillate in size until eventually collapsing to a small fraction of its original size, at

which point the entrapped gas dissipates into the surrounding liquid via a rather violent mechanism, releasing a significant amount of energy in the form of an acoustic shock-wave. In the case of a metal slurry containing suspended particles of reinforcement, the particles act as a point where the initiation of cavitation is benefited by the energy concentrated on their interface with the matrix, enhancing the wetting between both materials. This phenomenon was used by Genma et al. (1997) to obtain homogeneous dispersion of Al_2O_3 particles in Al-Mg alloys, while Yang et al. (2004) used the same process to homogeneously disperse 2 mass percentage of SiC nanoparticles in molten Al-7 mass percentage of Si alloys.

Another way to improve the wetting of reinforcement in metals is coating its surface with a more compatible material with the matrix, like in the case of graphite fibers coated by Si to wet magnesium alloys, reported to produce better tensile properties than uncoated fibers (Chin and Nunes, 1988), while SiC-coated fillers were reported to have better wettability than uncoated graphite in aluminum alloys containing high weight percentage of Mg (59^o) at 750^{o}C (Candan, 2002). In the present work, mechanical stirring compocasting followed by ultrasonic agitation was used to disperse Si surface-coated carbon nano-fibers into AZ91D magnesium alloy in order to enhance its mechanical and tribological properties. Composites obtained were also post-processed by thixoforming and extrusion.

2. EXPERIMENTAL PROCEDURES

2.1. Materials

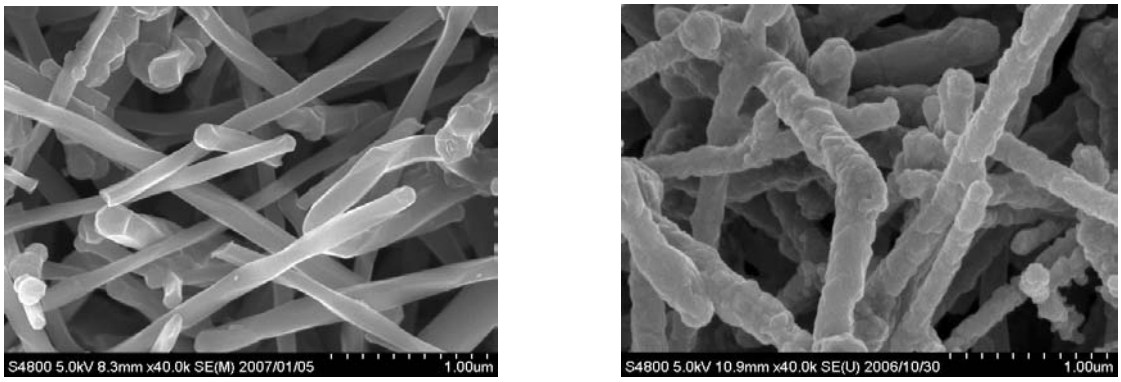
The material used as matrix in the present work was a commercial AZ91D magnesium alloy, which is a cast alloy with wide melting temperature interval (approximately 175^{o}C), having also good castability, corrosion resistance and mechanical properties. It is a commercial alloy used mainly in die-cast parts, being readily available at suitable costs in the market. The nominal chemical composition specifications for this alloy and some of its nominal properties are shown in Tab. 1 (ASM, 1993).

Table 1. Nominal chemical composition, physical and mechanical properties of AZ91D magnesium alloy.

Element	Range (wt %)	Properties*	Values
Al	8.3-9.7	Density	1.8 g/cm^3
Zn	0.35-1.9	Melting Interval	429^{o}C -595^{o}C
Mn	0.15min.	Thermal Conductivity	72 W/m.K
Si	0.10max.	Tensile Strength	230 MPa
Cu	0.030max.	Tensile Yield Strength	160 MPa
Ni	0.002max.	Elongation	3%
Fe	0.005max.	Hardness	70 HV
Mg	balance		

*Properties of Cast AZ91D-F magnesium alloy.

The material used to reinforce AZ91D was Carbon Nano-Fibers (CNF), which are extremely thin fibers 15μm long and 150nm in diameter, with real density of 2.0g/cm^3. The CNF used in this work were produced by Showa Denko Tokyo Co. Ltd. (Tokyo, Japan) denominated as Vapor Grown Carbon Fiber (VGCF-H), similar to multi-walled carbon nano-tubes. A thin coating of silicon was deposited on the surface of CNF and after annealing turned into a SiC coating, resulting in reduction of the contact angle of the fibers with AZ91 from 155^o to 42^o. The CNF coated with SiC will be denominated hereinafter as CNF_{Coat}, while as-received CNF will be referred to as CNF. Both CNF and CNF_{Coat} are shown in Fig.1.



(a) (b)

Figure 1. Carbon nano fibers observed by SEM (a) before and (b) after receiving Si coating.

2.2. Production of Composites

AZ91D/CNF composite materials were produced by a compocasting method modified with an inclined cooling plate, which has been utilized successfully by other authors (Motegi et al., 2003; Tanabe et al., 2003; Yano et al., 2002) in the production of semi-solid alloys, including AZ91D. The method consisted of melting AZ91D magnesium alloy and when it reached 605°C, a bath level controller was introduced into the molten metal at constant speed of 2mm/s. The level of the bath rose and the liquid metal was forced into the pouring tube, flowing down it onto a water-cooled plate with 160mm in length and inclination of 60° placed at its end. This method produced magnesium alloy slurry with primary solidification nuclei, which was collected in a metallic mould kept in the tundish at constant temperature of 595°C. Mechanical stirring was started 10 seconds after pouring, in order to allow the solidification nuclei generated on the cooling plate to grow and stabilize in the tundish. While the slurry was agitated, 1, 2 and 3 volume percentage of CNF were added from its surface going into the semi-solid metal through the vortex generated around the paddle (edged turbine).

After the reinforcement material was completely poured into the slurry, stirring proceeded for 20min, at constant stirring speed of 1750rpm. Argon gas was used to protect the Mg alloy from oxidizing or burning. The temperatures of the furnaces, bath and tundish were monitored by K-type thermocouples. The temperature of the slurry during stirring varied from 588 to 592°C, being monitored at uniform intervals along the experiment, since keeping the thermocouple constantly inside the mould would bring interference to the flow of the metal. At the end of mechanical stirring process, the paddle, still in movement, was extracted from the tundish, which was immediately followed by ultrasonic agitation.

The ultrasonic agitation equipment was placed above the tundish furnace and the pre-heated probe was lowered in the center of the mold containing the slurry until its tip had sunk 10mm in the slurry. Ultrasonic waves of 20μm amplitude were applied during 210sec., after which the process was stopped, the probe raised and the mold containing the slurry rapidly cooled in water, freezing the microstructure obtained at the end of the process, i.e., avoiding the possibility of microstructure coarsening. The tip of the probes were previously heated to about 600°C with the aid of a gas (propane) burner before being introduced in the bath to prevent metal near the surface from solidifying and sticking to the probe. A schematic representation of the equipment utilized and the modified compocasting process is illustrated in Fig. 2 (Mussi et al., 2006).

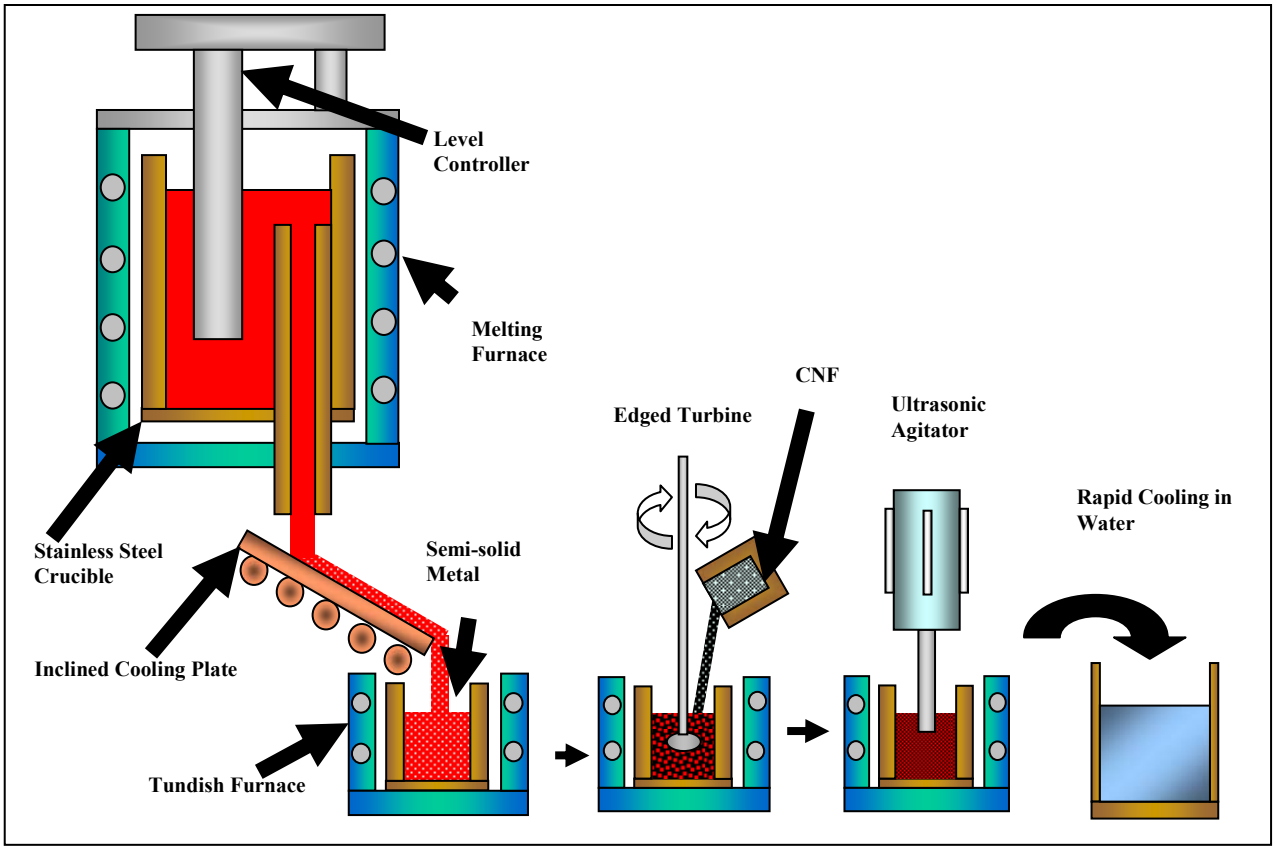


Figure 2. Schematic illustration of the compocasting using an inclined cooling plate followed by ultrasonic agitation.

2.3. Post-Processing

Composites containing 1, 2 and 3 volume percentage CNF_{Coat} produced by compocasting and ultrasonic agitation were separately processed by high pressure thixocasting, thixoinjection and hot extrusion. AZ91D alloy produced by mechanical stirring and ultrasonic agitation, as well as commercial rheocast AZ91D billets with globular primary phase, called MagBall, were also post-processed at the same conditions to serve as comparison samples.

High pressure thixocasting experiments were performed in a press adequate for pressure casting with maximum capacity of 200MPa. In order to fit the mould, composites produced by mechanical stirring and ultrasonic agitation as well as comparison samples were machined to cylinders measuring Ø51 x 60mm, which were placed in a steel cylinder and heated to 585°C in an arc image furnace, protected by artificial atmosphere consisting of a mixture of SF_6 and CO_2. Heating process was divided in two parts: eight minutes pre-heating of the sample from room temperature to 450°C, at which it was kept for 30 seconds to homogenize the temperature through all the material; subsequent 11 minutes to heat it to 585°C that is maintained for 5 minutes before the sample was placed inside the mould at 300°C. The press was then activated at 200MPa and high pressure thixocasting was performed producing samples with Ø60 x 45mm.

Thixoinjection experiments were performed in an injection machine denominated FMg7000-85X adapted to perform injection of semi-solid magnesium alloy, comprising a preheating resistance furnace where the sample material previously machined to a billet measuring Ø60 x 100 mm was placed and pre-heated to 400°C, and a melting chamber where the sample was transferred to be heated and kept for 20 minutes at 585°C. In this case, the temperature of the melting recipient was measured, since the temperature of the sample could not be directly measured due to the configuration of the equipment, but 20 minutes holding time should suffice to homogenize the temperature through the whole sample. The semi-solid sample was then injected into a metallic mould preheated to 280°C at a rate of 1004.8ml/s and pressure of 100MPa. The speed of the injector arm was 200mm/s and the mass injected in each shot was 500g. The mixture of SF_6 and CO_2 was again used to avoid oxidation and combustion of the material.

Hot extrusion experiments were performed using billets with Ø60 x 20 mm extracted from the samples obtained in high pressure thixocasting experiments. The temperature of extrusion (mould temperature) was 350°C, and the section area of the sample was reduced in a ratio of 25:1 in relation to the initial billet. The speed of extrusion utilized was 4mm/s and the rod produced measured about Ø12 x 500 mm.

2.4. Characterization of Materials

2.4.1. Microstructural Analysis

Samples for optical and scanning electronic microscopy were extracted from the center of the ingots obtained at each experiment. The samples were fixed in thermoplastic resin and superficially ground using 1200 grit paper. After that, they were polished with 1μm and 0.06μm alumina buffer, and subsequently etched for 20 seconds with 2 mass percentage oxalic acid ($H_2C_2O_4*2H_2O$) diluted in water to allow microstructure observation. Scanning electron microscopy (SEM) was also performed on fractured surface of samples after tensile tests.

2.4.2. Hardness Tests

Samples for hardness tests were extracted from the center of the ingots obtained at each experiment. After being fixed in thermoplastic resin, the samples were superficially ground using 1200 grit paper, polished with 1μm alumina buffer to avoid noise in the results due to roughness. Vickers hardness tests were carried out in a flexible hardness tester machine with constant load of 20kgf applied for 15 seconds. For each sample 10 marks were made being the average of results considered as representative of the hardness value of that sample. The imprinted marks were homogeneously distributed over the surface of the sample and adequately separated from each other to avoid interference of eventual hardening on the surrounding and imprinted point in the following measurement.

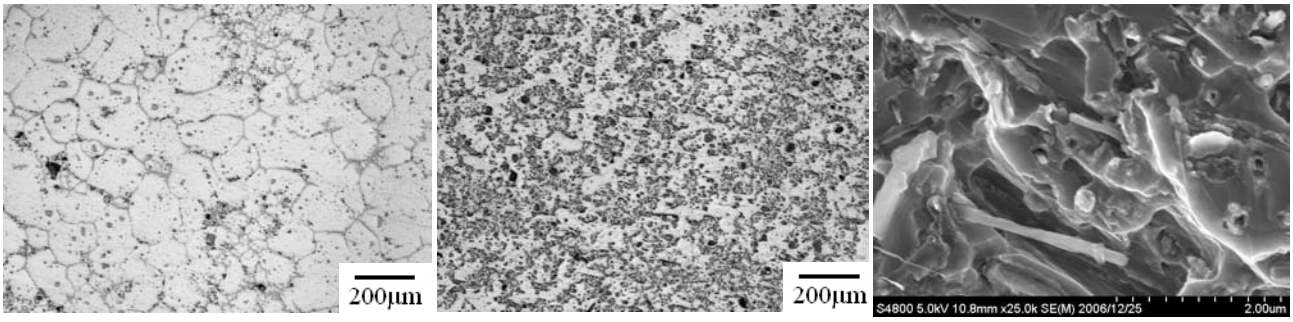
2.4.3. Tensile Tests

Tensile test pieces were machined from the samples obtained after each of the post-processes. The tests were performed at room temperature, using a universal testing machine with a crosshead speed of 1mm/min. Two ingots were produced at each condition, being four test pieces extracted from each ingot, totalizing 8 test pieces for each condition. The procedures are in accordance to Japanese Industrial Standards referred as JIS Z 2201 (1998).

3. RESULTS AND DISCUSSIONS

3.1. Microstructures

As it can be observed in Fig. 3, microstructure of high pressure thixodie-cast AZ91D reinforced with 3 volume percentage CNF_{Coat} consisted of finer primary phase α (lighter phase in the pictures) than in the unreinforced material. The size of α phase particles decreased with increase in quantities of filler, which also produced less globular primary phase in the composite materials. According to previous work (Mussi, 2007), CNF_{Coat} was dispersed exclusively in the secondary phase of the composite material, which seems to have acted as a barrier to coalescence of globules during its initial process of reheating and its final solidification. Besides, it promoted further refinement during process of HPTD, since turbulent flow of semi-solid slurry into the mold reinforces interactions among the secondary phase consisting of liquid and solid fibers with solid primary globules, which could cause their rupture and dispersion into the material. Therefore, the existence of filler in semi-solid AZ91D allowed the material to keep the refined microstructure produced during ultrasonic agitation (Mussi et al., 2006) before being pushed into the mold, and avoided coarsening of primary globules during solidification inside the mold.

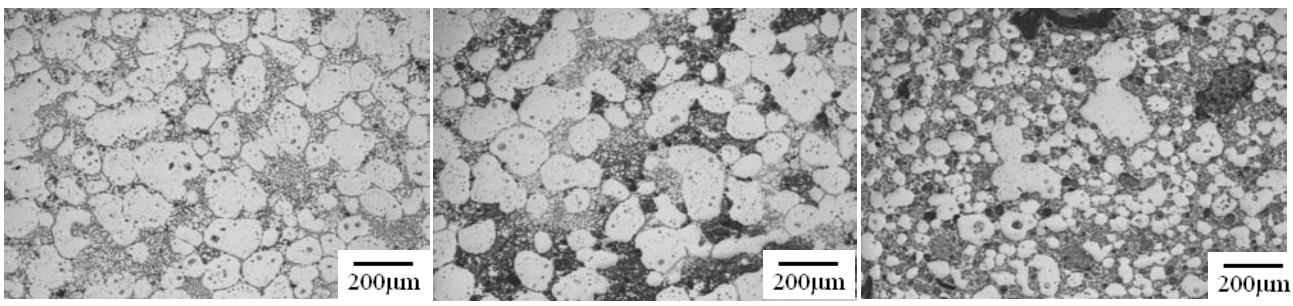


AZ91D + 0vol% CNF_{Coat} **AZ91D + 3vol% CNF_{Coat}**

Figure 3 – Microstructures of unreinforced AZ91D and AZ91D/3 volume percentage CNF_{Coat} pressure thixodie-cast and the SEM photos of fractured surface of the later.

In the SEM of Fig. 3 showing a fractured surface of AZ91D/3 volume percentage CNF_{Coat} post-processed by HPTD, the holes from where fibers were pulled can be seen, with a layer of as-brittle material around their internal cavity in contact with the matrix. This layer is probably the SiC coating that remained attached to the matrix after the fiber was pulled, meaning poor cohesion between fiber and coating, at least in part of the interface. In the cases when fibers remained attached to the matrix, some torn or thinned fibers could be found in the same area, meaning that they possibly had their properties damaged at some point of the coating process or during dispersion into the magnesium alloy. In both cases, interaction of coating with matrix appeared to be adequate, being evident the necessity for improvement and homogenization of fiber-coating interface properties. Another important factor that must be noticed in the same Fig.3 is the random orientation of the CNF_{Coat} on the fractured surface, i.e., the non-alignment of fibers during the HPTD.

The microstructures of thixoinjected samples are shown in Fig. 4. In this case, despite the presence of some coarser primary globules probably resulting of coalescence occurring during the process, the microstructure of thixoinjected composites presented similar behavior to that of high pressure thixodie-cast samples, with increase in refinement with increased quantity of filler. However, the shape of primary α in this case is closer to globular, due to lower pressure and filling speed, producing, respectively, less turbulence in the slurry during process and giving more



AZ91D + 0vol% CNF_{Coat} **AZ91D + 2vol% CNF_{Coat}** **AZ91D + 3vol% CNF_{Coat}**

Figure 4 – Microstructures of unreinforced AZ91D and AZ91D/2 and 3 volume percentage of CNF_{Coat} thixoinjected.

time to the globules to grow and coalesce. Moreover, unreinforced material showed distinct distribution of primary and secondary phases compared to HPTD samples, with larger quantity of second phase separating primary globules, due to differences in reheating methods and to the different re-heating periods utilized in each process, which could have caused further remelting of primary phase. The distribution and orientation of CNF_{Coat} were similar to those of HPTD samples.

Microstructure of extruded samples consisted of two distinct layers alternately disposed parallel to the direction of extrusion, as shown in Fig. 5. The lighter phase in the picture is a refined primary phase that suffered recrystallization during warm deformation, with grain size slightly decreasing with larger quantity of filler, varying from an average 18µm for unreinforced AZ91D to 13µm for 3 volume percentage of CNF_{Coat}, which suggests that the presence of fibers dispersed in the secondary phase avoided again coalescence during extrusion, reinforcing the deformation of grains recrystallized in finer new grains. The dark layers seen in microstructures of Fig. 5 are the secondary phase containing also, in the case of composites, the filler dispersed. The quantity of these layers increased with larger addition of fibers, reducing the thickness of layers of α phase. SEM of fractured surface of extruded AZ91D/CNF_{Coat} shows alignment of fibers obtained after processing. Although tearing of fibers could still be noticed, the number of empty holes decreased in comparison to HPTD.

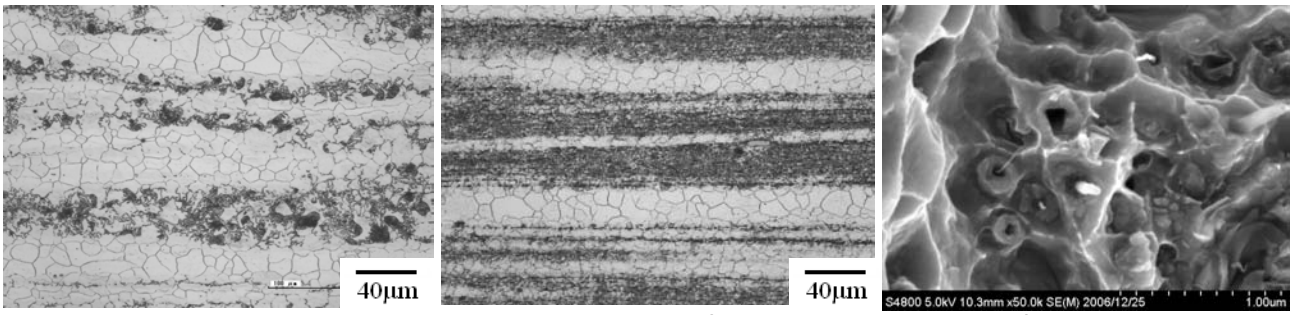
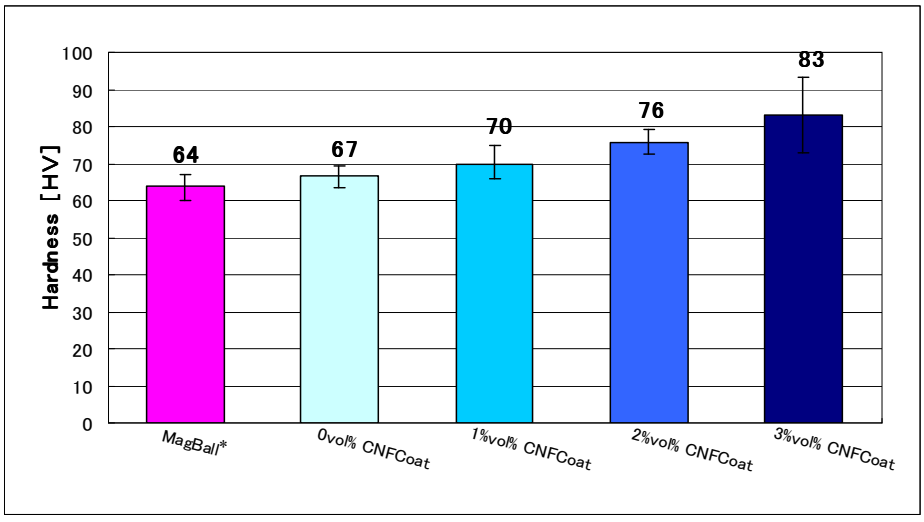


Figure 5 – Microstructures of unreinforced AZ91D and AZ91D/3 volume percentage of CNF_{Coat} extruded and SEM photo of fractured surface of the later.

3.2. Hardness

Average Vickers hardness of AZ91D reinforced with CNF_{Coat} presented similar behavior for all post-processes utilized, showing increased hardness with increase in quantity of filler. For samples produced by high pressure thixodie-casting, for example, the influence of the reinforcement on the hardness of the composites became clear, as shown in Fig. 6. This is explained by the increasing refinement of primary globules obtained for composites and by the presence of harder filler (SiC coating) adequately wetted and distributed into the AZ91D matrix, combined with the reduced porosity of the material after being high pressure thixocast, which improved also the hardness of unreinforced AZ91D compared to values obtained for as-cast AZ91D without post-processing, of approximately 61HV (Mussi, 2007).



*Commercial AZ91D with globular microstructure.

Figure 6 – Vickers hardness of MagBall, ultrasonically agitated AZ91D and AZ91D containing 1, 2 and 3 volume percentage of CNF_{Coat} post-processed by high pressure thixodie-casting.

Composites added with 1 volume percentage of CNF_{Coat} reached the nominal values for hardness of commercial AZ91D (ASM, 1993), while larger quantities of filler resulted in average increase of up to 13HV which means a 20% improvement compared to the original alloy. Larger values for Vickers hardness were obtained in extruded samples, ranging from average 78HV in unreinforced AZ91D to over 110HV for 3 volume percentage of CNF_{Coat}.

3.3. Tensile Strength

The results of tensile tests performed on samples of post-processed AZ91D/ CNF_{Coat} are shown in Fig. 7, where bars represent average UTS and yellow lines represent the average elongation, in relation to which 5% variation must be considered. Composites produced with 3 volume percentage of CNF_{Coat} and post-processed by HPTD had their UTS improved up to 50MPa compared to unreinforced material similarly processed, representing an increase of approximately 40% in UTS due to addition of 3 volume percentage of CNF_{Coat}, without any significant decrease in elongation. Similar behavior was observed in UTS and elongation values measured for thixoinjected samples. The strengthening obtained in the composite materials can be explained primarily by their refined microstructure, allied to the presence of resistant SiC-coated nano-fibers adequately dispersed and wetted by AZ91D matrix, working as a barrier to diffusion and movement of dislocations, hindering consequently deformation of the material during tensile stress. Although fibers were found to be pulled out or torn apart on the fractured surface of tensioned samples, SiC coating remaining attached to the matrix could suffice to raise the stress necessary to deform the material.

Thixoinjected and high pressure thixodie-cast composites reinforced with CNF_{Coat} had their Young`s modulus increased of about 5GPa compared to unreinforced samples, varying from 25GPa for 0 volume percentage of CNF_{Coat} to 30GPa in the case of samples containing 3 volume percentage of CNF_{Coat}, proving the gain in resistance against deformation with the addition of these carbon nano-fibers. The shape of primary phase α allied to the presence of some impurity and agglomerations of filler in the secondary phase of thixoinjected samples seems to be the cause of the lower elongation obtained for these samples.

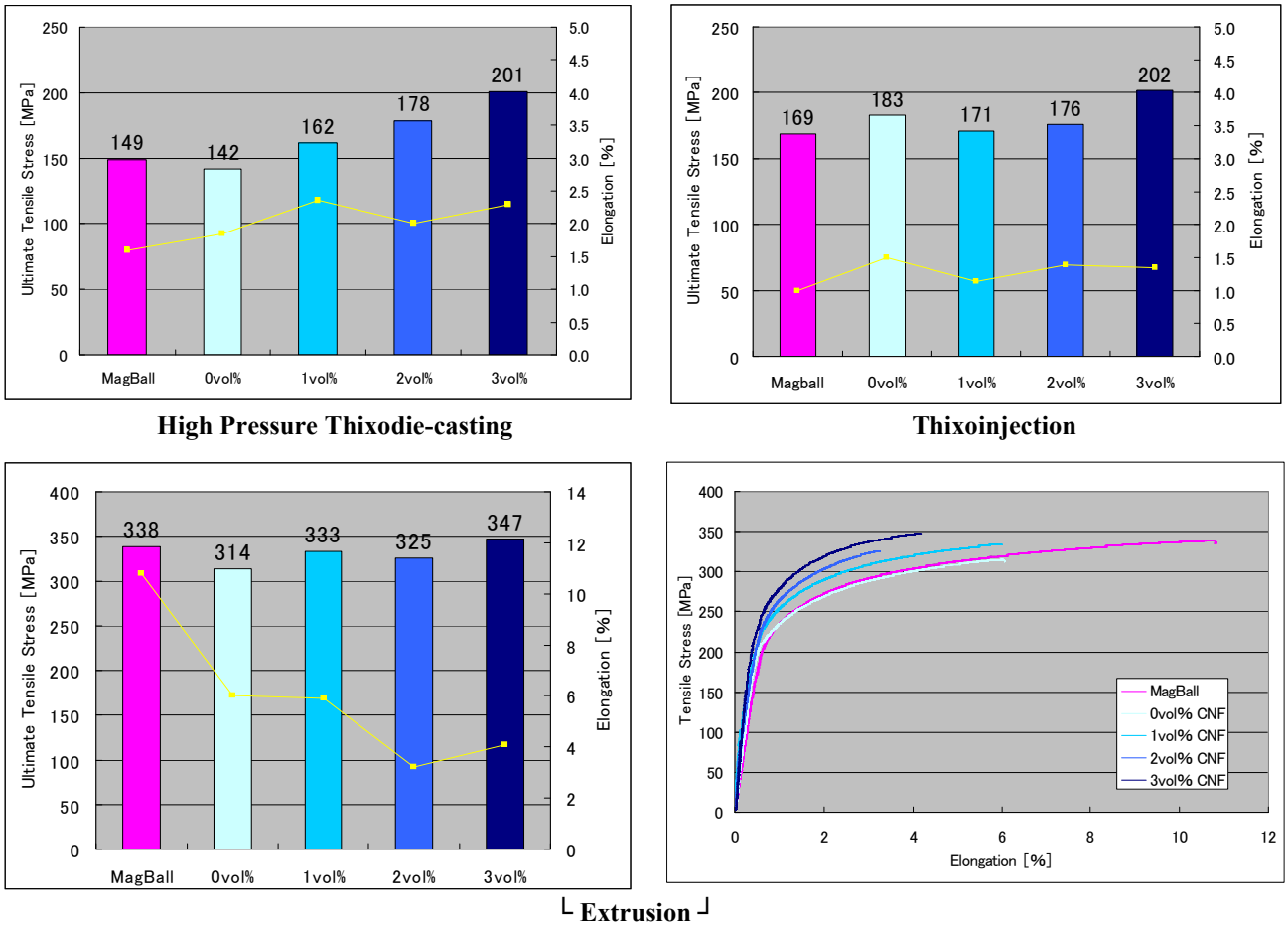


Figure 7 – UTS and elongation of AZ91D and AZ91D/CNF_{Coat} produced by modified compocasting with ultrasonic agitation, post-processed by: HPTD, thixoinjection and extrusion, with bars representing average UTS and yellow lines the elongation; a representative sample of curves obtained from tensile tests for extruded samples.

For extruded samples, the presence of CNF_{Coat} does not seem to have significantly influenced the UTS of the samples, although AZ91D/3 volume percentage of CNF_{Coat} presented slightly larger values. Since recrystallization of grains occurred during extrusion, the size of primary grains in unreinforced samples became similar to those of composites, resulting in similar resistance against tensile stress. Considering that recrystallization played the main role in strengthening of extruded samples, the presence of larger quantity of hard coated fibers arranged in strips parallel to extrusion direction, isolated the primary phase grains in narrow strips as well, accelerating the recrystallization inside each strip. This, nonetheless, resulted in significant decrease of elongation in the case of composites compared with unreinforced samples, since recrystallization process reached its limit faster in composites. Values obtained of Young`s modulus reinforce this theory, as an increase of up to17GPa could be measured for AZ91D containing 3 volume percentage of CNF_{Coat}, varying from 38GPa to 55GPa for unreinforced and 3 volume percentage of reinforced sample, respectively, despite their similar UTS. This means that the addition and alignment of coated carbon nano-fibers by extrusion improved the stiffness of the material, although it became brittle compared to the original alloy. It can be observed by the behavior of stress/strain representative curves of Fig. 7 that the composites resisted significantly more to deformation, reaching the best UTS and Young`s modulus for 3 volume percentage of CNF_{Coat}.

4. CONCLUSIONS

It can be concluded from the present work that:

1. Utilization of a SiC coating on the surface of CNF allied to high pressure thixodie-casting, thixoinjection or extrusion as post-processes of AZ91D/CNF_{Coat} composites produced by modified compocasting with ultrasonic agitation improved the wetting and dispersion of the filler into the matrix;

2. The presence of CNF_{Coat} in the secondary phase of the matrix alloy hindered phenomena of coalescence and coarsening from happening, resulting in more refined primary phase α for larger amounts of filler for HPTD, thixoinjected and extrusion samples, being in the later, arranged in the form of narrow strips oriented parallel to the direction of extrusion, alternating with strips containing primary grains;

3. Vickers hardness increased with larger quantities of reinforcement, reaching up to 15HV increase for HPTD and thixoinjection, and over 20HV increase for extruded samples containing 3 volume percentage of CNF_{Coat};

4. UTS of AZ91D alloy was increased in approximately 50MPa by reinforcing it with 3 volume percentage of CNF_{Coat}, without significant decrease in elongation, besides obtaining increase in Young`s modulus in about 5GPa compared to unreinforced material for HPTD and thixoinjected samples, mainly due to refined microstructure;

5. In extrusion post-processing recrystallization seems to be the most important mechanism in the determination of final mechanical properties of the samples. The presence of larger quantities of CNF_{Coat} seems to accelerate the process to reach the limit of recrystallization, resulting in slightly larger UTS and significant increase in Young`s modulus (17GPa) of composites reinforced with 3 volume percentage of CNF_{Coat} in detriment of elongation.

5. ACKNOWLEDGEMENTS

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