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Microstructural and mechanical properties of nanometric magnesium oxide particulate-reinforced aluminum matrix composites produced by powder metallurgy method †

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Abstract

In this research, aluminum alloy (A356.1) matrix composites reinforced with 1.5, 2.5 and 5 Vol.% nanoscale MgO particles were fabricated via powder metallurgy method. Pure atomized aluminum powder with an average particle size of 1μm and MgO particulate with an average particle size between 60 to 80 nm were used. The specimens were pressed by Cold Isostatic Press machine (CIP), and were subsequently sintered at various sintering temperatures, viz. 575, 600 and 625°C. Optimum amount of reinforcement and sintering temperature were determined by evaluating the density, microstructure and mechanical properties of composites. The composites were characterized by scanning electron microscopy (SEM) and X-ray diffraction (XRD). Hardness and compression tests were carried out in order to identify mechanical properties. Reinforcing the Al matrix alloy with MgO particles improved the hardness and compressive strength of the alloy to a maximum value of 44 BHN and 288 MPa, respectively. The most improved compressive strength was obtained with the specimen including 2.5% of MgO sintered at 625°C. According to the experiments, a sintering temperature of 625°C showed better results than other temperatures. A good distribution of the dispersed MgO particulates in the matrix alloy was achieved.

Keywords: Al-matrix composite; Mechanical properties; Nanoscale MgO; Powder metallurgy; Reinforcement

1. Introduction

Discontinuously reinforced aluminum composites are being recognized as an important class of engineering materials that are making significant progress. The reasons for their success are related to their desirable properties including low density, high hardness, high compressive strength, wear resistance, etc. Casting and powder metallurgy are the two major fabrication methods of aluminum matrix composites, though powder metallurgy is more complicated than casting, it yields a better interface between the reinforcement and matrix alloy, improving mechanical properties of the composite [1].

Nowadays, demands for developing metal matrix composites for use in high performance applications have been significantly increased. Aluminum alloy matrix composites attract much attention due to their lightness, high thermal conductivity, moderate melting point, etc. [1, 2]. Among these composites, metal matrix nanocomposites are a new class of nanostructured materials, consisting of nano-scale particles used as reinforcements. It is of interest to use nano-sized ceramic particles to strengthen the metal matrix, while maintaining good ductility, high temperature creep resistance and better fatigue. However, the ductility of the MMCs deteriorates significantly with high ceramic particle concentration [3, 4]. Various kinds of ceramic materials, e.g. SiC, Al_2O_3, ZrO_2 and B_4C, are extensively used to reinforce aluminum alloy matrixes. Superior properties of these materials such as refractoriness, high hardness, high compressive strength, wear resistance, etc. make them suitable for use as reinforcement in a matrix of composites [5, 6]. Nevertheless, low wettability with molten metals and density differences increase their tendency toward agglomeration, which deteriorate mechanical properties [7].

Magnesium oxide (MgO) is a refractory material with a melting point of about 2780°C. MgO possesses properties such as good thermal shock resistance, high melting point, low thermal conductivity, and excellent thermodynamic stability. Its density, Young's modulus, and hardness are 3.58 g/cm^3, 320 GPa, and e910 HV, respectively [8]. There are different methods for the fabrication of aluminum matrix composites which are divided into three major categories: (a) smelting processing, (b) semi-smelting processing, and (c) solid state processing; the last one has been divided into two methods:

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Table 1. Chemical composition of A356.1 alloy.

Element	Al	Si	Fe	Cu	Mg	Mn	Zn	Ni
Mass %	91.73	7.23	0.32	0.18	0.38	0.02	0.05	0.05

(1) mechanical alloying and (2) powder metallurgy [9]. The sintering process in powder metallurgy method including sintering of nano-ceramic particles within metals matrix in comparison with other methods, such as casting method, has some important advantages, including better bonding between particles and matrix alloy and being easier to control the matrix structure. However, this method has some problems such as high complexity and expense [10]. In this study, nano MgO particles were used to reinforce A356.1 alloy by powder metallurgy method. Also, the optimum sintering temperature and reinforcement content were determined by analyzing its microstructure and mechanical properties.

2. Experimental procedures

Aluminum powder alloy (A356.1 with $D_{50}=1$ μm) and nano-sized MgO (with $D_{50}=70$ nm) were used for fabrication of composites. Chemical composition of A356.1 is shown in Table 1. After weighting the reinforcement and matrix powders and before preparing the composite samples, powders were mixed completely. First, powders were put into a mixer and mixing operation was performed for 30 minutes in Alcohol-Ethanol environment. After mixing, powders were completely dried by dryers. To evaluate the amount of reinforcement and the effect of sintering temperature on the properties, composites with 1.5, 2.5 and 5 volume percent of magnesium oxide were prepared. Then, samples were prepared by Appling 200 MPa load for CIP. Raw composite samples were put into an atmosphere control furnace and were sintered at different temperatures (575, 600, and 625°C) for 1 hour. Finally, cylindrical samples with 14 mm height and 10 mm diameter were prepared. The bulk density of the samples was determined by the Archimedes method. The phase analysis of the samples was investigated by XRD using Philips, PW-1800 diffractometer with Cu-k_α radiation. The morphology of the samples was analyzed by scanning electron microscope (SEM, Oxford CAMSCAN-MV2300), equipped with X-ray mapping system.

The compression test was conducted in air at room temperature (Instron Universal Testing Machine-1195 machine) according to ASTM-E9. At least five specimens were tested for each sintering conditions. After grinding and polishing, composite samples were tested using the Brinell method with a load of 306.56 N.

3. Results and discussion

3.1 Density measurements

The effects of sintering temperature and volume percent of nano MgO on density of the Al-nano MgO composites produced by CIP method are shown in Fig. 1. The highest density value in three temperatures belongs to the samples containing 1.5 and 2.5

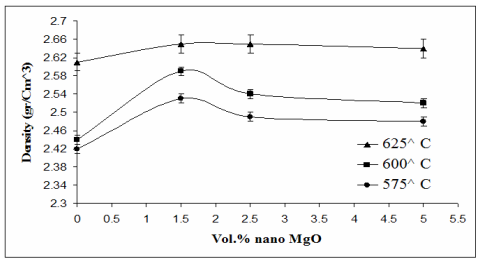

Fig. 1. The density results of the Al alloy and the composite specimens containing 1.5, 2.5 and 5 vol.% MgO sintered at 575, 600 and 625°C.

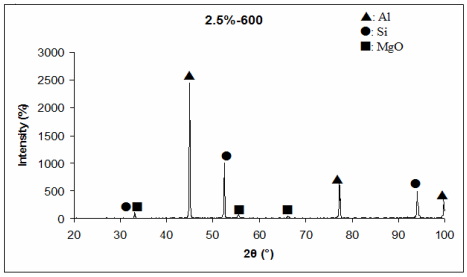

Fig. 2. XRD pattern of composite containing 2.5 vol.% MgO sintered at 600°C.

volume percent of nano MgO. According to the mixture law, by increasing the volume fraction of MgO particles in aluminum, density of samples should be increased because the density of magnesium oxide is higher than that of aluminum.

However, by increasing the volume fraction of refractory MgO particles, sintering of MgO by molten Al alloy decreased. Furthermore, particle agglomeration was expected and any of these factors and the incidence of created porosities will result in reduction of density. On the other hand, this change in decreasing trends of composites density in Fig. 1 is due to agglomeration of MgO particles during the sintering process. Because of the high melting point of MgO, it can be expected that in the sintering temperature, the MgO particles are totally dense and also, it can be expected that MgO particles form a dense network with random distribution which prevents the specimens to be dense [11].

This phenomenon can be attributed to the preventative effect of MgO particles on samples compaction during sintering. Because of the high melting point of magnesium oxide, MgO in sintering temperature is quite rigid and its strong lattice formation prevents samples compaction.

Increasing the sintering temperature enhanced the diffusion of atoms (which helps the better sinter ability of composites, and finally the density of composites) and resulted in higher density value.

3.2 XRD and microstructural analyses

The phases identified by XRD analysis were similar for all composites. Although the intensities of the peaks were different, only magnesium oxide (MgO), silicon (Si) and aluminum (Al) were detected. Fig. 2 shows the XRD pattern of a com-

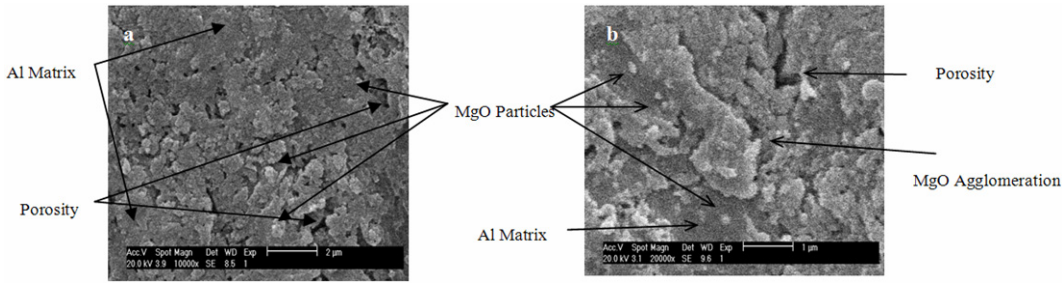

Fig. 3. SEM images of composite containing: (a) 2.5 vol.% MgO; (b) 5 vol.% MgO sintered at 575°C.

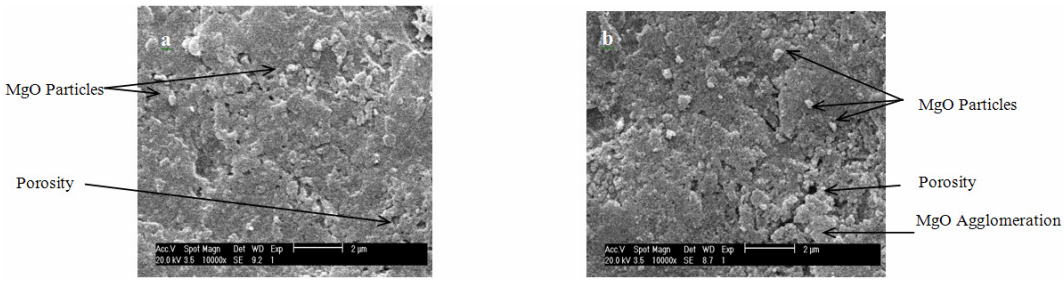

Fig. 4. SEM images of composite containing: (a) 1.5 vol.% MgO; (b) 5 vol.% MgO sintered at 600°C.

posite containing 2.5 vol.% MgO, fabricated at 600°C.

Microstructures of composites with the content of 2.5 and 5 vol.% MgO, sintered at 575°C are shown in Fig. 3. Because of difference between the densities of MgO and aluminum, the contrast of the micrographs is high enough for further investigations.

Dark grey Al matrix and bright particles of MgO can be clearly observed. The phases are indicated by arrows on the above images. It should be noted that nano-sized MgO particles were well dispersed in the matrix of aluminum and just a partial agglomeration in composites with high content of MgO can be detected in Figs. 3-5. Also, similar microstructures were observed for composites fabricated in various sintering temperatures and volume fraction of MgO. The only minor difference was related to the content of porosity and agglomeration.

As demonstrated, there are some black points representing porosities of composites which are formed during powder metallurgy process. Enhancing the MgO content of composites increases the amount of porosities. This could be because of the reinforcing particles aggregations which prevent the densification of composites.

On the other hand, increasing the volume percent of MgO particles decreases the uniformity and homogeneity of the specimens, and the number of MgO clusters tends to increase. These figures show that the distribution of MgO particles is fine, which causes more uniform composites. The reason for fine distribution of MgO particles is to determine the appropriate time and method of mixing.

At constant MgO content, increasing temperature causes more uniformity in microstructure with less porosity. This structure could be the result of the fact that raising temperature causes easier diffusion in microstructure (in addition to better wettability), and therefore, aluminum can diffuse faster and fill the pores of the whole specimen.

Sintering is one of the most important parts of powder metallurgy processing and higher temperatures could facilitate it. Also, the uniformity and compressive strength of the specimen is strongly dependent on sinterability of powders. Increasing the ability of specimens to sinter allows better mechanical properties such as compressive strength to be obtained [12].

Increasing temperature causes a decrease in microporosities of specimens, which is equivalent to lowering stress concentration regions. Therefore, the compressive strength of specimens gets better [13]. Eventually, higher temperatures cause better bonding between aluminum and MgO and this effect improves the mechanical properties of the composite [14].

Fig. 4 represents the microstructure of a composite containing 1.5 and 5 vol.% MgO sintered at 600°C. Fig. 5 indicates the morphology and corresponding X-ray mapping of the sample containing 5 vol.% MgO sintered at 625°C. X-ray maps confirmed the presence of MgO and their well distribution in matrix of composites.

The above-mentioned results, obtained from XRD and microstructural analyses, confirm feasibility of powder metallurgy method to produce such a kind of composite with well distribution of reinforcement. Lack of undesirable phases, which results from undesirable reactions, is another prominent result of this study. It can be mentioned that nano-particle MgO represents appropriate wettability with molten metal and good stability as well.

It should be noted that wettability does not solely determine

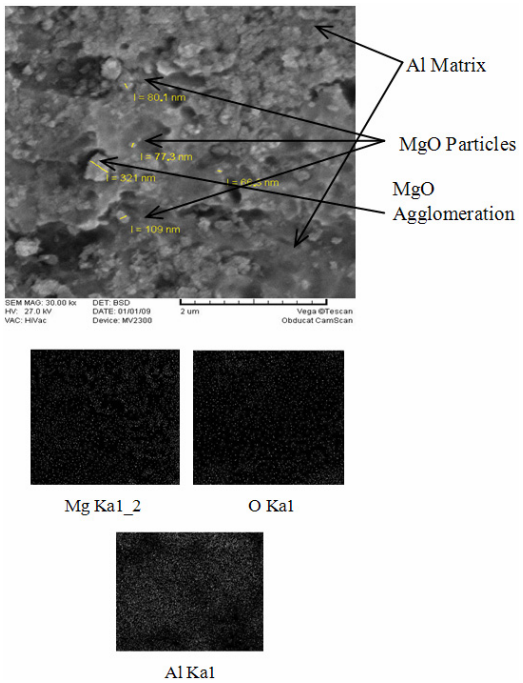

Fig. 5. SEM image of composite containing 5 vol.% MgO sintered at 625°C as well as corresponding X-ray maps.

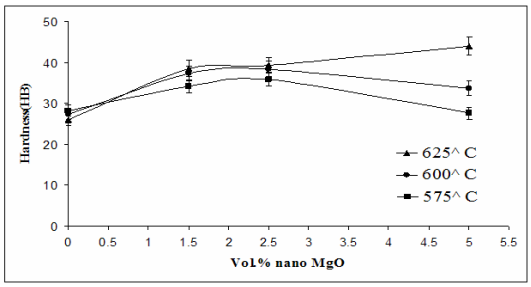

Fig. 6. The hardness of the Al alloy and the composite specimens containing 1.5, 2.5 and 5 vol.% MgO sintered at 575, 600 and 625°C.

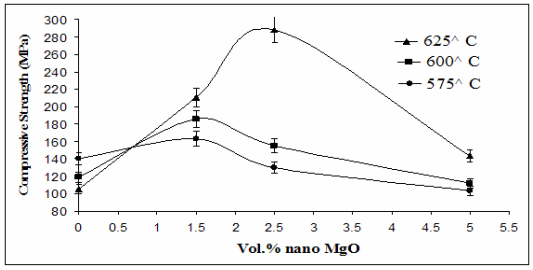

Fig. 7. The compressive strength of the Al alloy and the composite specimens containing 1.5, 2.5 and 5 vol.% MgO sintered at 575, 600 and 625°C.

the distribution of reinforcement particles in the matrix, but other factors such as sintering temperature and time, volume percent of reinforcing phase, reinforcement and matrix particles size, etc. also have profound influences on even distribution of MgO in the metal matrix fabricated by powder metallurgy [15-17].

4. Mechanical properties

4.1 Hardness test

The effects of the sintering temperature and volume percent of MgO nano particles on hardness properties of the prepared composites are shown in Fig. 6. As seen, increasing the volume fraction of magnesium oxide nano particles resulted in enhancement of hardness values. Considering that the hardness value of the magnesium oxide is higher than that of the matrix alloy, improvement in hardness properties of composites would occur by increasing the volume percent of MgO particles.

The mixture law in composite materials confirms this phenomenon. Thus, the properties of a composite are identified by the volume percentage of each material in the composite.

As seen in Fig. 6, at 625°C, increasing the amount of MgO particles up to 5 vol.% increases hardness values. Also, a sample containing 5 vol.% MgO which was sintered at this temperature has the maximum hardness value compared to other samples, and this behavior is related to increasing sintering temperature. Also, the hardness of an unreinforced sample reduced after increasing the sintering temperature. This behavior is a result of grain growth phenomenon due to increase of temperature, which has a great effect on all mechanical properties of materials such as hardness and strength properties.

However, the presence of MgO particles in the matrix alloy could be an obstacle for grain boundaries migration preventing grain growth. Consequently the effect of high temperature on increasing grain growth would be omitted. As mentioned before, increasing atomic diffusion due to raising the sintering temperature could improve sinterability. Therefore, properties of the samples, including density and hardness, would be enhanced. Increasing density improves many properties of the samples such as hardness. Moreover, porosity reduction due to increasing sintering temperature plays an important role in improvement of the hardness properties.

4.2 Compression test

The effects of the sintering temperature and volume percent of nano MgO on the compressive strength of Al-nano MgO composites produced by CIP method are shown in Fig. 7. Increasing the volume percent of magnesium oxide reinforcement particles initially increased the value of the compressive strength compared to unreinforced matrix alloy and then decreased at all three sintering temperatures. The initial enhancement seems to be due to work-hardening behavior.

This could be related to effects of elastic properties of ceramic particles and inhibition response of plastic deformation of matrix by them; ceramic particles can only deform elastically while aluminum matrix can deform plastically. So if the boundary is assumed to be strong, ceramic particles prevent plastic deformation of the matrix, and this leads to a higher

work-hardening rate. The difference between coefficients of thermal expansion of ceramic and metal matrices may result in stress concentrations, and therefore, high density of dislocations; the strength of the aluminum matrix increases accordingly. These confirm the obvious effect of MgO particles on strengthening of composites.

Increasing the volume fraction of magnesium oxide particles in the constant particle size decreased the distances between particles and resulted in enhancement of dislocation density and their pile-ups behind the MgO particles. Therefore, higher stress needs to move dislocations, which also has the effect on strength increment [18].

The following equation can explain this effect better:

$$\tau_0 = \frac{Gb}{\lambda} \tag{1}$$

where τ_0 is shear stress for a single crystal; G is shear module; b is Burger's vector of crystal and λ is the distance between reinforcement particles.

According to these equations, it can be concluded that if the distance between particles decreases, the stress for shearing of dislocations would increase and thus, the stress would be augmented. This trend continues until micro-porosity causes low mechanical properties in composites, since increasing the reinforcement content would cause the micro-porosity volume to elevate [19, 20].

It should be noted that increasing micro-porosities develops discontinuities in the matrix phase which act as stress concentration locations, and these phenomena cause severe decreases in sample properties. On the other hand, increasing the amount of reinforcement particles decreased the homogeneity in distribution of magnesium oxide particles in the matrix and in some areas formed the cluster assembly. These regions do not change during sintering. Thus, they remain without any binding agent between them. In the higher volume percentages of MgO, these regions could be seen near every boundary. The direct contact of these MgO regions causes weak binding between boundaries. Thus, the composite would not have desired mechanical properties such as compressive strength.

As seen in Fig. 7, compressive strength of the specimen with 5 vol.% MgO is the lowest at all three temperatures. This could be attributed to the aforementioned reasons. Also, low density of composites resulted from incorporation of higher content of MgO, and also, air absorption caused the decreasing trend of compressive strength.

Increasing temperature causes better wettability of MgO particles by aluminum, but can increase the internal stresses in composites with higher percentages of MgO particles, even causeing cracks in the samples.

From another point of view, increasing the temperature causes easier diffusion, and with easier diffusion, the properties of composites change, especially mechanical ones. Fig. 7 shows these effects clearly. It shows that the compressive strength curves of composites with increasing sintering temperature are located in upper positions. Therefore, at constant amount of MgO, composites which are sintered at lower temperatures have lower compressive strength values which cause poor mechanical properties.

The general trend of this figure (especially at 625°C) shows an improvement in compressive strength of specimens. Among composites with constant amount of MgO (e.g. 1.5, 2.5 and 5 vol.%) sintered at various temperatures the sample with maximum density shows higher compressive strength.

This phenomenon can be clearly seen in Fig. 7. The compressive strength of specimens with more than 1.5 vol.% MgO content changes abruptly (there is a sharp rise) with increasing temperature at highest temperature.

5. Conclusion

A356.1 aluminum alloy reinforced with nano-sized MgO was successfully fabricated via powder metallurgy method. SEM micrographs indicate that reinforcement particles were homogeneously distributed in the matrix of composites. However, partial agglomeration was observed in composites with high content of MgO.

With increasing MgO content of composites, the hardness of specimens increases to a maximum value of 44 BHN (increase of 69% in comparison to the A356.1 non-reinforced alloy). Increasing temperature facilitates this trend. Also, higher temperatures cause increase in density. Moreover, sintering temperature improves compressive strength. In this experiment, a sintering temperature of 625°C showed better results than both 600 and 575°C.

Compressive strength changes with increasing MgO content. At first it increases but then starts to decrease. The maximum compressive strength observed was 288 MPa, which belongs to the specimen with 2.5 vol.% MgO content in 625°C.

It looks like in this composite, MgO particles are responsible for preventing plastic deformation and also cause higher work hardening. In addition, the difference between the MgO content and Al matrix results in internal stress and higher density of dislocations, which will result in better mechanical properties.

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