Influence of nanosized Al_2O_3 weight percentage on microstructure and mechanical properties of Al–matrix nanocomposite

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Aluminium based metal matrix composites produced by powder metallurgy are used in the aerospace and automobile industries because of their high strength, light weight, etc. The properties may become much better when nanosized reinforcements are used. In this research, the morphological, microstructural and mechanical properties changes during nanosized alumina increment to Al powder were studied. Because nanosized particles are extremely prone to agglomeration, all of the samples have been milled for 12 h by a planetary ball mill. The process was conducted for Al–(0–20) wt-%Al_2O_3 powders to explore the role of reinforcement nanoparticles on the microstructure and mechanical properties. The results showed that the strength, ductility and hardness were increased by increasing the reinforcement nanoparticles weight percentage. Also, addition of hard particles accelerated the milling process, leading to faster work hardening rate and fracture of the aluminium powder.

Keywords: Alumina, Nanocomposite, Ball milling, Particle reinforcement, Mechanical properties

Introduction

Al based metal matrix composites (Al–MMCs), which possess high strength, high hardness, high specific elastic modulus, etc., are being widely used in the aerospace and automobile industries.[1,2] Recently, many Al–MMCs have been produced by *in situ* processes such as mechanical milling and powder metallurgy.[3,4] Powder metallurgy can produce metal matrix composites almost in the whole range of matrix reinforcement compositions without the segregation phenomena typical of the casting processes.[3–5] The first requirement for a composite material to show its superior performance is the homogeneous distribution of the reinforcing phase.[6,7] The homogeneity of the reinforcement in PM is very dependent on the particle size ratio used for the powders.[7] Most studies on powder metallurgy (PM) MMCs concentrated on a composite with high strength aluminium matrix.[7–13] Lai and Chung[14] presented one of the very few papers on MMC using pure aluminium as a matrix. In their work, the MMCs were manufactured by vacuum infiltration of liquid aluminium into a porous particulate perform under inert gas. Several studies on aluminium alloy matrix composites reported a decrease in mechanical strength of the composite compared to the unreinforced matrix.[7,11,15–18] This was attributed to the overloading of the brittle reinforcement particles during the load transfer process and fracture of them. Thus, these fractured particles cause deterioration of mechanical properties of the composite.

Manoharan and Lewandowski[15] explained that low strength matrices may benefit more from brittle reinforcement particulates than high strength matrixes.

The agglomeration of the reinforcement particles deteriorates the mechanical properties of composites. Differences in particle size, densities, geometries, flowing or the development of an electrical charge all contribute to particle agglomeration.[3] High energy ball milling has been used to improve particle distribution throughout the matrix.

According to literature survey, there are many articles which have been focused on production of composite materials using mechanical alloying method.[1,3,4,19] There are many main approaches among investigators. One approach pays attention to the effect of milling time on microstructure of composite powders. Others are focused on the improvement of efficiency of milling using surfactant, charging of mill, etc. But according to author's knowledge, there are a few articles which are concentrated on the effect of milling and weight percentage of particles on composite microstructure and mechanical properties thus, the main goal of this study is to investigate the effect of weight percentage of nanosized Al_2O_3 in Al–Al_2O_3 nanocomposite on the properties and improving reinforcement distribution by means of ball milling.

Experimental methods

Materials

To produce Al–Al_2O_3 nanocomposite, commercial aluminium powders with particle size smaller than

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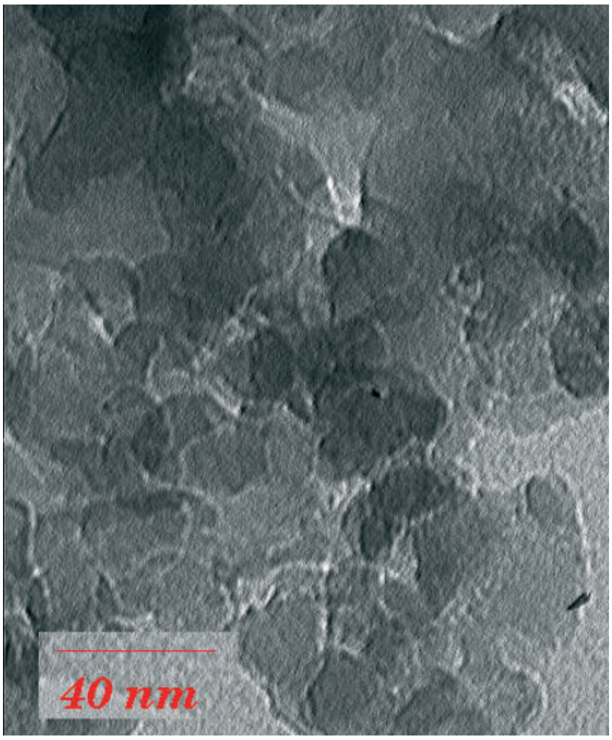


1 Image (TEM) of microstructure of α-alumina nanosized powders used

63 μm and α-alumina nanopowder with 99·5% purity and average size of about 27–43 nm have been provided. Size and shape of α-alumina powder were determined using transmission electron microscopy (LEO 912AB).

Milling and sample preparation

High energy planetary ball mill with stainless steel balls with different diameters (8·5–10 mm) were employed. Rotary velocity of the mill and ball to powder weight ratio were 250 rev min^{-1} and 15 respectively. Five different reinforcement weight fractions were used, i.e. Al–0, 2·5, 5, 10 and 20 wt-% $A1_2O_3$. The aluminium and alumina powders were milled for 12 h. Ethanol (C_2H_5OH) was also used as the process control agent (PCA) to prevent the powders from severe cold welding and contamination.

Then, cylindrical samples were produced using powder metallurgy method under 420 MPa. Sample diameter and height were chosen 10 and 15 mm respectively.

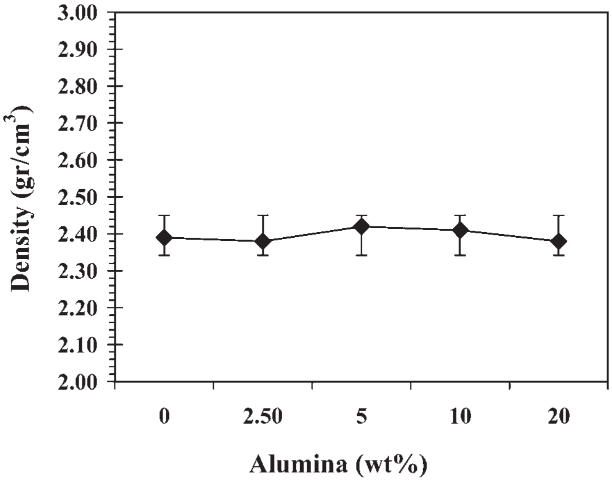
For sintering, the compressed powders were kept at 624–626°C for 45 min under argon gas and then the samples were cooled at the furnace.

Microstructure evaluation

After ball milling of the powders for 12 h, microstructure of the powder was studied by scanning electron microscopy (SEM) to investigate the effect of nanosized Al_2O_3 content on the microstructure of the nanocomposites. Metallographic studies were also performed on the compacted and sintered samples. Density measurement was performed on all compacted and sintered samples using Archimedes technique.

Hardness measurement

Hardness measurement was carried out using Vickers with 10 kg load on all sintered samples. Assessment of the effect of reinforcement particle contents on hardness of the nanocomposites was the main goal of this test. It is worth noting that after hardness test no any cracks were observed around the indentation effect.



2 Density variation of 12 h milled nanocomposites versus alumina weight percentage after compressing and sintering (0 wt-%Al_2O_3 means pure aluminium)

Compression test

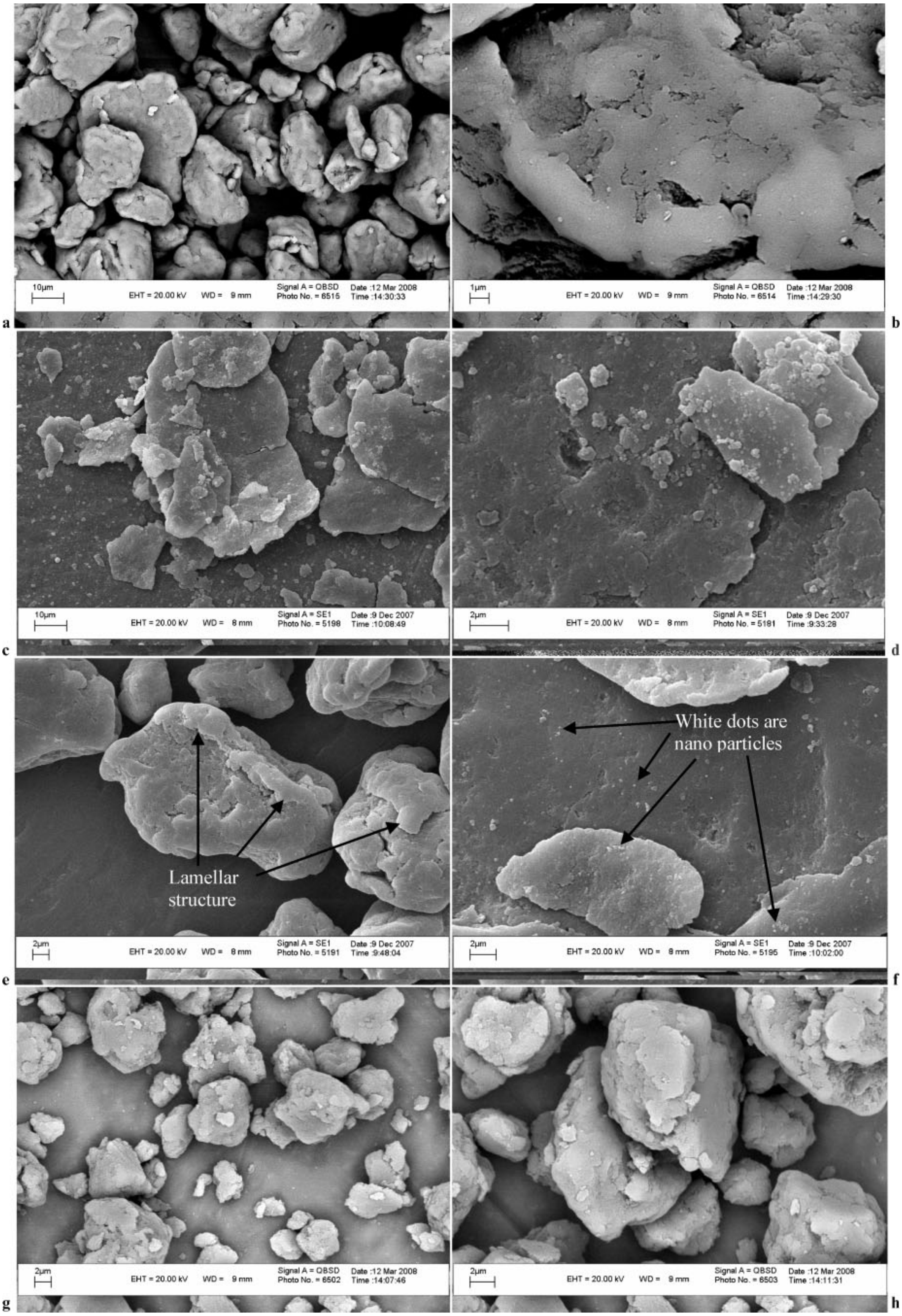
For evaluation of mechanical properties of the nanocomposites, compression test was carried out by Zwick (Z/250) device. Strain rate was chosen 5×10^{-4} s^{-1} in compression test and the test was continued to the splitting of samples. From each nanocomposite three samples were tested at room temperature and a lubricant was used for reduction of friction between surfaces of the sample and device jaws.

Results and discussion

Figure 1 shows micrograph of α-alumina powder taken by transmission electron microscopy. Average size of the equiaxed particles was measured as 27–43 nm. To ensure that all samples are produced with the same procedure and have the same amount of porosity, density of the samples were measured. Figure 2 shows that the sample densities are almost constant. Therefore, the mechanical properties of all samples can be compared together.

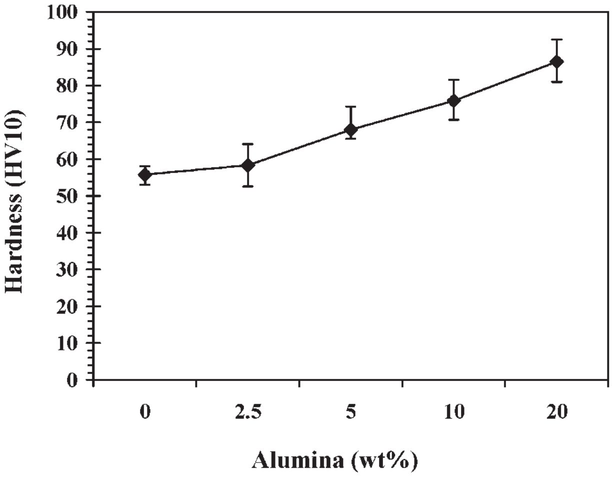
Figure 3*a* to *h* shows the SEM microstructures taken from Al–Al_2O_3 powder samples with different contents of the reinforcement after 12 h milling. As it was expected, at Al–2·5%Al_2O_3 alumina agglomerations are observed which have been welded on the aluminium surfaces (Fig. 3*a* and *b*). These agglomerations would be removed by increasing alumina weight percentage. The presence of alumina particles causes more work hardening due to ball milling consequently, there are more splitting of aluminium particles that result in disappearing of agglomeration. Comparison of Fig. 3*d* and *b* indicates that agglomerations are very much smaller and less in Al–5Al_2O_3 than in Al–2·5Al_2O_3 nanocomposite so that it can be said that in Al–5Al_2O_3 nanocomposite alumina particles dispersed very well in welded aluminium laminates without any agglomeration.

There is another noticeable difference between Al–2·5Al_2O_3 and Al–5Al_2O_3 nanocomposites. For Al–2·5Al_2O_3, as shown in Fig. 3*a*, because of low alumina



a, *b* 2·5 wt-%; *c*, *d* 5 wt-%; *e*, *f* 10 wt-%; *g*, *h* 20 wt-%

3 Images (SEM) of microstructures of Al–Al_2O_3 powders with different amounts of nanosized Al_2O_3 particles milled for 12 h



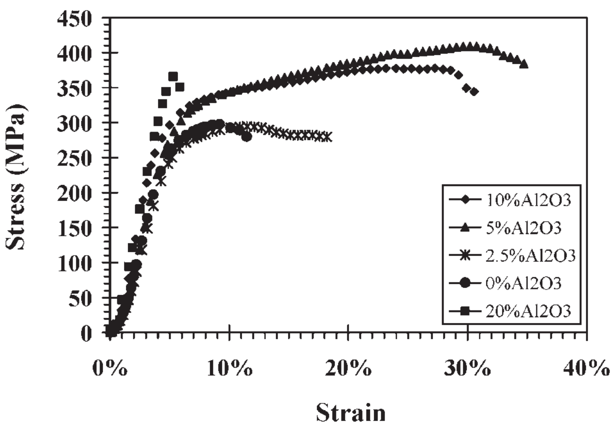
4 Hardness variation of 12 h milled composites versus alumina weight percentage (0 wt-%Al_2O_3 means pure aluminium)

content, plastic deformation and consequently, cold welding are extremely high, so particles morphology is spherical in shape. On the contrary, as shown in Fig. 3*c* for Al–5%Al_2O_3, the particles shape is flake like. Also, there is smaller particles size in Al–5Al_2O_3 than Al–2·5Al_2O_3, because fragmentation of the particles occurs more easily due to the enhanced work hardening.

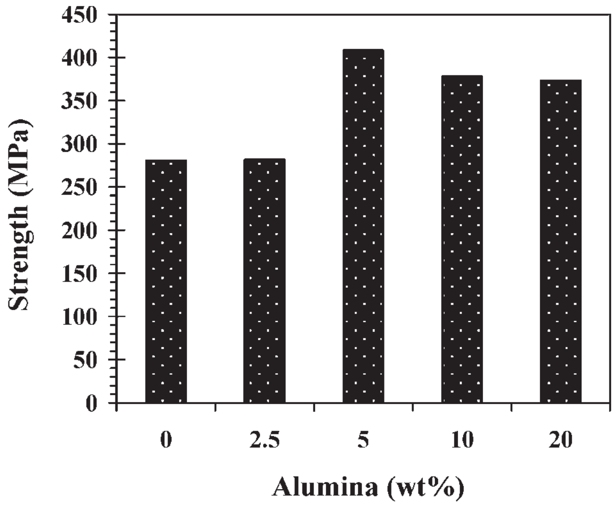
As it was mentioned, uniform distribution of alumina particles and disappearing of agglomeration are due to the alumina addition and splitting of aluminium particles by milling. However, the level of nanoparticles agglomeration was increased by increasing their weight percentage from 5 to 10%. Figure 3*e* and *f* shows that alumina agglomerations in Al–10Al_2O_3 nanocomposite appear again, although the agglomeration size is on the nanometre scale. Lamellar microstructure on the aluminium particles surface shows that milling process is not stabilised, yet. Also, because of alumina and work hardening increment in Al–10Al_2O_3, the particle size is smaller than the two past composites (Fig. 3*a*, *c* and *e*).

Particles size of Al–20Al_2O_3 nanocomposite was extremely reduced due to the large number of splitting particles (Fig. 3*g*). At this nanocomposite, like Al–10Al_2O_3, alumina agglomeration was observed.

Figure 4 shows hardness variation of 12 h milled samples versus alumina weight percentage. According to strengthening mechanisms related to the presence of reinforced particles in a soft matrix and work hardening, hardness was increased by alumina particles increment.



5 Stress–strain curves of nanocomposites with different amounts of alumina nanosized powders



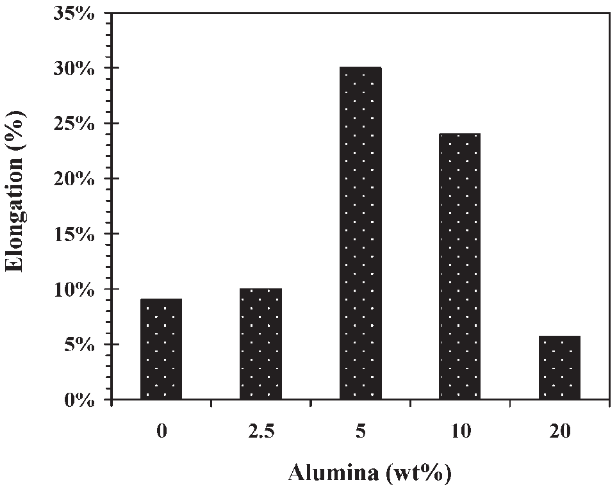
6 Compressive strength variation of 12 h milled composites versus alumina weight percentage (0 wt-%Al_2O_3 means pure aluminium)

Reinforcement particles in the matrix play useful roles in the nanocomposite, although they have harmful effects, such as stress concentration at the surface, too. The stress–strain curves of the nanocomposites with different amount of nanoparticles obtained from compression tests are shown in Fig. 5. The mechanical properties resulted from the tests, such as compressive strength and ductility, are discussed separately.

The compressive strength of the 12 h milled samples versus alumina weight percentage is indicated in Fig. 6. From the figure, it can be seen that the addition of about 2·5 wt-% Al_2O_3 is not so effective in increasing the compressive strength. Because of the low reinforcement amount in Al–2·5 wt-%Al_2O_3 nanocomposite, harmful effect dominates useful effect and prevent strength rising.

Increasing the nanosized alumina to 5 wt-% raises compressive strength substantially, because of operating of strengthening mechanisms in the presence of reinforcement particles. However, because of alumina agglomerations in Al–10Al_2O_3 and Al–20Al_2O_3, strength was decreased by increasing alumina weight percentage from 5 to 20 wt-%. Agglomeration of the alumina particles cause stress concentration and local fracture. Thus, on the one hand, strength is influenced by uniform distribution of the alumina nanoparticles and tends to increase, and on the other hand, strength is affected by alumina agglomeration and tends to decrease. Alumina agglomeration has little effect on the hardness, as on the strength, therefore, an increase in alumina causes hardness and brittleness of samples to enhance. The brittleness results in the fracture of samples at low stress and thus leads to a decrease in strength.

The weight percentage concentration of Al_2O_3 influences the ductility of the nanocomposite. Figure 7 shows the effect of 12 h milled samples with different alumina weight percentages on compressive ductility. As it is shown, the ductility is increased with ceramic particles amount increment up to 5 wt-%. Because of nanosized particles and very short distances between them, they



7 Compressive ductility variation of 12 h milled composites versus alumina weight percentage (0 wt-%Al_2O_3 means pure aluminium)

prevent the joining of cracks. Thus, nanoparticles inhibit crack formation and growth consequently, the ductility was increased by increasing the amount of ceramic particles. Beyond 5 wt-%Al_2O_3, agglomeration of the reinforcement particles in the matrix causes stress concentration and more work hardening consequently, the matrix can not release its strains and becomes brittle, so the ductility is reduced.

Conclusions

The results from this research show that the alumina weight percentage has effect on the mechanical properties of Al–Al_2O_3 nanocomposite. The conclusions of this research are as follows.

1. The amount of 2·5 wt-%Al_2O_3 did not strongly affect the strength and elongation in 12 h milled nanocomposite.
2. Hardness was increased by increasing the amount of alumina.
3. Strength was increased by increasing the amount of alumina up to 5 wt-%.
4. Because of nanosized reinforcements, the ductility was appreciably increased by increasing the amount of alumina up to 5 wt-%.
5. Al–5Al_2O_3 nanocomposite showed more uniform distribution of alumina particles and the best mechanical properties.

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