# Synthesis of Al-$Al_2O_3$ Nano-Composite by Mechanical Alloying and Evaluation of the Effect of Ball Milling Time on the Microstructure and Mechanical Properties

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## Abstract

*Aluminum matrix composites reinforced by nano-ceramic particles are widely used in military, airplane and automotive industries because of their high strength, modulus, wear resistance and low thermal expansion coefficient. However, low ductility and toughness have limited their applications. This research focuses on production of Al–$Al_2O_3$ nanocomposite materials by mechanical alloying method and evaluation of the milling time on their microstructures and mechanical properties. In this regard, Al-5wt%$Al_2O_3$ powders were ball milled at different times and microstructure of specimens were studied by SEM. Also, mechanical properties like: hardness, compression strength and ductility of the specimens were determined after pressing and sintering. Study of the microstructures indicated that more uniform distribution of nano-size alumina particles in aluminum matrix can be achieved by increasing of milling time. The results of hardness and compression tests showed that hardness, strength and ductility increased by augment of milling time.*

## I. Introduction

All Aluminum and its alloys are among the interested metals for producing metal matrix composites. This causes aluminum matrix composites to find extremely attention in the last two decades [1,2].

Recently, researches results denote, nano-sized reinforcement particles would increase strength, ductility and toughness[2,3,4,5]. Segregation and non uniform distribution of reinforcements are of cast method defects[2,6]. One of the main challenges inherent to this technique is to obtain a homogeneous distribution of the reinforcement in the metal matrix[7,8]. Powder metallurgy can produce metal matrix composites in the whole range of matrix-reinforcement compositions without the segregation phenomena typical of the casting process[6,7,8].

The first requirement for a composite material to show its superior performance is the homogeneous distribution of the reinforcing phase[9]. The agglomeration of the reinforcement particles deteriorates the mechanical properties of the composite[4,7-10]. Differences in particle size, densities, geometries, flowing or the development of an electrical charge all contribute to particle agglomeration[6,9]. Recently, high-energy ball milling has been used to improve particle distribution throughout the matrix[1,11-13].

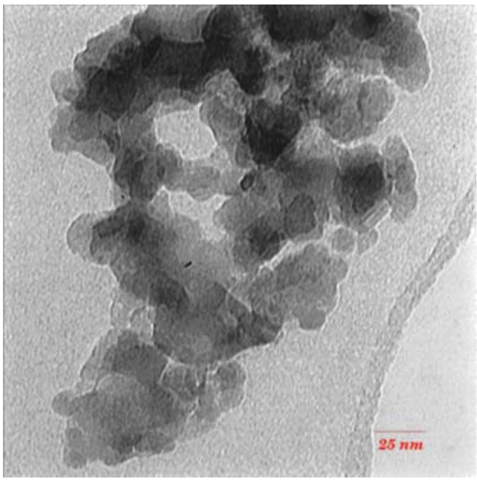
Process control agent (PCA) has been used to reduce cold welding and particles fragment augment and also has been used to prevent powder contamination[11,13,14].

According to literature survey there are many articles which have been focused on production of composite materials using mechanical alloying method. There are two main approaches among investigators. The first approach pays attention to the effect of milling time on microstructure of composite powders. The second approach focuses on the improvement of efficiency of milling using surfactant, changing mill and so on. But according to authors knowledge there are a few articles which are concentrated on the effect of milling-time on nanocomposite mechanical properties thus, the main goal of this study is to investigate the effect of this parameter on Al–$Al_2O_3$ nanocomposite mechanical properties and also, microstructure produced during ball milling.

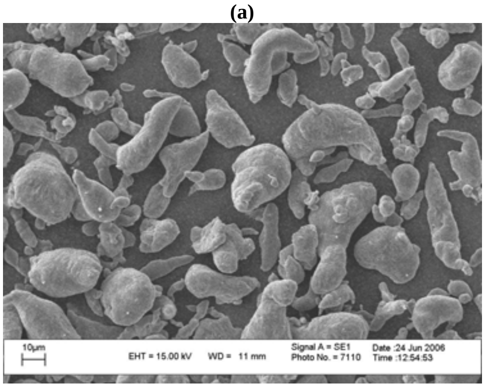
## II. Experimental methods

### A. Materials

To produce Al–$Al_2O_3$ nanocomposite, commercial aluminum powders with particle size smaller than 63 µm and nano-sized α-alumina powder with %99.5 purity and average size of about 27-43 nm have been provided. Figures 1(a) and (b) show micrographs of the α-alumina and aluminum powders taken by transmission electron microscopy (LEO 912AB TEM) and scanning electron microscope (LEO 1450VP SEM), respectively. It was found that the α-alumina particles are almost spherical and aluminum particles are irregular in shape.




**(a)**




**(b)**

Figure 1. Initial used powders: (a) α-alumina, (b) aluminum

### B. 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 rpm and 15, respectively. The aluminum and 5wt% alumina nano-powders were milled at different times, i.e., 0, 4, 8 and 12 hours. Process control agent (PCA) was also used to prevent powders from severe cold welding and contamination.

Diameter and height of the cylindrical samples produced using powder metallurgy method were 10 and 15mm, respectively. Samples were fabricated under 420MPa pressure and after five minutes, pressure was removed.

For sintering, the compressed powders were kept at 624-626ºC for 45min under argon gas atmosphere and then the samples were furnace cooled.

### C. Microstructure study

After ball-milling of the powders for a specific time, microstructure of the samples was studied by Scanning Electron Microscope (SEM) to investigate the effect of ball-milling time on the microstructure of the nanocomposites produced by mechanical alloying.

### D. Hardness measurement

Vikers hardness measurement was performed with 10Kg. load on all samples.

### E. Compression test

For evaluation of mechanical properties of the nanocomposites, compression test was done by Zwick (Z/250) device. Strain rate was chosen $5*10^{-4}$ $s^{-1}$ in compression test until sample was split. Three samples were tested at room temperature and grease was used for reduction of friction on the surfaces of the sample and device jaws.

## III. RESULTS AND DISCUSSION

### A. Milling time effect on the microstructure

Figures 2(a) to 2(h) show the SEM microstructures taken from Al–5%$Al_2O_3$ powder samples after different milling times. As it was expected at 0 hours milling time, the alumina agglomeration particles are extremely observed (Fig. 2(a) and 2(b)). These agglomerations would be removed by increasing milling time. At four hours milling time (Fig. 2(c) and 2(d)), alumina agglomerations were observed yet, but they are smaller and less than no milled powders also, a little of alumina particles agglomerations welded with aluminum matrix.

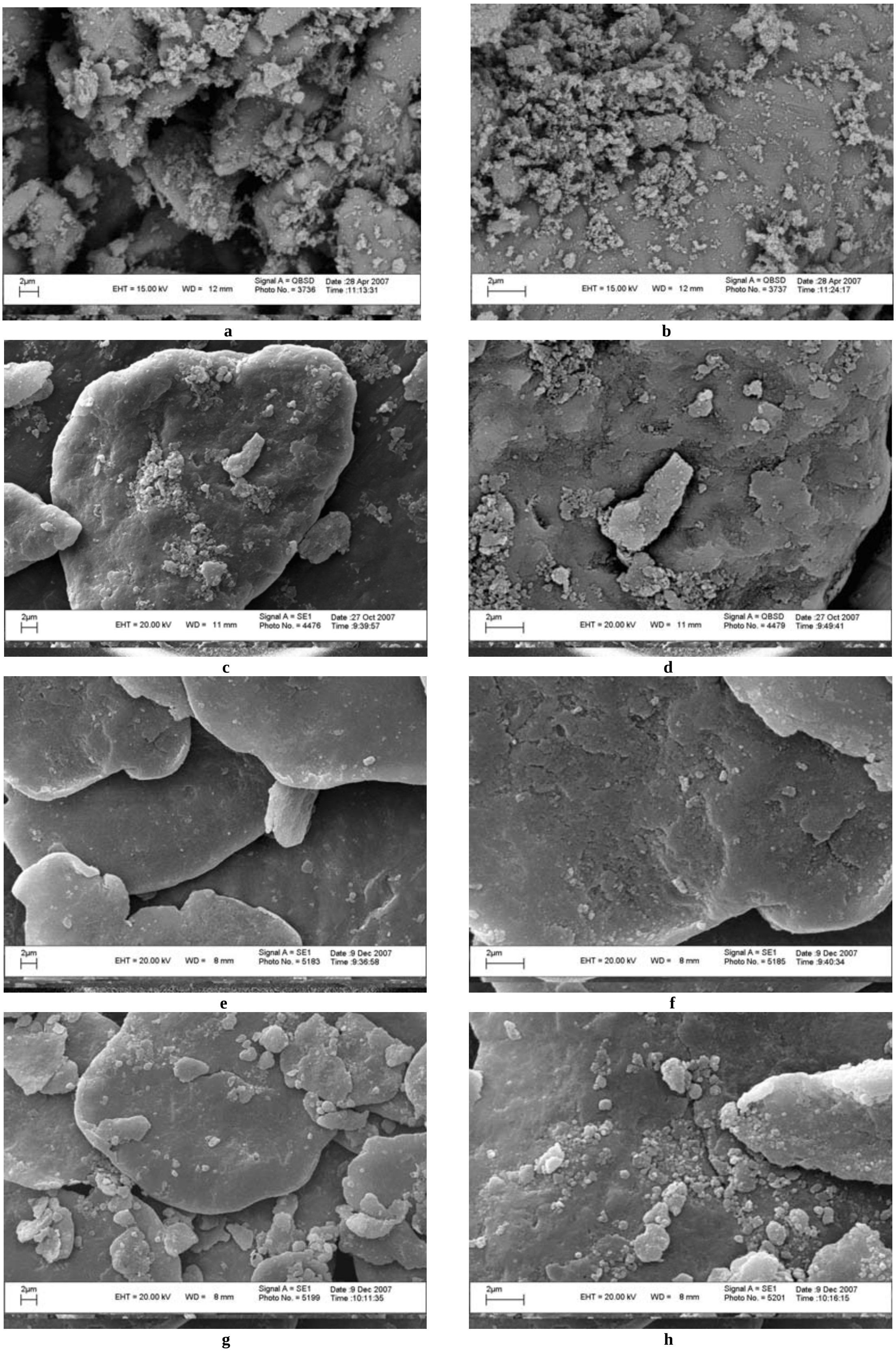



Figure 2. SEM Micrograph of Al–5 wt% Al2O3 powder particles at various milling time: (a,b) 0h, (c,d) 4h, (e,f) 8h and (g,h) 12h.

However, increasing milling time caused to uniform distribution of the reinforcement particles, dissolution of particles agglomerations and reduction of distances between them. Note that at this milling time particles have been under deformation and cold welding therefore, flattened particles with high aspect ratio were formed. This is because aluminum particles are soft and their sizes are increased by cold welding. Splitting of the particles was not observed during milling.

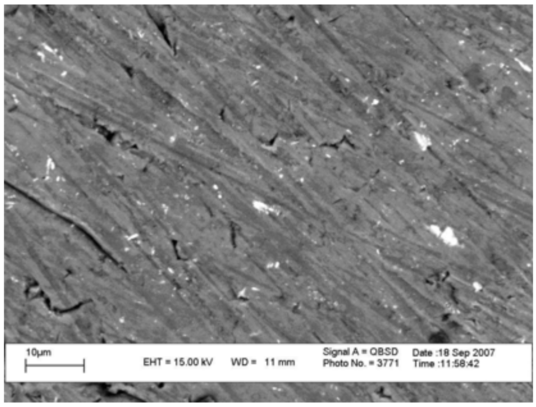
An eight-hour milling (Fig. 2(e) and 2(f)), resulted in a morphological change of particles from flattened to flake-like. At this milling-time because of work hardening of the soft aluminum powders, along with cold welding of the particles, fragment was happened in a large amount and particles sizes were reduced. Also, all of alumina particles have been moved in welded aluminum and confined between them so that, welded layers were obviously observed on the alumina particles surface (Fig. 2(d) and 2(f)). As the figures indicate, at this stage there are alumina clusters surrounded by aluminum particles. Certainly, such microstructures have undesirable effects on mechanical properties.

At twelve-hour milling time, because of excess work hardening and enhancement of brittleness of the particles by milling, splitting of aluminum particles has been occurred. Figures 2(g) and 2(h) show the resulted microstructure. In the microstructures, uniform distribution of the sub-micrometer aluminum particles is illustrated. At this milling time, particles morphology is like laminar and also, alumina clusters have been disappeared and distributed uniformly. Because of nanometer size of alumina particles and covering of them by aluminum particles, these particles haven't been observed at this stage.

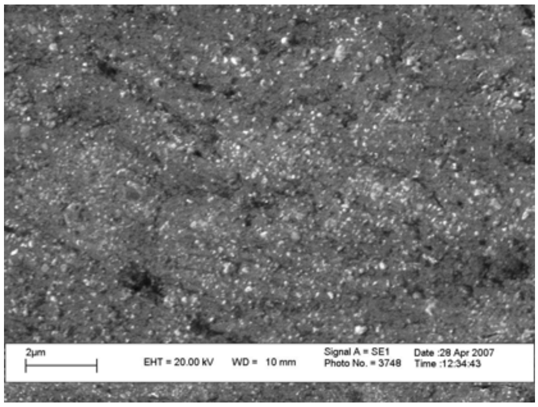
Figures 3(a) and 3(b) show the SEM microstructures taken from Al–5% $Al_2O_3$ sintered samples after 4 and 12 hours milling, respectively. As it is expected, at 4 hours milling time (Fig. 3(a)), the distribution of alumina particles (white particles) was not uniform and the distance between them was so high also, there are particle clusters. However, increasing milling time up to 12 hours caused a uniform distribution of the reinforced particles, reduction in particles size (even to nanometer), disappearing of particle agglomeration and reduction of distances between them (Fig. 3(b)

B. Milling time effect on mechanical properties

Figure 4 shows Al-5%wt. $Al_2O_3$ hardness versus the milling time. As it is illustrated, hardness was increased with milling time increment due to the strength mechanisms described in the next section. Also, hardness of un-milled sample is very low because there are a lot of alumina agglomerations (Fig. 2(a) and (b)) and residual porosities among the reinforcement particles which produce stress concentration sites.




(a)




(b)

Figure 3: SEM micrographs taken from Al–5% $Al_2O_3$ samples sintered after milling times of (a) 4 and (b) 12 hours.

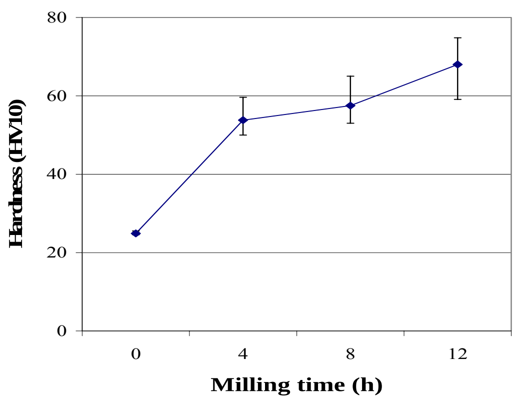



Figure 4. Hardness variation of Al-5%wt. $Al_2O_3$ versus milling time.

Researches show that ceramic nano-particles delay recrystallization or prevent it even up to the temperatures near to its melting point. Figure 5 shows with heating of sintered sample at 645°C temperature for 45 minutes, no change in hardness were observed because recrystallization has not been occurred. Thus, work hardening effect of the milled samples hasn't been removed by high temperature sintering.

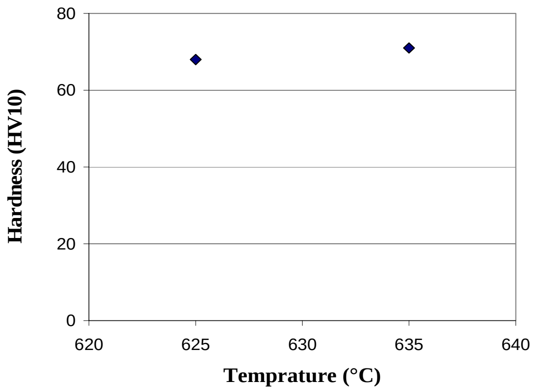



Figure 5. Heating effect on the Recrystallization of Al-5%wt. $Al_2O_3$ powders milled for 12 hours and tempered at 625ºC.

Figure 6 shows the strength variation of the Al-5%wt. $Al_2O_3$ nano-composite versus milling time. As it is observed increasing milling time causes a raise in the strength.

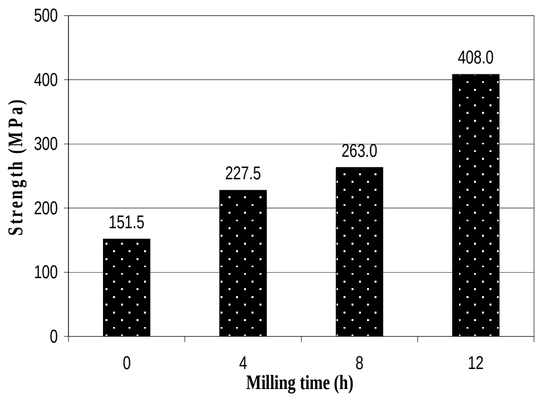



Figure 6. Strength variation of the Al-5%wt. $Al_2O_3$ versus milling time.

The raise in the composite strength was influenced by a few factors such as:

- Milling consequent deformation and work hardening;
- Grain refinement and sub grains production because of increasing in dislocations density;
- Increasing in dislocations density because of difference in thermal expansion coefficient of aluminum and alumina;
- Uniform distribution of alumina particles.

High sintering temperature (625ºC) and difference in thermal expansion coefficient of aluminum and alumina produce thermal stress. The stress will be disappeared by dislocations production and cause increasing in dislocations density, this is important for strength enhancement.

Agglomeration of the alumina particles cause stress concentration and unexpected fracture thus, strength is increased by uniform distribution of the alumina particles.

As it is obvious from Figures 2 and 3 with increasing of milling time more uniform microstructure is occurred and stress concentration is reduced, thus strength and elongation should be improved, although Fig. 7 shows that elongation is constant, relatively. The behavior is the result of the opposite effects of the two factors. On one hand, milling causes work hardening, which reduces the elongation and on the other hand, causes uniform distribution of alumina particles, which improves ductility. It is worth mention that uniform distribution of reinforcement and very short distances between nano particles prevent crack growth and their interconnection therefore, elongation and strength will increase. In comparison, un-milled sample has low strength and elongation that is because of reinforcement particles agglomeration and stress concentration (Fig. 2(a) and (b)).

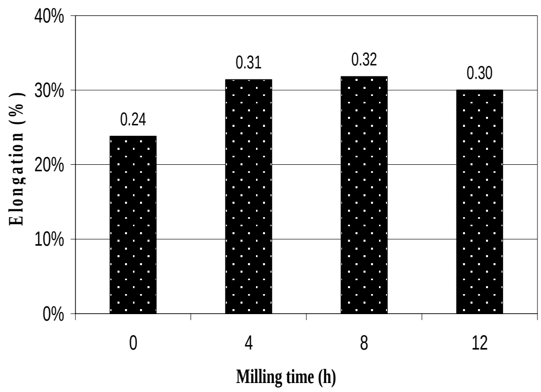



Figure 7. Elongation variation of the Al-5%wt. $Al_2O_3$ versus milling time.

Comparison of Figures 4 and 6 indicates that milling is very effective at first 4 hours milling and mechanical properties change suddenly. Only in Fig. 6, strength severely raised at 12 hours milling. This trend can be due to the activating Orowan strength mechanism. Because Orowan mechanism only activate for very small particles about nano size and after 12 hours milling and splitting of alumina agglomerations, alumina particles distribute in nano scale (Fig. 3b). Also, the uniform distribution of ultra-fine alumina particles inhibits stress concentration and crack growth.

## IV. CONCLUSIONS

The results of the research show that Al- $Al_2O_3$ nano composite mechanical properties concern to milling time. Receiving results at this research is:

1. The microstructure becomes more uniform and fine as a result of milling.
2. Strength and hardness were increased by increasing of milling time but the elongation was almost constant.
3. Al-5%wt. $Al_2O_3$ composite strength was appreciably increased by 12 hours milling, due to the uniform distribution of nano-size alumina particles.
4. Because of nano size reinforcements, alumina particles inhibit elongation to reduce.

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