



## Effect of Mechanical Milling on the Morphology and Structural Evaluation of Al-Al<sub>2</sub>O<sub>3</sub> Nanocomposite Powders

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### ABSTRACT

The morphological and microstructural changes during mechanical milling of Al powder mixed with 2.5, 5 and 10 wt.% Al<sub>2</sub>O<sub>3</sub> particles were studied. The milling was performed in a planetary ball mill for various times up to 20h. The produced composite powders were investigated using X-ray diffraction pattern (XRD) to elucidate the role of particle size, secondary phase content and milling time on grain size and lattice strain of Al matrix. The aluminum crystallite size estimated with broadening of XRD peaks by Williamson–Hall formula. The morphological changes were studied by SEM technique. The results show that the addition of hard Al<sub>2</sub>O<sub>3</sub> particles accelerates the milling process, leading to faster work hardening rate and fracture of the aluminum matrix. Furthermore, Al becomes smaller crystallite size during ball milling of Al powder in the presence of Al<sub>2</sub>O<sub>3</sub> particles. The results revealed that the grain size of milled powders was about 45nm with a noticeable presence of agglomerates. Uniform distribution of nano-sized Al<sub>2</sub>O<sub>3</sub> particles in the Al matrix could be achieved with increasing milling time.

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## 1. INTRODUCTION

Particle reinforced aluminum matrix composites possess high-specific elastic modulus, high-specific strength, good wear resistance and excellent properties at elevated temperature over conventional aluminum alloys. They are considered as potential light weight and high-performance materials to be used in aerospace craft, aircraft and engine parts in automobiles [1].

This has caused aluminum matrix composites to find extreme attention in the last two decades [1, 2]. In recent years, many researchers have paid attention to new fabrication techniques for making aluminum matrix composite. The most important challenge in production of a composite material is the homogeneous distribution of the reinforcing phase [3]. Differences in particle size, densities, geometries, flowing or the development of an electrical charge all contribute to particle agglomeration [3].

Mechanical milling is a unique process in which a solid state reaction takes place between fresh powder surfaces of the reactant materials at room temperature

[4]. Consequently, it can be used to produce alloys and compounds which are difficult or impossible to be obtained by conventional melting and casting techniques [5, 6]. Recently, high-energy ball milling has been used to improve particle distribution throughout the matrix [7-9]. High energy ball milling is a simple and useful technique for attaining a homogeneous distribution of the inert fine particles within a fine grained matrix [7]. Addition of ceramic reinforcements into a ductile matrix has a great effect on the structural evolution during ball milling. Mechanical milling is a process involving repeated deformation, welding and fracture. Many parameters such as milling type, ball to powder weight ratio, characteristics of the balls and their speed, milling atmosphere and temperature, and process control agent influence the stages of milling [8]. Upon milling of composite powders, the volume fraction, particle size and type of the reinforcement also affect the process [10, 11].

According to literature survey, there are many articles which have focused the production of composite materials using mechanical milling method. The goal of the current work is fabricating by mechanical milling of Al-Al<sub>2</sub>O<sub>3</sub> nanocomposite powders. The influence of

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milling time and weight fraction of  $\text{Al}_2\text{O}_3$  particles on the properties of prepared powders as well as on morphology, grain size and lattice strain of Al- $\text{Al}_2\text{O}_3$  composites were studied.

## 2. EXPERIMENTAL PROCEDURE

Commercial pure aluminum powder with an average particle size of  $80\mu\text{m}$  and 99.5% purity (see Figure 1) and  $\text{Al}_2\text{O}_3$  (99.5% purity  $80\mu\text{m}$  size) (see Figure 2) were used as raw materials for composite fabrication. Composite preparations were done from mixtures of  $\text{Al}_2\text{O}_3$  and Al with 2.5, 5 and 10 wt.%  $\text{Al}_2\text{O}_3$ , which were processed with a high energy Fritsch planetary ball mill using a hardened stainless steel vial and hardened steel balls of 5–10mm in diameter. The ratio of balls to powder weight and rotational speed were 20:1 and 250 rpm, respectively and kept the same for all experimental runs. Selected milling intensities were 5, 10, 15 and 20h. Stearic acid (3 wt. %) was also used as process controlling agent to prevent the agglomeration of the powder mixture during milling.

The powders produced after different stages of milling were investigated by using SEM Model Quanta 250 FEG (Field Emission Gun) attached with EDX unit (Energy Dispersive X-ray Analyses), with accelerating voltage of 30 kV. Morphology, size and particle distribution of the milled powders were quantified by visual basic software using several SEM images and their morphology was characterized by scanning electron microscopy.

X-ray diffraction (XRD) patterns were carried in a Rigaku-DXR 3000 X-ray diffractometer using  $\text{Cu K}\alpha$  radiation ( $\lambda=0.15406\text{nm}$ ) at 30 kV and 30 mA settings. The XRD patterns were recorded in the  $2\theta$  range of  $20\text{--}70^\circ$  with a step size of  $0.02^\circ$  and a scanning rate of 1.5 degs./min.

The crystallite size and lattice strain of milled aluminum powders were estimated by XRD peak broadening using William–Hall method as follows [12]:

$$B \cos \theta = \frac{0.9\lambda}{D} + 4\varepsilon \sin \theta \quad (1)$$

where  $B$ ,  $\lambda$ ,  $\theta$ ,  $D$  and  $\varepsilon$  are full width at half maximum (FWHM), the wave length, peak position, crystallite size and lattice strain, respectively.

## 3. RESULT AND DISCUSSION

**3.1. Particles Size Distribution** Figure 3 shows the variation of average particle size versus milling time for Al–10wt.%  $\text{Al}_2\text{O}_3$  powder mixture. It is observed that the trend shows a decrease in particle size as milling time increased. The effect of milling time on the particles size of ductile powders has been studied on

pure and composite powders [3]. In all cases a similar trend in powder particle size was observed, i.e. an initial increase is followed by a decrease, and then steady state in powder size was observed. This behavior can be attributed to the cold welding of initial ductile particles followed by work hardening, and thus the fracturing of powder particles. When the rate of cold welding and fracturing processes equals, the steady state is achieved. The presence of reinforcement particles mixed with aluminum changes the mechanical milling classification to a ductile-brittle component system.

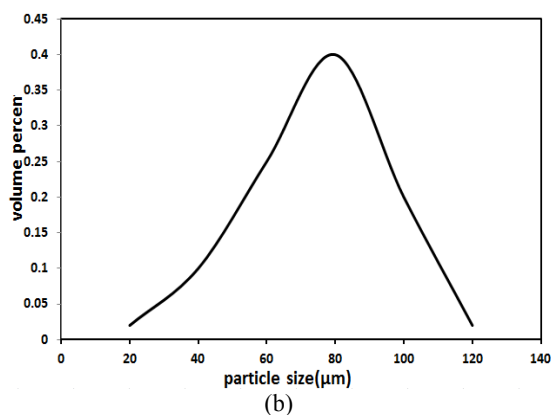
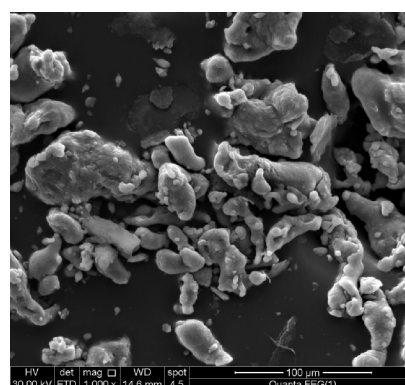
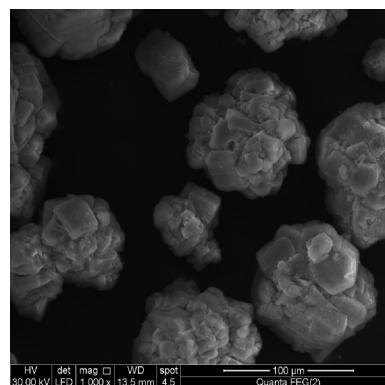


Figure 1. (a) SEM of Al pure powder (b) average particle size distribution



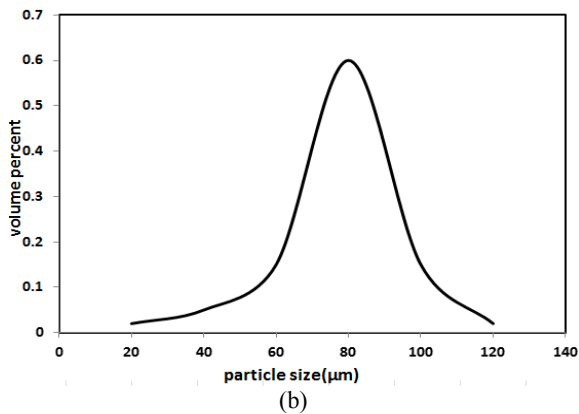


Figure 2.(a) SEM of  $\text{Al}_2\text{O}_3$  pure powder (b) average particle size distribution

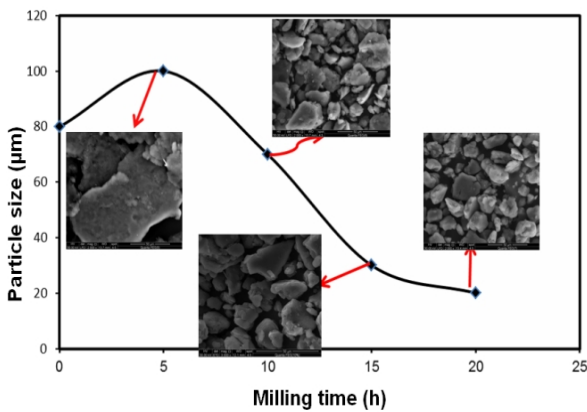


Figure 3. Variation of particles size as a function of milling time of Al-10% $\text{Al}_2\text{O}_3$  powder

In Figure 4, one possible scheme of the mechanical milling process of this system is proposed. In the first stage of milling, the ductile particles undergo deformation while brittle particles undergo fragmentation. Then, when ductile particles start to weld, the brittle particles come between two or more ductile particles at the instant of the ball collision. As a result, fragmented reinforcement particles will be placed in the interfacial boundaries of the welded metal particles, and the result is the formation of a real composite particle. As welding is the predominant mechanism in the process, the particles change their morphology by piling up the laminar particles. These phenomena, deformation, welding and solid dispersion harden the material and increase the fracture process, which also contributes to the equiaxed morphology. Welding and fracture mechanisms then reach equilibrium, promoting the formation of composite particles with randomly orientation interfacial boundaries. At the steady state, the microstructure undergoes a great refinement, and the interfacial boundaries are no longer visible by optical microscopy.

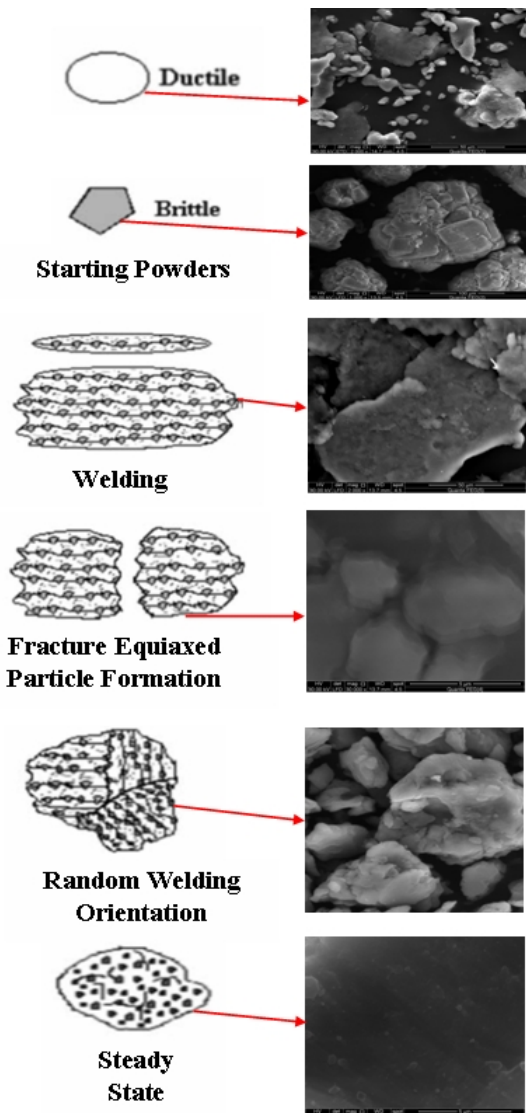
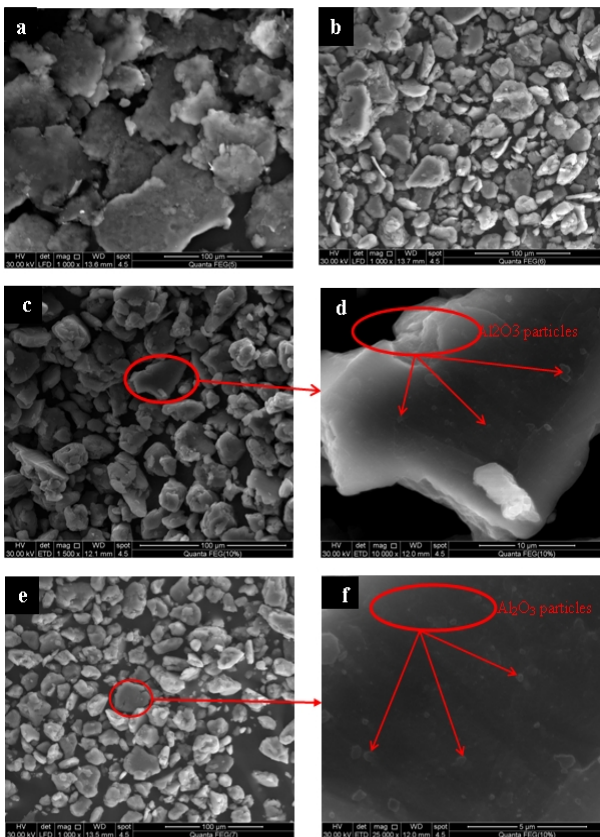


Figure 4. The various stages of a ductile-brittle system during mechanical alloying and experimental SEM images for Al- $\text{Al}_2\text{O}_3$  nanocomposite powder

**3. 2. Morphology Analysis** The change in morphology of powder particles was studied by SEM. Figure 5 shows the morphology of mechanically milled Al-10wt.% $\text{Al}_2\text{O}_3$  powder for different times. The powder particle size is changing with milling time, as a result of the two opposing factors of cold welding and fracturing of powder particles. While cold welding increases the particle size, fracturing reduces the size. In the early stages of milling time (5h), the powder particles are still soft and cold welding predominates. Consequently, the particle size increases (Figure 5a). Particle shape has become flattened due to cold working effects during milling [13]. When the milling time increased to 10h (Figure 5b), the particles appear like irregular particles with high aspect ratio whilst many small and irregular particles with relatively low aspect ratio also exist.



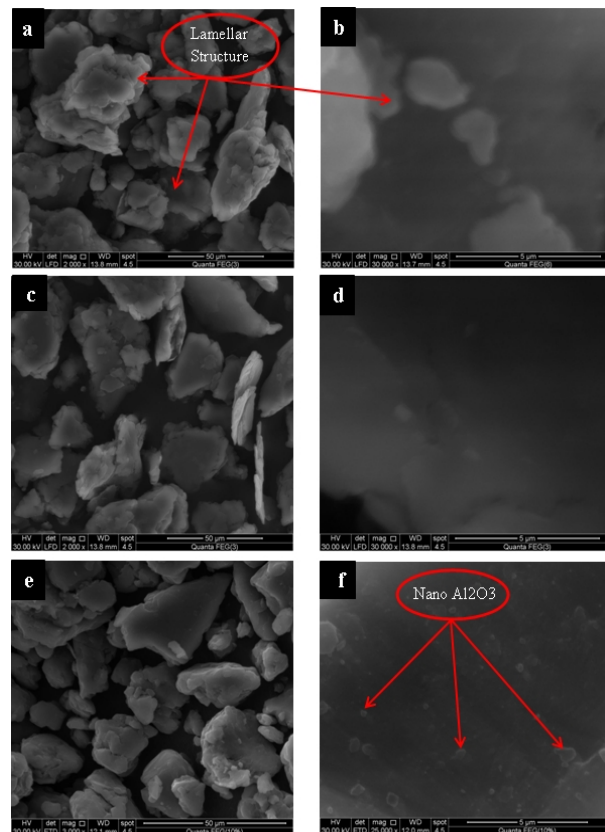
**Figure 5.** SEM micrograph of Al-10vol.%Al<sub>2</sub>O<sub>3</sub> powders milled for different times; (a) 5 h, (b) 10 h, (c) 15 h, (d) high magnification 15 h, (e) 20 h, (f) high magnification 20 h

After 15 hours, a progressive decrease in the aspect ratio of irregular particles can be noticed in Figure 5c, while almost the high magnification shows the formation of alumina particles on the grains of aluminum (Figure 5d). At longer milling time (20h), provide a balance between welding and fracture, with the morphological transformation from lamellar to equiaxed particle morphology (Figure 5e). It may be noted from the high magnification micrograph shown in Figure 5f that the distribution of Al<sub>2</sub>O<sub>3</sub> particles in the Al matrix is very uniform after 20 hours of milling. Further, even after the steady-state condition has been achieved, mixing between Al and Al<sub>2</sub>O<sub>3</sub> can continue to improve due to the dynamic process of welding and fracturing [13].

Figure 6 (a-f) shows the SEM micrograph taken to Al-Al<sub>2</sub>O<sub>3</sub> powder samples with different contents of the reinforcement after 20h milling. As it was expected, at Al-2.5%Al<sub>2</sub>O<sub>3</sub> alumina agglomerations are observed which have been welded on the aluminum surfaces (Figures 6a and b). These agglomerations would be removed by increasing alumina weight percentage [13]. The presence of alumina particles causes more work hardening due to ball milling. Consequently, there are more splitting of aluminum particles that result in

disappearing of agglomeration. Comparison of Figures 6d and b, indicates that agglomerations are very much smaller and less in Al-5%Al<sub>2</sub>O<sub>3</sub> than in Al-2.5%Al<sub>2</sub>O<sub>3</sub> nanocomposite. There is another noticeable difference between Al-2.5Al<sub>2</sub>O<sub>3</sub> and Al-5Al<sub>2</sub>O<sub>3</sub> nanocomposites. For Al-2.5Al<sub>2</sub>O<sub>3</sub>, as shown in Figure 6a, because of low alumina content, plastic deformation and consequently, cold welding are extremely high, so particle morphology is spherical in shape. On the contrary, as shown in Figure 6c, for Al-5%Al<sub>2</sub>O<sub>3</sub>, the particle shape is flake like.

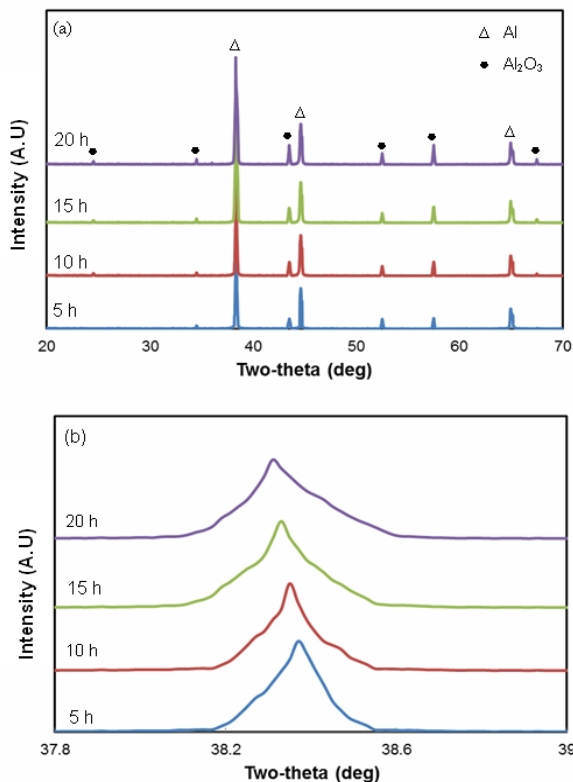
Also, there is smaller particle size in Al-5Al<sub>2</sub>O<sub>3</sub> than Al-2.5Al<sub>2</sub>O<sub>3</sub>, because fragmentation of the particles occurs more easily due to the enhanced work hardening. As it was mentioned, uniform distribution of alumina nanoparticles and disappearing of agglomeration are due to the alumina addition and splitting of aluminum particles by milling (Figures 6e and f). Moreover, the level of nanoparticles was increased by increasing their weight percentage from 5 to 10%. So, it can be said that in Al-10Al<sub>2</sub>O<sub>3</sub> nanocomposite, alumina particles dispersed very well in welded aluminum laminates without any agglomeration.



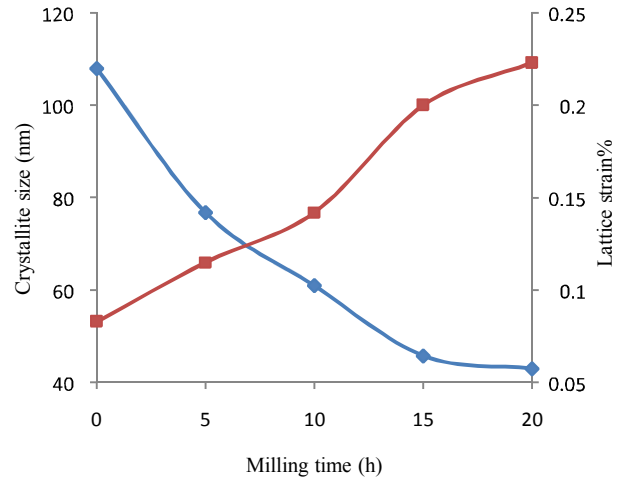
**Figure 6.** SEM micrograph of Al-Al<sub>2</sub>O<sub>3</sub> powders milled for 20 h; (a) Al-2.5%Al<sub>2</sub>O<sub>3</sub>, (b) high magnification of Al-2.5%Al<sub>2</sub>O<sub>3</sub>, (c) Al-5%Al<sub>2</sub>O<sub>3</sub>, (d) high magnification of Al-5%Al<sub>2</sub>O<sub>3</sub>, (e) Al-10%Al<sub>2</sub>O<sub>3</sub>, (f) high magnification of Al-10%Al<sub>2</sub>O<sub>3</sub>

**3. 3. Structural Analysis** Figure 7a shows the XRD patterns of Al-10wt.%Al<sub>2</sub>O<sub>3</sub> powders milled for various milling times (5, 10, 15 and 20h). Only two phases, i.e. Al and Al<sub>2</sub>O<sub>3</sub> were detected in the patterns of milled powders. With increasing milling time, the diffraction peaks of Al and Al<sub>2</sub>O<sub>3</sub> became broader and their intensities became weaker. Full width at half maximum (FWHM) measured from X-ray diffraction patterns for Al-10wt.%Al<sub>2</sub>O<sub>3</sub> powders is shown in Figure 7b.

Lines broadening were observed with increasing milling time. This is due to severe lattice distortion and grain size refinement [14, 15]. Consequently, reduced crystal size and an elevated strain energy stored inside particles could be obtained because of the severe plastic deformation introduced during ball milling. Noticeable shift in the position (i.e., 2 $\theta$ angles) of the Al peaks was observed in Figure 7b with the increase of milling time. The shift was measurable and this could be related to the dissolution of atoms related to minor matrix alloying elements and reinforcement particles in the lattice of aluminum during high-energy ball milling. This suggests the formation of Al-based solid solutions and an increase in the solute concentration as the milling time increases. These observations are in agreement with [11, 16].



**Figure 7.** X-ray diffraction patterns of Al-10wt.%Al<sub>2</sub>O<sub>3</sub> powder mixtures milled for different times



**Figure 8.** Effect of milling time on Al-10wt.% Al<sub>2</sub>O<sub>3</sub> crystallite size and lattice strain

Crystalline size and lattice strain are important parameters for milled powders, since they have a significant effect on both compacting of the powders during sintering process and properties of the finely obtained Al matrix strengthened by fine dispersions. The average crystal size of Al matrix in the composites was estimated using broadening of XRD peaks. The effect of milling time on crystal size and lattice strain of examined powder particles was presented in Figure 8. It is indicated that the crystallite size decreased with increasing milling time. The reason of this variation can be referred to this point that with increasing milling time, severe deformation on powder particles applies and leads to increase the crystalline defects such as point defects, dislocations and so on [8, 16]. Indeed, the presence of defects makes an increase in system energy and the lattice strain.

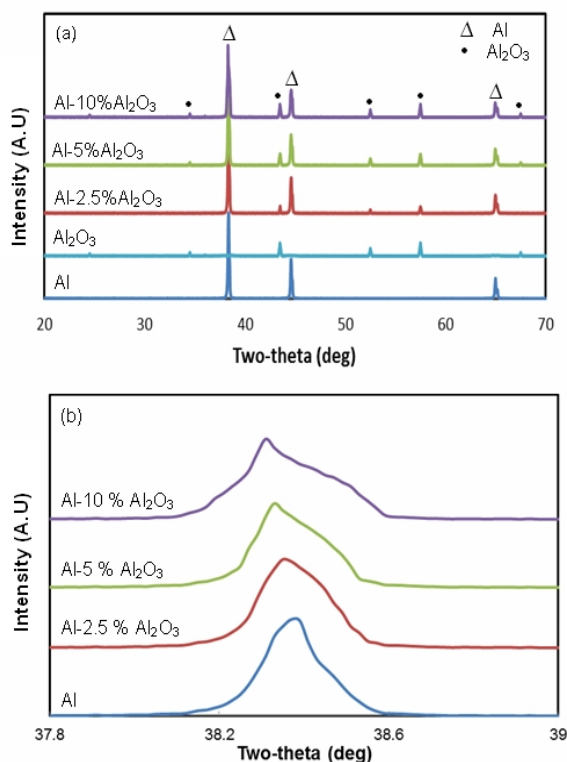
To compensate the mentioned effect, dislocations move to form a new orientation with lower energy-the so-called sub-grain boundary [8, 16, 17]. In the present work, the most intensive crystallite refinement occurs in the early stage of milling up to 10h. With prolonged time, the crystallite size of milled powders decreased slowly. The lattice strain increases with milling time due to the lattice distortion. The lattice strain increased while crystallite size reduced with increasing milling time due to distortion effect caused by dislocation in the lattice [18-20]. With increasing milling time, severe plastic deformation brings about a deformed lattice with high density of dislocations [18, 20].

Figure 9 shows X-ray diffraction patterns of milled powders with and without Al<sub>2</sub>O<sub>3</sub> addition for 20h. XRD peak intensities of the Al<sub>2</sub>O<sub>3</sub> phase noticeably increase with increasing weight fraction of Al<sub>2</sub>O<sub>3</sub> particles. Mechanical milling of soft aluminum powder is accompanied by sub- and micro-structural changes.

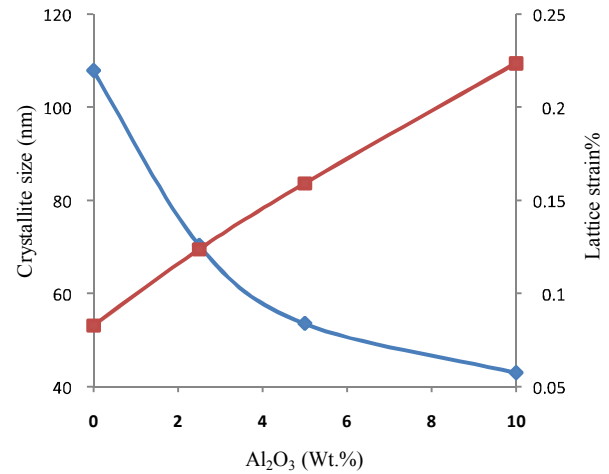
Severe plastic deformation of the particles can lead to grain refining, accumulation of internal stress, change of the lattice parameter, and formation of cell structure [8, 21]. Line broadening of the milled powders represents a decrease in the crystallite size and accumulation of lattice strain (Figure 9b).

It appears that the addition of ceramic particles to Al powder broadened the full-width at half-maximum (FWHM). A slight shift in the position of the XRD peaks was also noticed that can be related to the dissolution of impurities, particularly iron, in the lattice of aluminum [22].

Figure 10 shows the effect of  $\text{Al}_2\text{O}_3$  reinforcement addition on the particle size of the composite powder during mechanical milling. Since the mechanical milling of soft aluminum powder is accompanied by sub-micron structural changes, the plastic deformation of Al particles could be increased as the percentage of hard  $\text{Al}_2\text{O}_3$  increased. Hence, the intensity of fracture mechanism increased and dominated the soft ductile matrix particle-particle cold welding, resulting in the formation of very fine particles which are decreased by around two times compared with particle size of the Al matrix. Accordingly, the addition of hard reinforcement  $\text{Al}_2\text{O}_3$  particles would accelerate the fracture process of the matrix particles [23].



**Figure 9.** X-ray diffraction patterns of Al - $\text{Al}_2\text{O}_3$  powder mixtures milled for 20 h.



**Figure 10.** Effect of  $\text{Al}_2\text{O}_3$  weight fraction on crystallite size and lattice strain.

#### 4. CONCLUSION

The following remarks were concluded:

1. Al- $\text{Al}_2\text{O}_3$  nanocomposites have been fabricated using mechanical milling for different milling times up to 20h, in planetary ball mill. After milling, grain refinement took place and fine  $\text{Al}_2\text{O}_3$  particles were regularly distributed in the aluminum matrix.
2. The crystallite size decreased, while the lattice strain was found to be increased with increasing milling time due to distortion effect caused by dislocation in the lattice.
3. With increasing the  $\text{Al}_2\text{O}_3$  content, the crystallite size decreases and lattice strain increase for 20h milling due to the grain refinement process by adding alumina particles; this can be attributed to the generation of a high dislocation activity, owing to interaction between the hard particles and dislocations.
4. It was found that the milling stages include plastic deformation, micro-welding, and particle fragmentation. When hard  $\text{Al}_2\text{O}_3$  particles are added to aluminum powder, the fracture occurs earlier, and thus the steady-state condition, i.e. formation of equiaxed particles, is achieved after shorter milling time.

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Morphology

Particle size

تغییرات مورفولوژیک و میکروساختاری در طول آسیاکاری مکانیکی پودر Al مخلوط شده با ۲.۵، ۵ و ۱۰ درصد وزن ذرات Al<sub>2</sub>O<sub>3</sub> مورد مطالعه قرار گرفتند. آسیاکاری در آسیاب گلوله ایسیاره‌ای برای زمان‌های مختلف تا ۲۰ ساعت انجام شد. پودر کامپوزیت تولید شده با استفاده از الگوی پراش پرتو X (XRD) برای مشخص کردن اثر اندازه ذرات، درصد فاز دوم و زمان آسیاب بر اندازه دانه و کرنش شبکه آلومینیم بررسی شد. اندازه دانه آلومینیم تخمین زده شده با گسترش قله XRD با استفاده از فرمول ویلیامسون-هال انجام شد. تغییرات مورفولوژیک با استفاده از میکروسکپ الکترونی روبشی (SEM) مورد مطالعه قرار گرفتند. نتایج نشان می‌دهد که علاوه بر این از ذرات سخت Al<sub>2</sub>O<sub>3</sub> به تسریع فرآیند آسیاکاری، که منجر به سرعت بیشتر کارسختی و شکستن شبکه آلومینیم می‌شود. علاوه بر این، اندازه دانه‌های آلومینیم در طول آسیاکاری در حضور ذرات Al<sub>2</sub>O<sub>3</sub> کوچک‌تر می‌شود. نتایج نشان داد که اندازه دانه پودرهای آسیاکاری شده به حدود ۴۵nm با حضور قابل توجه از توده آگلومره می‌رسد. توزیع یکنواخت ذرات Al<sub>2</sub>O<sub>3</sub> به اندازه نانو در ماتریس Al را می‌توان با افزایش زمان آسیاب به دست آورد.

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