

Synthesis and Characterization of Al–Al₂O₃ and Al/ (Al₂O₃-ZrO₂) Nanocomposites Using High-Energy Milling

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Abstract

Aluminum metal matrix nanocomposites, Al-20 wt.% Al₂O₃, and Al-10 wt.% Al₂O₃-10 wt.% ZrO₂ systems, were synthesized by mechanical milling to obtain advanced engineering materials with new properties. A homogenous distribution of the reinforcements in the Al matrix was obtained after milling the mixed powders by a Planetary ball mill for a period of 45 h at a ball-to-powder ratio of 10:1. The uniform distribution of Al₂O₃ and Al₂O₃-ZrO₂ in the Al matrix was confirmed by characterizing these nanocomposite powders by SEM and XRD techniques. The powders were consolidated by cold pressing under 600 MPa pressure at room temperature followed by sintering at 600°C for 45 min under inert gas atmosphere. X-ray patterns were analyzed by using the Williamson–Hall treatment to determine the crystallite size and the lattice strain. Microhardness measurements and compression tests were performed to characterize the composite materials mechanical properties.

Keywords: Mechanical milling; Nanocomposites; Al-ceramic composites, Al/Al₂O₃/ZrO₂ nanocomposite, Mechanical properties, Sintering.

I. Introduction

Aluminum-based metal matrix composites (MMCs) produced by mechanical milling are used in the aircraft and automotive industries due to their light weight and high strength-to weight ratio. This causes Al matrix composites to find great attention in the last years [1,2]. Reinforcing the ductile aluminum matrix with stronger reinforcements like ceramics gives a combination of properties of both the metallic matrix and the ceramic reinforcement components resulting in improved properties of the composite [3,4].

High-energy ball milling is a powder processing method in which powder particles go through a repeated process of cold welding, fracturing, and rewelding [5]. These processes are repeated several thousands of times during the mechanical milling operation resulting in crystallite size decrease with processing milling time and uniform distributed particles in the metal matrix. From the advantages of using ball-milling process that, homogeneous nanocomposite powders can be produced from apparently immiscible elements and the drawbacks of the liquid metallurgy method for producing undesirable materials can be avoided because milling carried out at room temperature [6]. The aim of the present investigation is to synthesize and characterize aluminum reinforced with a uniform dispersion of specified weight fraction nanometer-sized alumina and alumina-zirconia particles and to investigate the effect of reinforcement type and milling speed in the properties of the produced nanocomposites.

II. Experimental Work

2.1 Milling and sample preparation

To synthesize two nanocomposite systems of (Al-20wt.% Al₂O₃) and (Al-10wt.% Al₂O₃ -10 wt.% ZrO₂). The materials used in the experiments were commercial aluminum powder with a particle size of (-210+90 μm), commercial alumina powder with particle size smaller than 44 μm and zirconia powder with particle size smaller than 38 μm. For the two systems; Planetary Monomill "Pulverisette 6" was used for the milling of the powder mixtures. Grinding is carried out under Argon gas atmosphere at 300 rpm milling rotation speed and ball-to-powder ratio of 10:1. Stearic acid was used as a process control agent (PCA) to prevent the aggregation of powders during milling. The duration of milling process is fixed at 30 min in order to avoid temperature rise and samples were taken at intervals of 7, 12, 15, 21, 30, 38 and 45 hours. To investigate the effect of milling speed and milling time on the properties of the composite, another two milling rotation speeds (200 and 400 rpm) were used in milling only for the powders of Al-Al₂O₃ nanocomposite system.

Consolidation of the milled powders was done in two stages including pressing and sintering of the compacts to achieve high dense samples. Pressing the powders was done in a hardened steel die, thus producing green compacts with 15mm diameter and 5mm height. The applied pressure was 600MPa pressure and five minutes of the load hold time. For sintering, the compressed powders were kept at 600°C for 45 min. To avoid oxidation of aluminum, all samples were protected using Nitrogen gas during

heating and then the samples were furnace cooled. The density was measured by a volumetric method. This method was employed through measuring the weight and dimensions of the compacts by using an accurate balance.

2.2 Structure analysis

XRD patterns were recorded from the asreceived and milled powders. During the powder milling process, the changes in peak shape are related to microstructural changes. Williamson–Hall (WH) [7] method is used to estimate the lattice strain and the crystallite size in the samples. In the WH treatment, the full width at half maximum (FWHM), β , is related to the crystallite size, D , and the distortion, ϵ , by the equation:

$$\beta^* = d^* \epsilon + 1/D \dots\dots\dots *$$

Where $\beta^* = \beta \cos \theta / \lambda$ and $d^* = 2 \sin \theta / \lambda$; θ is the Bragg angle and λ is the wavelength used. From the above equation, the intercept of the plot of β^* against d^* gives $1/D$ and the slope gives the strain. The experimental breadth of a given reflection was corrected for the peak breadth from the instrument by Voigt functions using the Originpro 8 software [8,9]. The powders were characterized for their microstructure and distribution of the reinforcements (Al_2O_3 & Al_2O_3 - ZrO_2) particles in the Al matrix using

a JEOL-JSM 5400 F scanning electron microscope (SEM) with a maximum magnification of 200,000X.

2.3 Mechanical characterization

Hardness measurement was performed on the sintered consolidated samples by using a laboratory MICROMETVickers hardness tester. Compression test determines the behavior of the material under compressive loads. The test specimens of 15 mm diameter and 22.5 mm height [$L/D = 1.5$] were loaded at a rate of 0.1 mm/min. From the diameter and the applied loading conditions, stress-strain response of the material is calculated.

III. Results and Discussion

3.1 XRD analysis, crystallite size, and lattice strain calculations

The X-ray diffraction patterns of the $Al-Al_2O_3$ and $Al-Al_2O_3-ZrO_2$ powders milled with 300 rpm rotation speed for different periods of time are shown in Figures 1,2. It can be seen that line broadening increases with milling time. The main contributions to the decrease in intensity and the broadening of the diffraction peaks are the grain size and the strain of the lattice, i.e., the reduction of crystallite size and lattice strain introduced by milling [9].

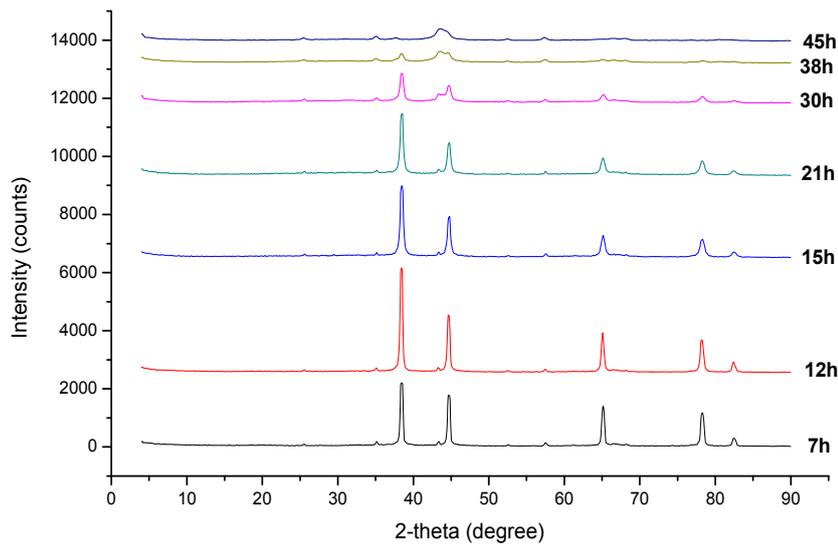


Fig.1: XRD patterns of $Al-Al_2O_3$ powders milled for different times; 7, 12, 15, 21, 30, 38 and 45 h.

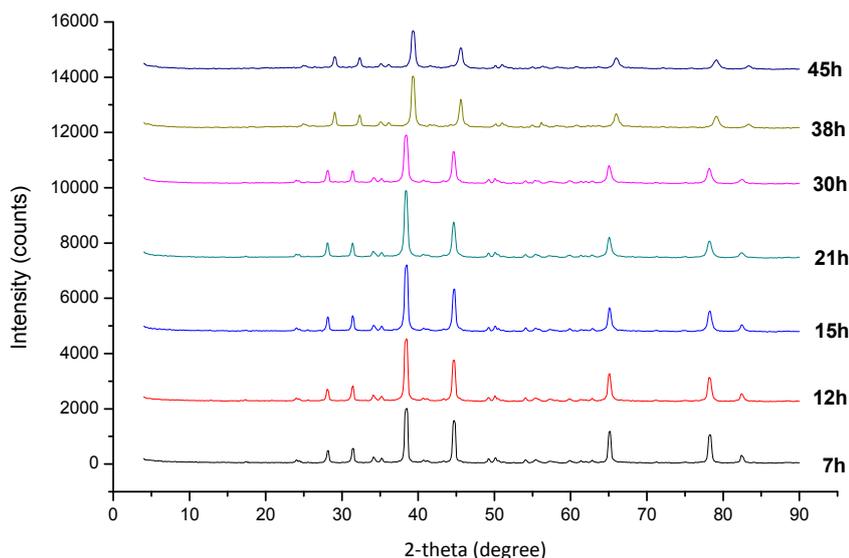


Fig.2: XRD patterns of Al-Al₂O₃-ZrO₂ powders milled for different times; 7, 12, 15, 21, 30, 38 and 45 h.

To estimate the crystallite size and the lattice distortion, an inspection of the shape of the diffraction peaks was performed. WH calculations procedure are carried out for every peak of the two nanocomposites samples of different times for Al and Al₂O₃ and for ZrO₂ in the Al-Al₂O₃-ZrO₂ nanocomposites.

milling time obtained by WH method for Al-Al₂O₃ and Al-Al₂O₃-ZrO₂ respectively. It can be seen that the rate of the grain refinement continuously decreases reaching, after 45h of milling, the value of 19.77 for Al-Al₂O₃ and 25.36 nm for Al-Al₂O₃-ZrO₂. On the other hand, the lattice strain shows a continuous increase to value of 1.16% for Al-Al₂O₃ and 0.139% for Al-Al₂O₃-ZrO₂.

3.1.1 Al crystallite size and the lattice strain values

Figure 3 shows the variation of the crystallite size and of the lattice strain of Al against

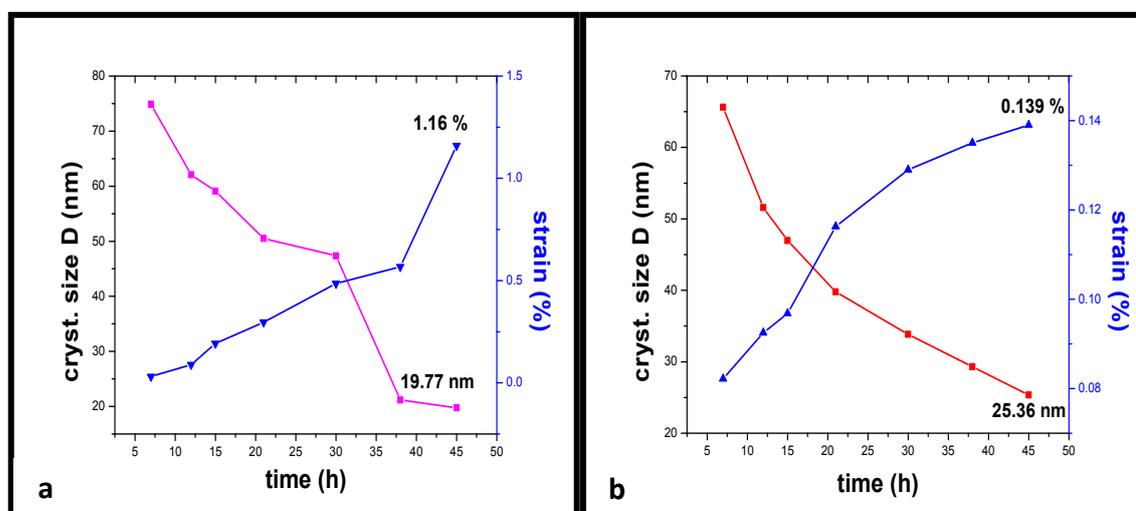


Figure3: Al crystallite size and lattice strain values for Al-Al₂O₃ and Al-Al₂O₃-ZrO₂ nanocomposite-300rpm ; 7, 12, 15, 21, 30, 38, 45 h. a) Al-Al₂O₃ nanocomposite b) Al-Al₂O₃-ZrO₂ nanocomposite.

3.1.2 Al₂O₃ crystallite size and the lattice strain values

The calculations procedure of different samples of the two nanocomposites to obtain the size and strain for alumina is as that in the Al. The

variation of crystallite size and lattice strain of the alumina against milling time obtained by WH method are shown in Figure 4.

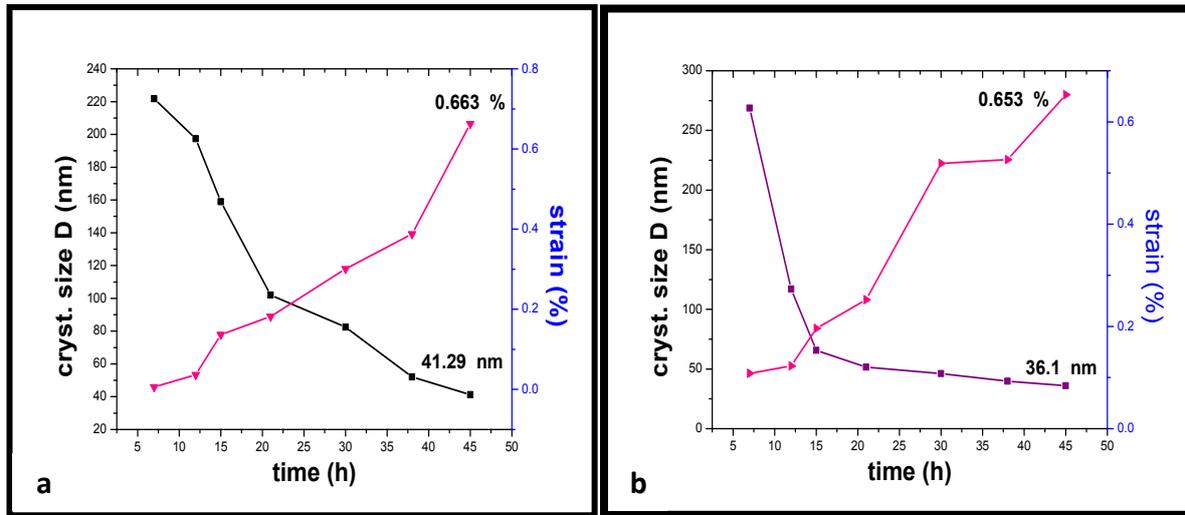


Figure4: Al₂O₃ crystallite size and lattice strain values for Al-Al₂O₃ and Al-Al₂O₃-ZrO₂ nanocomposite-300rpm ;7, 12,15, 21, 30, 38, 45 h. a)Al-Al₂O₃ nanocomposite b)Al-Al₂O₃-ZrO₂ nanocomposite.

It can be seen that for alumina in Al-Al₂O₃ powders the grain refinement reaches a value of 41.29 nm, and the lattice strain shows a continuous increase to value of 0.663 %, while for Al-Al₂O₃-ZrO₂ powders, the grain refinement for alumina reaches a value of 36.1 nm while the lattice strain reaches a value of 0.653 %.

3.1.3 ZrO₂ crystallite size and the lattice strain values

Figure 5 shows the variation of the ZrO₂ crystallite size and lattice strain against milling time obtained by WH method. It can be seen that for ZrO₂ in Al-Al₂O₃-ZrO₂ system, the rate of the grain refinement continuously decreases reaching, after 45 h of milling, a value of 58.14 nm. On the other hand, the lattice strain shows a continuous increase to values of 0.33 %.

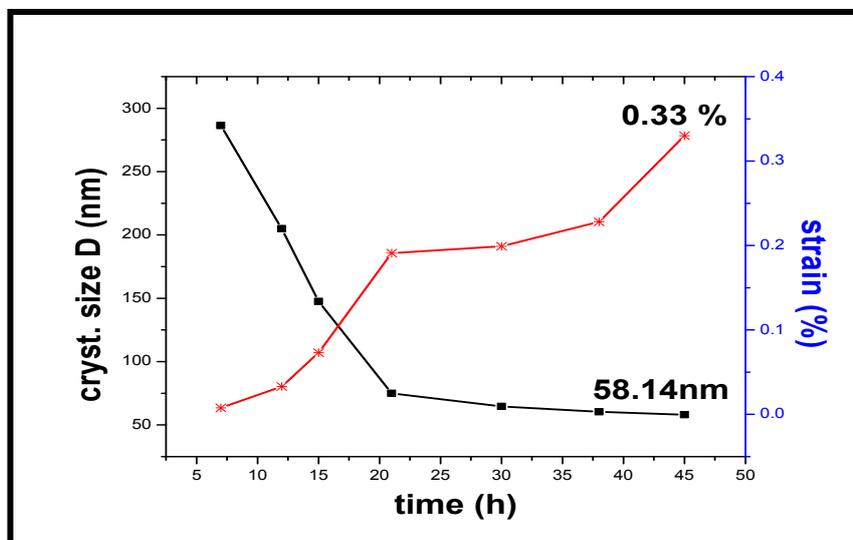


Figure5 : ZrO₂ crystallite size and lattice strain values against milling time for Al-Al₂O₃-ZrO₂ nanocomposite (7, 12, 15, 21, 30, 38, 45 h).

3.2 Milling speed effect on crystallite size and the lattice strain values

Two different experiments were carried out at the same operating conditions, but with two different rotation speeds; 200 and 400rpm to study milling speed effect. Milled samples were taken out at 3, 7, 12, 15, 30, 38, 45 and 60h for the 200 rpm rotation speed and at 3, 5, 7, 12, 15, 21, 27 and 30h for the 400 rpm rotation speed. Based on XRD results, the peaks tend to broaden as the milling time increases. Therefore, the X-ray line broadening analysis has been used to characterize the microstructure in terms of crystallite size and lattice strain. WH calculations procedure are carried out for every peak of the two nanocomposites samples of different times for Al and Al₂O₃. The results of

crystallite size and lattice strain calculations for Al-Al₂O₃ powders (200 and 400 rpm) are as follows;

3.2.1 Al-Al₂O₃- 200rpm

Figure 6 shows the variation of the Al and Al₂O₃ crystallite size and lattice strain against milling time obtained by WH method. In this figure, it can be seen that the rate of the Al grain refinement continuously decreases reaching, after 60 h of milling, a value of 22.1nm. On the other hand, the lattice strain shows a continuous increase to values of 0.20% for Al₂O₃ and the rate of Al₂O₃ grain refinement decreases reaching, after 60 h of milling, 55.2 nm and the lattice strain increases reaching 0.15 %.

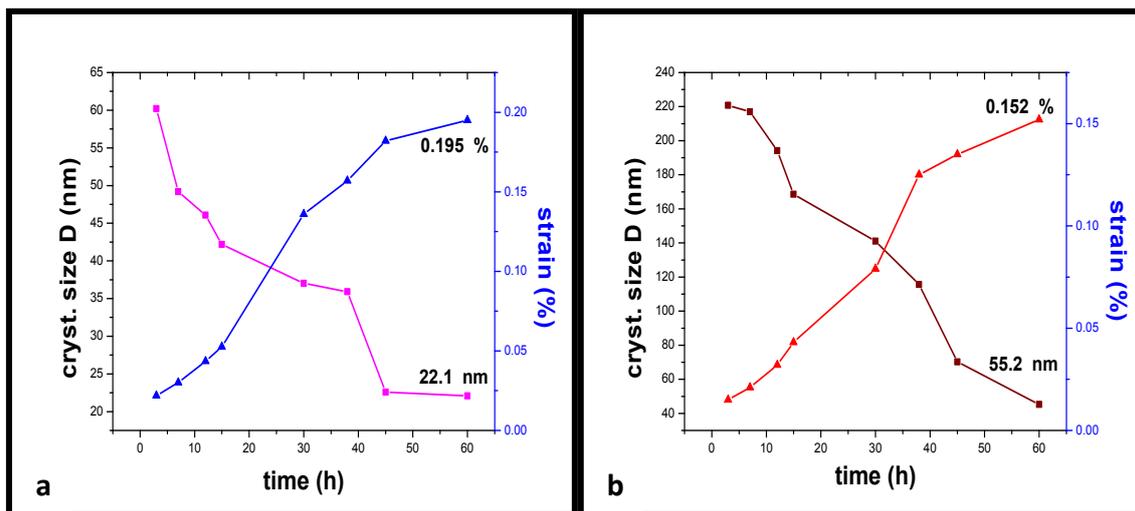


Figure6: Al & Al₂O₃ Crystallite size and strain for Al-Al₂O₃ -200 rpm (3, 7, 12, 15, 30, 38, 45, 60 h),
 a) Al b) Al₂O₃

[3.2.2 Al-Al₂O₃-400rpm

Figure 7 shows Al and Al₂O₃ crystallite size and strain values for different times of 300rpm nanocomposite. It can be seen that Al crystallite

size decreases to 29.3 nm and the lattice strain increases to 0.8 %. For Al₂O₃ the crystallite size decreases to 47.9 nm and lattice strain increases to 0.3 %.

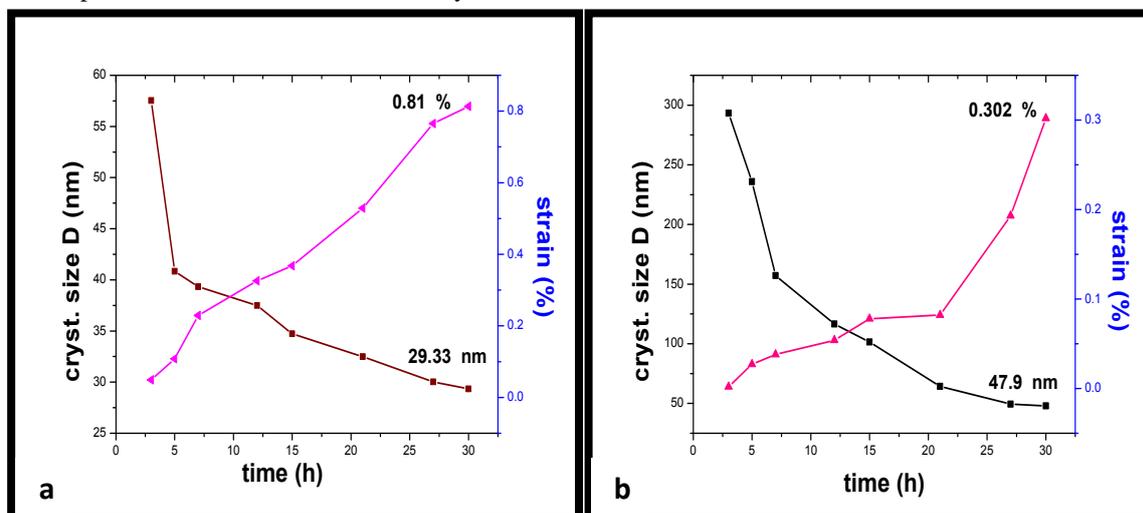


Figure7: Al & Al₂O₃Crystallite size and strain for Al-Al₂O₃ -400 rpm (3, 5, 7, 12, 15, 21, 27, 30 h)
 a) Al b) Al₂O₃

In Al-Al₂O₃nanocomposite system three sets of powders with three different milling speeds for which the grain size is calculated by using WH method. Figure 8shows Al Crystallite size values (22.1, 19.77, 29.33 nm for 200, 300, 400 rpm

respectively) and also, Al₂O₃crystallite size values (55.2, 41.29, 47.9 nm for 200, 300, 400 rpm respectively). These values give an indication about the effect of milling speed on the crystallite size.

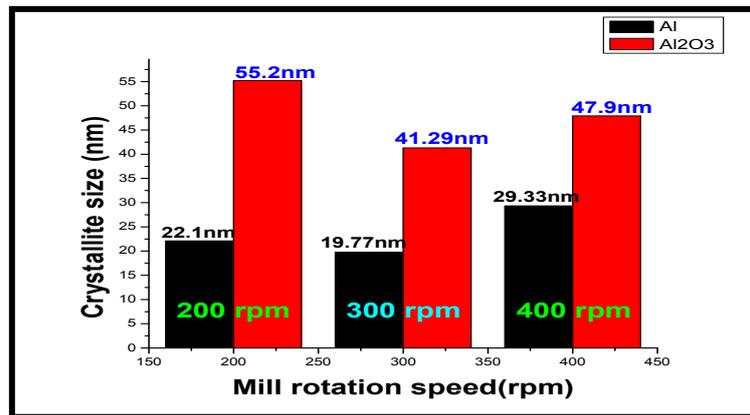


Figure 8: Al and Al₂O₃ Crystallite size values with three different milling speeds in Al-20 wt.% Al₂O₃nanocomposites

Milling speed has an important role in the process of mechanical milling. The higher the speed the higher is the rate of energy transfer to the powder and lower is the milling time to achieve the desired size. However, there is a limit to the maximum speed that can be used. For example, at higher speeds the balls tend to stick to the walls of the vial and thus are incapable of transferring energy to the powder particles. At higher speeds the temperature of the system may increase and may accelerate the transformation process and results in crystallization of the amorphous phase [10], thus the maximum speed selected should be lower than this critical value. From Al-Al₂O₃nanocomposite system, it is found that 300 rpm milling rotation speed is the proper milling speed to achieve the desired size, and it is used in Al-Al₂O₃-ZrO₂ system.

The crystallite size decreases due to the local deformation from milling, which accelerating the work hardening of the matrix and thus, increase the grain refinement. The lattice strain increases due to plastic deformation which increases the defect

density as well as dislocations and lattice defects in the initially defect-free material[8,11].

It is noticeable from crystallite size values that crystallite size of alumina is bigger than that of Al for the three rotation speeds. That is may be related to a degree of coverage of the reinforcement (Al₂O₃) particles by the aluminum, i.e., encapsulation of reinforcement particle with Al. This explanation is in good agreement with others[12,13].

3.3 Microstructural observations

SEM analysis was applied on the milled powders to check the uniform distribution of the reinforcement particles in the Al matrix. SEM analysis for the Al-Al₂O₃-ZrO₂ milled at 300rpm was studied elsewhere, by Dohiem et al.[14]. So, we will suffice by mentioning SEM analysis for the Al-Al₂O₃, milled at 300 rpm. The samples were taken for analysis after different milling times. Figure 9 shows the SEM image of Al-Al₂O₃ powder particles before milling i.e. at 0 hours milling time. The agglomerated alumina particles are observed. This agglomeration can be removed by increasing the milling time.

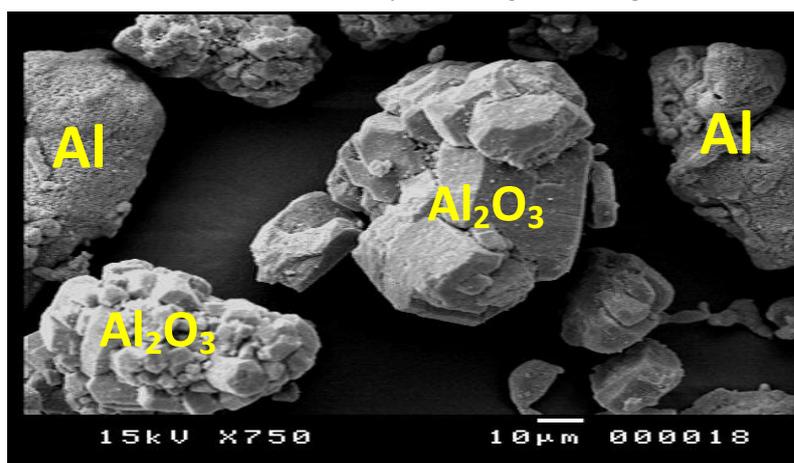


Figure 9: SEM image of Al-Al₂O₃-300rpm powder particles before milling (0 hours)

Figure 10 shows the SEM images of Al- Al_2O_3 powder particles at different milling times for 300 rpm milling speed. The powder particle size is changing with milling time, at 7 hours milling time, Figure 10 (a), the agglomerations of alumina are still observed, but they are smaller. It should be noted that at this milling time, the Al and Al_2O_3 particles are under deformation and cold working, so flattened particles were formed. This is because Al particles are soft and their sizes are increased by cold welding which becomes predominant and leading to an increase in particles size.

After 30 hours milling time, a change in particles morphology from flattened to flake-like occurred. At this milling time, due to work hardening

of the ductile Al powders, along with cold welding of the particles, fragmentation of particles happened with particle size reduction. Also, alumina particles have been moved in the cold welded Al and confined between them so that, welded layers of Al and Al_2O_3 were obviously observed at this stage and there are alumina clusters surrounded by aluminum particles as shown in Figure 10 (b).

Figure 10(c) shows the resulted microstructure, at this milling time alumina clusters have been disappeared and distributed uniformly. Nanometer size of alumina particles are covered by Al particles. The powder particles were more uniform in size compared to the early stages of milling.

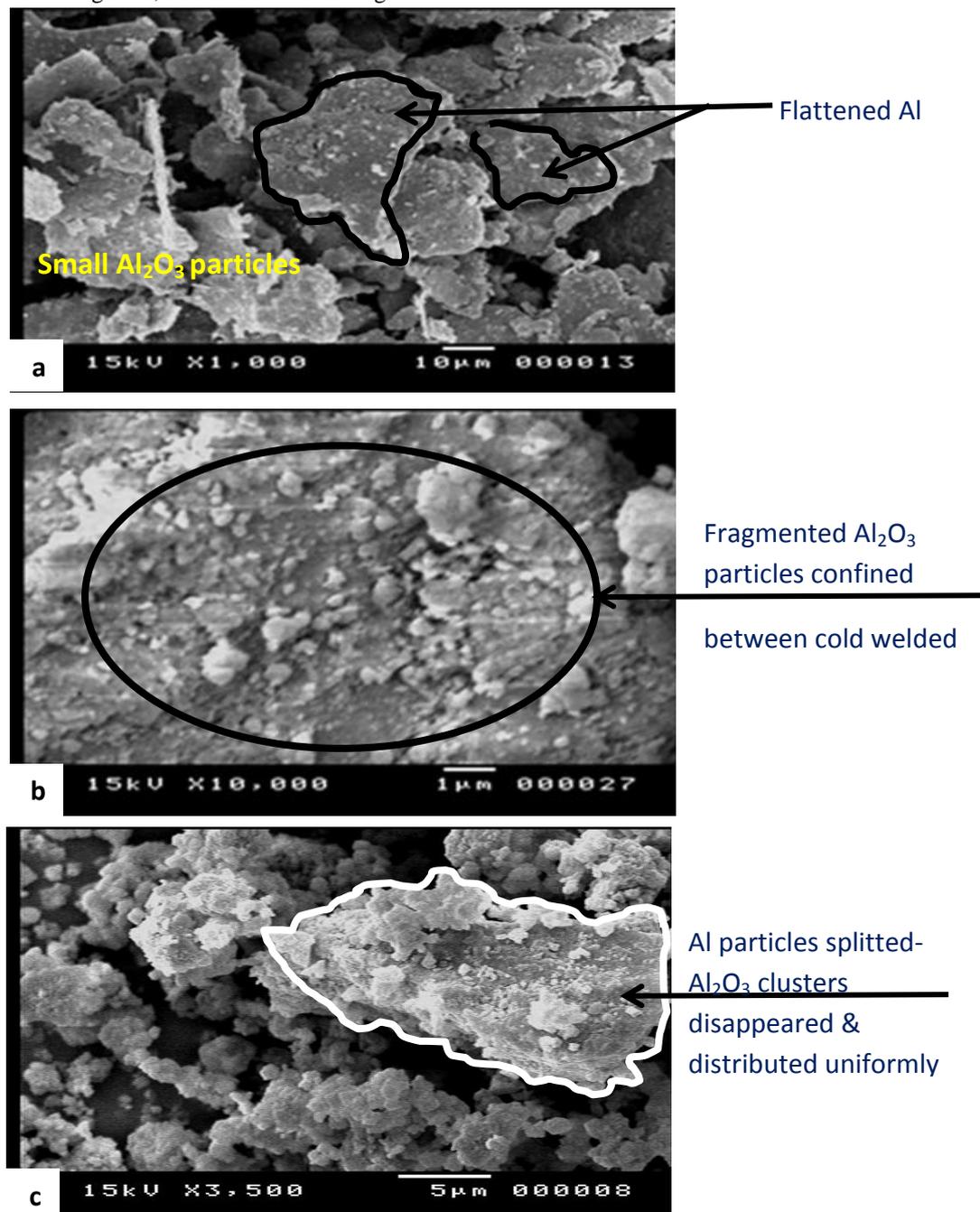


Figure 10: SEM micrographs of Al- Al_2O_3 powders - 300rpm after (a) 7 h, (b) 30 h, (c) 45h.

3.4 Characterization of the consolidated powders

3.4.1 Density measurement

The density was measured by a volumetric method before and after sintering. Measured density was compared to the theoretical density which

calculated by using rule of mixture according to the weight fraction of powders[15]. The results of density calculations for the pressed samples (Al-Al₂O₃ and Al-Al₂O₃-ZrO₂) before and after sintering are indicated in table 1.

Table 1: The results of density calculations for the compacted samples before and after sintering.

Sample		Measured Density (g/cm ³)	Theoretical density (g/cm ³)	Relative density (%)
Al-Al ₂ O ₃ 300rpm	Before sintering	2.5966	2.9511	87.9863
	After sintering	2.6553	2.9511	89.9754
Al-Al ₂ O ₃ -ZrO ₂ 300rpm	Before sintering	2.7995	3.1289	89.4733
	After sintering	2.8331	3.1289	90.5462

As it is seen the density of the green compacted powders is lower than the density of sintered compacted powders. This increase may be attributed to reduction of pore space during sintering process and/or due to grain refining which can play an important role in obtaining higher density materials. This result is in agreement with results which also observed by others [6,16].

3.4.2 Mechanical characterization

Mechanical testing is applied on the sintered consolidated specimens to make an evaluation of mechanical properties. That is to estimate the effect of milling speed and milling time on the mechanical behavior of the obtained Al-Al₂O₃ nanocomposites and also to estimate the influence of using both the

alumina and zirconia particles as reinforcement for aluminum matrix on the properties of the formed hybrid composite material.

3.4.2.1 Microhardness

The values of microhardness, after compaction and sintering processes, for Al-20 wt.% Al₂O₃ nanocomposite system are; 78, 83 and 80 HV at 200, 300 and 400 rpm respectively. For the Al-10 wt.% Al₂O₃-10 wt.% ZrO₂ system at 300 rpm, the value is 115 HV. Microhardness of aluminum is about 30 HV. From the obtained results (Figure 11), it is clear that; microhardness increased after the addition of reinforcement particles and milling which can be referred to some reasons. Increasing the high energy ball milling duration increases the

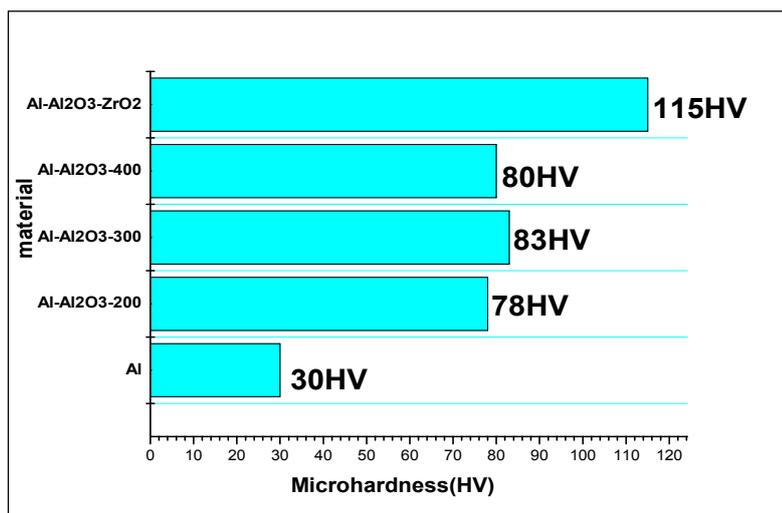


Figure 11: Microhardness values of Al, Al-Al₂O₃ and Al-Al₂O₃-ZrO₂ nanocomposites

deformation and work hardening of powders. Moreover, the presence of hard reinforcing particles might enhance the work hardening rate of the matrix resulting in increase in hardness. The general trend was observed by others [11,17,18].

3.4.2.2 Compression test

Compression tests were performed and from the diameter and the applied loading conditions, stress-strain response of the material is calculated. The point of yielding is determined by drawing a line parallel to the initial linear region of the curve at a strain offset of 0.2 % strain. The stress corresponding

to the intersection of this line and the stress-strain curve as it bends over in plastic region is defined as yield strength (σ_y). Compression test for sintered samples, Al-20wt% Al_2O_3 , 300rpm and Al-10wt% Al_2O_3 -10wt% ZrO_2 , 300rpm are carried out.

For Al- Al_2O_3 - ZrO_2 nanocomposite, the compressive strength (CS = 760 MPa) and the yield strength is calculated as ($\sigma_y = 630$ MPa).For Al-20wt.% Al_2O_3 nanocomposite, CS=530 MPa and $\sigma_y=405$ MPa,while CS =300MPa and $\sigma_y=200$ MPa, for Al.

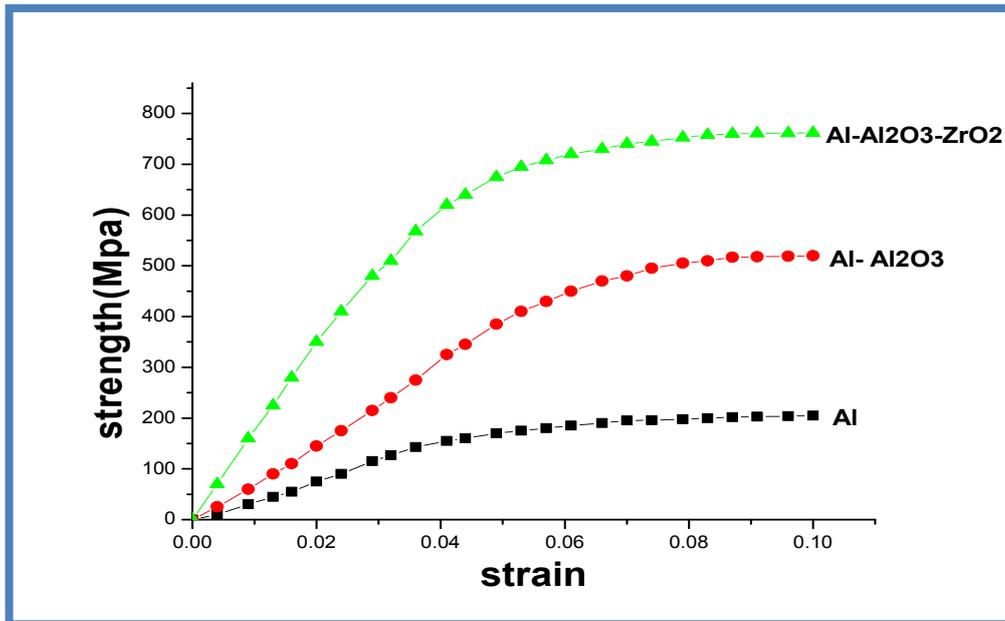


Figure 12:Engineering stress-strain response of different nanocomposite systems and Al

Figure 12 shows the maximum stress on the stress-strain curve for Al- Al_2O_3 and Al- Al_2O_3 - ZrO_2 nanocomposite. The increase in the composite strength is influenced by a some factors such as: milling, consequent deformation and work hardening, grain refinement and sub grains production because of the increase in the dislocations density. The general trend was observed by others [19,20].

Similar to microhardness results, a higher gain in strength is obtained, with 20wt.% Al_2O_3 addition in Al- Al_2O_3 nanocomposite system, strengthens the aluminum by about 230 MPa. While (Al_2O_3 - ZrO_2) addition increased aluminum strength by about 500 MPa. Also a significant increase in Young's modulus is clearly observed as shown in Figure 12.

The significant increase in hardness and compressive strength is attributed to that Al_2O_3 and ZrO_2 nanoparticles are hard and non-deformable, they are working as a barrier to the dislocation movement leading to an increase in dislocation density and according to Orowan bowing mechanism this leads to dislocation multiplication.

IV. Conclusions

Al-20 wt. % Al_2O_3 and Al-10 wt. % Al_2O_3 -10 wt. % ZrO_2 nanocomposites are synthesized by milling the powder mixture with a uniform distribution of thereinforcement phase in Al matrix. The crystallite size decreases with milling time to steady values of 22.1, 19.77 and 29.33 nm after 60, 45, 30h of milling for Al- Al_2O_3 powders milled with

200, 300 and 400 rpm respectively and the value of 25.36 nm for Al- Al_2O_3 - ZrO_2 powder after 45h milling. The values of microhardness, for Al- Al_2O_3 nanocomposite system are; 78, 83 and 80 HV at 200, 300 and 400 rpm respectively. For the Al- Al_2O_3 - ZrO_2 system at 300 rpm, the value is 115 HV. Use of zirconia raised the microhardness by about 40 %. Compressive strength has a value of 530 MPa and yield strength of value 405 MPa for Al- Al_2O_3 sample. For Al- Al_2O_3 - ZrO_2 sample compressive strength = 760 MPa and the yield strength = 630 MPa. Use of ZrO_2 increased the yield strength by about 56 %. Advanced material of Al- Al_2O_3 - ZrO_2 hybrid nanocomposite with improved properties could be fabricated, which may be the first study on such nanocomposite hybrid systems with achieved new properties.

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