Preparation and Characterization of Al Matrix Composites Reinforced with (20-x) wt.-% Al_2O_3 - (x) ZrO_2

Mahmoud F. Zawrah [a,*], Mohammed A. Taha [b], F. A. Saadallah[b], A.G. Mostafa[c], M.Y. Hassan[c], Mahmoud Nasr [b]

[a] Ceramics Department, National Research Center , Dokki, Cairo, Egypt.
[b] Solid State Physics Department, National Research Center, Dokki, Cairo, Egypt.
[c] Physics Department, Faculty of Science, Al-Azhar University, Nasr City, Cairo, Egypt.

Abstract

Metal matrix nanocomposites composed of Al-(20-x) wt.-% Al_2O_3 - (x) ZrO_2, x=0, 1, 2 & 4, were prepared by mechanical alloying technique. The powders' mixture was milled in a planetary ball mill up to 7h. The effect of milling time on properties of obtained powders was studied by using X-ray diffraction analysis (XRD) and transmission electron microscopy (TEM) to investigate phase composition, crystal size and morphology of the milled powders. In order to study the sinterability, the milled nanocomposite powders were cold pressed and sintered in argon atmosphere at different firing temperatures i.e. 300, 370 and 470°C for 1h. Physical properties, namely, bulk density and apparent porosity of sintered bodies were determined by Archimedes method. Phase identification and microstructure of the sintered composites were investigated by using XRD and scanning electron microscope (SEM) as well as energy dispersive spectrometer (EDS). Microhardness of sintered composite was also examined using Vickers hardness indentor. The results were discussed in terms of the effect of milling time on the properties of the prepared powders and sintered composites. The results revealed that the grain size of milled powders was about 30 nm with a noticeable presence of agglomerates. Uniform distribution of nano-sized alumina-zirconia particles in the aluminum matrix could be achieved with increasing milling time. The density of the sintered composites was affected by milling time of starting powders and firing temperature. It increased with increasing milling time and firing temperature. Microhardness of sintered bodies was found to be progressively increased with increasing of milling time of starting powders.

Key words: metal-matrix composites (MMCs); mechanical properties; mechanical alloying

---

***Corresponding author:** M.F. Zawrah, National Research Center, Al-Behooth St., Cairo, Egypt, Email: mzawrah@hotmail.com, Fax: +20233370931.

1. Introduction

Owing to low density, low melting point, high specific strength, high hardness, high-specific elastic modulus and high thermal conductivity of aluminum, it has been used as engineering material in the aerospace, automobile industries, electric wires and sports **machines [D.B. Miracle, D.B et al 2005; Torralba, G.M et al 2003; Woo. K et al 2007].** A wide variety of reinforcement particulates such as SiC, B_4C, Al_2O_3, ZrO_2, AlN, Si_3N_4, TiC, TiO_2, TiB_2 and graphite have been used to reinforce aluminum**[Fogognolo, J.B et al 2003] .**

High energy ball milling is a simple and useful technique for attaining a homogeneous distribution of inert fine particles within a fine grained matrix [**Prabhu, B et al 2006; Cullity, B.D et al 2001; Estrada-Guel, I et al 2009].** During ball milling, two essential processes occur, cold welding between the different particles and fracturing of cold welded particles due to high energy collision **[Lu, L et al 1999].** The cold welding minimizes diffusion distance between atoms of different components. The fracturing of welded particles impedes the clustering of particles promoting transfer of high ball collision energy to all particles and produces new clean surfaces without the oxide layers accelerating diffusion **[Lu, L et al 1999; Gubicza, J et al 2004; Rosenberger, M.R et al 2005].** Reinforcing of ductile Al matrix with hard particles such as oxides provides a suitable combination of the properties of both phases which, in turn, results in an improvement of physical and mechanical properties of composites **[Fogognolo, J.B et al 2003; Zebarjad, S.M et al 2006; Suryanarayana, C et al 2013; ; Zebarjad, S.M et al 2007] .** On the other hand, it has been shown that nano-crystalline matrices strengthened by nano-sized reinforcement are expected to have much better microstructural stabilities and performance than nano-crystalline materials **[Fogognolo, J.B et al 2003],** because of the concurrence of strengthening by both grain boundary and nanoparticle reinforcements **[[Kaufman, J.G 2002; Khakbiz, M et al 2009; Liu, Y.Q et al 2009]** . Uniform dispersion of fine reinforcements and a fine grain size of the matrix contribute to improve the mechanical properties of the **composite [Khakbiz, M et al 2009; Hassan, S.F et al 2008] .** The main goal of this work is to investigate the effect of milling time on the properties of Al - (20-x) wt.-% Al_2O_3 - (x) ZrO_2, x=0, 1, 2 &4 composite powders and sintered bodies. The effect of milling time on the crystallite size, lattice strain, relative density, apparent porosity, microhardness, and morphological properties was considered.

2. Materials and Methods

Firstly, a mixture of Al and Al_2O_3 powders with purities 99 & 98.2% and average particle sizes ≥74 & 2μm, respectively, were transferred into the milling machine (Desk-top high energy planetary ball mill, type MTI SFM-1 (QM - 3SP2)) to prepare Al-20wt.-%Al_2O_3 nanocomposite at different milling times (1,3,5 and 7h). Stearic acid was also taken as process controlling agent to prevent agglomeration of the powder mixtures

during milling. The milling process conditions were: ZrO_2 and Al_2O_3 balls with different diameters (6-20mm), 500 rpm rotating speed and 10:1 ball-to-powder weight ratio.

Secondly, in order to examine the properties enhancement of Al-Al_2O_3 composite, different amounts of zirconia powder (1, 2 & 4 wt.-%, average particle size; 35 nm) was added on the starting mixture (Al - 20wt. % Al_2O_3 at the expanse of alumina and was milled for 7 h.

For the milled powder, X-ray diffraction data was collected at ambient temperature in step scanning mode using a computerized controlled X-ray diffractometer (PAnalytical Empyrean, Netherlands) with Cu k_α- radiation (λ k_α= 1.5406 Å) operated at 30 mA and 45 kV. The powder diffraction pattern was scanned in 2Θ range of 20-80° with scan step 0.013 and counting time 20 s/step. The data obtained by XRD was used to calculate the crystallite size and lattice strain. Transmission electron microscope (TEM) type JEOL JEM-1230, operating at 120 kV and attached with a CCD camera, was employed to investigate the morphology and particle size of milled powders at different milling times.

The composite powder was compacted into small compacts of desired size at room temperature. Specimen of 10 mm diameter and 4 mm height were compacted in a hardened steel pre-compaction die set using hydraulic compressor at 10 MPa. The compacted samples were sintered at different temperatures i.e. 300, 370 and 470^oC, in argon atmosphere for one hour and heating rate 8^oC/min.

The relative density and apparent porosity of the sintered samples were determined by Archimedes method. Theoretical density of compacted samples was calculated using the simple rule of mixtures, considering the fully dense values of Al and Al_2O_3 are 8.96 and 3.95 g/cm^3, respectively. An optical imaging was performed for all samples in order to support the structural study. Optical microscope model OLYMPUS-BX51, equipped with digital DP12cam was used. Scanning electron microscope (SEM), attached with energy dispersive spectroscopy unit (EDX), model "Philips XL30" was also used to investigate the microstructure of sintered samples.

The reinforcement behavior of sintered composites was estimated by microhardness measurement (Vickers hardness machine-model: Shimadzu corporation hardness tester) by applying 1.961N load for 10 sec. during measuring hardness. The hardness value of the investigated materials was measured as average of 5 readings along the cross section surface of the specimens.

3. Results and Discussion

3.1. Characterization of the prepared powders

3.1.1 Phase composition of prepared powders

XRD patterns of mechanically alloyed Al-20wt. % Al_2O_3 composite milled for 1, 3, 5, and 7h are shown in Figure 1. The expected two phases Al and Al_2O_3 are obtained and compared with the data in cards (88-0826&03-0932) [18-19][Youssef, K.M et al 2006; Liu, R.S et al 1997. With increasing milling time, the peaks tend to get broadened

associated with weakens in their intensities. Similar results were reported for Al-Al_2O_3 composite [Moustafa, M.M et al 2013; Aboraia, M.S et al 2013].

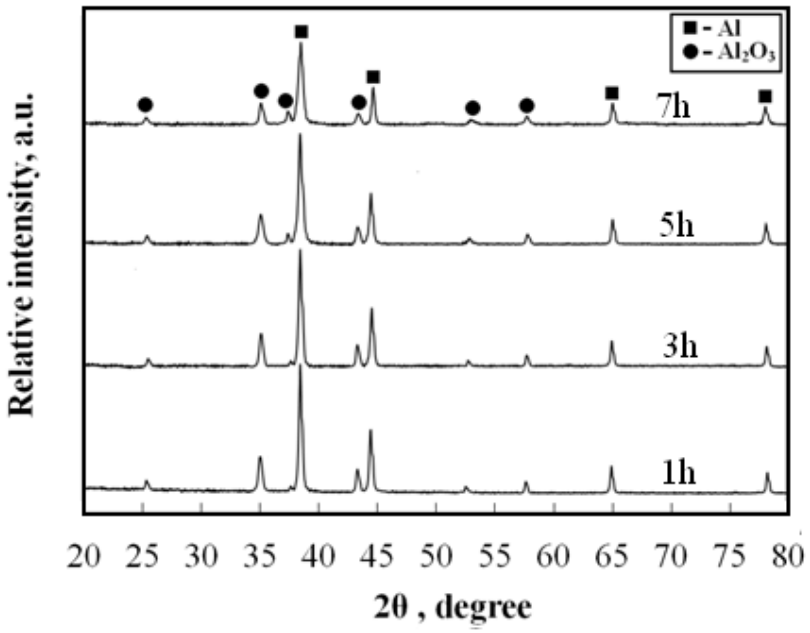


Fig. 1: XRD patterns of mechanically alloyed Al-20wt.-% Al_2O_3 composites milled at 1, 3, 5, and 7h.

Fig. 2 shows full width at half maximum (FWHM) calculated from X-ray diffraction patterns for Al-20wt.-% Al_2O_3 powders. The broadening in peaks with increasing milling time is due to the severe lattice distortion and grain size refinement **[Rajkovic, V et al 2008; Lonnberg, B et al 1994] .**

Fig. 3 depicts crystallite size and lattice strain of milled powders calculated from XRD peak broadening, versus milling time. It is indicated that the crystal size (D) decreases with increasing milling time (t) according to the equation: $D=Kt^{-2}$, where K is a constant **[Tousi, S.S.R et al 2009; Suryanarayana, C 2001].** The reduction happened in Al crystal size and an elevated strain energy stored inside particles could be obtained because of the severe plastic deformation introduced during ball milling by hard Al_2O_3 particles. It can be, in a part, attributed to hindering the dislocation movement by Orowan bowing mechanism, leading to an increase in the dislocation density thereby accelerating the crystal refining progress. Consequently, the lattice strain is found to be increased with increasing milling time (Fig.3). Similar results have been reported by many researches **[Tousi, S.S.R et al 2009; Zhou, F et al 2001; Zhou, U et al 2006; Hesabi, Z.R et al 2006; Maurice, D et al 1995] .** Oxidation of Al and formation of solid solution between metals and ceramics causes changes in the lattice parameters.

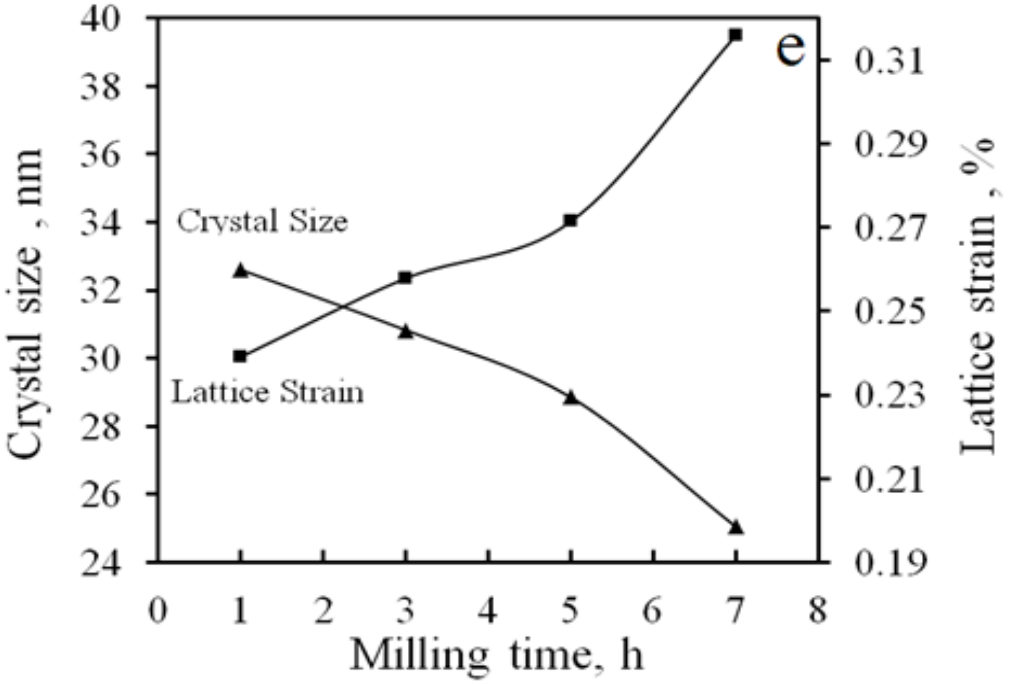


Fig. 2: Full width at half maximum (FWHM) of Al-20 wt. % Al_2O_3 versus milling time.

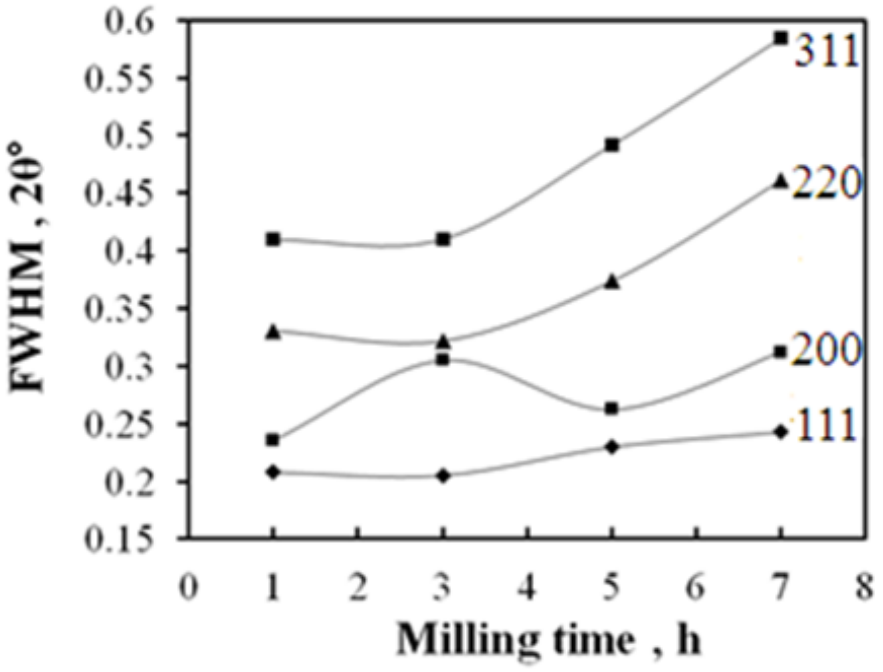


Fig. 3: Crystal size and lattice strain of Al-20 wt. % Al_2O_3 milled at different milling times.

Fig.4 shows the relationship between lattice parameters calculated for the principle planes (h k l) i.e. 111, 200, 220 & 311, using obtained XRD data and milling time. The results obtained indicate that the lattice parameter remains constant with increasing milling time. This means that neither oxidation of Al nor formation of solid solution between Al and Al_2O_3 has been took place.

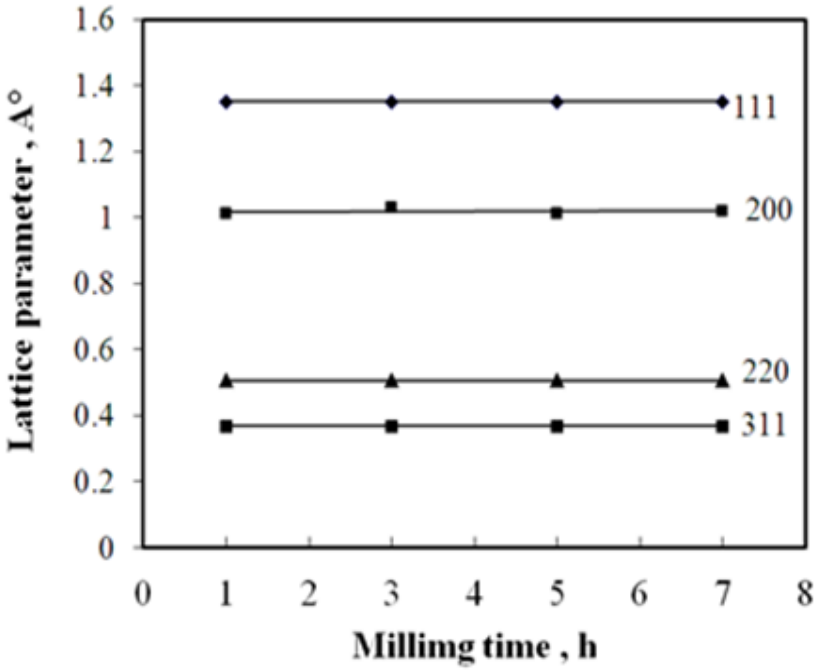


Fig. 5: XRD patterns of Al - (20-x) wt.-% Al_2O_3 - (x) ZrO_2, x=0, 1, 2 & 4 composites milled for 7 h.

Fig. 5 shows XRD of Al-(20-x) wt.-% Al_2O_3 - (x) ZrO_2 composites containing 1, 2 and 4 wt.-% ZrO_2. In case of addition 1 and 2 wt.-% zirconia, no new peaks beside Al and Al_2O_3 are observed, but when the percentage increased to 4wt.-%, small peak related to zirconia (101) is detected at $2\theta = 30.27^o$, (X-ray card no. 88-1007) **[Francisco, A.T.G et al 2009; Zhao, Z et al 2005; Rao, P.G et al 2003] .**

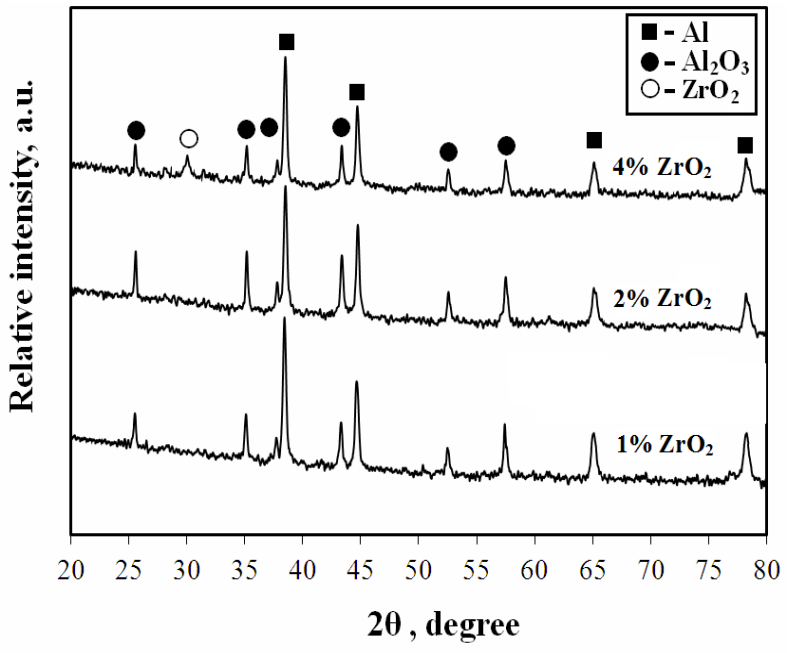


Fig. 5: XRD patterns of Al - (20-x) wt.-% Al_2O_3 - (x) ZrO_2, x=0, 1, 2 & 4 composites milled for 7 h.

Fig. 6 shows relationship between lattice strain & crystal size versus zirconia's concentration. Decreasing in crystal size and increasing in lattice strain are indicated with increasing zirconia content. Beside the above mentioned reason, the decreasing in crystal size may be also related to the more hardened ZrO_2 particles embedded in Al-Al_2O_3 matrix which cases more plastic deformation.

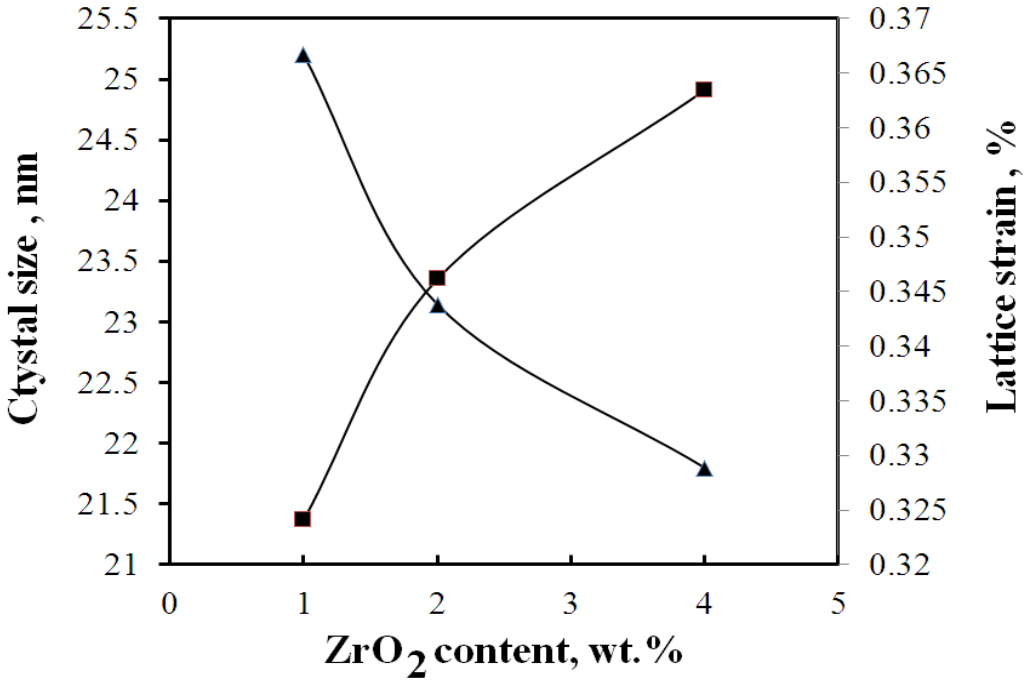


Fig. 6: Lattice strain and crystal size versus of Al - (20-x) wt.-% Al_2O_3 - (x) ZrO_2, x=0, 1, 2 & 4 composites.

3.1.2 Morphology of prepared powders

In order to investigate the morphology and particle size of the composite powder, transmission electron microscopy (TEM) was used. Fig.7 shows TEM images of Al-20wt. % Al_2O_3 composite powder milled for 1, 3, 5, and 7hrs. As regards particle size, it is evident from TEM images that it decreases with increasing of milling time. After 1 and 3h milling, agglomerated alumina particles are extremely observed (Fig.7a & 7b). Such particles' agglomeration could be removed by increasing milling time. After 5h milling (Fig.7c), alumina agglomerates are also observed but they are smaller and less than that obtained after 1 and 3h milling. After 7h milling, it seems like a few amounts of alumina particles welded with aluminum matrix (Fig.7d). It is well known that at the first stage of milling, the ductile particles undergo deformation (plastic 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 ball collision. However, no evidence of particle coarsening could be obtained through TEM. The raw powder of aluminum and alumina become continually intermixed leading to a homogenous material with a uniform second phase dispersion, i.e. alumina. This is because planetary mill is a high-energy mill which operates under high velocity and the

collision with milling balls enhances the powder refinement. During that, the powder particles are repeatedly deformed, cold welded and fractured by colliding balls. Fine and hard alumina particles are also act as milling agent, which help in reducing the powder**[Fogognolo, J.B et al 2003; Rajkovic, V et al 2008; Tousi, S.S.R et al 2009]** . In this situation, with more fracturing occurred, a large amount of fresh particle surfaces was produced. It is noted that finer alumina particle is distributed more homogeneously in MA composite. This is because mechanically alloying breaks up and continually embeds the alumina particles into aluminum matrix by repeated fracturing and cold welding of the powder charge **[Hussain, Z et al 2008].** The average particle size increases until the welding process dominates the milling process, and oppositely decreases after the fracture process becomes dominant. By achieving the balance between fracture and welding processes, the particles are rather uniform and equiaxed (i.e. no new changes) **[Maurice, D et al 1995, Rajković, V et al 2004] .**

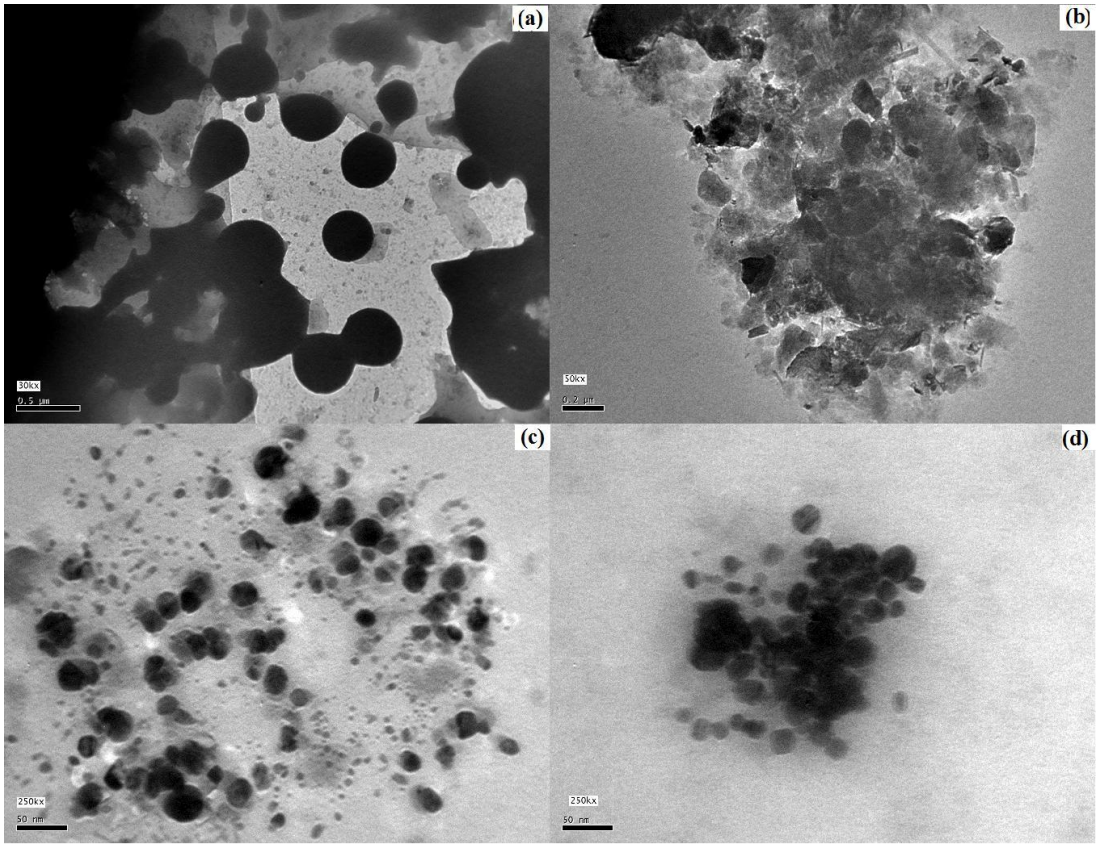


Fig. 7: TEM micrographs of Al-20 wt.-% Al_2O_3 composites milled at, (a) 1h, (b) 3h, (c) 5h, and (d) 7 h.

Fig. 8 shows TEM image of ZrO_2 powder, it used to enhance the properties of Al-Al_2O_3 composite matrix. the size of ZrO_2 about 35 nm with sphere-like shape. Such shape is likely to produce more force agent on the other particles (i.e. Al and Al_2O_3) and acting as milling balls in milling process and reaching by aluminum and alumina particles to smaller size in the whole mixture when added, specially 4 wt.-% (Fig. 9c). In fact, the more hardness the more behaving milling agent, the less hardness the less milling effect.

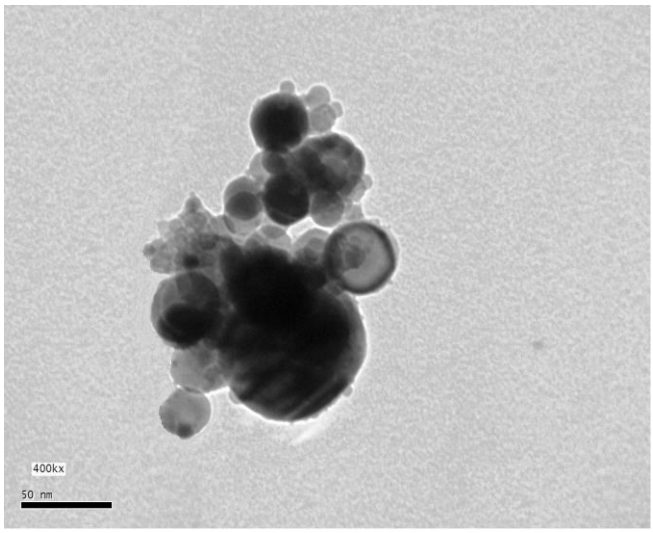
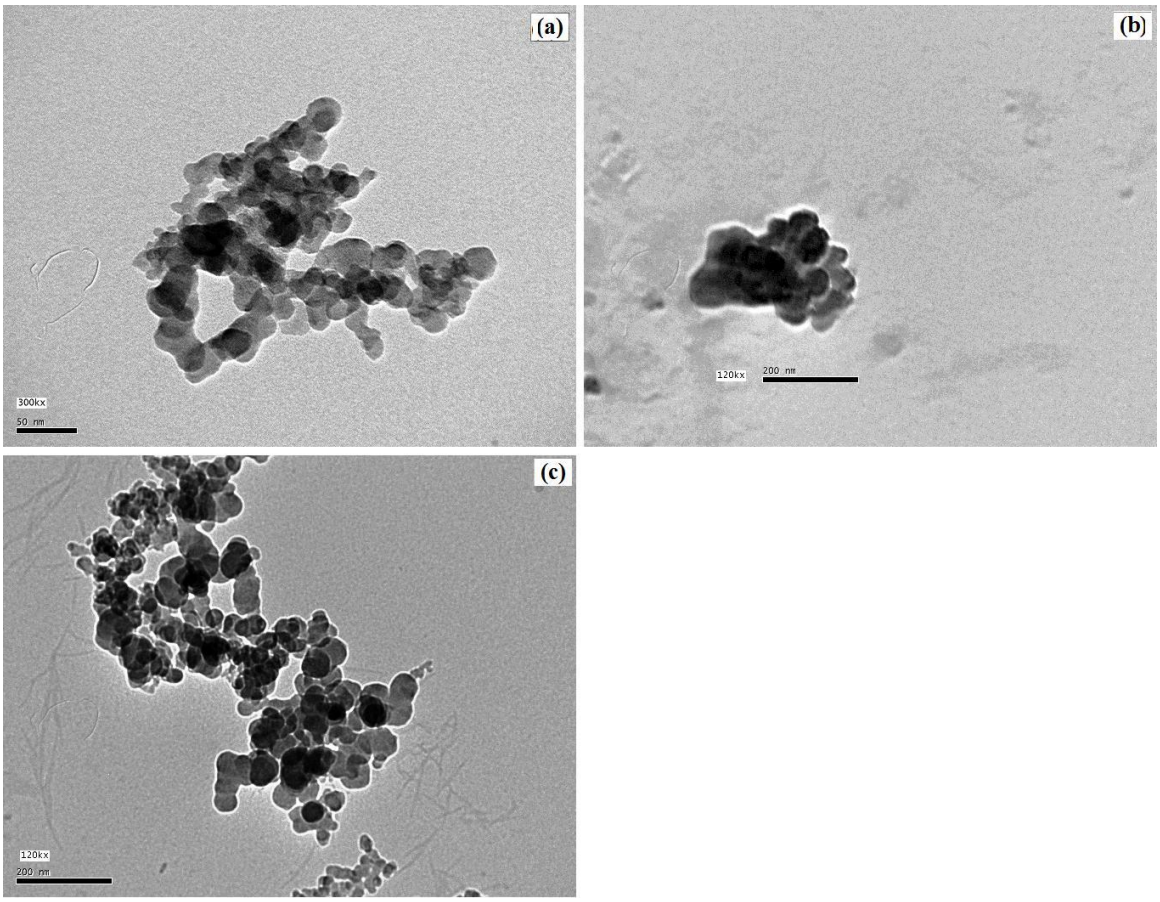


Fig. 8: TEM image of ZrO_2 powder.

Fig. 9: TEM micrographs of Al - (20-x) wt.-% Al_2O_3 - (x) ZrO_2, x= 1, 2 & 4



composites milled for 7h.

3.2. Sintering of prepared nanocomposite powders

3.2.1. Relative density and apparent porosity

Fig. 10 shows the relative density and apparent porosity of Al-20wt.-% Al_2O_3 nanocomposites sintered at different sintering temperatures i.e. 370, 400 and 470 ºC, for 1 h in argon atmosphere. The relative density of sintered samples increases with increasing both milling time and sintering temperature. This is due to increasing of surface area and decreasing of particle sizes, reaching to nano scale, which associate with aluminum matrix and the reinforcement material "alumina" after milling for long time. In deeply interpretation, with increasing sintering temperature, the actions of complex diffusion mechanisms become more intense, directly affecting the formation of surface contacts between the particles forming closed pores and grain growth **[Zawrah, M.F et al 2013; Korać, M et al 2007]** , are the matter causes the relative density increasing tendency. On the other hand, apparent porosity is found to be decreased with the increase in both milling time and sintering temperature (Fig.11). These results have been observed in many previous researches **[Hanumanth, G.S et al 1993] .**

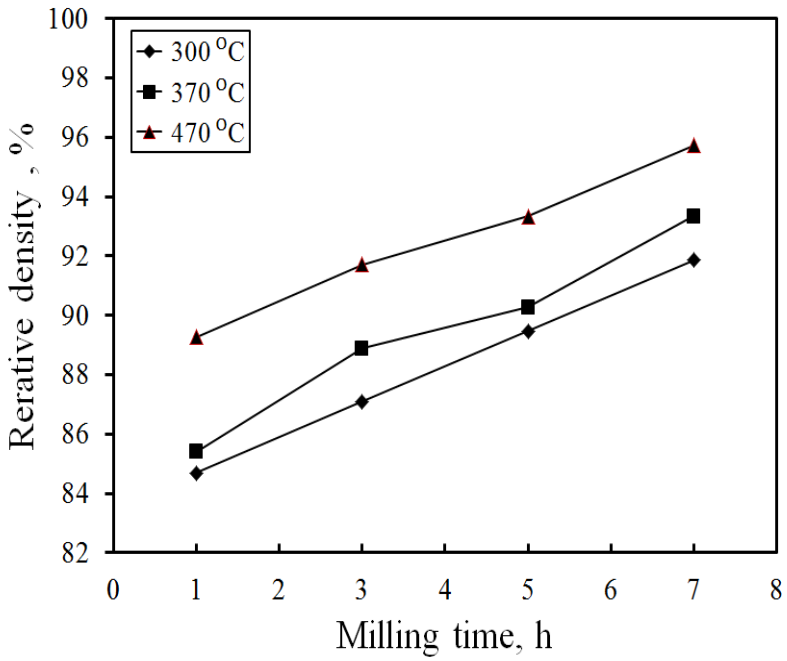
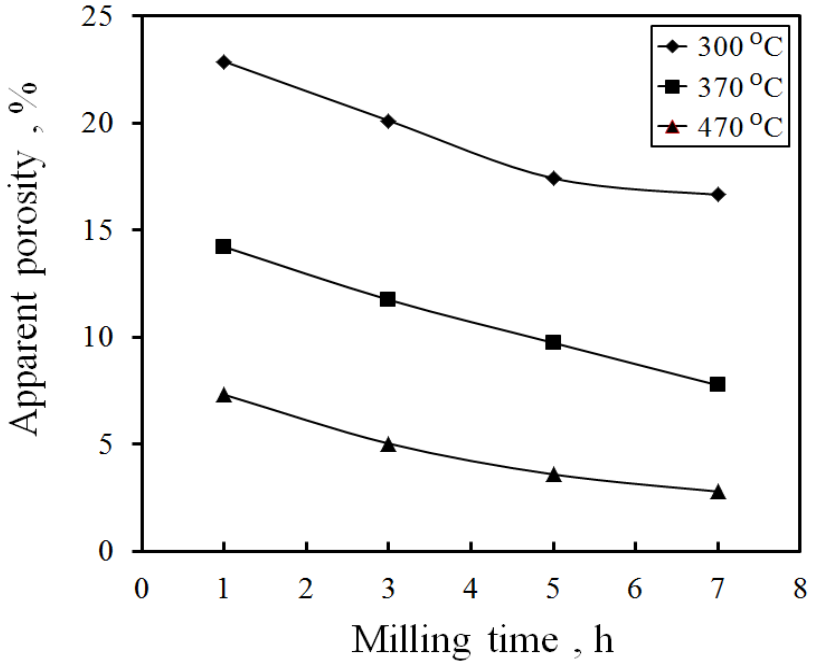


Fig. 10: Relative density vs. milling time for Al-20 wt. % Al_2O_3 composites sintered at different temperatures



Apparent porosity vs. milling time for Al-20 wt. % Al_2O_3 composites sintered at different temperatures.

Fig. 12 depicts the relative density and apparent porosity of Al - (20-x) wt.-% Al_2O_3 - (x) ZrO_2 composites sintered at 470°C. It is indicated that the relative density increases with increasing ZrO_2 content. Also, the relative density of the composite containing ZrO_2 is higher than that of the composite without zirconia (Fig. 11). This is attributed to two factors; the first is the higher theoretical density of zirconia as compared to aluminum and alumina, while the second is the small size of zirconia which achieves more filling for the grain boundaries. Consequently, apparent porosity is found to be decreased from 27.5% to 23% with zirconia addition. Guimara˜es et al. have been recorded similar results for Al_2O_3-ZrO_2 matrix [Francisco, A.T.G et al 2009].

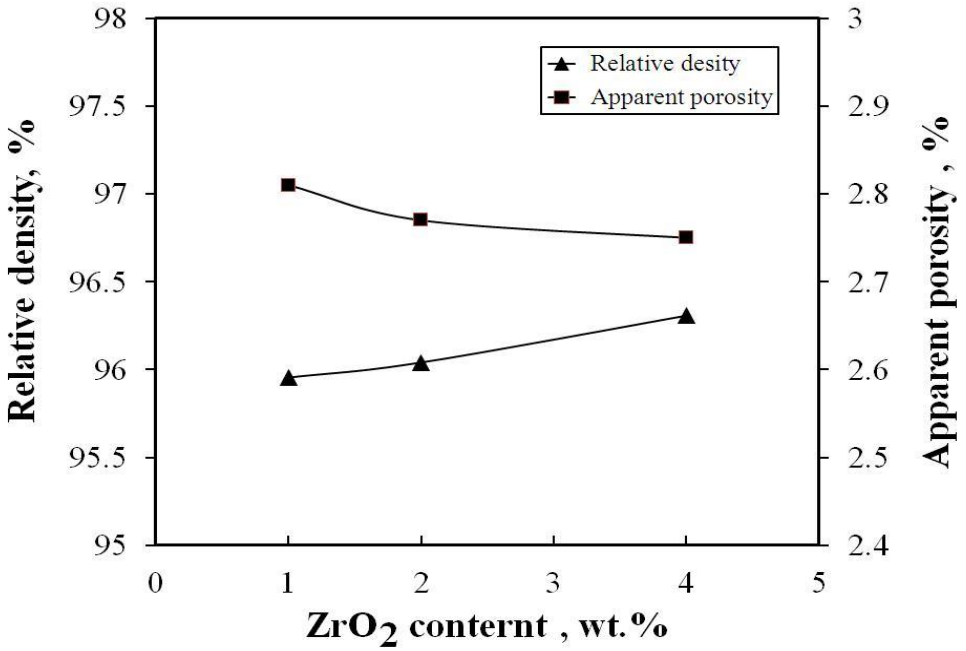


Fig. 12: Relative density and apparent porosity of Al - (20-x) wt.-% Al_2O_3 - (x) ZrO_2, x= 1, 2 & 4 composites milled for 7h and sintered at 470°C.

3.2.2. Microstructure of the sintered composites

Fig.13 shows optical micrographs of Al-20wt.-% Al_2O_3 sintered in argon atmosphere at 470 ºC for 1 h and prepared from powders milled at 1, 3, 5 and 7 hrs. A homogeneous microstructure with uniform distribution of alumina in the aluminum matrix is detected. Its homogeneity is increased with increasing milling time of starting

powders. The usage of mechanical alloying technique leads to obtain appreciable grain size reduction with increasing in milling time and less agglomerated regions.

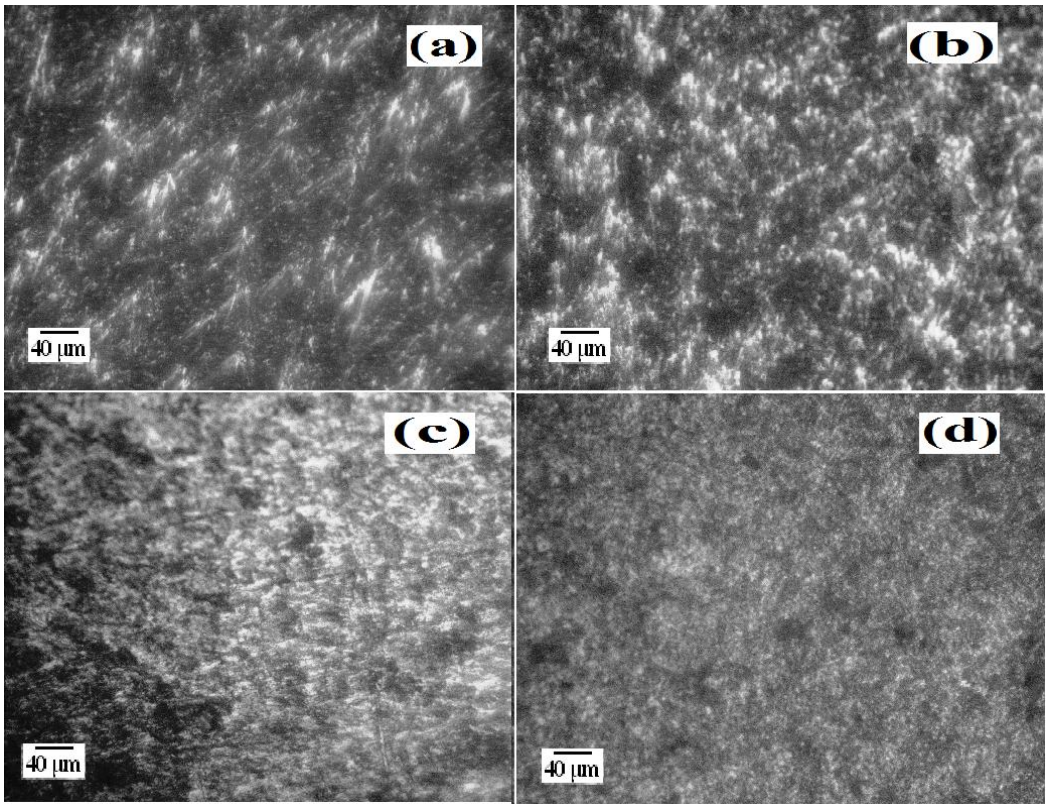


Fig. 13: Optical micrographs for Al-20 wt. % Al_2O_3 composites sintered for 1 h at 470 ºC in argon and prepared from powers milled at 1,3,5 and 7h.

For more details about the microstructure, SEM was conducted for Al-20 wt.-% Al_2O_3 prepared form powder milled for 7hrs and sintered in argon atmosphere at 470 ºC for 1h. Nearly homogeneous microstructure with good distribution of the reinforcement (alumina) is appeared in the image. Some agglomerated particle with grain growth is also detected in a dense microstructure. The particle agglomeration may cause by reduction of distances between the composite particles. Energy dispersive spectroscopy (EDX) area analysis of Al-20 wt.-% Al_2O_3 composite prepared from powder milled for 7h and sintered at 470 °C for 1 h is shown in Fig. 15. Form the figure, it is indicated that the weight percent of Al and O are 71.51 and 28.49, respectively, which are corresponding to aluminum and alumina.

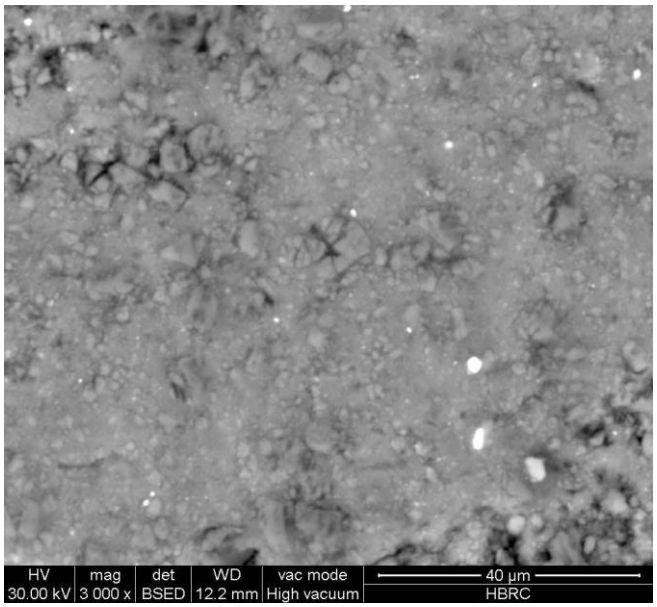
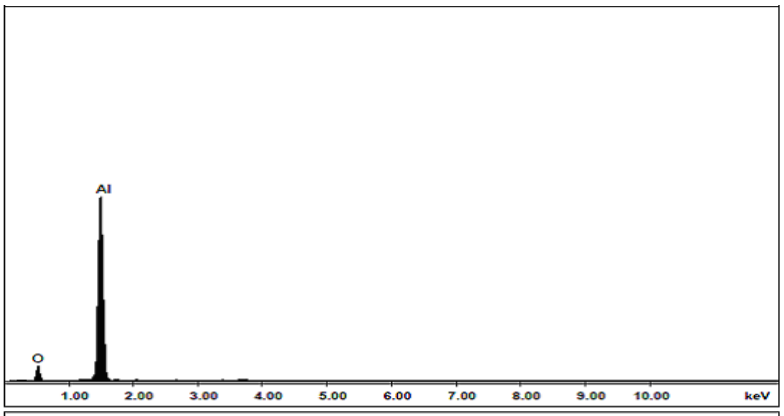


Fig. 14: SEM micrographs of Al-20 wt.-% Al_2O_3 composite sintered for 1 h at 470°C prepared from powder milled for 7h.



EDAX ZAF Quantification (Standardless)
Element Normalized
SEC Table : Default

Element	Wt %	At %	K-Ratio	Z	A	F
O K	28.49	40.19	0.0778	1.0470	0.2605	1.0014
AlK	71.51	59.81	0.5074	0.9807	0.7236	1.0000
Total	100.00	100.00				

Element	Net Inte.	Bkgd Inte.	Inte. Error	P/B
O K	26.97	1.66	2.44	16.28
AlK	370.80	1.94	0.62	190.85

Fig. 15: EDAX of Al-20 wt.-% Al_2O_3 composite sintered for 1 h at 470°C prepared from powder milled for 7h.

Fig.16 shows optical imaging of Al - (20-x) wt.-% Al_2O_3 - (x) ZrO_2; x= 1, 2 & 4, prepared from powder milled for 7 hrs and sintered in argon atmosphere at 470^{o}C for 1h.

More fine and homogeneous microstructure is detected after addition of zirconia as compared that without zirconia.

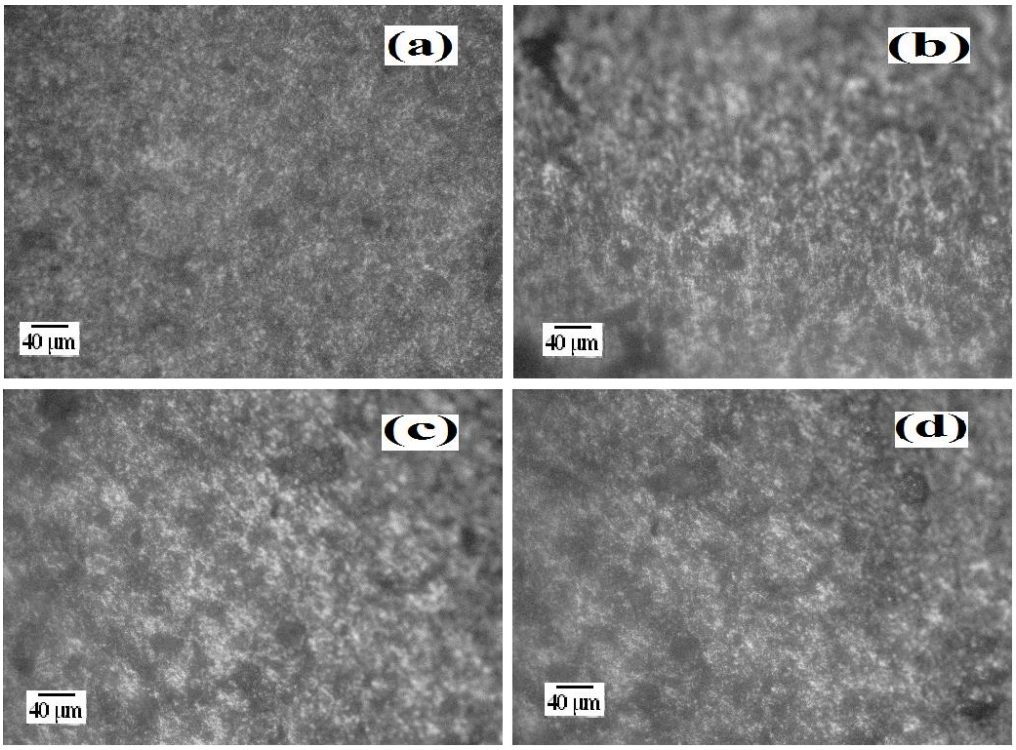


Fig. 16: Optical micrographs Al-(20-x)wt.-%Al_2O_3-(x)ZrO_2, x=1, 2&4, composites sintered for 1 h at 470 ºC in argon and prepared from powers milled for 7h.

Fig.17 show SEM micrograph of Al - (20-x) wt.-% Al_2O_3 - (x) ZrO_2; x=4, composite prepared from powder milled for 7h and sintered at 470 °C for 1 h. It is indicated from the image that more compacted, homogeneous and dense microstructure without grain growth is obtained. The porosity is mostly disappeared in this microstructure as compared that without zirconia. EDAX analysis shown in Fig. 18 indicates that the composite contains 71.98% Al, 24.79% O and 3.23% Zr.

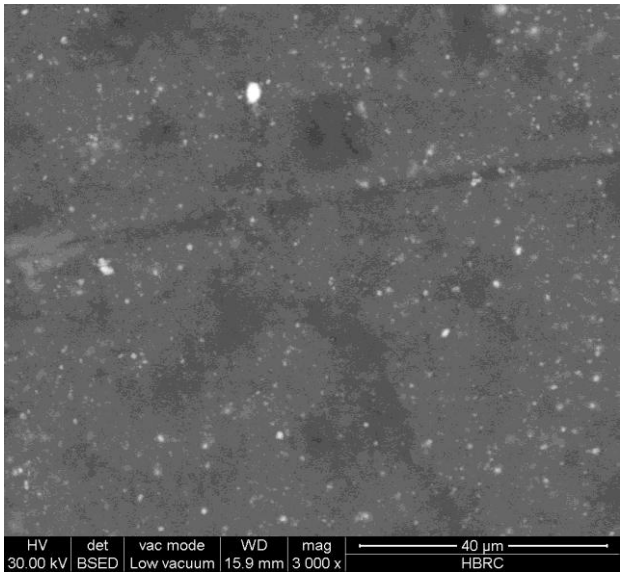


Fig. 17: SEM micrographs Al - (20-x) wt.-% Al_2O_3 - (x) ZrO_2, x= 4, composite sintered for 1 h at 470 ºC in argon and prepared from powers milled at 7h.

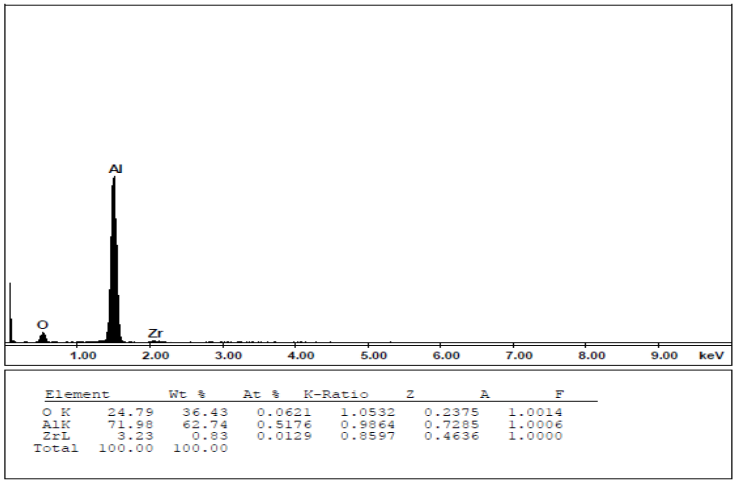


Fig. 18: EDAX of Al - (20-x) wt.-% Al_2O_3 - (x) ZrO_2, x= 4, composite sintered for 1 h at 470 ºC in argon and prepared from powers milled at 7h.

3.2.3 Microhardness of sintered composites

Fig.19 shows microhardness of Al-20 wt.-% Al_2O_3 prepared from powder milled for different milling times and sintered in argon atmosphere at 470 °C for 1 h. Microhardness of sintered composites increased with increasing milling time of starting powders **[Alizadeh, M et al 2011; Shehata, F et al 2009; Belhadjamida, A et al 1991;** Hernández, J.L.R et al 2012]. This increase in microhardness is a consequence of fine Al_2O_3 particles dispersion in Al matrix-compacts; and also due to crystal size refinement of starting powders after milling which led to increasing the sinterability and lowering the porosity. Before milling, the particle distribution isn't uniform and the distance between particles is so high, but increasing milling time causes break of big and brittle alumina powders, decreases the distance between particles and indent them into the ductile aluminum powders. This leads to increasing the sinterability and consequently the hardness of composites. The maximum value of microhardness of compacts processed after 7h milling and sintering at 470^{o}C attains 979 MPa.

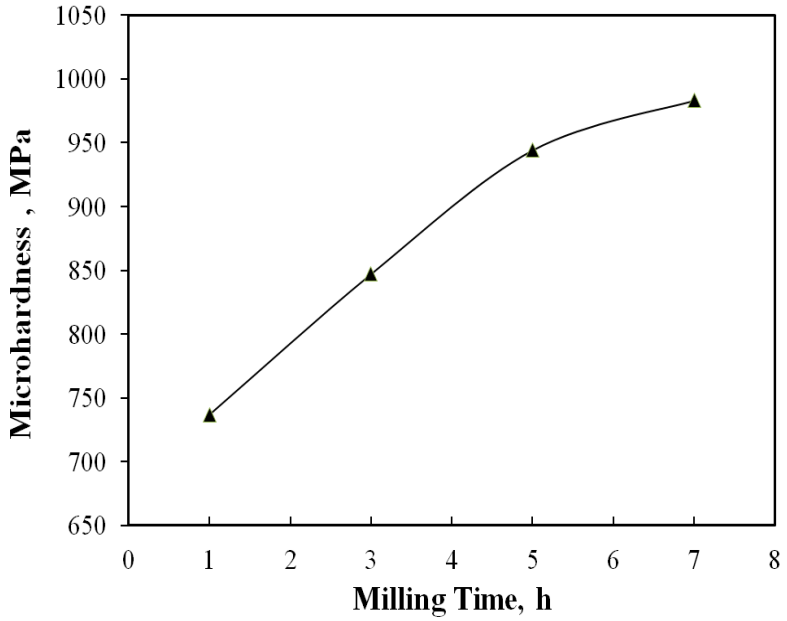


Fig. 19: Microhardness of Al-20 wt.-% Al_2O_3 composites sintered for 1 h at 470°C prepared from powder versus milling time of starting powder.

It is well known that the hardness of ZrO_2 is higher than that of Al_2O_3 and also it improves the microstructure though fining of the grains and limiting the grain growth. This leads to improving the hardness of the composite [Francisco, A.T.G et al 2009; Zhao, Z et al 2005; Liu, G.J et al 1998; Tuan, W.H et al 2002]. Fig. 20 shows microhardness of Al - (20-x) wt.-% Al_2O_3 - (x) ZrO_2; x= 1, 2 & 4, sintered in argon atmosphere at 470 ºC for 1 h and prepared from powders milled for 7 hrs. It is indicated

that the value of hardness increases with increasing zirconia's content reaching its maximum value with 4% zirconia (1070 MPa).

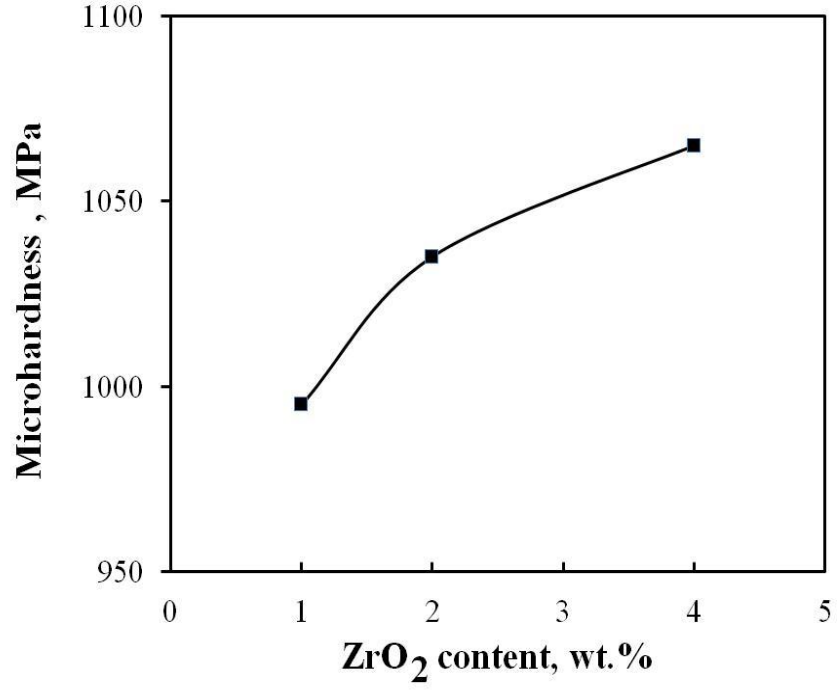


Fig. 20: Microhardness of Al - (20-x) wt.-% Al_2O_3 - (x) ZrO_2, x=1, 2 & 4, composites sintered for 1 h at 470 ºC in argon and prepared from powers milled for 7h.

4.Conclusion

The following remarks are concluded:

- Al - 20 wt.-% Al_2O_3 nanocomposites have been successfully fabricated using mechanical alloying at different milling time (1, 3, 5 up to 7 h), in planetary ball mill. Another series of Al - (20-x) wt.-% Al_2O_3 - (x) ZrO_2; x= 1, 2 & 4 were fabricated after milling for 7h.
- Crystal size was found to be decreased while the lattice strain was found to be increased with increasing milling time due to distortion effect caused by dislocation in the lattice. The lattice parameters were found to stay constant during milling. With increasing milling time, severe plastic deformation brings about a deformed lattice with high density of dislocations

- Both morphology and particle size of nanocomposite powders were changed with increasing the milling time; the particle size has been decreased with increasing the time.
- Relative density of sintered nanocomposites was increased with increasing both milling time of the starting powders and sintering temperature. While, apparent porosity was found to be decreased.
- The hardness of sintered composites was affected by refining of grains and dispersion of Al_2O_3 and/or ZrO_2 reinforcing particles in the composite with increasing milling time of starting powders. Microhardness of sintered bodies was found to be progressively increased with increasing of milling time of starting powders reaching 979 MPa in case of composite without ZrO_2 and 1070 MPa for composite with 4% ZrO_2.

Acknowledgement

Special thanks for late **Prof. Dr. Raghieba Abdel Wahab Esawey**, Prof. of Solid State Physics, National Research Center, Physics Division, Solid State Physics Department, for her supervision and fruitful discussion

5.References

Alizadeh M, Aliabadi M.M, High-energy mechanical alloying (MA) of a micrometer-scaled W and TiC powder mixture was performed to prepare TiC/W nanocomposites, Journal of Alloys and Compounds 509, 4978-4986, (2011).

Aboraia M.S., Abdalla G. A., Wasly H.S., Synthesis and Characterization of Al–$Al_2O_3$and Al/ (Al_2O_3-ZrO_2) Nanocomposites Using High-Energy Milling, Int. Journal of Engineering Research and Applications ISSN : 2248-9622, Vol. 3, Issue 6, Nov-Dec, pp.1654-1663(2013).

Belhadjhamida A and German R.M, "Tungsten and tungsten alloys by powder metallurgy: a status review," in Tungsten and Tungsten Alloys Recent Advances, A. Crowson and E. S. Chen, Eds., pp. 3–20, TMS, Warrendale, Pa, USA, 1991

Cullity B.D., Elements of x-ray diffraction, third ed., Prentice Hall, London, 2001.

Fogognolo J.B., Velasco F., Robert M.H., Torralba J.M., "Effect of mechanical alloying on the Morphology, microstructure and properties of aluminum matrix composite powder", Mat. Sci. Eng. A., Vol. 342, , 131-143(2003).

Francisco A.T.G., Ka´tia L.S, Trombini V, Juliano J.P., Jose´ A.R, Tomasi R, Eliria M.J.A.P. , Correlation between microstructure and mechanicalproperties of Al_2O_3/ZrO_2 nanocomposites, Ceramics International 35 741–745 (2009).

Guel I.G., Gallardo C.C., Ruiz D.C.M., Yoshida M.M., Rangel E.R., Sanchez R.M., J. Alloys Compd 483 173-177(2009).

Gubicza J., Kassem M., Ribarik G., Ungar T., The microstructure of mechanically alloyed Al–Mg determined by X-ray diffraction peak profile analysis, Mater. Sci. Eng., A 372 115–122(2004).

Hernández J. L. R, Cruz J.J. R, Pazdel V., Febles G, Alonso V. O. C, Sánchez R. M, Materials and Design 37 96–101(2012).

Hassan S.F., Gupta M., Effect of submicron size Al_2O_3 particulates on microstructural and tensile properties of elemental Mg. J. Alloys Compd. 457, 244–250 (2008).

Hesabi Z.R, Simchi A and Reihani S.M.S, Structural evolution during mechanical milling of nanometric and micrometric Al2O3 reinforced Al matrix composite. Materials Science and Engineering A 428 159–168 (2006).

Hanumanth G.S., Irons G.A., J. Mater. Sci. 28 2459-2465 (1993).

Hussain Z, Kit L.C, Properties and Spot Welding Behaviour of Copper-Alumina Composites through Ball Milling and Mechanical Alloying, Materials and Design 29 1311-1315 (2008).

Kaufman J.G., Properties of aluminum alloys; tensile, creep, and fatigue data at high and low temperatures, ASM International,(2002).

Khakbiz M., Akhlaghi F., Synthesis and structural characterization of Al–B_4C nano-composite powders by mechanical alloying, J. Alloys Compd., 479 334–34 (2009).

Korać M, Andić Z, Tasić M and Kamberović Ž, Sintering of Cu-$A1_2O_3$ nano-composite powders produced by a thermochemical route J. Serb. Chem. Soc. 72 (11) 1115-1125 (2007).

Liu G.J., Qiu H.B., Todd R., Brook R.J., and Guo J.K., Processing and mechanical behavior of Al_2O_3/ZrO_2 nanocomposites, Materials Research Bulletin, Vol. 33, No. 2, pp. 281–288(1998).

Lu L., Zhang Y.F., Influence of process control agent on inter diffusion between Al and Mg during mechanical alloying, J. Alloys Compd. 290 279–283(1999).

Liu R.S., Shi W.C., Cheng Y.C., Huang C.Y., Crystal structures and peculiar magnetic properties of alpha- and gamma-(Al_2O_3) powders. Mod. Phys. Lett. B , 11, 1169–1174 (1997).

Lonnberg B, Characterization of milled Si_3N_4 powder using X-ray peak, broadening and surface area analysis. J. Mater. Sci. 29 3224-3230 (1994).

Liu Y.Q., Cong H.T., Wang W., Sun C.H., Cheng H.M., Mater. Sci. Eng. A 505 151-156 (2009).

Miracle D.B., Metal matrix composites - from science to technological significance, Compos. Sci. Technol. 65 2526–2540 (2005).

Moustafa M.M.M., Omayma A.E., Abdelhameed A.W., Open Journal of Metal, 3 72-79 (2013) .

Maurice D, Courtney T.H., Modeling of mechanical alloying: part III. Applications of computational programs. metall., Metallurgical and Materials Transaction, 26A 2437 (1995).

Prabhu B., Suryanarayana C., An L., Vaidyanathan R., Synthesis and characterization of high volume fraction Al–Al_2O_3 nanocomposite powders by high-energy milling, Mater. Sci. Eng., A 425 192–200 (2006).

Razavi S.S.T, Yazdani R.R, Salahi E, Rahimipour M.R., Kazemzade A. and Razavi M., *Materials and Energy Research Center* Vol. 22, No. 2, August 177(2009).

Rajković V, Erić O, Božić D, Mitkov M, Romhanji E , Characterization of Dispersion Strengthened Copper with 3wt% $A1_2O_3$ by Mechanical Alloying, Science of Sintering, 36 205-211(2004).

Rosenberger M.R., Schvezov C.E., Forlerer E., Wear 259 590-601(2005).

Rajkovic V, Bozic D, Jovanovic M.T, Properties of copper matrix reinforced with nano- and micro-sized $A1_2O_3$ particles. Journal of Alloys and Compounds 459 177-184(2008).

Rao P.G., Iwasa M., Tanaka T., Kondoh I., Inoue T., Preparation and mechanical properties of Al_2O_3–15wt.%ZrO_2 composites, Scripta Materialia 48 437–441(2003).

Suryanarayana C., Al-Aqeeli N., Mechanically alloyed nanocomposites, Prog. Mater. Sci. 58 383–502 (2013).

Shehata F., Fathy A., Abdelhameed M., Moustafa S. F., Materials and Design 30, 2756-2762, (2009).

Suryanarayana C, Mechanical alloying. Prog Mater Sci; vol.46, pp.1-184, (2001).

Tousi S.S.R, Rad R.Y, Salahi E, Mobasherpour I, Razavi M. Production of Al–20 wt. % $A1_2O_3$ composite powder using high energy milling, powder Technology 192 346-351(2009).

Torralba J.M., da Costa C.E., Velasco F., P/M aluminum matrix composites: an overview, J. Mater. Processing Technol. 133 203–206 (2003).

Tuan W.H., Chen R.Z., Wang T.C., Cheng C.H., Kuo P.S., Mechanical properties of Al_2O_3/ZrO_2 compositesJournal of the European Ceramic Society 22 2827–2833 (2002).

Woo K., Lee H.B., Fabrication of Al alloy matrix composite reinforced with sub sieve-sized Al_2O_3 particles by the in situ displacement reaction using high-energy ball-milled powder. Mater. Sci. Eng., A 449–451, 829–832 (2007).

Youssef K.M., Scattergood R.O., Murty K.L., Koch C.C., Scripta Materialia 54 251–256 (2006).

Zawrah M.F., Zayed H.A, Essawy A.S, Nassar A.H, Taha. M.A, Preparation by mechanical alloying, characterization and sintering of Cu–20 wt.% Al_2O_3 nanocomposites, Materials and Design 46, 485–490 (2013).

Zebarjad S.A., Sajjadi S.A., Microstructure evaluation of Al–Al_2O_3 composite produced by mechanical alloying method, Mater. Des. 27 684–688 (2006).

Zebarjad S.M., Sajjadi S.A., Dependency of physical and mechanical properties of mechanical alloyed Al–Al_2O_3 composite on milling time, Mater. Des. 28 2113–2120 (2007).

Zhou F, Lee J, Lavernia E.J, Grain growth kinetics of a mechanically milled nanocrystalline Al. Scripta Mater. 44 2013-2017 (2001).

Zhou Y, Li Z.Q., Structural characterization of a mechanical alloyed Al–C mixture.J. Alloys Compd. 414 107-112 (2006).

Zhao Z, Zhang L, Jian Zheng J, Bai H, Zhang S, Xu B, Microstructures and mechanical properties of Al_2O_3/ZrO_2 composite produced by combustion synthesis, 1 Scripta Materialia 53 (2005) 995–1000.

الملخص باللغة العربية

تحضير وتوصيف بعض المتراكبات من ُلحمة فلزية مدعمة بأكسيد الالومنيوم والزركنيوم

محمود فرج زورة[1] ،احمد جمال الدين مصطفى [2]، فردوس احمد سعدالله[1] ، محمد يسرى حسان [2]
محمد عبد العزيز طه [1]، محمود نصر الدين محمد [1]

١. قسم الكيمياءغير العضوية وقسم الجوامد المركز القومي للبحوث ،2 قسم الجوامد جامعة الأزهر

تم تحضير متراكب نانومترى من لحمة فلزية مدعمة باكسيد الالومنيوم واكسيد الزركونيوم بطريقة التحضير الميكانيكى حتى زمن طحن ٧ ساعات. تم دراسة تأثير زمن الطحن على خواص المسحوق المحضر بواسطة نموذج حيود الاشعة السينية والميكروسكوب الالكترونى النافذ وذلك للتأكد من النسب الكيميائية للمتراكب الناتج ووجود اكثر من طور للمادة ودراسة حجم البللورات واشكالها. تم بعد ذلك كبس المسحوق الناتج فى صورة اقراص واخضاعها لحرارات حرق عند ٣٠٠ و ٣٧٠و ٤٧٠درجة مئوية لمدة ساعة فى وجود غاز الارجون.
تم دراسة الخواص الفيزيائية للعينات مثل الكثافة النسبية والمسامية الظاهرية بطريقة ارشميدس.
كما تم عمل دراسة بالميكروسكوب الالكترونى الماسح وتحليل طيفى بواسطة حيود الاشعة السينية للتأكد من نسب العناصر. كما تم عمل دراسة لخاصية الصلابة الميكانيكية للمتراكبات ومن خلال الدراسة وجد ان قيمة الصلابة تزداد بازدياد ساعات الطحن وتم تفسير النتائج. ووجد ان حجم الحبيبات قد وصل الى 20 نانوميتر بعد زمن طحن ٧ ساعات مع ملاحظة وجود بعض التكتلات الحبيبية. ووجد ان توزيع حبيبات كلا من اكسيد الالومنيوم واكسيد الزركونيوم فى لحمة الالومنيوم يزيد بانتظام مع زيادة عدد ساعات الطحن. كما وجد ان الكثافة النسبية تزداد ايضا بازدياد عدد ساعات الطحن وحرارة الحرق.