PROCEEDINGS OF THE INTERNATIONAL CONFERENCE NANOMATERIALS: APPLICATIONS AND PROPERTIES
Vol. **1** No 1, 01PCN33(4pp) (2012)

Thermal Stability and Mechanical Properties of Al-Al_2O_3 Nanocomposite Produced by Mechanical Milling and Hot-Pressing

F. Karimzadeh*, V. Rastar†, M.H. Enayati‡

Department of Materials Engineering, Isfahan University of Technology, Isfahan 81736-55953, Iran

(Received 20 June 2012; published online 24 August 2012)

In this study, Al-Al_2O_3 nanocomposite powders containing 5, 10 and 15 Wt% of nanopowder were produced by mechanical alloying. For comparing, Al-Al_2O_3 composite powder containing 5Wt% of micrometric Al_2O_3 was also produced. The powder was then hot-pressed in a mold to produce bulked parts. The effect of Al_2O_3 content on grain growth, density, hardness and bending strength of bulked composite was discussed and microstructures were investigated by optical, scanning and transmission electron microscopy. The results revealed that when nanometric particles were used instead of micrometric particles the grain growth was reduced, while the increase of particle weight percentage did not affect the grain growth. The results also showed that when weight percentage of nanometric particles was increased, although hardness of bulked parts was increased but relative density and bending strength was reduced severely.

Keywords: Nanocomposite; Mechanical milling; Al_2O_3; Thermal stability; Bending strength.

PACS numbers: 81.20.Wk, 62.23.Pq

1. INTRODUCTION

Particulate reinforced metal matrix composites remain interesting field of materials, such as high specific modulus, high specific strength, low density and easy fabrication [1]. Amongst ceramic particle reinforced metal matrix composites we can name Al, Ti and Mg matrix composites which are reinforced with SiC, B_4C, TiB_2, TiC, Al_2O_3 and etc. For good properties of Aluminum, Aluminum matrix composites have a very wide range of applications [2,3]. Aluminum Oxide (Al_2O_3) is the most widely used material in the family of engineering ceramics because of its high strength and high wear resistance. Coefficient of thermal expansion of Al_2O_3 is similar to Aluminum, therefore Al_2O_3 is a good reinforcement for improving mechanical and wear properties of Aluminum matrix [4,5].

Ceramic/Metal interfaces play an important role in understanding most composite properties and stability of these interfaces are so significant in order to achieve good mechanical properties. If composite phases are finely made, they make better and stronger bounds with adjacent matrix and therefore mechanical properties and hardness will be increased. In fact, conversion of composite to nanocomposite usually causes increase in hardness and mechanical properties. Metal matrix nanocomposites are subset of nanostructured materials and at least one of their phases (reinforce particles or metal matrix grains) is in nanometric scale [6-8]. Hosseini *et al*. investigated tribological properties of Al6061/Al_2O_3 composite and observed that when Al_2O_3 particle size is 30nm wear rate becomes about 9 times smaller compared to 60μm particle size [9]. Tavoosi *et al*. fabricated Al-Zn/Al_2O_3 nanocomposite by mechanical alloying and showed that hardness of this nanocomposite is 6 times higher than original Aluminum [10].

In most composite manufacturing techniques final product is powder. Therefore, a pressing stage is necessary in conversion of powder to bulked parts. The success in pressing nanostructured powder is not easy because neither grain growth nor inducing porosity should happen [11,12]. Powder forging, powder rolling, hot extrusion of powder and cold and hot pressing of powder are some of the pressing methods of Aluminum powder for achieving dense products [13]. For fabrication of nanocomposite by hot-pressing method, the growth of nanometric grains is the most important factor. In fact, high temperature may be a cause for grain growth and the nanostructured matrix destruction. Nevertheless, in many cases due to presence of nano particles in grain boundaries, grain growth does not destroy nanostructured matrix because nano particles lock the boundaries. Tavoosi *et al*. revealed that crystalline grain size in Al-Zn/Al_2O_3 nanocomposite stays constant at 40nm after 30 minutes heat treatment in 550°C [10].

The goal of this study was to fabricate Al-Al_2O_3 nanocomposite powders by mechanical alloying with different reinforcement percentage and then produce bulked parts by hot pressing. In order to investigate the effect of reinforcement particle size, Al-Al_2O_3 composite containing 5 Wt% of micrometric Al_2O_3 was also produced.

2. EXPERIMENTAL

Aluminum powder (with 99.99% purity), nanometric Al_2O_3 (20-30 nm) and micrometric Al_2O_3 (120-130 μm) were used in this study. In order to produced nanocomposite and microcomposite powders containing 5 Wt% reinforcement (10 and 15 Wt% reinforcement only for nanocomposite powder), Aluminum powder was mixed with appropriate amounts of nanometric and micrometric Al_2O_3 powder and then samples were milled in room temperature and Argon gas. Mechanical alloying was

* karimzadeh_f@cc.iut.ac.ir
† vahhab.rastar@gmail.com
‡ ena78@cc.iut.ac.ir

performed in a steel chamber containing 19 steel balls with a diameter of 20 mm. The MA conditions employed in this experiment were: 5 h, 250 rpm, 10/1 balls/load ratio (by Wt.). Composite powders were granulated by sieve and powders with less than 100 μm in size were used for bulked parts. Hot-press mold was consisting of resistive element as a heat source, temperature control equipments and piston and matrix made of H13 steel. The hole dimensions of the mold were: length = 4.2 cm, Width = 1.2 cm and Height = 5 cm. In order to produce each part 16.3 g of powder was poured into the mold and temperature was raised to 500 °C and then using a hydraulic press, pressure of 450 MPa was applied on samples for 15 min. Finally all produced samples with 1.2×1.2×4.2 cm dimensions, were converted to rods with 0.9 cm in diameter using a lathe.

Structural changes and grain growth were studied by X-rays diffraction. Grain size was estimated by XRD patterns and Williamson-Hall equation. Since usually a lot of strain is formed by mechanical alloying in crystalline lattice, the Williamson-Hall method for calculating the grain size is more accurate than other methods [14]. Microstructures were investigated by OM, SEM and TEM. In order to ensure appropriate compaction and to investigate the effect of reinforcement on the compression level, bulked part densities were measured by soaking method (Archimedes method). Hardness of bulked parts was also determined by Vickers hardness testing machine for 10sec with 10kg load. Also bending strength of nanocomposites measured by three point bending method.

3. RESULT AND DISCUSSION

3.1 Thermal stability and grain growth

X-ray diffraction experiments were performed on all composite powders and bulked parts. The XRD patterns of composite powders and bulked parts with different amounts of nanometric reinforcement are shown in Fig. 1. As shown in this figure, none of the Al_2O_3 peaks is visible. This is due to very small size of Alumina particles. In many cases when nanometric phases exist in a structure, they can not be investigated well by XRD and this method can not distinguish these phases in all cases. This has been expressed by many researchers as well [11-13].

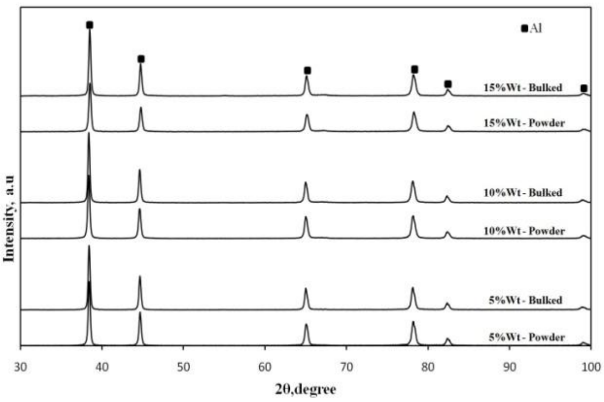

Fig. 1 – Al-Al_2O_3 composite XRD patterns containing 5, 10 and 15 Wt% of nanometric reinforcement in powder and bulk forms

The XRD patterns of composite powder and bulked part containing 5%Wt of micrometric Al_2O_3 are shown in Fig. 2. As shown in this figure, Al_2O_3 peaks are visible due to large size of particles. Crystalline grain sizes for powders and bulked parts were estimated by XRD patterns and the Williamson-Hall equation. The results revealed that when nanometric particles were used, the grain sizes were about 75 nm in all cases. This means that not only produced nanocomposites have sufficient thermal stability at 500°C but also weight percentage of reinforcement has no effect on crystalline grain size after milling. The grain size for the composite powder containing 5%Wt micrometric Al_2O_3 was also estimated to be about 75 nm. This also revealed that reinforcement size has no effect on crystalline grain size after milling. But the grain size for the bulked composite containing 5%Wt micrometric Al_2O_3 that was estimated to be about 105 nm is indicative of grain growth due to heat of hot-press.

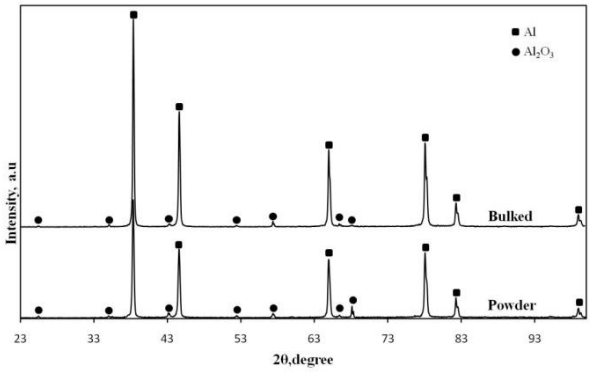

Fig. 2 – Al-5%Al_2O_3 composite XRD patterns in powder and bulk forms.

In order to examine more accurately, (111) peaks were compared to each other. As shown in Fig. 3, these peaks are very similar for composite powders and bulked parts. This proves that, neither in composite powders nor in bulked parts, is weight percentage of nanometric reinforcement a factor in grain size.

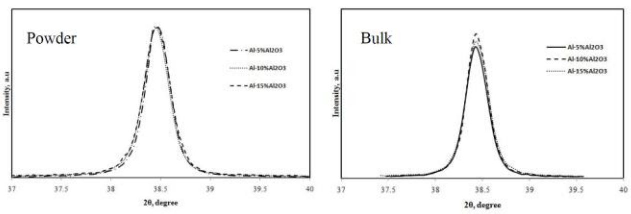

Fig. 3 – Al-Al_2O_3 composite XRD (111) patterns in powder and bulk forms

For composites containing 5%Wt of nanometric and micrometric Al_2O_3, (111) peaks are compared in Fig. 4. As shown in Fig. 4.a, when nanometric reinforcements were used, (111) peaks are similar in composite powder and bulked part and therefore, grain sizes should also be the same for both. But as shown in Fig. 4.b, when micrometric reinforcements were used, intensity has increased and width has decreased in bulked part. This reduction in peak width indicates that the grain size has increased. The reason that grain sizes are not increased with nanometric particles is the presence of nano particles in grain boundaries. In fact these nano particles can lock the boundaries and prevent their movements.

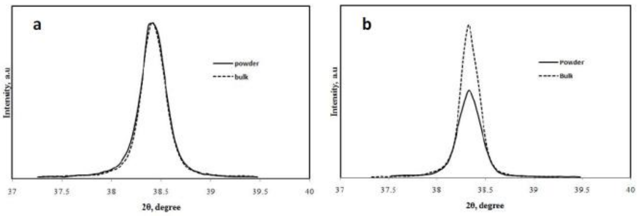

Fig. 4 – Al-5%Al_2O_3 composite XRD (111) patterns in powder and bulk forms a) nanometric Al_2O_3, b) micrometric Al_2O_3

3.2 Microstructure

Microstructures of composites containing 5%Wt micrometric and nanometric Al_2O_3 are shown in Fig. 5. Since the grain sizes in both composites are very fine (about 50nm), crystalline grains are not visible by OM and SEM. As shown in Fig. 5a., Micrometric Al_2O_3 particles can easily be observed. This figure shows that micrometric Al_2O_3 particles are crushed to 10-40µm due to milling and they are uniformly distributed in Aluminum matrix. Fig. 5.b shows the SEM micrograph of etched Al-5%Al_2O_3 containing nanometric reinforcements. In this figure nano particles can not be seen because of their very small sizes. But boundaries of the pressed powder particles are very well visible because they are etched more due to their higher energy. This figure also shows that powder particles were fully sintered during hot-press.

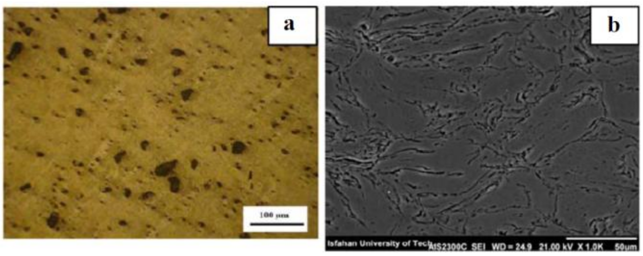

Fig. 5 – a) Optical microstructure of Al-5%Al_2O_3 containing micrometric reinforcement b) SEM micrograph of Al-5%Al_2O_3 containing nanometric reinforcement

Nanometric grains and nano Al_2O_3 particles were observed by transmission electron microscopy (TEM). As shown in Fig. 6. approximately nanometric Al_2O_3 particles are uniformly distributed in Aluminum matrix.

3.3 Hardness

Fig. 7. Shows hardness of Al-Al_2O_3 composites containing 5, 10 and 15Wt% of nanometric reinforcements compared to Al-Al_2O_3 composite containing 5Wt% of micrometric reinforcement. As shown in this figure, composite hardness increases with increasing Al_2O_3 weight percentage. Additionally, there is 95Hv difference between composites containing nanometric and micrometric reinforcements at 5%Wt Al_2O_3. This difference in hardness is the effect of using nanometric Al_2O_3 particles.

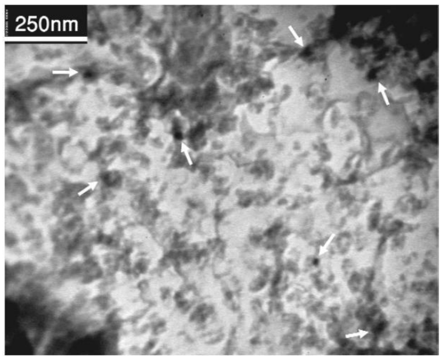

Fig. 6 – TEM micrograph of Al-5%Al_2O_3 (Nanometric Al_2O_3 particles are shown by arrows)

There are two reasons that use of nanometric reinforcements increases the hardness more than micrometric reinforcement. The first reason is that nanometric reinforcements are distributed more dispersed. The second reason is that there are more particles at the grain boundaries which can prevent boundary movements.

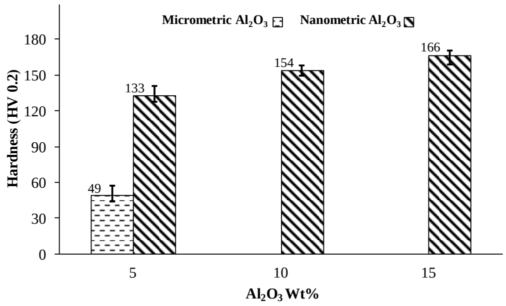

Fig. 7 – Hardness of Al_2O_3 composites

3.4 Density

Lathe ability of produced composites indicates very good powder compaction and highly dens composites. Fig. 8. Shows relative densities of Al-Al_2O_3 composites containing 5, 10 and 15Wt% nanometric reinforcement and they are 98.8, 97.8 and 94.4% respectively. The increase in reinforcement content makes it difficult to compress powder and therefore relative density is reduced.

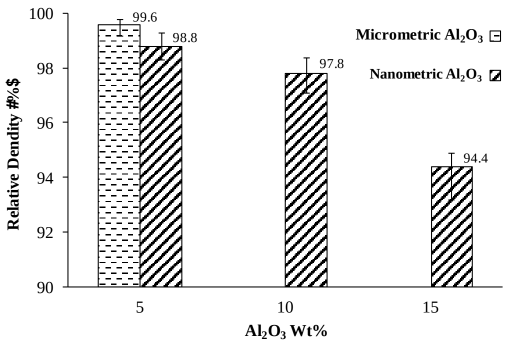

Fig. 8 – Density of Al_2O_3 composites

Relative density of Al-5%Al_2O_3 composite containing micrometric reinforcement is 99.6%. The higher density of this composite compare to Al-5%Al_2O_3 composite containing nanometric reinforcement is most likely due to less hardness of micrometric particle reinforced powder. In fact this powder has less flow stress and can be compacted easier in steel mold.

3.5 Bending strength

As shown in Fig. 9. bending strength of Al-Al_2O_3 composites decrease with increasing in reinforcement content. In fact decrease in relative density values is proportional to the porosity in composites. Therefore strength loss in nanocomposites can be caused by increased porosity.

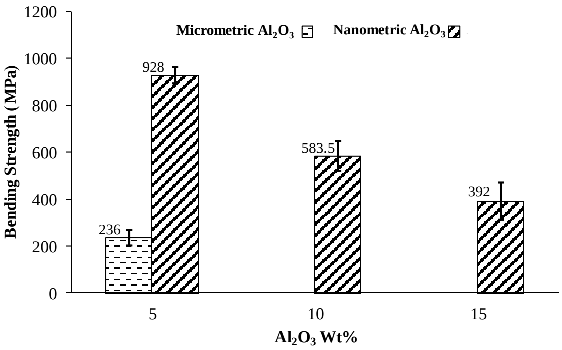

Fig. 9 – Bending strength of Al_2O_3 composites

4. CONCLUSIONS

1. In Al-Al_2O_3 powder and bulked part composites containing nanometric reinforcements, the grain sizes were about 75nm in all cases. This means that not only produced nanocomposites have sufficient thermal stability at 500 °C, but also weight percentage of reinforcement has no effect on crystalline grain size after milling.
2. Reinforcement size (micrometric or nanometric) has no effect on crystalline grain size after milling.
3. When nanometric Al_2O_3 particles were used, the grain sizes remained constant but in the case of micrometric Al_2O_3 particles the grain sizes were increased.
4. Micrometric Al_2O_3 particles were crushed to 10-40μm due to milling and they are uniformly distributed in Aluminum matrix.
5. TEM pictures prove that nanometric Al_2O_3 particles are uniformly distributed in aluminum matrix.
6. Composite hardness increases with nanometric Al_2O_3 weight percentage increase.
7. The increase in reinforcement content makes it difficult to compress powder and therefore relative density is reduced.
8. Bending strength of composites decreases with nanometric Al_2O_3 weight percentage increase.

REFERENCES

1. A. DiIlio, A. Paoletti, *Int. J. Mach. Tool Manuf.* **40**, 173 (2000).
2. N. Chawla, K.K. Chawl, *Metal Matrix Composites* (Springer Science: 2006).
3. T.W. Clyne, P.J. Withers, *An Introduction to Metal Matrix Composites* (Cambridge: 1995).
4. I.A. Ibrahim, F.A. Mohamed, E.J. Lavernia, *J. Mater. Sci.* **26**, 1137 (1991).
5. http://www.accuratus.com/alumox.html
6. Y.X. Gan, *Int. J. Molecul. Sci.* **10**, 5115 (2009).
7. J.S. Moya, S.L. Esteban, C. Pecharroma, *Prog. Mater. Sci.* **52**, 1017 (2007).
8. M. Tabandeh Khorshid, S.A. Jenabali Jahromi, M.M. Moshksar, *Mater. Design* **31**, 3880 (2010).
9. N. Hosseini, F. Karimzadeh, M.H. Abbasi, M.H. Enayati, *Mater. Design* **31**, 4777 (2010).
10. M. Tavoosi, F. Karimzadeh, M.H. Enayati, *Mater. Lett.* **62**, 282 (2008).
11. C. Suryanarayana, *Int. Mater. Rev.* **40**, 41 (1995).
12. C. Suryanarayana C.C. Koch, *Hyperfine Interact.* **130**, 5 (2000).
13. J.R. Pickens, *J. Mater. Sci.* **16**, 1437 (1981).
14. M. Sherif, E.L. Eskandary, *Mechanical alloying for fabrication of advanced engineering materials* (Cairo: Noes Publication: 2001).