

# CONSOLIDATION OF MECHANICALLY MILLED $\text{Al}/\text{Al}_2\text{O}_3$ AND $\text{Al-8Zn}/\text{Al}_2\text{O}_3$ COMPOSITE POWDERS

D. Hernandez-Silva, J. Gamez-Huerta, M. A. Garcia-Bernal and V. Sauce-Rangel

Department of Metallurgical Engineering, IPN-ESIQIE, Apdo. Postal 118-392, 07738 México, D. F., México

Received: March 29, 2008

**Abstract.** In this work, the consolidation of composite powders of  $\text{Al-xAl}_2\text{O}_3$  and  $\text{Al-8Zn-xAl}_2\text{O}_3$  (where  $x=0, 1, 2,$  and  $3$  wt.%) has been studied. Al and Al-8wt.%Zn powders with a mean particle size of  $5 \mu\text{m}$  were obtained by mechanical milling in a ball mill for 30 hours in hydrogen atmosphere. The milled powders were mixed with  $\alpha\text{-Al}_2\text{O}_3$  with a mean particle size of  $0.4 \mu\text{m}$  in several percentages and consolidated by hot pressing. The effect of zinc addition and alumina content on density and hardness of consolidated samples was evaluated.

It was found that zinc has a very strong effect on the sinterability of the composites, increasing the density of consolidated samples up to 97% of the theoretical density for  $\text{Al-8Zn-xAl}_2\text{O}_3$  composites compared to a density of up to 75% reached in  $\text{Al-xAl}_2\text{O}_3$  composites. Alumina particles increase the hardness of the Al-8Zn sample but decrease the density of the consolidated samples due to increased friction effects between particles.

## 1. INTRODUCTION

Parts made of sintered aluminum combine low weight, good corrosion resistance and high strength [1]. By introducing hard carbide or oxide particles into the aluminum matrix, strength at room and high temperatures as well as stiffness and wear resistance can be improved [2,3]. Silicon carbide and alumina particles have been used for this purpose.

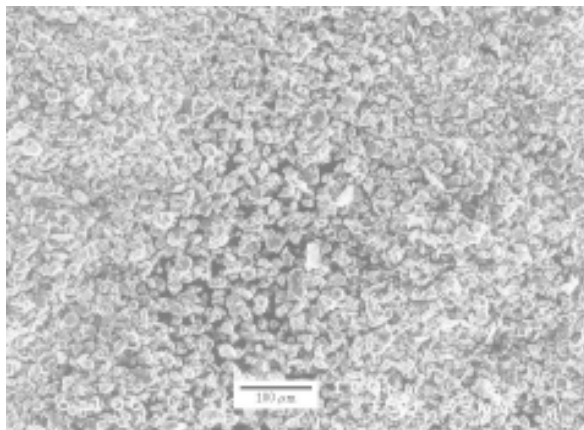
Several processing routes such as casting or powder metallurgy have been used to produce aluminum based composites. However, in casting, low wettability of the reinforcement particles by liquid aluminum frequently causes agglomeration of the particles resulting in non-uniform properties [4]. Powder metallurgy on the other hand produces better distribution of the reinforcement particles and therefore homogeneous properties.

The strengthening of the matrix is more effective if the reinforcement particles are small and are homogeneously distributed. Reducing the particle size of the powder of the matrix phase, e.g. through

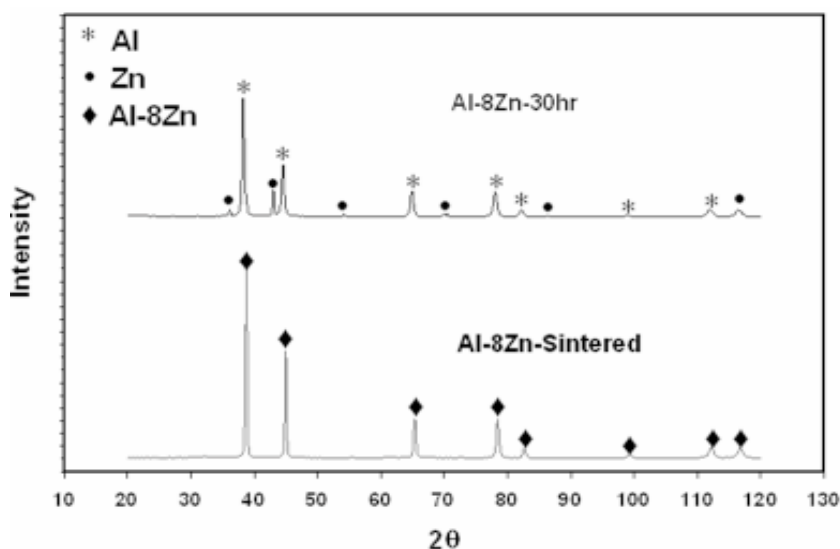
mechanical milling, can help to improve this distribution [5,6].

During mechanical milling of aluminum powder, aluminum oxide particles resulting from the naturally formed protective oxide film on aluminum surface, are introduced into the alumina matrix, helping to improve its strength through dispersion strengthening. However, this protective oxide film acts as a barrier during consolidation, affecting severely the final density. This film prevents the diffusion of atoms and the creation of bonds between particles. Some elements [1] have been added to aluminum powder before sintering to improve the sinterability. These elements do not form an oxide film on their surface, which helps to improve the bonding between particles. Some elements, such as zinc, are liquid at temperatures normally used for sintering of the aluminum powder, helping to improve the consolidation through capillary forces by filling the voids between the particles.

Corresponding author: D. Hernandez-Silva, e-mail: dhs07670@yahoo.com



**Fig. 1.** SEM-micrograph of the Al-8 wt.% Zn milled powder.



**Fig. 2.** X-ray diffraction patterns of the Al-8 wt.% Zn milled powder before and after sintering obtained using copper  $K_{\alpha}$  radiation.

In this work, the sinterability of aluminum powder with zinc and alumina additions was studied. Before sintering the particle size of both aluminum and aluminum-8 wt.% zinc powders was reduced by mechanical milling in order to improve the distribution of the reinforcement particles and therefore its strengthening effect. Density and hardness of consolidated samples were evaluated and the microstructure was studied by means of scanning electron microscopy.

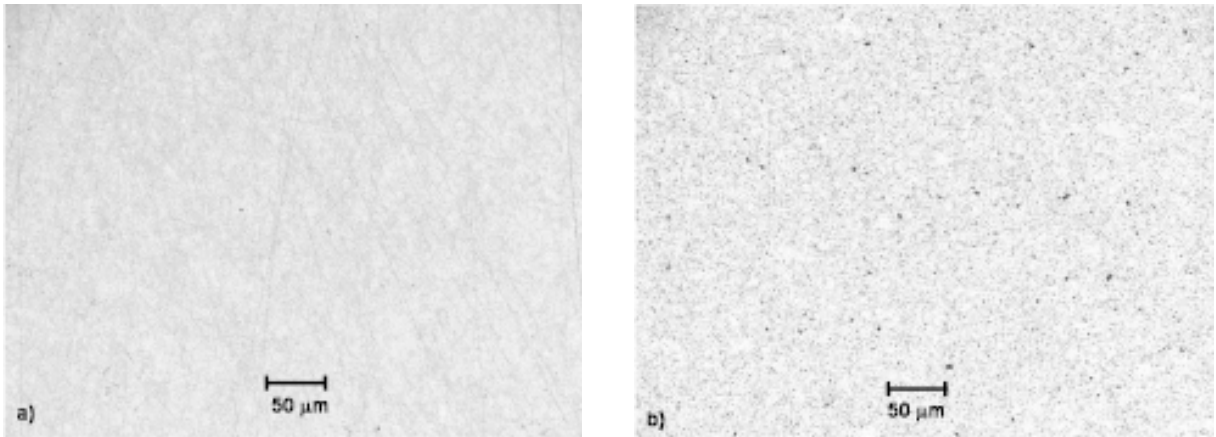
## 2. EXPERIMENTAL PROCEDURE

Pure aluminum (99.9%) and zinc (99.9%) powders with a mean particle size of 50  $\mu\text{m}$  and 40  $\mu\text{m}$ , respectively, were used in this work. Pure aluminum and a mixture of aluminum with 8 wt.% of zinc were mechanically milled using a tumbler ball mill

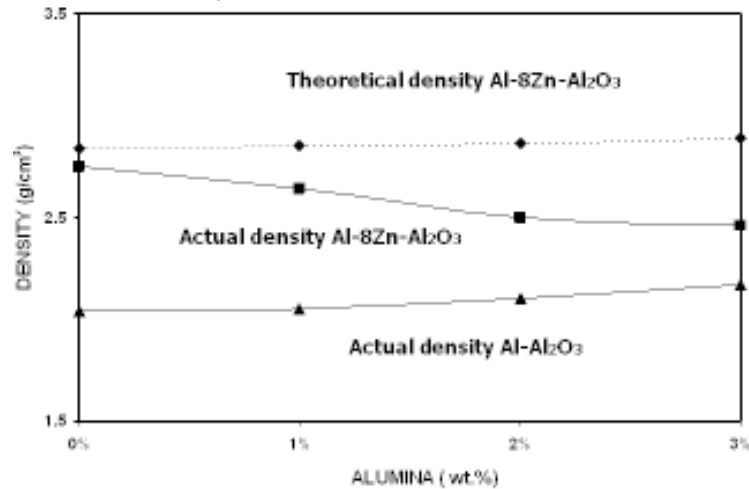
made of stainless steel (vial and balls) during 30 hours. The purpose of the mechanical milling was to reduce the particle size of the original powders of matrix in order to obtain more uniform distribution of  $\text{Al}_2\text{O}_3$  particles. The atmosphere inside the ball mill was hydrogen. Stearic acid was added as a process control agent. The mean particle size of the milled powder was measured using laser diffraction.

The milled powders were mixed with 1, 2, and 3 wt.% of  $\alpha\text{-Al}_2\text{O}_3$  with a particle size of 0.4  $\mu\text{m}$  in an alumina ball mill with alumina balls for three hours.

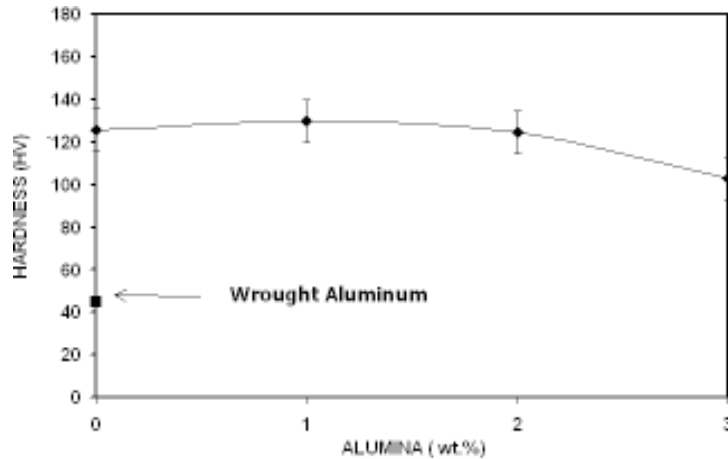
In order to consolidate the powders, samples of ten grams were put into a graphite die and heated in vacuum at 400  $^{\circ}\text{C}$  for 30 min in order to eliminate the stearic acid. After this step, the temperature was raised to 650  $^{\circ}\text{C}$ , a pressure of 10 MPa



**Fig. 3.** Optical microscope images of the microstructure of the consolidated samples, a) Al-8 wt.% Zn sample, b) Al-8 wt.% Zn-3 wt.% Al<sub>2</sub>O<sub>3</sub> sample.



**Fig. 4.** Density as a function of alumina content for Al-8Zn/Al<sub>2</sub>O<sub>3</sub>.



**Fig. 5.** Variation of hardness as a function of alumina content for Al-8Zn/Al<sub>2</sub>O<sub>3</sub>.

was applied for 1 hour at this temperature. The sample was then cooled at room temperature.

The compacted samples were characterized by means of scanning electron microscopy (SEM).

The density of the consolidated samples was measured by the Archimedes Method. The hardness of the consolidated samples was measured using

a Vickers hardness tester employing a load of 100 gf applied for 12 s.

### 3. RESULTS AND DISCUSSION

Fig. 1 shows a SEM-micrograph of the mechanically milled Al-8 wt.% Zn powder for 30 hours. It consists of rounded particles with a mean particle size of 5  $\mu\text{m}$ .

During consolidation at 650 °C zinc melts and dissolves in the aluminum matrix. The figure 2 shows the diffraction patterns of the Al-8 wt.% Zn before and after consolidation. Before consolidation aluminum and zinc are present as two distinct phases, what is demonstrated by the peaks of the fcc and hcp lattices of pure aluminum and pure zinc respectively. However after consolidation the peaks of zinc disappear and only the peaks of the Al-8 wt.% Zn alloy are left. The shift of the peaks of aluminum to the right shows that zinc was dissolved in aluminum.

The microstructures of the consolidated samples, (a) Al-8 wt.% Zn and (b) Al-8 wt.% Zn-3 wt.%  $\text{Al}_2\text{O}_3$  are shown in Fig. 3 (Optical microscope images). This figure shows that the particles of a- $\text{Al}_2\text{O}_3$  are homogeneously distributed in the aluminum-zinc matrix.

Fig. 4 shows the density of the consolidated powders as a function of alumina content. The density of the Al-Zn/ $\text{Al}_2\text{O}_3$  consolidated powder decreases continuously when the alumina content increases. This is believed to occur due to increased friction forces during consolidation of the powders produced by the hard alumina particles. This friction effect is increased by the submicrometer particle size of alumina. Density of the composites with pure aluminum matrix remains approximately constant at very low values. In this case the friction effect of alumina is not clear due to the very low density reached for samples without  $\text{Al}_2\text{O}_3$  particles. The upper line (theoretical density) indicates the expected values for a 100% dense material for the Al-8 wt.% Zn/ $\text{Al}_2\text{O}_3$  composite.

The variation of hardness as a function of alumina content for Al-8 Zn/ $\text{Al}_2\text{O}_3$  composites is shown in Fig. 5. It can be observed an initial increase of the hardness at low alumina content. However, at high alumina content hardness decreases as a consequence of the increased porosity of the sample. Hardness of sintered aluminum is not reported due to the great porosity of the samples making difficult to take a reliable measurement. In

this figure the hardness of pure wrought aluminum is shown for comparison.

Zinc forms a liquid phase at the sintering temperatures and it is alloyed to aluminum as is shown in Fig. 2. This liquid phase helps to fill the voids between particles by capillary forces and creates bonds between matrix particles, therefore increases density and hardness. The alumina particles increase the hardness but reduce density due to friction effects. Al and zinc form a solid solution, so additional increase in hardness is obtained in the Al-8Zn by solid solution strengthening.

### 4. SUMMARY

1. The zinc addition has a strong effect on consolidation behaviour. Zinc melts during sintering and helps to fill the pores and to create strong bond between particles of the matrix, therefore increases density and hardness of the consolidated samples.
2. Hardness and density were influenced by the alumina content. Increasing alumina content decreases density due to friction effects. Alumina increases hardness of the Al-8Zn until 2 wt.% but higher contents decreases the hardness due to increased porosity (low density).

### ACKNOWLEDGEMENTS

This work has been supported by Instituto Politécnico Nacional and CONACYT (México). One of the authors (DHS) is recipient of COFAA-IPN and SNI fellowship.

### REFERENCES

- [1] *ASM-Handbook, Vol. 7 Powder Metal Technologies and Applications* (ASM International, Materials Park, Ohio, 1998).
- [2] E. Nembach, *Particle Strengthening of Metals and Alloys* (John Wiley and Sons, 1997).
- [3] *Dispersion Strengthened Aluminum Alloys*, ed. by J. W. Kim and W. M Griffith (TMS, 1988).
- [4] Sang-Chul Kim, Moon-Tae Kim, Sungkyu Lee, Hyungsik Chung and Jae-Hwan Ahn // *Journal of Materials Science* **40** (2005) 4441.
- [5] C.C. Koch, In: *Processing of Metals and Alloys, Materials Science and Technology-A Comprehensive Treatment, Vol. 15*, ed. by R.W.Cahn (VCH, 1991).
- [6] C. Suryanarayama, R. Sundaresan and F.H. Froes, In: *Structural Applications of Mechanical Alloying*, ed. by F.H Froes (ASM, 1990).