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# Microstructural Analysis of Al/Al<sub>2</sub>O<sub>3</sub>/Gr Powder Composites Produced by Mechanical Alloying

Powder samples of Al/Al<sub>2</sub>O<sub>3</sub>/Gr hybrid composites with different weight percents were obtained by mechanical alloying in a high energy ball mill. The aim of this study is to investigate the effect of alumina and graphite particles content on the microstructure of Al/Al<sub>2</sub>O<sub>3</sub>/Gr hybrid composites. Results obtained using Scanning Electron Microscopy (SEM) as well as Energy-Dispersive X-ray Spectroscopy (EDS) show that the addition of alumina particles as the reinforcement has a drastic effect on the size and morphology of the composite powders. Also, the addition of graphite particles as one of the reinforcing components is presumed to improve tribological properties by forming a graphite-rich lubricant film between the sliding surfaces.

Keywords: composites, powder, microstructure, morphology.

# 1. Introduction

Mechanical alloying (MA) is a rapidly developing technology capable of producing a wide range of dispersion strengthened, energetic, nanocrystalline, and other advanced alloys. This technology is used for metals, ceramic and polymers. The most important advantage consists in the possibility for synthethizing of unique alloys that are not accessible by any other fabrication technique, for improvement of their mechanical and physical properties [1, 2].

Mechanical alloying is a simple and useful technique for attaining a homogeneous distribution of the inert fine particles within a fine grained matrix. A strongly influence during the milling process has the mill microdynamics which controls both the microstructural evolution and the final structure of the obtained material [3]. Agglomeration of the reinforcement particles is one of the main challenges towards achieving a homogeneous distribution of the ceramic phase in the metal matrix. When the size of the reinforcement is small and its volume fraction is high this becomes even more significant. These problems could be overcome by a rational usage of the processing technique [4].

Uniform dispersion of the fine reinforcements and a fine grain size of the matrix contribute to improve the mechanical properties of the composite. On the structural evolution during ball milling a great effect is the addition of ceramic reinforcements into a ductile matrix [3, 4]. This research is focused on production of Al/Al<sub>2</sub>O<sub>3</sub>/Gr hybrid composites by mechanical alloying method and on investigation of composites microstructures. These composites have been achieved through an advanced powder metallurgy technique known as high-energy milling.

High-energy ball milling (also known as mechanical alloying) involves repeated cold welding, fracturing, and rewelding of powder particles at the atomic level. During each collision the powder particles get trapped between the colliding balls, between the ball and the vial walls and suffering severe plastic deformation. The grain structure is refined by the continuous interaction between the fracture and welding events which leads to a uniform distribution of the reinforcement particles in the metallic matrix [5].

The objective of the present investigation is to synthesize and characterize aluminum matrix composites reinforced with different content of alumina and graphite particles.

## 2. Experimental procedure

The pure Al powder (99.5% purity and a size of 44  $\mu m$ ), Al\_2O\_3 powder (45  $\mu m$  in size and 98.5 % purity) and graphite powder (32  $\mu m$  in size) were mixed together and mechanically alloyed. Figure 1 shows the SEM micrographs of the initial powders.



**Figure 1.** SEM micrographs of as-received (a) aluminum, (b) Al<sub>2</sub>O<sub>3</sub>, and (c) Gr powders, magnification 500 X.

The pure component powders of Al,  $Al_2O_3$  and graphite in the desired volume fractions, were mixed under normal conditions. High-energy milling of powders was conducted in a RETSCH PM 400 high energy ball mill to produce the composite powders. Zinc stearate amount up to 1.0 wt. % of the total powder charge was used as the process control agent to avoid any unwarranted and excessive cold

welding of powder particles amongst themselves, onto the walls of the vial, and to the surface of the grinding medium during milling. Were used different contents of  $Al_2O_3$  particles (10, 15 and respectively 20 wt. %) and graphite was maintained constant (1 wt. %).

Alloying was performed in a high energy ball mill using a ball-to-powder ratio of 10:1. The milling time was two hours and the rotational speed was 300 rpm. Was analyzed the influence of  $Al_2O_3$  particles in the hybrid composites.

Aluminum alloys possess a number of mechanical and physical properties (low density, good resistance to corrosion, low thermal expansion) that make them attractive for automotive applications, but they exhibit extremely poor resistance to seizure and galling.

 $Al_2O_3$  was chosen as the reinforcement since it is chemically inert with AI and can also be used at higher temperatures, comparative with the un-reinforced aluminum, also having good benefits on the mechanical properties, especially on the creep resistance. Graphite is presumed to improve tribological properties by forming a graphite-rich lubricant film between the sliding surfaces. The graphite-rich lubricating film reduces the friction coefficient, disintegrates the wear products, accelerates heat abstraction and increases seizure resistance.

The obtained composites were characterized for their microstructure, size and distribution of  $Al_2O_3$  and graphite particles in the AI matrix using electron microscope (SEM) Philips XL30 ESEM type equipped with X-ray energy disspersive spectrometer (EDS) for compositional analysis.

#### 3. Analysis

The morphological and microstructural changes after mechanical alloying of Al mixed powder with alumina (10, 15 and 20 wt. %) and graphite (1 wt. %) particles were studied. The main objective of the investigation was to ensure that a homogeneous distribution of  $Al_2O_3$  and graphite in an aluminum matrix was achieved after MA. A uniform distribution of reinforcements could potentially result in composites with improved mechanical properties. However, achieving this uniform distribution requires careful synthesis since clustering of the reinforcement particles would be a major constraint.

Figure 1 (a, b and c) illustrates the morphology of the as-received particles which exhibit different shapes: aluminum particles have irregular shape, alumina is spherical shape and graphite particles appear like flakes. The microstructures of the obtained composites with different alumina contents are presented in the following figures. After two hours of milling, the initial particles were deformed and the changes from the initial shapes were noticed (figures 2, 4 and 6). EDS analysis of the hybrid composite are illustrated in figures 3, 5, and 7 respectively.

Figure 2 shows SEM micrographs of the Al/Al<sub>2</sub>O<sub>3</sub>/Gr hybrid composite containing 10 wt. % Al<sub>2</sub>O<sub>3</sub>, respectively 1 wt. % Gr, milled for two hours. In figure 3 is presented the EDS analysis of this composite.



**Figure 2.** SEM micrograph of the Al/10%Al<sub>2</sub>O<sub>3</sub>/1%Gr hybrid composite, magnification 500 X.



Figure 3. EDS analysis of  $Al/10\% Al_2O_3/1\% Gr$  hybrid composite.

Figure 4 shows SEM micrographs of the Al/Al<sub>2</sub>O<sub>3</sub>/Gr hybrid composite containing 15 wt. % Al<sub>2</sub>O<sub>3</sub>, respectively 1 wt. % Gr, milled for two hours. In figure 5 is presented the EDS analysis of this composite.



**Figure 4.** SEM micrograph of the Al/15%Al<sub>2</sub>O<sub>3</sub>/1%Gr hybrid composite, magnification 500 X.



Figure 5. EDS analysis of  $AI/15\% AI_2O_3/1\% Gr$  hybrid composite.

Figure 6 shows SEM micrographs of the Al/Al<sub>2</sub>O<sub>3</sub>Gr hybrid composites containing 20 wt. % Al<sub>2</sub>O<sub>3</sub>, respectively 1 wt. % Gr, milled for two hours. In figure 7 is presented the EDS analysis of this composite.



Figure 6. SEM micrograph of the Al/20%Al<sub>2</sub>O<sub>3</sub>/1%Gr hybrid composite, magnification 500 X.



The powder particles, in the early stages of milling, are still soft and cold welding predominates, consequently, the particle size increasing due to their coalescence. For increasing the brittleness of Al and consequently the rate of fractur-

ing are added the alumina particles. The particles get work hardened; become more brittle and their fracture leads to a reduction in particle size.

During mechanical milling of powders, the morphology and structure of the particles undergo continuous changes. Plastic deformation, welding, and fracture of the particles are dominant mechanisms which influence the characteristics of milled powders. When soft aluminum powder is milled, the milling energy deforms the particles and changes their morphology from equiaxed to flatten.

It is apparent that the plastic deformation, welding and fracture of the aluminum matrix are influenced by the addition of alumina. From the micrographs presented in figures 2, 4 and 6 respectively, it is clear that the particle sizes are fine and that the distribution also is uniform.

In order to ensure that the high-energy milling process did not introduce any contamination into the milled powder, EDS analysis of the as-milled powders was conducted (see figures 3, 5, and 7). The presence of only Al, O and C (graphite) is noticed from the peaks present in the spectrum, confirming that the milled powder did not contain any additional element due to contamination from the milling media (grinding vials and balls). From the absence of clear peaks from any other element, it is concluded that no significant contamination of the milled powders had occurred.

# 4. Conclusion

Three different  $Al/Al_2O_3/Gr$  powder composites were obtained:  $Al/10\% Al_2O_3/1\% Gr$ ;  $Al/15\% Al_2O_3/1\% Gr$ ;  $Al/20\% Al_2O_3/1\% Gr$ .

Using the mechanical alloying technique was obtained a homogenous distribution of the reinforcement phases in the Al matrix. This has been demonstrated for volume contents as high as 20% for alumina and particle sizes as small as 32  $\mu m$  (graphite) and 45  $\mu m$  (alumina).

The additions of alumina particles accelerate the milling process, leading to faster work hardening rate and fracture of the aluminum matrix.

By using the zinc stearate as process control agent we prevent the excessive cold welding of powder particles amongst themselves, onto the walls of the vial, and to the surface of the grinding balls during milling.

The obtained composite powders did not contain any additional element due to contamination from the milling media; this was observed from the EDS analysis.

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