

## OBTAINING AND CHARACTERIZATION OF Al/Al<sub>2</sub>O<sub>3</sub>/Gr COMPOSITES BY MECHANICAL ALLOYING

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*Lucrarea prezintă rezultatele cercetărilor experimentale pentru obținerea unor pulberi compozite de tipul Al/Al<sub>2</sub>O<sub>3</sub>/Gr folosind metoda alierii mecanice. Elaborarea acestor compozite prin metode convenționale este dificilă datorită fatului că rezultă produse cu proprietăți mecanice scăzute datorită structurii neomogene a materialului obținut. Pulberi elementare de aluminiu, alumina și grafit au fost aliate mecanic într-o moară de mare energie tip RETSCH PM 400. Amestecurile de pulbere au fost măcinate timp de două ore. Probele prelevate fiind apoi caracterizate prin analiză SEM și EDS. Experimentele au indicat faptul că prin metoda folosită se pot obține pulberi compozite cu o bună omogenitate.*

*The paper presents the results of experimental research in production of Al/Al<sub>2</sub>O<sub>3</sub>/Gr powder composites using mechanical alloying. Obtaining of these composites by conventional methods is difficult because the resulting products have low mechanical properties due to the structural unhomogeneity of the obtained material. Aluminum, alumina and graphite elemental powders have been mechanically alloy in a high-energy ball mill RETSCH PM 400. Powder mixtures were mill for two hours. The resulted samples then been characterized by SEM and EDS analysis. Experiments indicate that this method is appropriate for obtaining composites with better homogeneity.*

**Keywords:** mechanical alloying, composites, powder, characterization

### 1. Introduction

Mechanical alloying is a very useful method for producing advanced materials that implies powder metallurgical processes and it results in homogeneous distribution of the fine particles within a fine grain matrix [1]. Processing in solid state (powder metallurgical processes) is the most suitable

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method for making metal matrix composites. In comparison with the melting methods, its most important advantage is low processing temperature [2]. PM processes involve pressing and sintering and/or forging of powder mixtures and composite powders, extrusion or forging of metal-powder particle mixtures, and extrusion or forging of spraying compatible precursor materials [3].

Mechanical alloying (MA) involves repeated welding, fracturing, and rewelding of powder particles at the atomic level. By this route, one can obtain submicron or nanocrystalline powders, amorphous phases, intermetallics and composites at room temperature [4, 5].

Ceramic reinforcement's addition into a ductile matrix has a great effect on the structural evolution during ball milling. Reinforcement of aluminum (matrix phase) with solid lubricants (graphite) and non-metallic ceramic particles ( $\text{Al}_2\text{O}_3$ ) present in volume fraction less than 30% when used for structural and wear resistance applications, results in advanced aluminum metal-matrix composites (AMC) [6]. The reinforcement elements in the hybrid composite leads to improvement of the tribological properties by forming a graphite-rich film between the surfaces in friction, giving a coefficient of friction and low wear rate due to graphite and obtaining of tribological characteristics and hardness much better than the alloy matrix in the case of alumina particles [7].

Mechanical alloying of aluminum alloys of various compositions generally brings an increase of grain hardness. Strengthening does not depend on the initial hardness of the alloying component but, rather, on the kinetics of mechano-chemical reactions defining the formation of the strengthening phases [8].

The paper presents the technique of mechanical alloying (MA) on producing  $\text{Al}/\text{Al}_2\text{O}_3/\text{Gr}$  composites on a high energy ball mill. Investigations were made on elemental powders (Al,  $\text{Al}_2\text{O}_3$ , Gr) and alloyed powders using microstructural analysis and following the microstructural changes. For these measurements SEM and EDS analysis was used.

## 2. Materials and methods

To obtain  $\text{Al}/\text{Al}_2\text{O}_3/\text{Gr}$  composite the milling technique in a high energy planetary mill RETSCH type (Fig. 1 a) was used. The vials used for grinding (Fig. 1 b) are made of sintered alumina, as well as the grinding balls (Fig. 1 c). The planetary mill has a rotational speed from 30 to 400 rpm, speeds that can ensure a very high finesse of powders. The mill is able to reverse the direction of rotation, which helps to prevent the powder deposit on the walls of the vial. The milling chamber is automatically force ventilated, which prevents excessive temperature increase in the milling chamber.

The volume fraction of reinforcement was 20% for  $\text{Al}_2\text{O}_3$  and for graphite was 1 and 3 % respectively. The work parameters used for mechanical alloying of

the powder mixture of Al/Al<sub>2</sub>O<sub>3</sub>/Gr: rotation speed of 300 rpm, the milling time was two hours, the ball-to-powder weight ratio was 10:1 and the filling grade was approximately 80%. Since milling atmosphere can also influence the nature of the final phase, mechanical milling of Al/Al<sub>2</sub>O<sub>3</sub>/Gr powders experiments were conduct in normal atmosphere.



Fig. 1. a) High energy planetary mill RETSCH PM400; b) Milling vial; c) Milling balls

The obtained composites (Al/20%Al<sub>2</sub>O<sub>3</sub>/1%Gr and Al/20%Al<sub>2</sub>O<sub>3</sub>/3%Gr) were analyzed using a scanning electron microscope (SEM) equipped with X-ray energy disspersive spectrometer (EDS).

### 3. Results and discussion

Figs. 2 (a), 3 (a) and 4 (a) are present the SEM morphologies of aluminum, alumina and graphite powders before mechanical alloying processing. EDS analysis of the powder particles before mechanical alloying processing are presented in the Figs. 2 (b), 3 (b) and 4 (b). In tables 3, 4, 5 are present the indexed chart of the EDS analysis of aluminum, alumina and graphite powder particles before mechanical alloying processing.

Aluminum powder particles morphology is shown in Fig. 2 (a). It can be observ the elongated form of Al particles.

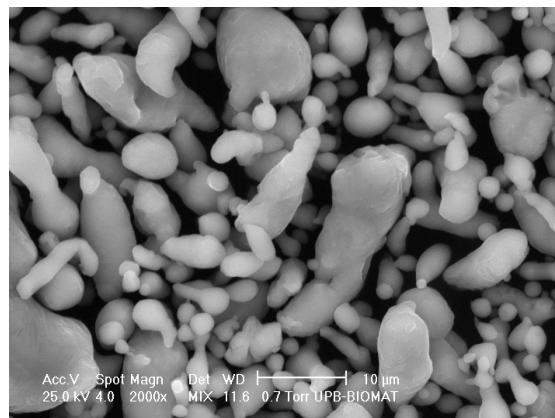


Fig. 2(a): Morphological aspect of Al powder before mechanical alloying, magnification 2000 X

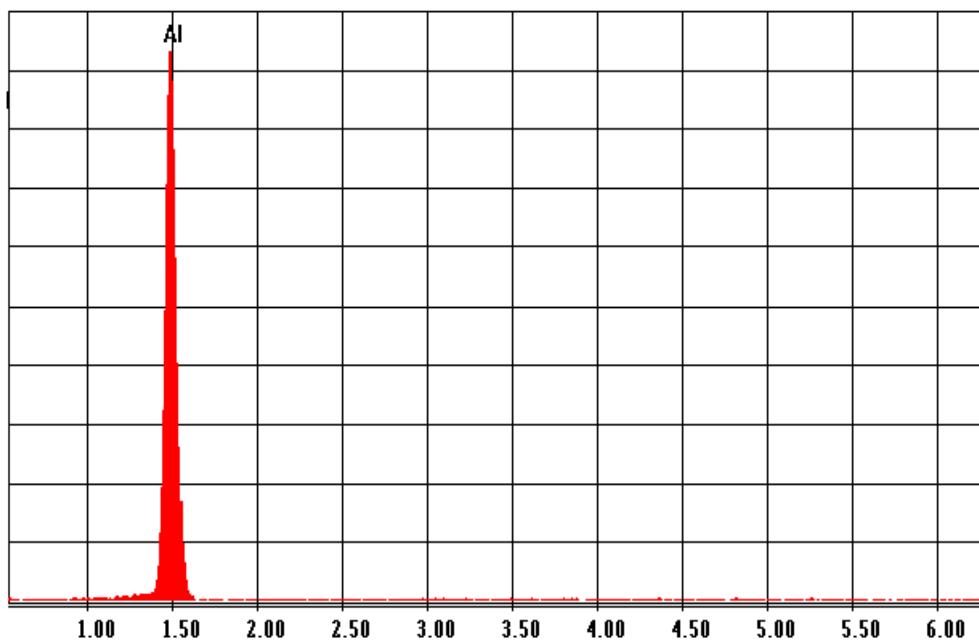


Fig. 2(b): EDS analysis of aluminum powder particles before mechanical alloying processing

Table 3

**Indexing chart of the Fig. 2 (b) diffractogram of aluminium powder**

Element	Wt.%	At. %	K-Ratio	Z	A	F
Al	100	100	1	1	1	1
Total	100	100				

Alumina powder particles morphology is shown in Fig. 3 (a). It can be observed the polyhedral shape and fragile appearance of Al<sub>2</sub>O<sub>3</sub> particles.

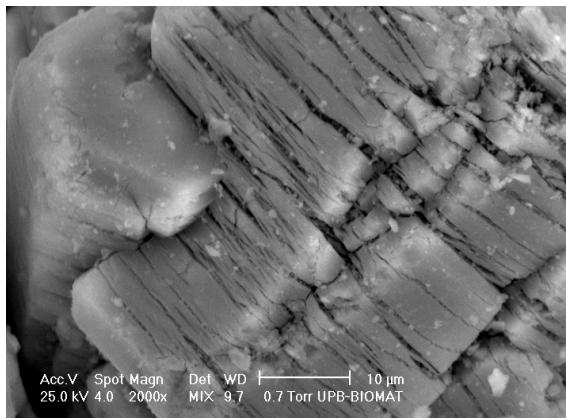


Fig. 3(a): Morphological aspect of Al<sub>2</sub>O<sub>3</sub> powder before mechanical alloying, magnification 2000 X

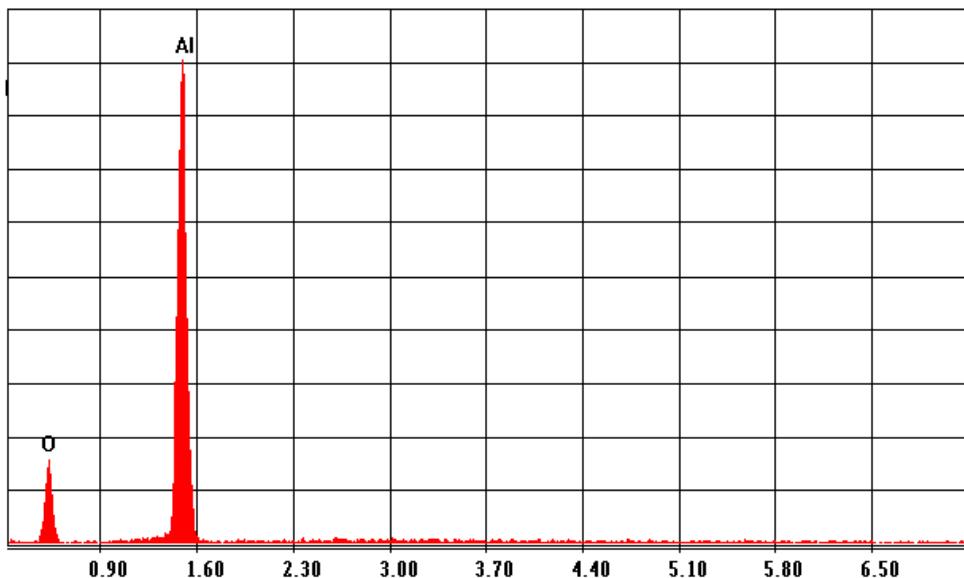


Fig. 3(b): EDS analysis of alumina powder particles before mechanical alloying processing

**Indexing chart of the Fig. 3 (b) diffractogram of alumina powder**

*Table 4*

Element	Wt.%	At. %	K-Ratio	Z	A	F
O	41,51	54,48	0,1402	1,0394	0,3247	1,001
Al	58,49	45,52	0,4007	0,9712	0,7054	1
Total	100	100				

Graphite powder particles morphology is shown in Fig. 4 (a). It can be observed the flake shape of graphite particles.

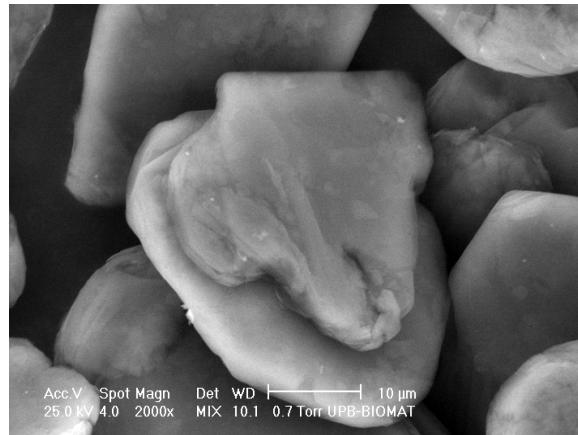


Fig. 4(a): Morphological aspect of Gr powder before mechanical alloying, magnification 2000 X

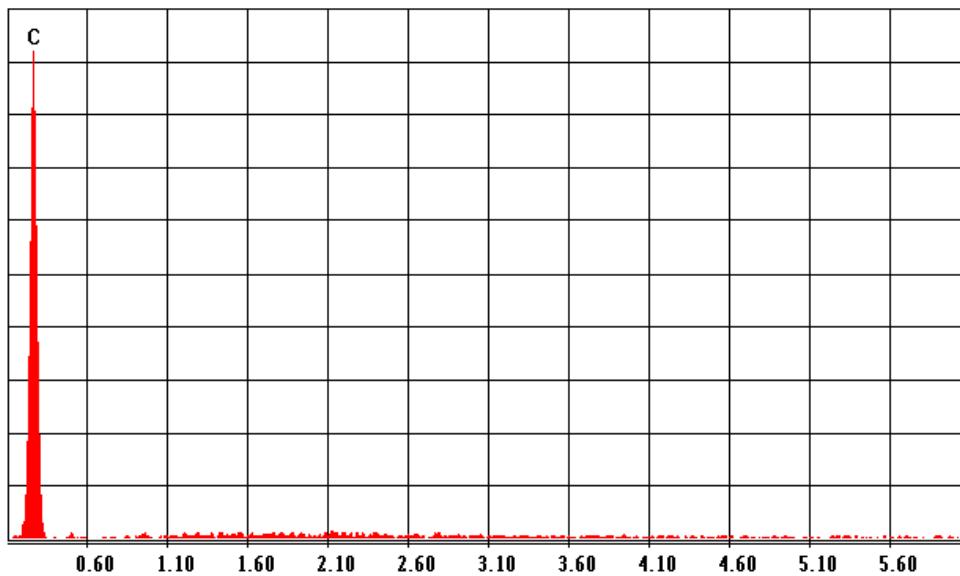


Fig. 4(b): EDS analysis of graphite powder particles before mechanical alloying processing

**Indexing chart of the Fig. 4 (b) diffractogram of graphite powder**

*Table 5*

Element	Wt.%	At. %	K-Ratio	Z	A	F
C	100	100	1	1	1	1
Total	100	100				

The results of the SEM microscopy investigation on the obtained Al/20%Al<sub>2</sub>O<sub>3</sub>/1%Gr and Al/20%Al<sub>2</sub>O<sub>3</sub>/3%Gr composites are presented in fig. 5 (a) and 6 (a). It can be remarked from fig. 5 (a) the irregular particles with sizes between 5  $\mu$ m and 25  $\mu$ m and from fig. 6 (a) respectively the particles with rounded edges and dimensions between 2  $\mu$ m and 20  $\mu$ m.

EDS analysis of the composites powder particles after mechanical alloying processing are presented in the Figs. 5 (b) and 6 (b). In table 6 and 7 are presented the indexed chart of the EDS analysis of Al/Al<sub>2</sub>O<sub>3</sub>/Gr composite powder particles after mechanical alloying processing.

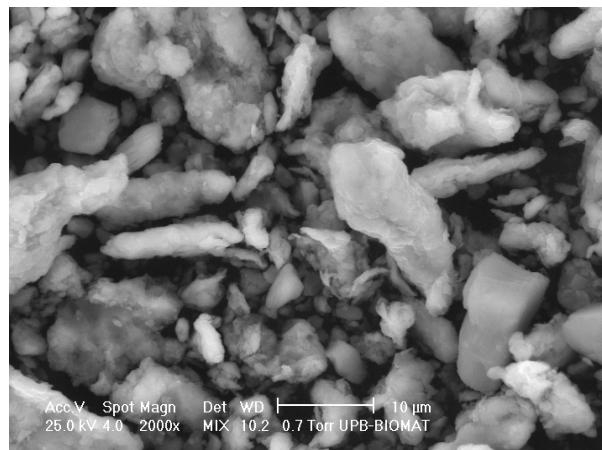


Fig. 5(a): Morphological aspects of Al/20% Al<sub>2</sub>O<sub>3</sub>/1% Gr composite, magnification 2000 X

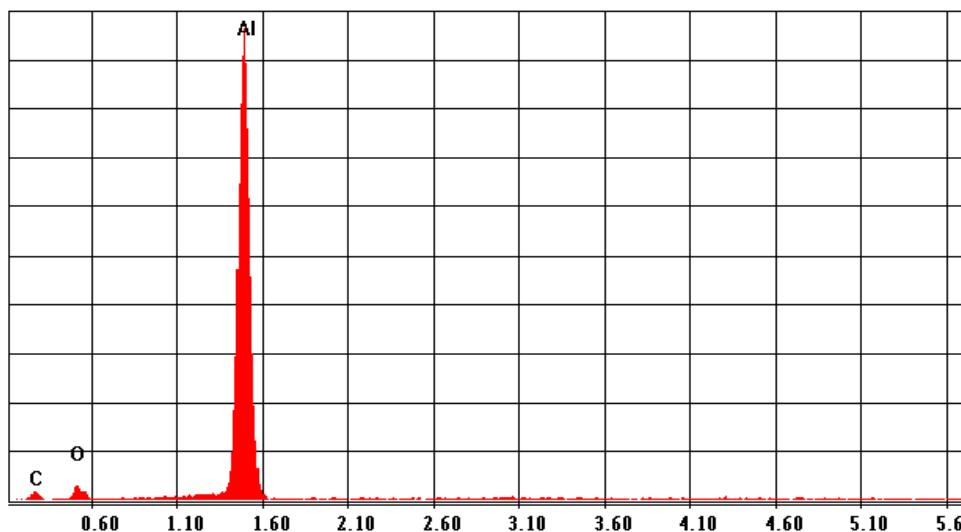


Fig. 5(b): EDS analysis of Al/20% Al<sub>2</sub>O<sub>3</sub>/1% composite after mechanical alloying

**Table 6**  
**Indexing chart of the Fig. 5 (b) diffractogram of Al/20% Al<sub>2</sub>O<sub>3</sub>/1% composite powder**

Element	Wt.%	At. %	K-Ratio	Z	A	F
C	30,24	46,33	0,041	1,0488	0,1292	1,0002
O	13,02	14,98	0,0251	1,0328	0,1864	1,001
Al	56,76	38,69	0,4575	0,9651	0,8356	1
Total	100	100				

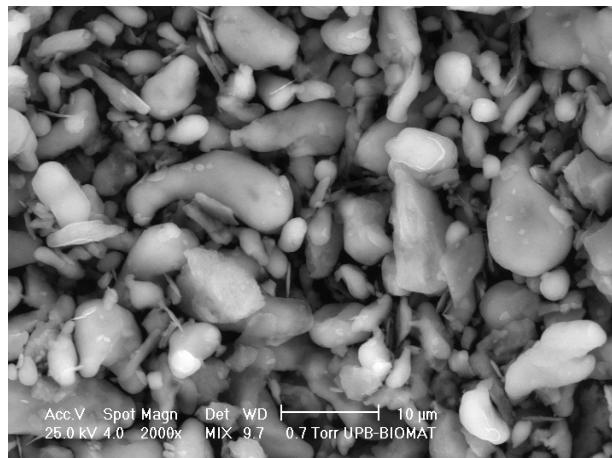


Fig. 6(a): Morphological aspects of Al/20% Al<sub>2</sub>O<sub>3</sub>/3% Gr composite, magnification 2000 X

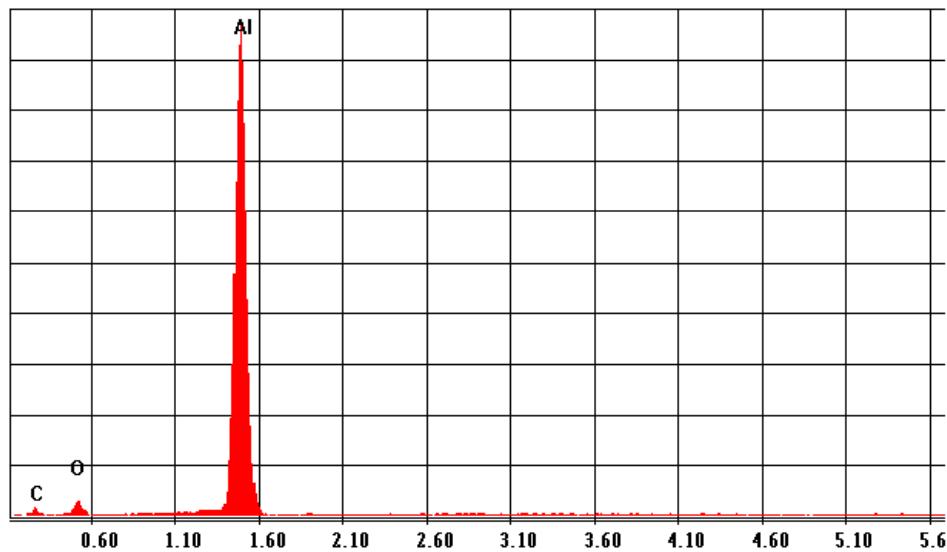


Fig. 5(b): EDS analysis of Al/20% Al<sub>2</sub>O<sub>3</sub>/3% Gr composite after mechanical alloying

Table 7

Indexing chart of the Fig. 5 (b) diffractogram of Al/20% Al<sub>2</sub>O<sub>3</sub>/3% Gr composite powder

Element	Wt.%	At. %	K-Ratio	Z	A	F
C	24,73	39,7	0,0312	1,0535	0,1197	1,0002
O	13,3	16,03	0,0269	1,0375	0,1948	1,0011
Al	61,96	44,27	0,5065	0,9694	0,8432	1
Total	100	100				

From these figures it could be noticed that the Al<sub>2</sub>O<sub>3</sub>, Gr particles and, composite particles are clearly defined. After mechanical alloying of Al/Al<sub>2</sub>O<sub>3</sub>/Gr composites, the shape of the particles become irregular or with rounded edges also. There is uniform repartition of the components with a very fine granulation.

#### 4. Conclusions

The results obtained in the present work can be used to draw the following conclusion:

- ❖ The scanning electron microscopy images have emphasized that the particles obtained had mainly a spherical or ellipsoidal shape.
- ❖ Al/Al<sub>2</sub>O<sub>3</sub>/Gr powders with a very fine granulation and a uniform repartition has been obtained by mechanical alloying.
- ❖ The above mention aspects proved the possibility to manufacture Al/Al<sub>2</sub>O<sub>3</sub>/Gr submicronic powders by mechanical alloying.
- ❖ Repeated welding, fracturing, and rewelding of powder particles at the atomic level by mechanical alloying lead to a better uniformity of the particles structure.
- ❖ The method presented for manufacturing of aluminum matrix composite materials reinforced by ceramic particles ensures required structure and can be apply in practice.

Advantageous of powder metallurgy method is possibility of manufacturing small parts with near net shape.

–The application of aluminum reinforced with Al<sub>2</sub>O<sub>3</sub> and/or Gr particles is in automotive industry for part like pistons, engine blocks, brake rotors, drums, calipers, connecting rods, drive shafts, snow tire studs and other parts (valves, crankshafts, gear parts and suspension arms) where the mechanical and tribological properties of the material are very important.

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