STUDIES REGARDING TECHNOLOGICAL PROPERTIES OF $Al/Al_2O_3/Gr$ HYBRID COMPOSITES

Gheorghe IACOB[1], Gabriela POPESCU[2], Mihai BUZATU[3]

This paper presents experimental results regarding fabrication through powder metallurgy of $Al/Al_2O_3/Gr$ hybrid composites. The effects of mechanical alloying time (2 and 4h) and Al_2O_3/Gr content (15/1 and 15/3 wt. %) on the technological properties of the composites were investigated. The powder mixtures were mechanical alloyed in a high-energy ball mill and then pressed into cylindrical performs with a diameter of 40 mm under 150 MPa pressure in order to achieve green compacts. Sintering experiments were conducted under controlled atmosphere at 620°C for 1 h. The characterization of composite powders ($Al/Al_2O_3/Gr$) was performed using analysis techniques specific for P/M process and chemical composition analysis.

Keywords: characterization, mechanical alloying, powders compacting, sintering

1. Introduction

The powder metallurgy route for manufacturing composites materials offers some advantages compared with ingot metallurgy or diffusion welding, the most important being the low manufacturing temperature that avoids strong interfacial reaction, minimizing the undesired reactions between the matrix and the reinforcement [1]. Composites that use particles as reinforcement can be obtained easily by powder metallurgy route; moreover, particles are cheaper than continuous/discontinuous fibers of the same composition [2]. Al and its alloys were for some time the most widely used materials as the MMCs matrix in research and development and in industrial applications due to its lower density and cost compared with other low density alloys. Some of the most widely used materials for reinforcing aluminum and aluminum alloys are ceramic particles (e. g. Al_2O_3 and SiC). Al_2O_3 compared with SiC it is more stable and inert and has better corrosion and high temperature behavior [3, 4].

Mechanical alloying (MA) is a high-energy ball milling technique using various types of mills in which a mixture of different powders is subjected to

[1] PhD. student, Materials Science and Engineering Faculty, University POLITEHNICA of Bucharest, Romania, e-mail: iacob_gh@yahoo.com
[2] Prof., Materials Science and Engineering Faculty, University POLITEHNICA of Bucharest, Romania, e-mail: gabriela.popescu@.upb.ro
[3] Prof., Materials Science and Engineering Faculty, University POLITEHNICA of Bucharest, Romania, e-mail: buzatu.mihai@yahoo.com

highly energetic compressive forces. By the repeated fracture and cold welding of the constituent powder particles, it is possible to make alloys from normally immiscible components [5]. It has been reported that aluminum matrix composites obtained by mechanical alloying and subsequent hot extrusions have exhibited desirable mechanical properties at room and elevated temperatures. This is due to uniformly distributed micron scale and/or nano-sized particles together with extremely fine grained Al matrix formed during mechanical milling process [6].

MA process starts with the blending of elemental and/or prealloyed powders in the correct proportion and loading of this blend into a mill along with the grinding medium. The powder and balls are then agitated for the time required to reach a steady state, at which point the composition of all of the powder particles is the same [7]. The powder is then consolidated into a bulk shape and heat treated to obtain the desired microstructure and mechanical properties. Thus, the important components of MA are the raw materials, the ball mill, and the milling, consolidation, and heat treatment parameters [8]. In some cases, especially when milling ductile metal powders (such as Al), a process control agent (PCA) is added to minimize the extent of particle agglomeration [9].

P/M manufacturing technology consists in mixing elemental or alloyed powders, compacting of those powders in a die at room temperature and then sintering or heating the shape in a controlled atmosphere furnace for particles bonding. The designer can adjust the chemistry and other powder metallurgy characteristics, such as density, to provide a custom result, uniquely suited for the application [10, 11]. The compacted or “green” part has the size and shape of the finished part when ejected and has sufficient green strength to be handled and transported to the sintering furnace. Then the sintering step transforms compacted mechanical bonds between the powder particles into metallurgical bonds by a solid state transformation process [12]. In addition to the metallurgical and chemical properties derived from the materials there are a number of other properties that are important to a P/M material. It includes particle size distribution, particle shape, flow rate, bulk density, compressibility, green strength and the spring-back characteristics of the powder [13].

In the present study, $Al/Al_2O_3/Gr$ composites with different content of component powders were produced via mechanical alloying, compacting and sintering. The effects of milling time and of Al_2O_3/Gr content (wt. %) on the technological properties values of the composite materials were evaluated.

2. Materials and Methods

The elemental powders were mixed together and mechanically alloyed in a RETSCH PM 400 high-energy ball mill. The reinforcement content was 15 wt. % in case of Al_2O_3 and respectively 1 and 3 wt. % for graphite. The working parameters used for MA of powders were: rotation speed 300 rpm; milling time was varied from 2 to 4 hours; BPR (Ball to Powder Ratio) was 10:1; filling grade was approx. 75%; normal atmosphere. Mechanical alloying is carried out with elemental powders and an organic process control agent (PCA), such as zinc stearate, to balance the cold-welding and powder-fracture processes. No dispersoid is added because the oxide on the surface of the powders and process control agent are consolidated during mechanical alloying as hydrated oxides and carbonates. For all mixtures 1 wt. % zinc stearate powder lubricant was added and milled together with the constituent powders in order to reduce friction between the powder mass and the surface of the die and obtain a good compaction. To obtain $Al/Al_2O_3/Gr$ composite experimental plan was pursued following Table 1.

Table 1

Chemical composition of the powder mixture

Composite	Matrix, (wt. %)	Reinfocement, (wt. %)		
	Al	Al_2O_3	Gr	Zn stearate
Al/15wt.%Al_2O_3/1wt%	83	15	1	1
Al/15wt.%Al_2O_3/3wt%	81	15	3	1

Apparent density, tap density, porosity, compressibility and green strength are referring to the physical-technological properties of powders. Knowledge of these technological parameters coupled with the physical properties enables evaluation of the powders behavior during further processing that is accomplish by their consolidation/ sintering.

The apparent density of the powder is a very important parameter that depends on the physical characteristics and the degree of porosity of the particles. The apparent density was determined by flowing mass of powder into a container of know volume and measuring the weight of powder which completely fills the space, according to SR EN 23923-1: 1998 standards.

The tap density is a mass of loose powder that is mechanically taped (SR EN 23923-2: 1999). Theoretical density of powder mixtures represent the maximum density of material attained in the final stage, where is calculated by the rule of mixtures, after the following relationship:

$$\rho_{mixture} = \frac{100}{\sum_{i=1}^{n} \frac{x_i}{\rho_i}} \qquad (1)$$

where x_i is the fraction of component i in mixture (i being Al, Al_2O_3, and Gr); ρ_i is the component density (g/cm^3).

The measured porosity (the pore volume fraction) P of compacted respectively sintered parts was determined by the equation:

$$P = \left(1 - \frac{\rho_u}{\rho_{mixture}}\right) \cdot 100 \qquad (2)$$

where ρ_u is the measured density of green compact or sintered part, g/cm^3 and $\rho_{mixture}$ - the theoretical density, g/cm^3.

The consolidation of powders was done at room temperature in an Atlas™ 15T Manual Hydraulic Press from SPECAC. The cavity of the die was filled with a specified quantity of milled powder, the necessary pressure was applied and then the compacted part was ejected resulted a green compacted composite.

The compaction of milled powders was performed in small compacts of 40 mm diameter, at room temperature. The green density of the compacts was determined by physical measurements. The sintering operation was realized in a heating induction furnace, Balzers type, the sintering operation parameters being: sintering temperature: 620ºC; 60 minute exposure time to sintering temperature.

3. Results and discussions

The determined technological properties specific to powder metallurgy process for the constituent powders and for composite powders are presented in table 2 and respectively table 3.

Table 2

Technological properties of elemental powders

Powder	Apparent density (g/cm^3)	Tap density (g/cm^3)
Al	0.7879	1.2151
Al_2O_3	1.0475	1.3898
Gr	0.665	0.8532

Table 3

Technological properties of composite powders

Composite powders	Apparent density (g/cm^3)	Tap density (g/cm^3)
Al/15wt.%Al_2O_3/1wt%Gr - milled 2 h	0.6192	0.8317
Al/15wt.%Al_2O_3/3wt%Gr - milled 2 h	0.7898	1.0866
Al/15wt.%Al_2O_3/1wt%Gr - milled 4 h	0.7067	0.9480
Al/15wt.%Al_2O_3/3wt%Gr - milled 4 h	0.7421	0.8590

The graphical representation of the evolution of Al/Al_2O_3/Gr composites technological parameters (apparent and tap density) with different component powders content was presented in figure 1.

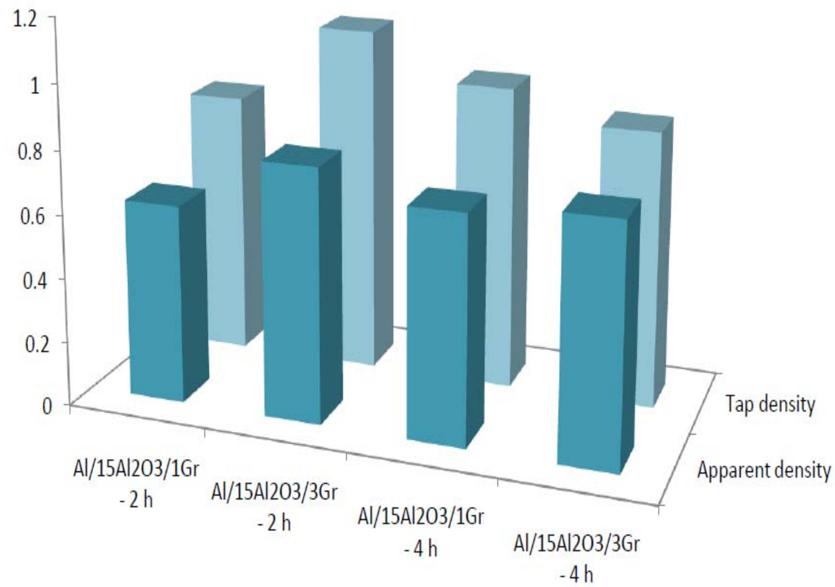

Fig. 1: Evolution of green and sintered density of composite compacts.

The highest densities were achieved in the case of Al/15wt.%Al_2O_3/3wt%Gr milled 2 h.

To ensure that high-energy milling process did not introduce any contamination into the milled powder was conducted the EDS analysis on the obtained composites (see figure 2 and 3). Compositional analysis was performed with X-ray energy dispersive spectrometer (EDS).

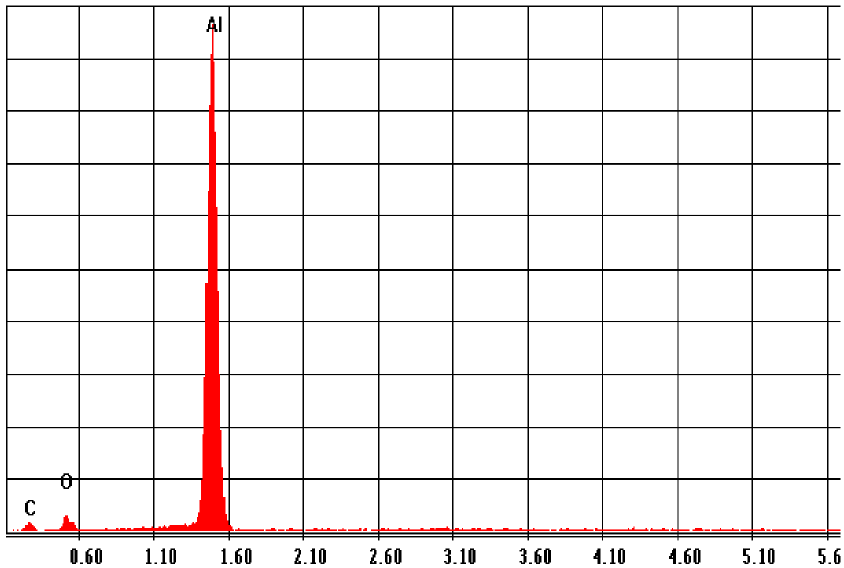

Fig. 2: Compositional analysis of Al/15wt.%Al_2O_3/1wt.%Gr hybrid composite

The presence only of Al, O and C (graphite), confirmed from the chart peaks present, indicate that the milled powders was not contaminated by additional element resulting from mechanical alloying process.

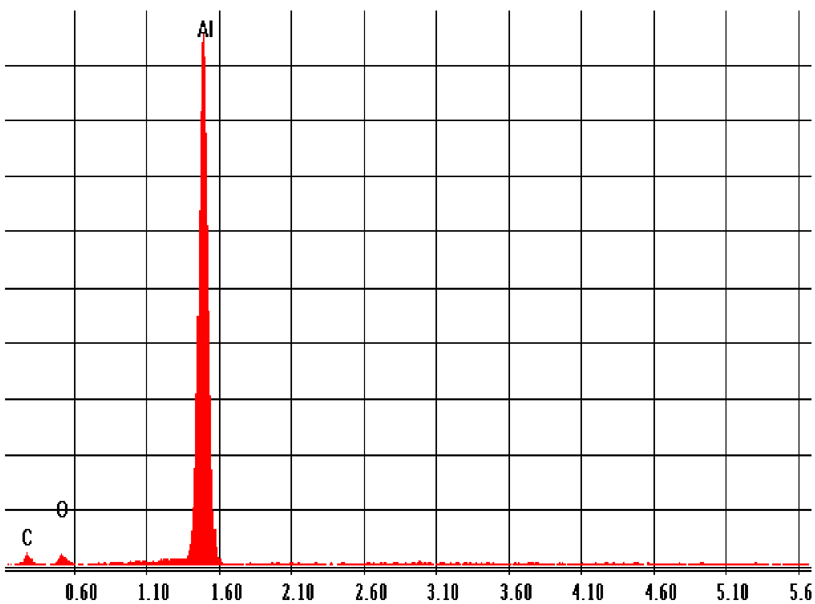

Fig. 3: Compositional analysis of Al/15wt.%Al_2O_3/3wt.%Gr hybrid composite

The technological characteristics of green compacts ae presented in table 4. A typical compacted and sintered billet is shown in fig. 4.

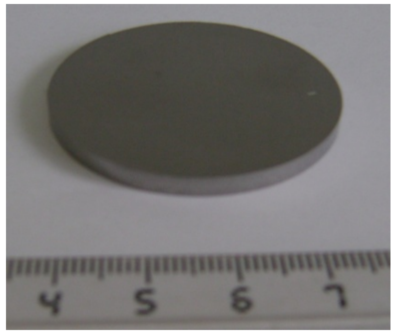

Fig. 4: $Al/Al_2O_3/Gr$ compacted and sintered composite

It can be seen that the compact has smooth surfaces with a disc shape conformed to the cavity of the compacting die.

Table 4

Technological properties after compacting process

Compacted Composite	Compact thickness (mm)	Green density (g/cm^3)	Compressibility /Porosity (%)
$Al/15wt.\%Al_2O_3/1wt\%Gr$ - milled 2 h	4	1.57	57.72 / 42.28
$Al/15wt.\%Al_2O_3/3wt\%Gr$ - milled 2 h	3.5	1.77	62.32 / 37.68
$Al/15wt.\%Al_2O_3/1wt\%Gr$ - milled 4 h	3.8	1.67	61.40 / 38.60
$Al/15wt.\%Al_2O_3/3wt\%Gr$ - milled 4 h	3,5	1.8	63.38 / 36.62

Density of the composite material after sintering processing step is summarized in table 5.

After cold compaction and after sintering, the density is lower for billet with small amount of graphite after 2 and 4 hour of milling. Sintered density is lower than green density and has an upward trend with increasing content of graphite and milling time. During sintering process releasing of gas previously trapped in the green compact cause’s slight volume expansion of the billet.

Table 5

Density after sintering process

Composite	Sintered billet thickness (mm)	Sintered density (g/cm^3)	Calculated density (g/cm^3)
Al/15wt.%Al_2O_3/1wt%Gr - milled 2 h	4.4	1.23	2.72
Al/15wt.%Al_2O_3/3wt%Gr - milled 2 h	4	1.32	2.84
Al/15wt.%Al_2O_3/1wt%Gr - milled 4 h	4.3	1.34	2.72
Al/15wt.%Al_2O_3/3wt%Gr - milled 4 h	4	1.39	2.84

The highest green densities were achieved in the case of Al/15wt.%Al_2O_3/3wt%Gr milled 4 h. The reduced compressibility of the Al/15wt.%Al_2O_3/1wt%Gr composite powder milled 2 h is also confirmed by the obtained low green density and sintered density of this compact.

During the sintering of the green compacts different shrinkages (figure 5) were observed as a result of the change of density from the green to sintered part.

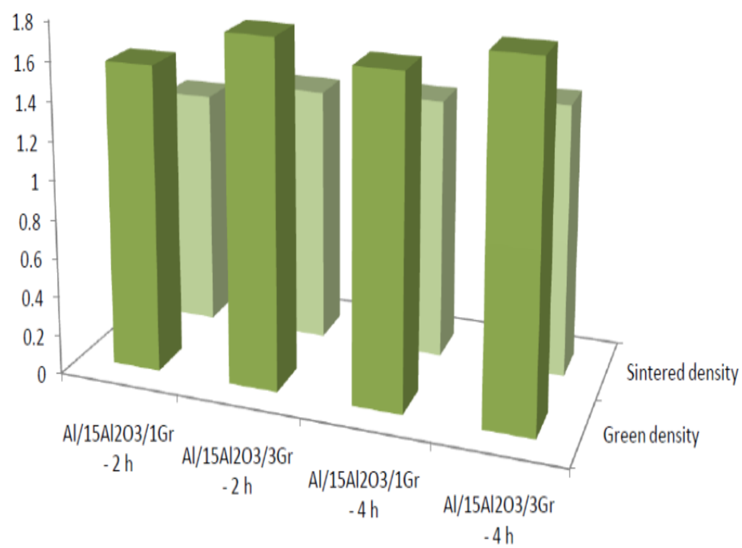

Fig. 5: Evolution of green and sintered density of composite compacts.

4. Conclusions

Two different Al/Al_2O_3/Gr composites were obtained: Al/15%Al_2O_3/1%Gr; Al/15%Al_2O_3/3%Gr on different milling time (2 and 4 hours).

Apparent and tap densities of composite powders increase with increasing Gr content in the mixture, and with increasing the milling time.

Using zinc stearate as PCA 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.

Density of green compacts and sintered billet is lower for small amount of graphite after 2 and 4 hour of milling. Introducing graphite in the powder mixture has led to the reduced density of composite material.

Sintered density is lower than green density and increase with graphite content and milling time. Al/15wt.%Al_2O_3/3wt%Gr milled 4 h has the highest green density. There was no contamination of milled powders that was confirmed by EDS analysis.

Aluminum matrix composites reinforced with ceramic particles have a weak relation between weight and volume, but high wear resistance, thermal conductivity and diffusivity, properties that are used in automotive and aerospace industry.

Acknowledgement

The work has been funded by the Sectoral Operational Programme Human Resources Development 2007-2013 of the Romanian Ministry of Labour, Family and Social Protection through the Financial Agreement POSDRU/88/1.5/S/61178.

R E F E R E N C E S

[1] *J. M. Torralba, C. E. da Costa, F. Velasco*, "P/M aluminum matrix composites: an overview", Journal of Materials Processing Technology, vol. 133, issues 1-2, 2003, pp. 203-206.

[2] *Al-Rashed, S. Holecek, M. Prazak and M. Procio*, "Powder metallurgy route in production of aluminium alloy matrix particulate composites", Journal de Physique IV, vol. 3, no. C7, nov. 1993, pp. 1821-1823.

[3] *B. Ralph, H. C. Yuen, W. B. Lee*, "The Processing of Metal Matrix Composites – an Overview", Journal of Materials Processing Technology, vol. 63, issues 1-3, 1997, pp. 339-353.

[4] *J. W. Kaczmar, K. Pietrzak, W. Wlosinski*, "The production and application of metal matrix composite materials", Journal of Materials Processing Technology, vol. 106, 2000, pp. 58-67.

[5] *L. R. Vishnyakov, N. P. Onis'Kova, A. N. Gribkov and I. M. Romashko*, "Effect of mechanical alloying on the properties of powder metallurgy composites of the Al-SiC system", Powder Metallurgy and Metal Ceramics, vol. 36, nos. 11-12, 1997, pp. 599-603.

[6] *J. B. Fogagnolo, F. Velasco, M. H. Robert, J.M. Torralba*, “Effect of mechanical alloying on the morphology, microstructure and properties of aluminium matrix composite powders”, Materials Science and Engineering: A, vol. 342, issues 1–2, 2003, pp. 131-143.

[7] *G. Le Caër et. al.*, “High-Energy Ball-Milling of Alloys and Compounds”, Hyperfine Interactions, vol. 141/142, 2002, pp. 63–72.

[8] *C. Dhadsanadhep, T. Luangvaranunt, J. Umeda, K. Kondoh*, “Fabrication of Al/Al_2O_3 Composite by Powder Metallurgy Method from Aluminum and Rice Husk Ash”, Journal of Metals, Materials and Minerals, vol.18, no. 2, 2008, pp. 99-102.

[9] *Y. Saberi, S. M. Zebarjad, G. H. Akbari*, “On role of stearic acid on morphology of Al–SiC_p composite powders produced by mechanical alloying method”, Powder Metallurgy, vol. 52, no.1, 2009, pp. 61-64.

[10] *C. Suryanarayana*, “Recent developments in mechanical alloying”, Rev. Adv. Mater. Sci., vol. 18, 2008, pp. 203-211.

[11] *T. S. Srivatsan, I. A. Ibrahim, F. A. Mohamed, E. J. Lavernia*, “Processing tehnique for particulate-reinforced metal aluminium matrix composites”, Journal of Materials Science, vol. 26, no. 22, 1991, pp. 5965-5978.

[12] *Z. R. Hesabi, H.R. Hafizpour, A. Simchi*, “An investigation on the compressibility of aluminum/nano-alumina composite powder prepared by blending and mechanical milling”, Materials Science and Engineering A, vol. 454–455, 2007, pp. 89–98.

[13] *M. Rahimian, N. Parvin, N. Ehsani*, “The effect of production parameters on microstructure and wear resistance of powder metallurgy Al–Al_2O_3 composite”, Materials and Design, vol. 32, 2011, pp. 1031–1038.