THE PRESSING-SINTERING PROCESS FOR THE PRODUCTION OF $Al/Al_2O_3/Gr$ HYBRID COMPOSITES

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The paper presents experimental results regarding the fabrication of $Al/Al_2O_3/Gr$ hybrid composite through powder metallurgy route. These composites represent a unique class of advanced materials. Powder pressing for sintering is a fairly important process, because the tablets resulting from pressing require special mechanical strength and porosity properties to give very good properties to the samples obtained after the sintering process. The pressing and sintering processes aimed to obtain a density as close as possible to the theoretical one. The compression densification process depends primarily on the granulometric distribution of the powder to be pressed. The characterization of composite powders ($Al/Al_2O_3/Gr$) were performed using analysis techniques specific for P/M processes and also other types of analysis to better understand the obtained properties: chemical composition, SEM-EDS, SEI-BSED, XRD.

Keywords: hybrid composites; aluminum based; powder metallurgy; processes

1. Introduction

Metal matrix composites (MMCs) are technical materials in which a tough ceramic component is dispersed in a ductile metal matrix in order to obtain superior characteristics to conventional alloys [1, 2]. The main area where the need for these types of composite has started was the aerospace industry for the

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spacecraft and aircraft body structure. More recently, the automotive, electronics and recreation industries have begun to use these types of materials [3-5].

The driving force behind the development of these metal matrix composites is their ability to be designed to provide the various required material behaviors depending on the chosen range such as improved strength and stiffness, excellent corrosion resistance, friction resistance and wear, high electrical and thermal conductivity and high mechanical high temperature behavior [6, 7].

Composite materials technologies provide a unique opportunity to adapt the properties of aluminum. This includes increased strength, weight reduction, higher use temperature, improved wear resistance, higher elastic modulus, thermal expansion coefficient control, improved fatigue properties, etc [8-11].

The use of complementary particulate material has been widely expanded since it has some important advantages [12-14], such as:

- low cost (compared to fibers, particles are much cheaper);
- simple incorporation and dispersion of particles in matrix;
- the possibility of obtaining isotropic materials (local isotropy or the entire section of the composite).

Regarding hybrid composite materials, the international literature contains a series of data to demonstrate the scientific applicability of Al-MMC reinforced with several types of particles (Al_2O_3, SiC, Grafit, TiC, B_4C, etc) with different granulometric sizes or shapes [1, 5-9, 13, 14].

A rapidly, simple and useful powder metallurgy technique used for metals, capable of producing a wide range of advanced alloys is mechanical alloying (MA) by high energy ball milling. Using this method, it can be obtained submicron or nanocristalline powders, amorphous phases, intermetallics and composites materials. Most important advantage of this technology consists in the synthesizing of unique composite material with improved mechanical and physical properties [15-17].

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. Knowledge of technological parameters coupled with the physical properties enables evaluation of the behavior of powders during further processing that is accomplish by their consolidation and sintering [18-20].

The process of powder metallurgy, by manufacturing the sintered parts, is fundamentally different from the classical metallurgy technology, where semi-finished products obtained by casting metals and molten alloys are processed by rolling, forging, molding, chipping, etc. thus arriving at pieces made through a large number of pretentious, costly and long-lasting operations [21-22].

Powder metallurgy provides sintered products and materials with a precise and uniform composition with a high constancy of properties, making it possible to replace expensive or deficient materials with others cheaper and easier to obtain [23].

2. Materials and methods

Mechanical alloying process starts with the loading of elemental powders in the correct proportion and of this blend into a mill along with the grinding medium. The powder and balls are then agitated for the time required [24].

Powders of Al, Al_2O_3 and graphite in the desired volume fractions (10, 15 and 20 wt. % Al_2O_3 and 1 and 3 wt. % Gr), the content difference being the material matrix, pure Al powder, were mixed together and mechanical alloyed in a RETSCH PM 400 high-energy ball mill in the following conditions: ball-to-powder ratio 10:1; milling time 2 hours and the rotational speed was 300 rpm, and led for obtaining of 6 different hybrid composite types, as seen in Table 1.

Table 1

Compositions selected for experiments

Notation	**Al (wt. %)**	**Al_2O_3 (wt. %)**	**Graphite (wt. %)**
HyCo A	89	10	1
HyCo B	84	15	1
HyCo C	79	20	1
HyCo D	87	10	3
HyCo E	82	15	3
HyCo F	77	20	3

The composite material powders of various compositions, obtained by mechanical alloying, were pressed in the form of tablets having dimensions of 40 mm, according to the inner diameter of the mold. The pressing was carried out on a hydraulic press, with nominal forces up of 15 tf. Initially, two pressing forces were established to obtain the green compacts, namely 5 and 8 tf, but the pressure was not sufficient to obtain high resistance samples (Fig. 1a and b) to sinter them, so we chose the maximum pressing force to be sure to get quite resilient samples, which were obtained as shown in Fig. 1(c). It can be seen that the obtained green compacts have a smooth surface with well-defined edges.

Physical dimensional, weight and technological characteristics (density, compaction, porosity, etc.) were determined for the samples obtained from the pressing process at a force of 15 tf. The measurements were made by volumetric methods, weighing, and measuring the tablets using a precise balance (± 0.0001), and a micrometer (± 0.1).

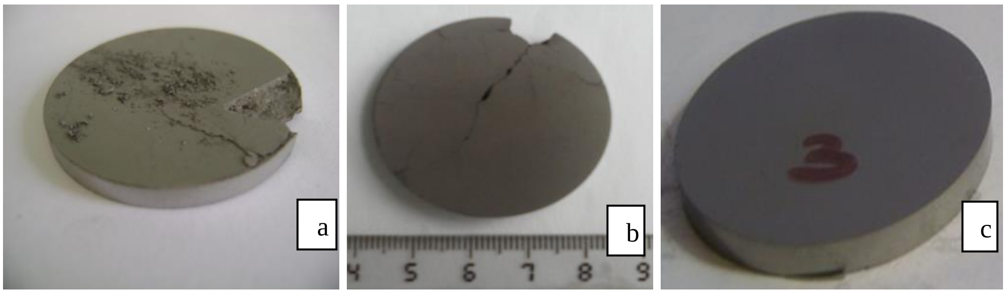

Fig. 1. Green compacts from mechanically alloyed powders, obtained at a pressing force of: a) 5 tf; b) 8 tf and c) 15 tf

Also, for a better understanding of the pressing-sintering process was performed a secondary electron image analysis (SEI), with the use of the scanning electron microscope Quanta Inspect F50, and XRD analysis using a Panalytical X'PERT MPD X-ray diffractometer with a Cu K-a radiation source (λ = 1.5418 Å) in the range of 2θ = 10–90°.

3. Experimental results and discussion

3.1. Technological properties of pressed composites

The technological properties of the hybrid composites obtained by mechanical alloying in which the ratio of the Al_2O_3 content (10, 15 and 20% by weight) and the graphite content (1 and 3% by weight) at different times of mechanical alloying (2h) subjected to the experimental pressing process were determined according to the powder metallurgy specific procedures are shown in Tables 2 and 3.

Table 2

Determination of the green compact density

Sample	***Pressing force,*** [tf]	***Tablet weight,*** [g]	***Height of the compacts h_p*** [mm]	***Volume,*** [cm^3]	***Density of the compacts*** [g/cm^3]	***Theoretical density*** [g/cm^3]
HyCo A	15	8.007	3.2	4.0212	1.9912	2.875
HyCo B	15	8.019	3.1	3.8955	2.0585	2.884
HyCo C	15	8.011	3.6	4.5238	1.7708	2.824
HyCo D	15	8.015	3.5	4.3982	1.8223	2.836
HyCo E	15	8.001	3.5	4.3982	1.8192	2.776
HyCo F	15	8.012	3.6	4.5238	1.7711	2.788

Table 3

Determination of the green compact specific technological properties

Sample	*Compactity,* [%]	*Porosity,* [%]	*Fill height,* [mm]	*Filling factor,* [mm]
HyCo A	69.26	30.74	9.65	3.02
HyCo B	71.38	28.62	8.18	2.64
HyCo C	62.70	37.30	9.24	2.56
HyCo D	64.26	35.74	7.78	2.23
HyCo E	65.53	34.47	8.49	2.42
HyCo F	63.53	36.47	7.59	2.11

The density of the tablets obtained by pressing the various $Al/Al_2O_3/Gr$ powder compositions shows a slight increase of the values, except for the samples HyCo E and F where was observed a decrease, where we can attribute this to the higher content of 20 wt.%. Compactity of the pressed tablets of mechanically alloyed $Al/Al_2O_3/Gr$ powders for 2 hours exhibits an oscillating values with increasing Al_2O_3 content from 10 to 20 wt% by and maintaining a constant content of 1 to 3 wt.% graphite, and a decreasing trend with the increase of Al_2O_3 content from 10% to 20 wt.% by and maintaining a constant volume fraction of 3 wt.% Gr. The maximum value is held by sample HyCo A and the minimum value by HyCo C. Porosity is complementary to compactity.

3.2. Technological properties of sintered composites

Thermal sintering treatment has resulted in the thermal activation of green compacts in order to carry out the diffusion processes, leading to the consolidation and increase of the bonding bridges between the particles, the densification (by decreasing the porosity) and the recrystallization of the structure.

Knowing the properties of the sintered material is necessary to evaluate, on the one hand, the correctness of the application and observation of the parameters of the operations in the technological flow of production and, on the other hand, to predict its behavior in exploitation. The sintering process was realized in a heating induction furnace, Balzers type, at a sintering temperature of 620ºC and a maintaining time of 60 minute. The heating rate was 15°C/min; the cooling of samples was carried out in the furnace at a cooling rate of 4°C/min. The technological characteristics (density, compactness, porosity, etc.) were determined. The physic-technological properties determined on the hybrid composites samples with different compositions, determined according to the specific powder metallurgy procedures, and are presented in Table 4.

Densities after sintering show a significant increase compared to the density after pressing. Densities after sintering the HyCo A to F samples show a decreasing trend with the increase in Gr content of 1 to 3 wt.% and constantly maintaining the Al_2O_3 content.

Table 4

Determination of the specific technological properties of sintered hybrid composites

Sample	***Weight,*** [g]	***Thickness*** [mm]	***Volume,*** [cm^3]	***Density of composites,*** [g/cm^3]	***Theoretical density,*** [g/cm^3]	***Compactity,*** [%]	***Porosity,*** [%]
HyCo A	7.980	2.8	3.5185	2.2680	2.875	78.89	21.11
HyCo B	7.932	2.9	3.6442	2.1766	2.884	75.47	24.53
HyCo C	7.983	3.1	3.8955	2.0493	2.824	72.57	27.43
HyCo D	7.955	3.2	4.0212	1.9783	2.836	69.76	30.24
HyCo E	7.979	3.3	4.1469	1.9241	2.776	69.31	30.69
HyCo F	7.947	3.3	4.1469	1.9164	2.788	68.73	31.27

The samples with the best density values after de sintering process are sample HyCo A and HyCo B.

Compactity of the sintered composites exhibits an decreasing trend of values with increasing Al_2O_3 content from 10 to 20 wt% and also with the increase of the graphite content from 1 to 3 wt.%. The maximum value is held by sample HyCo A and the minimum value by HyCo F. Porosity is complementary to compactity. The linear contraction values after sintering the green compact samples obtained from mechanically alloyed $Al/Al_2O_3/Gr$ powders depending on the reinforcement content are shown in Figs. 2 and 3.

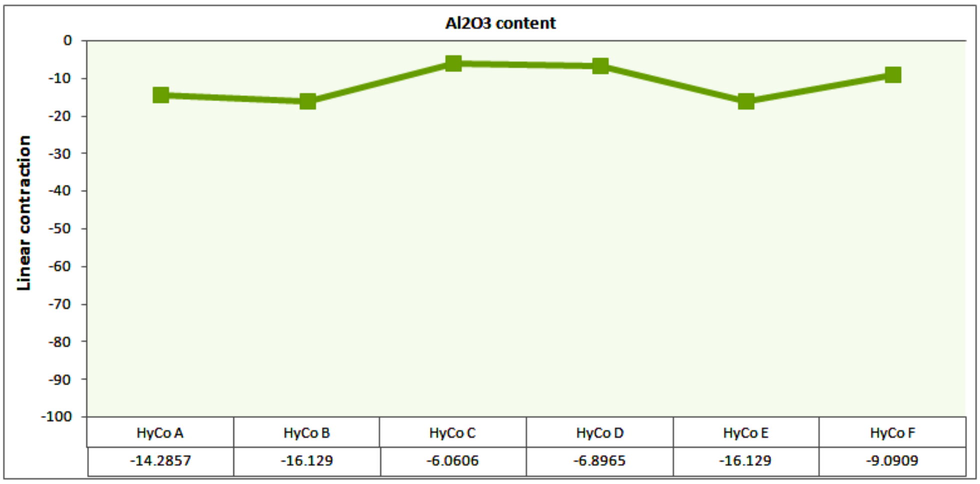

Fig. 2. Variation of the linear contraction of sintered samples depending on the alumina content

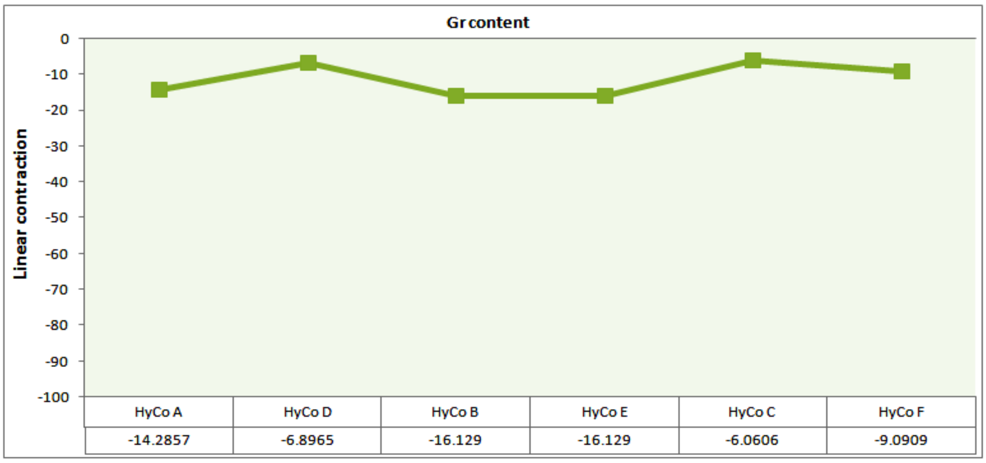

Fig. 3. Variation of the linear contraction of sintered samples depending on the graphite content

The values of the linear contraction after the sintering process have a tendency to decrease, as the graphite content increases, the samples HyCo B and HyCo E have the same values, and samples HyCo C and HyCo F show a growing trend. These microstructural analyzes were performed in order to observe the formation, appearance and diameter of the grains, as well as phases identification. It was also possible to determine precisely the distribution of the constituent elements of the examined samples. In this purpose we select the samples HyCo D, HyCo E and HyCo F with 10, 15 and 20 wt. % content of Al_2O_3 and 3 wt. % Gr. Microstructural analyzes are presented as follows: the SEI-BSED analysis of sample HyCo D in Fig. 4, the SEM-ETD analysis of samples in Fig. 5 and the comparison of diffraction pattern of samples in Fig. 6.

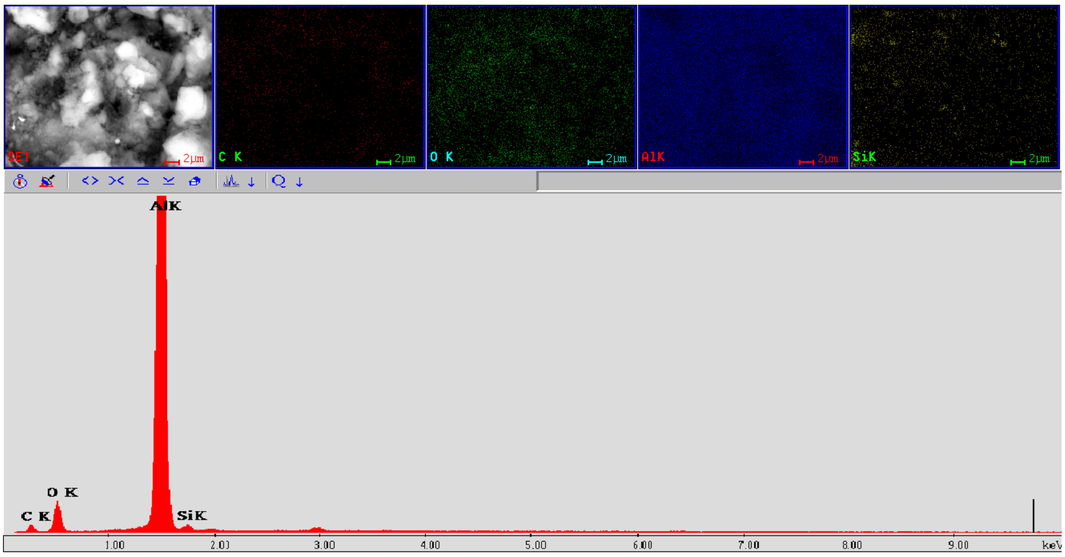

Fig. 4. SEI-BSED images and EDAX analysis of the HyCo D hybrid composite

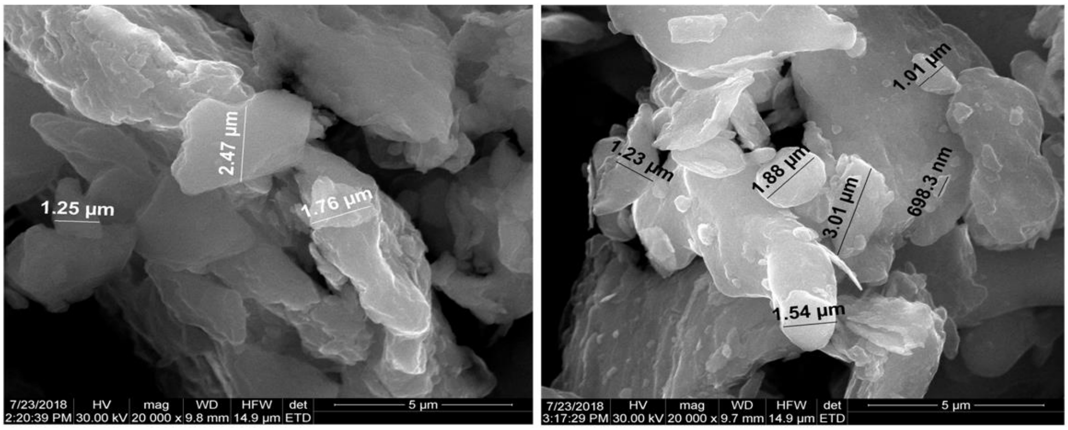

Fig. 5. SEM-ETD image of the HyCo D (a) and HyCo E (b) hybrid composites

SEM-ETD microscopy reveals nano-sized grains. The grain size was approximately between 698.3 nm - 3.01 µm. Aggregates of nanoparticles can also be observed.

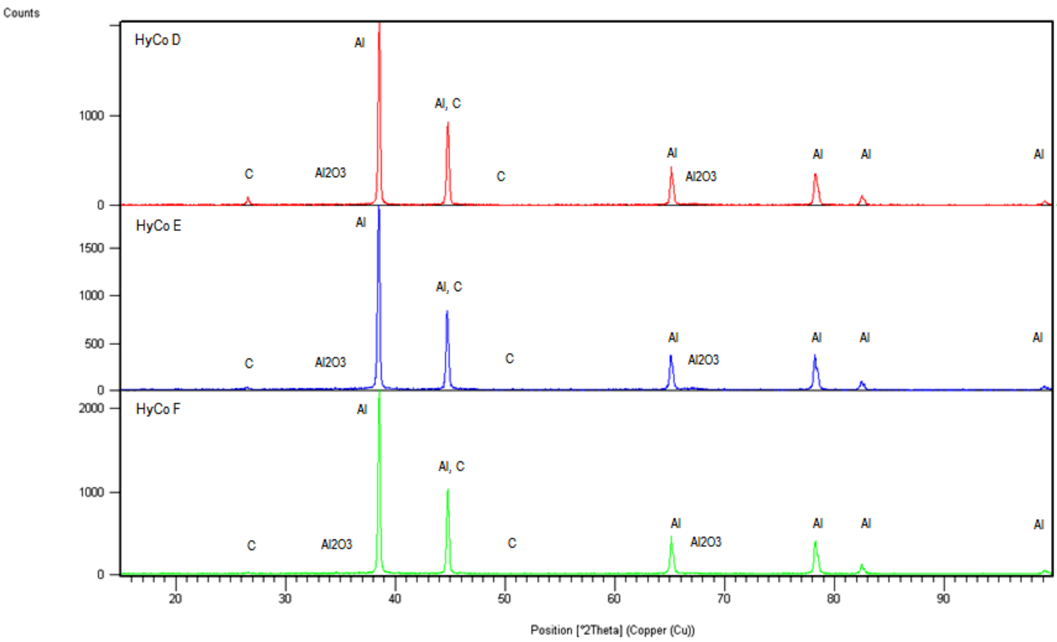

Fig. 6. XRD analysis of the HyCo D, E and F hybrid composites

Acknowledgments

This work has been supported by a grant of the University *Politehnica* of Bucharest, Program for research - Excellence Research Grants – GEX, project number 27/2017. We hereby acknowledge the research founds project UPB-GEX

2017 (internal no: SM 35-17-04, ctr. no: 70/2017) for providing the infrastructure used in this work and the project.

4. Conclusions

In this paper was investigated the influence of the reinforcements content on the technological parameters and microstructural aspects of the $Al/Al_2O_3/Gr$ hybrid composites produced through powder metallurgy methods. Various compositions of $Al/Al_2O_3/Gr$ composite powders were produced using mechanical alloying, which were pressed and subsequently sintered, thus completing a complete powder metallurgy cycle. The technological properties and microstructural analyses are demonstrating promising results.

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