Fabrication through P/M of ecological aluminum based composite materials. Part 2 - Densification and microstructural development during the sintering

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Abstract: In this second part of researches the compacted composites was presintering at 400°C/30 minutes and sintering at different temperatures between 520 and 620°C/ 1 hour maintaining, both thermal treatment made in protective atmosphere for achieved advanced matrix homogeneity and maximum densification. Sintered samples were investigated by parallel techniques, in terms of density/ porosity, dimensional changes during sintering and mechanical and microstructural point of view. The microstructure of composite has been studied by electron microscopy with electron Electron Probe Micro Analyzer and for identifiy the present phases after presintering –sintering treatment was analyzed using X-rays diffraction. The aim of investigations are obtaining the optimal combination of characteristics in concordance with requires of the European market: to obtaining a compacted, wear resistant and non-pollution for the environment material, for automotive destination.

Key-Words: Ecological AMC obtained by P/M, Dimensional changes during sintering, Densification, Electron Probe Micro Analyzer, X-ray diffraction

1 Introduction

The increased demand for new high-performance materials, ecological, for the automobile industry, in the last decades, led to the intensification of researches in this field [1-6]. The materials which accomplish in many parts these characteristics are the light weight composites discontinuous reinforced with ceramic particles, like aluminum based composites.

In order to achieve more compact material, the homogenized and pressed samples are dewaxed to remove the lubricant and sintered in order to strengthen the material (increasing the physical and mechanical properties of it). Taking into account that dewaxing - sintering is a very complex process, and for understand these physico-chemical processes is necessary to study the composites at different sintering temperatures and taking in account the experimental processing and results of similar Al-based composites reinforced with ceramic particles, especially with SiCp. In the scientific papers of other researcher is specified that in the case of aluminum or aluminum alloy reinforced with SiCp composite materials, dewaxing-degassing opperation took place at 400°C-420°C temperature for 30-60 minuteand for the unreinforced aluminum used like matrix took place at 300°C for 3 hours [7]. Sintering parameters (sintering temperatures, isothermal sintering time ant the sintering atmosphere) varies in very wide limits, owing to the diversity of materials used as matrix and the shape, size and distribution of silicon carbide. Sintering temperatures used in aluminium and its alloys is between 520 and 640°C, while the isonthermal maintaining time at sintering temparatures is between one and 16 hours [8-17]. The sintering atmosphere could be hydrogen, nitrogen, and dissociated ammonia with dew point of very low – 40°C or vacuum [8, 11, 13-17]. For example, in the the scientific research of D. Săbăduş [18], for processing of Al /SiCp from elemental powder mixtures by P/M, different sizes of initial powder under the same conditions of compaction pressure, the sintering temperatures chosen for research were between 530 and 610°C and the optimal sintering condition was established for sintering temperature between 580 and 590°C, sintering time at 4 hours for a mixture of different grain size fractions. W.F. Calley and collaborators [10] studied the obtain of Al-based composites reinforced with 14%wh. SiCp by cold izostatic pressure at 187 MPa. They use the folows processing temperatures: 400°Cfor dewaxing of prealloyed powders Al-0.5Mg-4Cu(AA2014) and for sintering temperatures ranged from 605-620°C, maintenance at sintering temperature for 1, 2, 4 hours. They demonstrate that the optimum temperature of sintering is 620°C, at a sintering time of 4 hours.

J. Zhou and J. Duszczyk [17] obtained by powder metallurgy (151MPa, uniaxial cold pressing pressure , delubricating temperature of 420°C for 25 min, degassing between 420 and 590°C and sintering temperature at 590°C for 30, 60 respectively 90 minutes) a composite AA2014/10% vol SiC. They decidied that the best properties (strength and elongation) is obtained at sintering temperature of 590°C, holding at sintering temperatures for 60 minutes and the heat-treated at T6 condition. For better consolidation these materials were subjected of extrusion[17].

2 Problem Formulation

Sintering of aluminum powders has presented special problems to researchers first of all because aluminum oxides is not reduced by common furnace atmospheres at the temperatures of sintering and second, because internal lubricants often generate decomposition that affect solid sinter bonds. For reduced or eliminate these problems is necessary to alloying aluminum with elements which have a high solubility in aluminum, to generate low-melting intermetallic phases, such as copper, magnesium, silicon zinc or combination of these elements and find the optimal manufacturing parameter who give us the best combination rapport between physico-mechanical and technological properties. By adding copper to aluminum the very stable Al_2O_3 layers is broken and penetrate the oxide layers and facilitate a good sinterization. The evolution of the sintered composites microstructure during sintering is studied by a combination phisical-mechanical and microstructural analysis.

2.1. Experimental Procedure

Al-Cu matrix composite materials with different proportions of SiC hard particles and un-reinforced Al-Cu alloys obtained by powder metallurgy (P/M), in the same homogenization – pressing -presintering conditions and at different sintering temperstures were analyzed in terms of microstructural and physical-mechanical characteristics. In order to minimize the processing costs, the dewaxing, degassing and sintering were combined into a single operation. The compacts were dewaxed for 30 min, degassing during heating up from 420°C to 520°C with duration of 30 min and sintered at 520-620 °C for 60 min in presintered-sintered furnace (Siemens-Plania type) in a protective atmosphere (hydrogen) and than furnace cooled. The existence of good bonding between metallic matrix and ceramic particles at interface and the morphology and distribution of pores and carbides in the matrix we can appreciate. The green density of the compacts was determined by physical measurements. The density of the sintered coomposites was measured using the Archimedes method (ASTM B 328-92). The sintered Al-Cu/SiCp composites were analyzed from physical, mechanical and microstructural point of view. The physical characterization consists in determine the density and porosity of composites and unreinforced matrix alloy and the mechanical characterization was Brinell hardness measurements. The Brinell hardness test was done on a device FRANKOSCOP universal type by pressing on the surface material with a ball of 2.5 mm and load of 62.5 Kgf for 30 seconds. For investigate the dimensional changes and the physical characteristics exhibited by Al-Cu/ SiCp compacts we made dimensional measurements - the Volume of compacted samples (Vc) and sintered samples (Vs) - before and after sinterization of samples at all 6 temperatures ranges between 520-620°C.
The sintered density of specimens was measured using the Archimedes techniques. The microstructural analysis was made with Electron Probe Micro Analyser JXA-5A JEOL type. With this apparatus we could investigate the morphology and distribution of particles, the omogenity of material, etc. The X-ray diffraction was made in order to put in evidence the presence of phases α solid solution, θ (CuAl2) and SiC hard particles.

3 Problem Solution

3.1. Dimensional and porosity changes during sintering of composite samples

Dimensional changes, in volume of sintered composites and his un-reinforced matrix alloys at different sintering temperature for Al-4Cu aluminum alloys and Al-4Cu/SiCp composites (5-20wt.%SiC as reinforcement in matrix) are presented in Figure 1. To observe the evolution of porosity depending on addition of ceramic particle tough material, and sintering temperature was drawn graph in Figure 2 presented below.
Brinell hardness values obtained at different sintering temperatures on aluminum alloy sintered composite materials that are presented in Table 1.

Table 1

Sinte-ring T, oC	Brinell Hardnesss				
	Al-4Cu 0%SiC	Al-4Cu 5%SiC	Al-4Cu 10%SiC	Al-4Cu 15%SiC	Al-4Cu 20%SiC
520	30	28	28,5	30	27.5
540	36	29	27,5	28	29
560	40	45	38	36	35
580	33	29,5	26	28	28,5
600	50,5	52	51	55	52
620	58	57	59	62	59

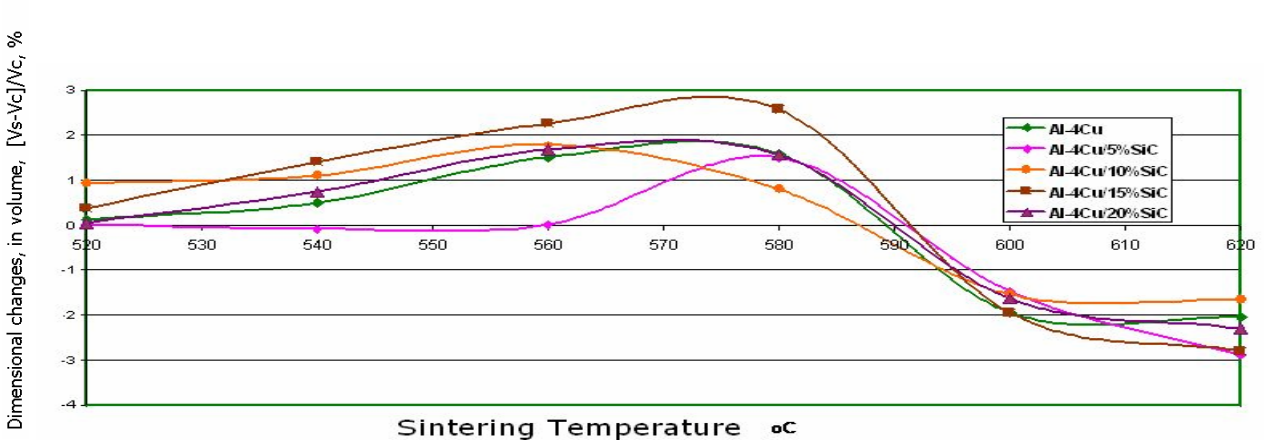


Fig. 1 Dimensional changes, in volume of sintered composites and alloys

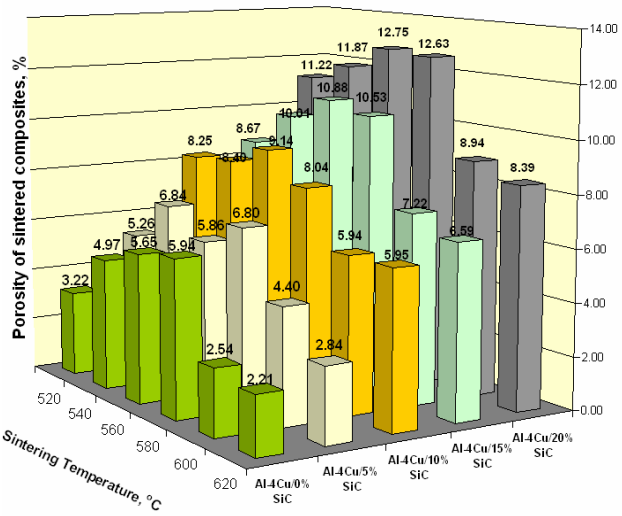


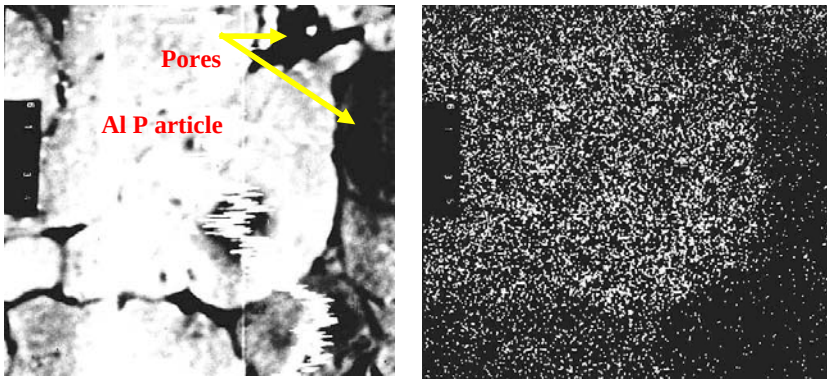
Fig. 2 Porosity of sintered samples depending on sintering temperature and proportion of SiC

After analyzing of the porosity evolution respectively dimensional variation of sintered materials we observed:
The appearance of progressive dilatation of all materials at temperatures between 520 and 580oC, and at temperatures above 580oC, the differentiate contraction of materials took place, according to the SiC particles proportion in the matrix.
Analyzing the porosity in function of the sintering temperatures and the proportion of SiCp was found that for sintered alloy matrix Al-4Cu (0% SiCp) and for the composites Al-4Cu / 5% SiCp at a 580oC sintering temperature, the maximum porosity were 5.94% respectively 6.80% and for composites with the same matrix (Al-4Cu) and 10, 15 respectively 20% SiCp at 560oC sintering temperature, the porosity were recorded between 9.13% and 12.63%.

It was found that the best densification was obtained at temperatures of 600 and 620oC in the presence of a sufficient quantity of eutectic liquid phase when the lowest values of porosity was 4.39% and 3.53% for composites with 5% SiCp. The values of porosity increase with the proportion of SiCp at 8.94%, at 600 oC respectively 8.38% at 620oC for composites with 20% SiCp;
High porosity values for composites with high content of SiCp in the first stages of sinterization it is explained, on the one hand, by existing of a large initial porosity of compacts due to a lower pressure in the packaging of mixtures containing large SiC particles (Fig. 2and 4) and on the other hand by reducing of self-diffusion between solid particles of Al-Al and reducing of diffusion between Al-Cu by hard particles of SiC. The minimum values obtained at 600 oC respectively at 620oC temperature are explained by the presence of the liquid phase in sufficient quantity (6% at 600oC and 20% at 620oC) to allow a maximum densification of the materials.

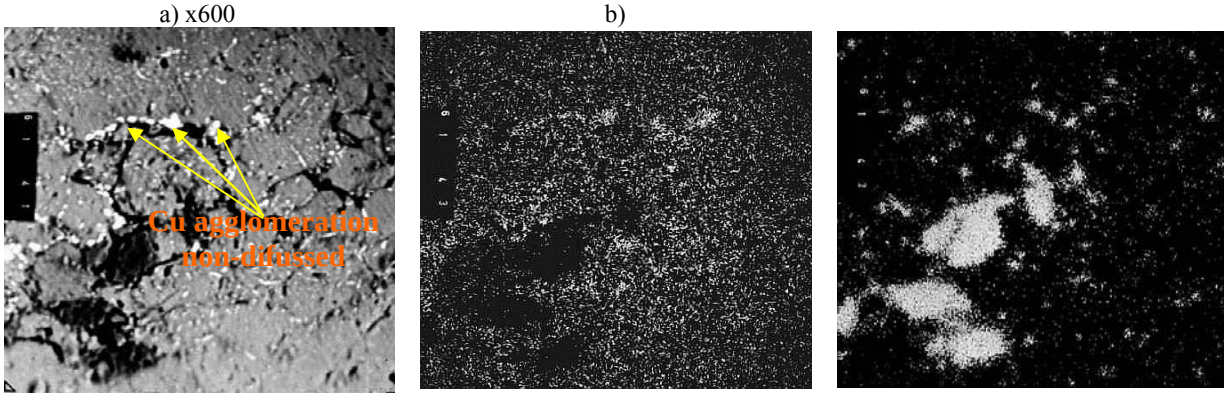
3.2. Microstructural anaysis of sintered composite samples with the Electron Probe Microanalyzer and X-ray investigations

The microstructural analysis with the Electron Probe Analyzer of pressed and sintered materials that confirm that the presence of SiC particles prevents proper densification of composites (Fig. 3). It appears that the SiC particles are located around pores, the size and quantity of these are proportional to the amount of SiC and sintering temperature. In Figure 4 is given the diffractometry of composite material with a high content of SiC (20%) sintered at 620oC.We observed in Figure 5 the presence of α-Al phase, θ (Al2Cu) and β-SiC carbide, which confirms that copper was completely dissolved in the material.



a) x600 b)

Fig. 3 Al-4Cu alloy sintered at 620°C/60'/ H_2 ; a) Compo image (X600) and concentration of copper lines. The concentration of profile lines indicates different composition gradients which confirms the presence of copper compounds b) The X-ray distribution map of Cu.



a) b) c)

Fig 4. Al-4Cu/20 % SiC sintered samplesata 580 °C/60'/.H_2 a) Compo image; b) The X-ray distribution map of Cu; c) The X-ray distribution map of Si; X150 Maginification

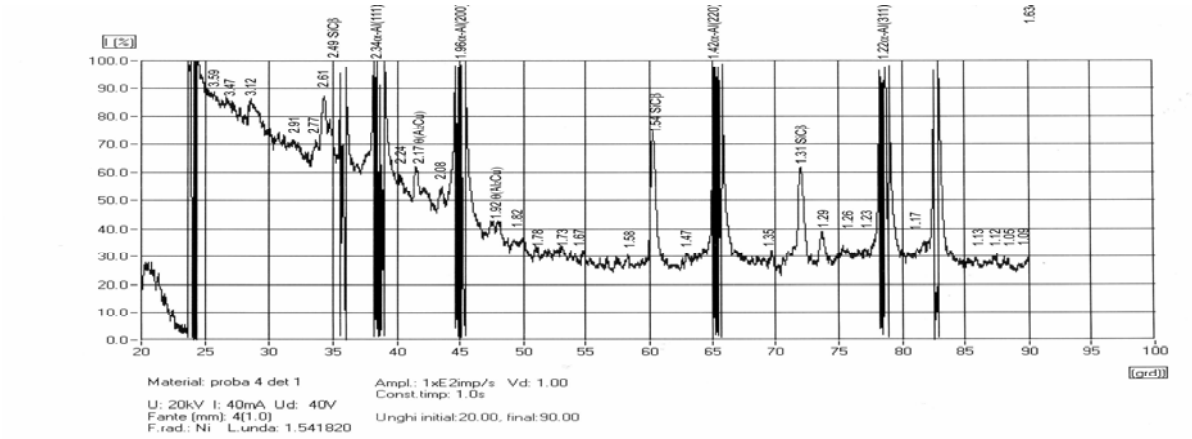


Fig. 5. X ray diffraction of composite with 20%SiC sintered at 620oC/60 min/ hydrogen atm.

Conclusion

It was found that the best densification was obtained at temperatures of 600 and 620°C in the presence of a sufficient quantity of eutectic liquid phase when the lowest values of porosity was 4.39% and 3.53% for composites with 5% SiCp, (Fig 1 and 2) in correlation with Brinell hardness . The microstructural analysis with the ElectronProbe Analyzer of sintered materials confirms that the presence of SiC particles prevents a proper densification of composites (Fig. 3). The diffractometry of composite material with 20% SiC sintered at 620oC shows the presence of α-Al phase, θ (Al2Cu) and β-SiC carbide, which confirms that copper was completely dissolved in the material.

After investigated sintered samples by parallel techniques, in terms of density/ porosity, dimensionalchanges, mechanical and structural point of view we concluded that the optimum sintering temperature is 620^{o}C and optimal proportion of SiC in ensuring an optimum compactness / hardness characteristics is 10 and 15% by weight SiC.

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