PROCESSING OF ZA-27 BASE COMPOSITES REINFORCED WITH CARBONACEOUS AND β-SiC PARTICLES

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SUMARY: The compocasting of two base ZA particulate composites is described in this paper. The used metallic alloy during the experimentations is known commercially as ZA-27. This alloy was reinforced in one case with calcined fine coke particles with low sulfur content which comes from processes of drying and sifting of hydrocarbon remainders, and in a second case, with fines particles of β-SiC obtained by pyrolysis of rice husk (RH).

For pyrolysis and compocasting process a special rheocaster and gas furnace equipment were designed. The variety of parameters related to the process and others related to the used raw materials, made us plan the work statistically with the purpose of reducing the magnitude of experimentation and arriving at the optimization of the pyrolysis of RH and compocasting process. The SEM and micro analysis for the β-SiC and mechanical tests and microscopic observation are reported for both composites.

KEYWORDS: Base ZA particulate composites, carbonaceous particles, SiC, compocasting.

INTRODUCTION

Even after almost half century of research, the particulate metal matrix composites (PMMCs) have begun to have a wide use in the marketing. In some applications, these materials can outperform continuous fiber reinforced composites, basically because of their isotropic properties, low cost and feasibility process [1].

Due to its high resistance, easily machining, low fusion temperature and good tribiological behavior [2], Zinc alloys are feasible matrix materials. Within the ZA alloy group (ZA-8, ZA-12 and ZA-27), the ZA-27 alloy (ASTM B669-82: 25-28%Al, 2.0-2.5%Cu, 0.01-0.02%Mg and Zn as balance) has the highest mechanical and wear resistance [3].

Some of the most popular materials that have been used as reinforcement for the ZA-27 alloy includes Al_2O_3 [4], glass [5], graphite [6], and SiC [7]. In all cases and especially graphite and SiC cases, the world research has dealt with commercial particles. In this work the ZA-27 alloy was reinforced with calcined fine coke particles with low sulfur content which comes from processes of drying and sifting of hydrocarbon remainders, and in a second case, with fines particles of β-SiC obtained by pyrolysis of rice husk (RH).

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The processing of two base ZA particulate composites was made by Compocasting. This technique entails the incorporation of reinforce particles into a semi-solid metal alloy, so the mechanical stirring action improve the contact and the reinforcement wetting by the metal. Additionally, the semi-solid state of the matrix conveys a more laminar flow, preventing the migration of reinforce particles due to density differences, as long as a homogeneous distribution of the particulates in the matrix [8].

PRODUCTION OF REINFORCEMENTS

The common equipments for thermal decomposition of inorganic and organic materials are electrical furnaces. The energy consumption and cost associated with these equipments are high, because it is necessary to get elevated temperatures. In this sense, the use of gaseous fuels (propane) offer excellent possibilities as on less expensive alternative due to the high calorific power of the fuel and the better economy that can be reached. In order to predict the remainders temperature evolution, a mathematical model for a one-dimensional and dependent time system was developed. This model considers the propane combustion with air excess (Eqn. 1) and also, a global energy balance in a transient state (Eqn. 2), which accounts the energy transferred among combustion gases, the sample and the internal walls, and the energy stored in the gases. Fig. 1 is an image of the gas furnace and ashes separation equipment builds during the researches.

$$C_3H_8 + 5(1 + e)(O_2 + 3.76\, N_2) \rightarrow 3CO_2 + 4H_2O + 5(1 + e)3.76\, N_2 + 5eO_2 \qquad (1)$$

$$\begin{aligned}
&\frac{F_{GN}}{M_{C_3H_8}}\Delta\overline{H}^{\circ}_{f,C_3H_8} + 5\cdot(1+e)\frac{F_{GN}}{M_{C_3H_8}}Cp_{O_2}\left(T_e - T_{ref}\right) + 5\cdot 3.76\cdot(1+e)\frac{F_{GN}}{M_{C_3H_8}}Cp_{N_2}\left(T_e - T_{ref}\right)\\
&= 3\frac{F_{GN}}{M_{C_3H_8}}\left[\int_{T_{ref}}^{T_{LL}} Cp_{CO_2}\,dT + \Delta\overline{H}^{\circ}_{f,CO_2}\right] + 4\frac{F_{GN}}{M_{C_3H_8}}\left[\int_{T_{ref}}^{T_{LL}} Cp_{H_2O}\,dT + \Delta\overline{H}^{\circ}_{f,H_2O}\right]\\
&+ 5e\frac{F_{GN}}{M_{C_3H_8}}\int_{T_{ref}}^{T_{LL}} Cp_{O_2}\,dT + 5\cdot 3.76\cdot(1+e)\frac{F_{GN}}{M_{CH_4}}\int_{T_{ref}}^{T_{LL}} Cp_{N_2}\,dT + h_M A_M\left(T_{LL} - T_M\right)\\
&+ h_M A_M\left(T_{LL} - T_M\right) + h_W A_W\left(T_{LL} - T_W\right) + \rho V\overline{C}p_{LL}\frac{dT_{LL}}{dt}
\end{aligned} \qquad (2)$$

For production of coke particles, about 30g of carbonaceous remainders were subjected to isothermal heating at 500°C and 600°C in air atmosphere. The expulsion of volatile matter and surface mordentation from particles when they are heat-treated was confirmed by SEM (Fig. 2a). According to Cruz [9], this morphology is useful in order to get a superior surface energy and better wettability by the matrix alloy.

On the other hand, in order to optimize the pyrolysis of RH, the experimental design was reduced by means factorial analysis, commonly known as Taguchi design. According to this design, the best conditions processes were [10]: 1370°C as a temperature process, 1.5 lt/min of argon flow and a residence time of 40 minutes. The used RH had a mesh No. 8 and it was catalyzed with $FeCl_2.4H_2O$. SEM micrograph, Fig. 2b, shows that the short fibers are constituted by separated crests structures of 60µm approximately and perfectly aligned with waved and spongy contours. This morphology is very useful and the same one can allows a good mechanical interaction with the matrix.

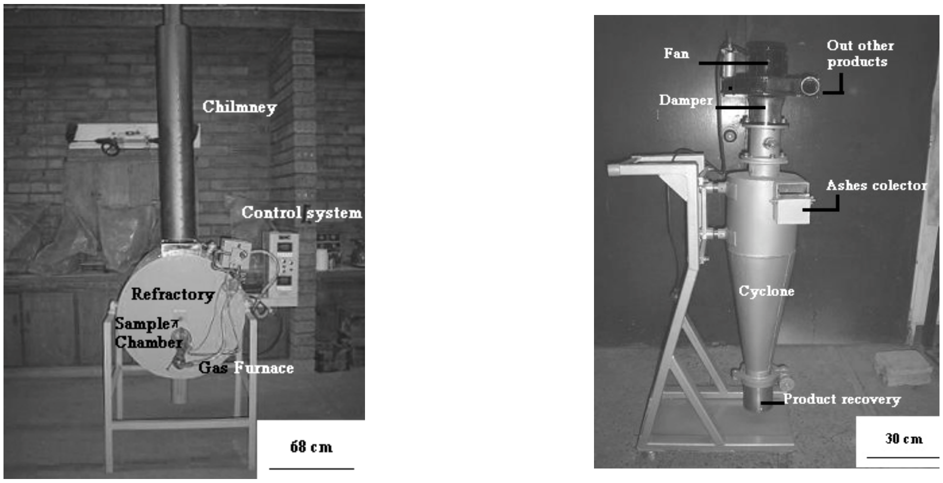

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Fig. 1: Equipments for reinforcements production, (a) special gas furnace, (b) separation equipment

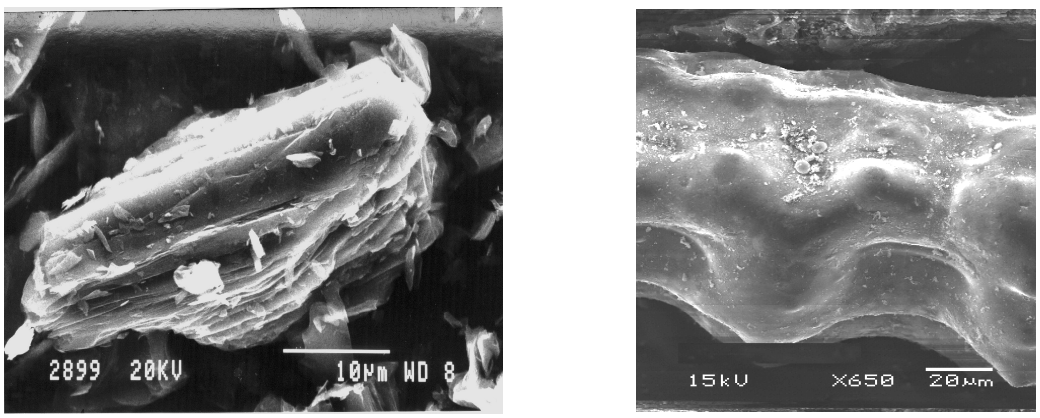

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Fig. 2: SEM micrographics, (a) Carbonaceous particles and (b) SiC particles

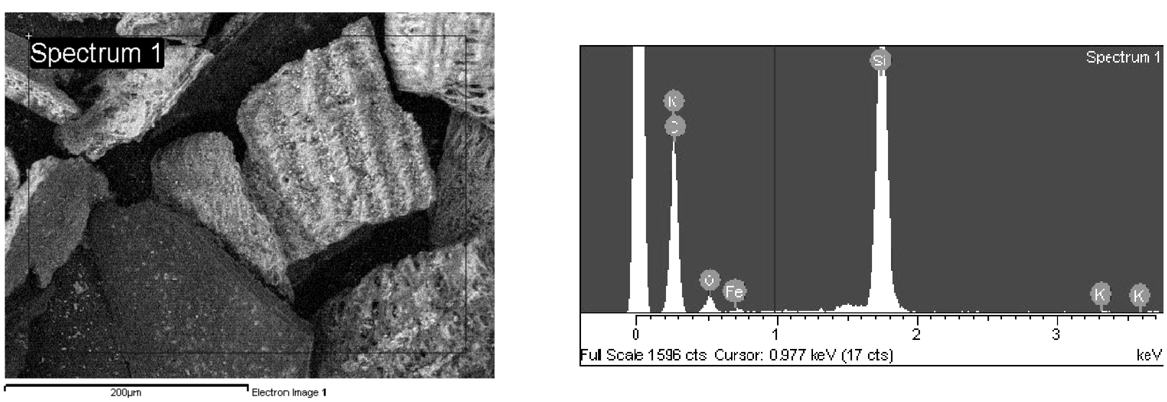

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Fig. 3: EDS for RH ashes

Fig. 3a and 3b shows the EDS qualitative analyses. The WDS results on the other hand, showed that the catalyzed ashes have mainly C and Si, the oxygen presence is also evident and the Fe content is product of the used catalyst. The general content in weight was: 58.45%-C, 9.23O%-O, 30.21%-Si, 2.10%-Fe and 0.01%-K.

SYNTHESIS OF COMPOSITES

The experimental equipment used during the research (Fig. 4), consisted in a crucible within the semi-solid processing and dispersion of reinforcements take place. Initially 1000g approx. of base alloy were placed in the graphite crucible, the material was then heated at a temperature higher than liquidus (>492°C). In this point, the stirrer was introduced into the metal. Subsequently, the furnace temperature was lowered at an average rate of 3°C/min until the desired semi-solid working temperature. The particles were added afterward and the stirring was maintained in order to have a good shearing action. Once the mixing was over, the composite material was foundry in a permanent mold. Finally, samples were cutting from ingot in order to carry out different characterization tests.

1. Elevation and stirring system
2. Particle feeder
3. Graphite crucible
4. Electric furnace
5. Permanent mold
6. Protection chamber
7. Inert gas (Nitrogen atmosphere)
8. Cooling water

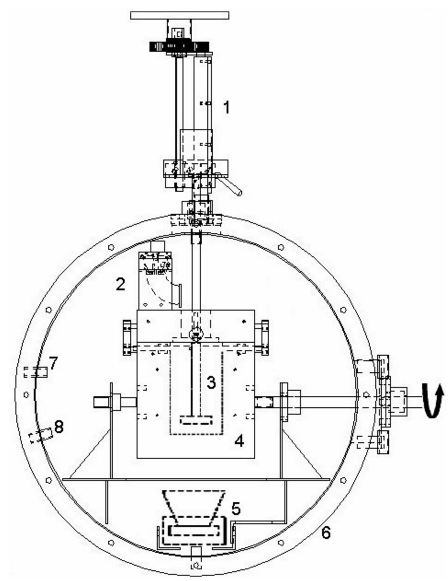

Fig. 4: Experimental equipment used for the fabrication of composites

The knowledge about the synthesis and process phenomenology for PMMCs, is not complete because each metal-ceramic system has a particular set of process variables (as solidification temperature, fraction and size of reinforcement, mixer geometry, cooling speed from the liquid, shear index and process time). In such a way, to optimize the value of each one of these variables for a particular a metal-ceramic system requires statistical and experimental tools for production of materials with high mechanical and microstructural quality. In this work, the Compocasting process was study statistically; again using a Taguchi design, the following factors were taken in consideration for both composites: Working temperature within the semisolid state (A), stirring speed (B), height of mixer respect to crucible's base (C), process time (D), volumetric fraction of reinforcement material (E) and size of reinforcement particles (F). The used process variables for each composite are listed in table 1.

Table 1: Conditions process for the composites

Factors		Composite		Unit
		ZA-27/C	ZA-27/β-SiC	
Process	A	470	455	°C
	B	450	400	rpm
	C	15	15	mm
	D	30	30	min
Reinforcements	E	5	12	%
	F	<60	37	μm

MECHANICAL STRENGTH AND MICROESTRUCTURAL ANALYSIS OF THE OPTIMIZED COMPOSITE MATERIALS

Mechanical behavior

Tension tests were performed at a constant deformation speed of 0.5 mm/min. The results for both composites are listed in table 2, and the values for the base alloy foundry in permanent mold are also listed.

For both composites the UTS increases with respect to the base alloy. Pillai et.al [11] reports similar increases in aluminum composites reinforced with graphite particles. In the case that was studied, this increase can be explained as a mechanical union at the interface, according to the particular surface morphology obtained for carbonaceous and β-SiC particles.

Table 2: Mechanical properties for the composites

Material	σ_y (Mpa)	UTS (Mpa)	ε_o (%)	BHN
Base alloy	66.5	307.5	3.3	106.3
Composites	ZA-27/C_p/5_p			
	380.0	392.7	8.80	93.1
	ZA-27/β-SiC_p/12_p			
	250	350	7.89	85.3

On the other hand, it has been reported that the ductility of PMMCs increases according to the percentage of reinforcement. This can be established by means of the unitary deformation (ε_o), which displayed an increase when it was compared with the same one obtained for the foundry base alloy. Nevertheless, ε_o is not very representative, since the foundry alloy without reinforcement has generally a high porosity degree, which degrades enough its properties, in special the ductility.

The hardness of the composite ZA-27/C_p is about 12.42% smaller than the base alloy. Pai *et.al* [12], as well as Murali *et.al* [13], reports similar hardness reductions in aluminum composites reinforced with carbonaceous and graphite particles.

In aluminum composites reinforced with particles of SiC, Lim [14] have found greater hardness and better wear behavior in comparison with the base alloy without reinforcement. Nevertheless, in spite of the greater volume content, the composite ZA-27/β-SiC_p does not confirm this result. In this case, the hardness can be improved with additional processes of cleaning and refinement of the reinforcement material before coming to incorporate it in the matrix.

Optical microscopy

The micro-structural evaluation of alloy ZA-27 by optical microscopy (Fig. 5a) basically showed a dendritic morphology, rich in aluminum (α-fcc) and surrounded by a very fine eutectoid structure (α+η). For this microstructure, the aluminum concentration decreases from the dendritic core until reaching a critical value in the inter-dendritic zone. This α phase is also a primary phase, that is easily transformed into a semi-globular microstructure (Fig. 5b) during the semi-solid process.

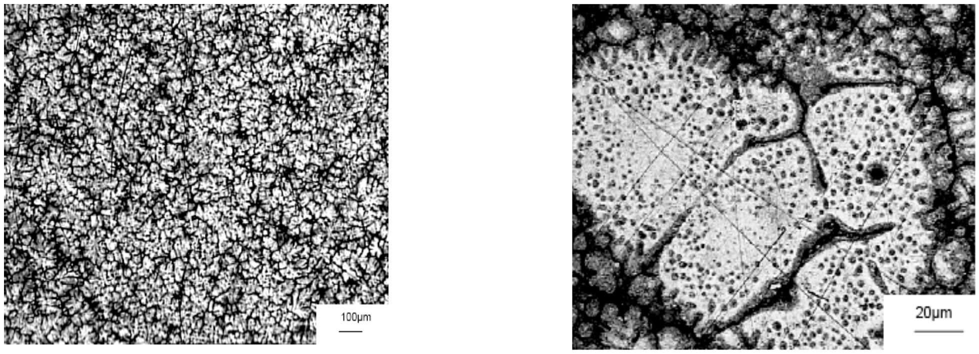

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Fig. 5: (a) Base foundry alloy ZA-2, (b) Seudoparticle. (Attack with CrO_2 (200g), Na_2SO_4 (15g) and distilled H_2O)

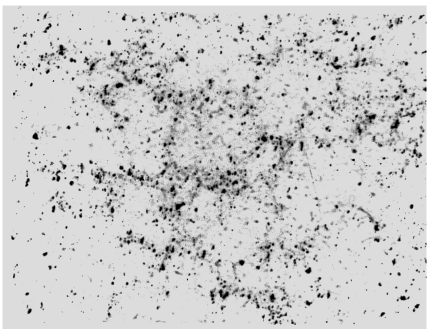

Fig. 6a: Optical micrograph of ZA-27/C_p/5_p composite processed under optimal conditions. (Without attack. 200X)

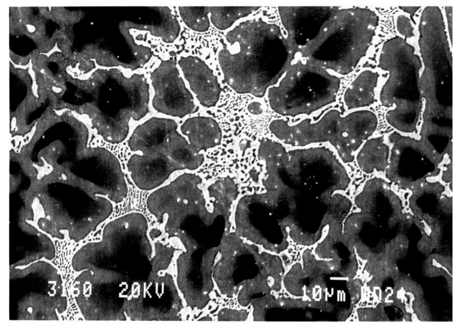

Fig. 6b: SEM micrograph of ZA-27/C_p/5_p composite. (Attack with Nital 2%)

The optical micrograph on Fig. 6a on a sample of ZA-27/C_p/5_p composite material shows that there is not any segregation, conglomerates or union among the reinforcement particles. It is also possible to appreciate that the particles form a network.

The corresponding micrograph SEM of Fig. 6a appears in Fig. 6b. On this figure, the continuous and connected grains are the primary α grains. In the center of the grain there is plenty of Al with darker appearance. In the periphery of these primary grains, the gray zones (Zinc and Cu), contain more white particles (carbonaceous) than the rich zone in Al. Comparing Fig. 6a and 6b it is possible to appreciate that the trajectory of the reinforced particles is the same as that one formed by the residual liquid in the matrix Therefore, it is possible to say that the great majority of carbonaceous particles are within the residual liquid and that a small part of this is caught by the primary solid phase of metallic alloy.

Fig. 7a-7d illustrates the ZA-27/β-SiC/12_p composite micrographs. After the composite is casting it was noted that the solidification of the remaining liquid is accelerated producing a fine dendritic micro-structure, where the primary α particles as a rosette form, are strongly bonded to the matrix (Fig. 7a).

In general, it was found a homogeneous distribution of the reinforced material in a matrix with no chemical attack (Fig. 7b). When the chemical attack is performed, it is subsequently observed that the particles followed a path marked by the eutectoid phase α+η (Fig. 7c). This distribution is not always efficient and it is the result of proper wetting and ideal mixture conditions. On the other hand, the formation of reactive zones at the interface, which evidence the potential chemical interactions, is not clearly seen in the optical microscopic tests for both composites (Fig. 7c).

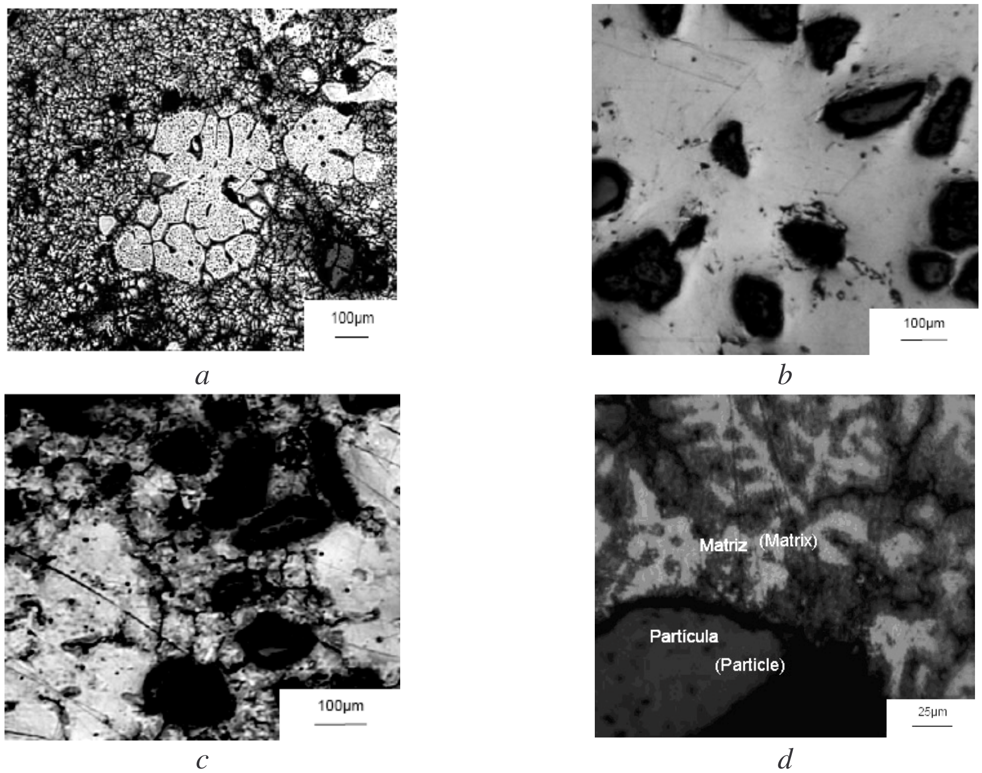

Fig. 7: Optical micrographics of ZA-27/β-SiC/12$_{p.}$ (Attack with CrO_2 (200g), Na_2SO_4 (15g) and distilled H_2O)

CONCLUSION

It has been processed two base ZA-27 composites. These materials showed a superior mechanical strength in comparison with the base alloy. Both composites were reinforced with materials obtained from remainders. In one case with carbonaceous particles of petrochemical origin and in a second case with carbides obtained from the rice husk. Both materials offer an application field where becomes useful the improved mechanical behavior and the environmental advantage that represents the use of remainder materials like reinforcement of metallic alloys.

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