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ASSESMENT OF AlTiB AND AlTiC GRAIN REFINERS PERFORMANCE USING THE ALCOA TEST

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Lucrarea prezintă cercetările experimentale efectuate pentru determinarea fenomenelor care au loc la finisarea granulației aliajelor AlMgSi şi AlSi cu prealiaje finisoare AlTi5B1 şi AlTi3C0,15.

Rezultatele experimentale confirmă rolul nucleanților TiB_2 *şi TiC (paradigma nucleantului) precum şi rolul elementului dizolvat Ti (paradigma elementului dizolvat). Pentru determinarea eficienței finisorilor de granulație aliajele au fost supuse testului ALCOA şi analizei microscopice şi macroscopice cantitative.*

The paper presents the investigations that has been conducted in order to evaluate the phenomena, which take place at grain refining of AlMgSi and AlSi alloys with Al-5%Ti-1%B and Al-3%Ti-0.15%C master alloys. The experimental results confirm both nucleant (the key role of TiB_2 *and TiC particles (nucleant paradigm) and the role of solute elements Ti (solute paradigms) during the grain refining process. ALCOA grain refining test and quantitative metallography were used in order to evaluate the effectiveness of the grain refiners used in experiments.*

Keywords: AlTiB, AlTiC, grain refinement, nucleant, dissolved element.

1. Introduction

For the grain refining of aluminum alloys, with a wide range of utilization in the industry, several types of master alloys are used depending on the type of alloy, the field where the parts are used, the alloy's resistance to the harmful action of some elements, the filtering and degassing methods [1, 2].

The specialty literature has developed two main trends with regard to the manner in which the aluminum alloys can be processed for grain refining: by increasing the number of crystal nuclei (nucleant paradigm) and by adding several elements that can create a high grain growth restriction factor (solute paradigm)[3].

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The paper presents the comparative experimental results obtained on grain refining the AlMgSi wrought alloy and Al12Si cast alloy using a two-stage master alloy addition. The first stage consists in the utilization of the binary Al-Ti master alloys for the Ti input (solute element), and the second stage consists in inoculating the melt with the ternary master alloys (Al-5Ti-1B or Al-3Ti-0.15C) in the melt in order to increase the number of nuclei [4, 5].

2. Experimental

The grain refining degree determination was performed by means of the ALCOA test, using a unidirectional solidification unit consisting of a mould and a copper device for water cooling (primary cooling).

In the upper part of the melt inside the mold has been introduced a copper device cooled with water, kept inside until the total solidification of the samples. The cooling water flow varies in function of desired solidification rate.

The moulds were made of various materials (copper, steel, firebrick, alumina and graphite) in order to establish the influence of the secondary-cooling rate on grain refining.

Alloy melting was performed in an electrical furnace with graphite melting crucible, of 3 kg capacity and 3.3 kW power, in argon atmosphere. The temperature was automatically adjusted, with $\pm 5^{o}C$ precision.

There have been chosen the refining binary and ternary master alloys, which contain $TiAl_3$ particles with structure that would ensure high dissolving velocity of titanium in the liquid alloys (Fig. 1). Binary master alloys Al5Ti and Al10Ti were obtained in the laboratory while the ternary ones were purchased from AB Ferrolegeringar Stockholm (AlTiC) and Asturiana de Aleaciones S.A. (AlTiB).

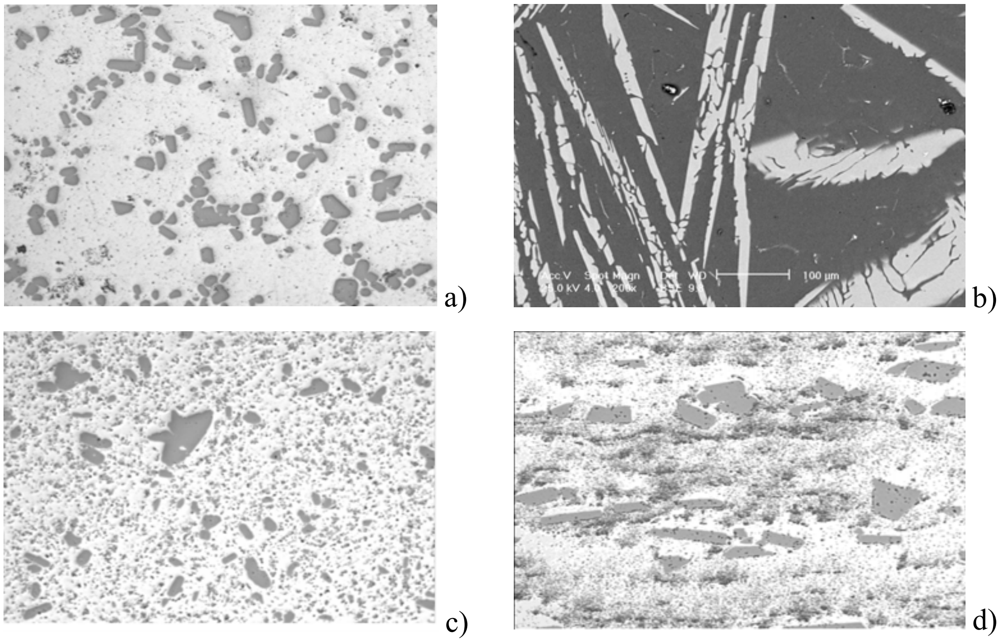


Fig. 1. Optical micrographs of the master alloys used on grain refining:
a) Al5Ti x100; b) Al10Ti x100; c) Al5Ti1B x100; d) Al3Ti0.15C x100

The master alloys were introduced in the melt previously overheated with 70^{o}C, and then they were hand mixed by using a graphite rod for a homogenous solubilization and distribution in the melt.

3. Experimental results

There are few studies in the specialty literature regarding the grain refining mechanism of the cast aluminum alloys, Al-Si type. This is because the alloys comprise a series of elements with grain growth restriction factors (GRF) of high values, and the silicon (over 3%) is an element that “poisons” the grain refiners.

The alloys used to evaluate the behavior – both of the dissolved elements (Ti) as well as of the carbides and borides – when grain refiners act, have the chemical composition presented in Table 1. For the non-refined alloys, the titanium comes from the load materials. For the refined materials, with 0.02% Ti and 0.03% Ti, the loss on oxidation both with respect to titanium and the other elements in the charge composition was considered. The optimum properties of the cast product are obtained when the ssα-Al grains are small and uniform. To achieve this desire, it is necessary to provide the conditions for the forming of as many crystals as possible at the deformable alloy and of dendrites in the initial solidification stage of the cast alloy, which should increase at low speed. To achieve this, the nucleation must be performed as close to the lichidus temperature as possible, i.e. the undercooling should be minimum.

Table 1

Nominal chemical composition of the alloys 6063 (AlMgSi) and Al12Si

Alloys		Ti	Fe	Si	Mg	Mn	Others	Al
6063	Unrefined	0.001	0.20	0.43	0.50	0.015	< 0.001	Bal.
	Refined with Al5Ti	0.020	0.19	0.45	0.45	0.027	< 0.001	Bal.
	Refined with Al5Ti + Al5Ti1B	0.031	0.17	0.42	0.44	0.034	< 0.001	Bal.
Al12Si	Unrefined	0.001	0.34	11.86	0.005	0.06	< 0.001	Bal.
	Refined with Al10Ti	0.021	0.37	12.01	0.007	0.06	< 0.001	Bal.
	Refined with Al10Ti + Al3Ti0.15C	0.032	0.35	11.97	0.004	0.07	< 0.001	Bal.

The values of the coefficients that characterize the solid-liquid interface are presented in Table 2.

Table 2

The segregation potential (GRF - Q) of some elements in aluminum

Element	k_i	m_i	$(k_i-1)m$
Ti	~9	30.7	245.6
Fe	0.02	-3.0	2.9
Si	0.11	-6.6	5.9
Mg	0.51	-6.2	3.0
Mn	0.94	-1.6	0.1

Calculus for growth restriction effect was estimated by index (Q - GRF):

$$Q = \sum m c_0 (k-1) = Q_{[Ti]} + Q_{[Fe]} + Q_{[Si]} + Q_{[Mg]} + Q_{[Mn]} \qquad (1)$$

where: m is the gradient of liquidus slope;

c_0 – the concentration of the solute element;

k – the repartition coefficient.

The results of the growth restriction factor Q calculation given by the main alloying elements are presented in Table 3.

Table 3

The calculated values of the growth restriction factor (GRF-Q) for 6063 and Al12Si alloys

Alloy 6063	Q_{6063}		Alloy Al12Si	Q_{Al12Si}
Unrefined	4.8626		Unrefined	71.2266
Grain refined with Al5Ti	9.468		Grain refined with Al10Ti	77.1166
Grain refined with Al5Ti + Al5Ti1B	11.905		Grain refined with Al10Ti + Al3Ti0.15C	79.5162

Fig. 2 presents the influence of the alloying and micro-alloying elements in the AlMgSi and AlSi alloys on the growth restriction factor. For both alloys we can notice the very significant share that the titanium has on the calculated value of the GRF-Q factor. The very high difference between the GRF values for the Al12Si alloy and 6063 alloy is given by the silicon content [5, 6].

By adding refining master alloys in the Al-Si and Al-Mg-Si alloys, uniform grains are obtained which lead to the increase of the values of the mechanical properties (R_m, A and endurance strength). Moreover, the grain refiners improve the supply of liquid alloy in the moulds, especially in the thinner sections of the parts.

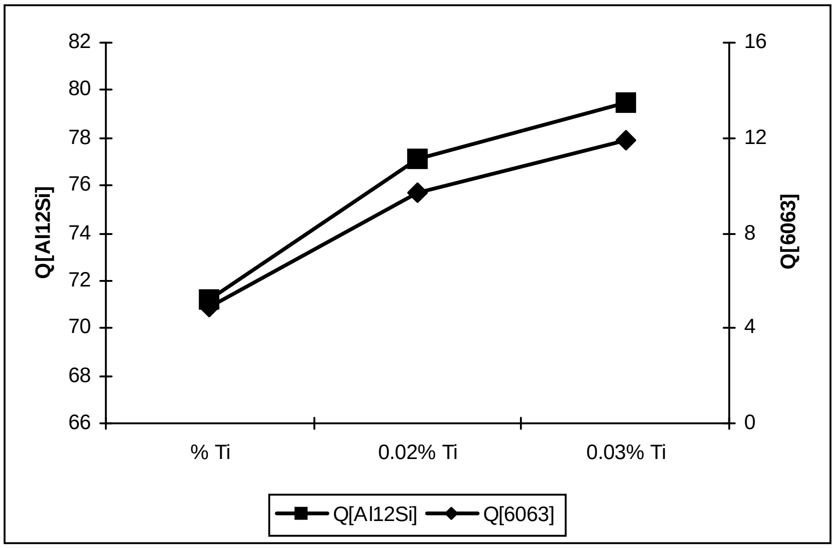


Fig. 2. The influence of the dissolved element Ti on the restrictive factor of the increasing GRF-Q in the alloys under study

Through the ALCOA test, we have emphasized both the influence of the dissolved element Ti and TiB_2 and TiC over the studied alloy grains. In this way, we can notice from the macrostructures presented in the Figs. 3 and 4, the fact that both paradigms of the refining grains are confirmed.

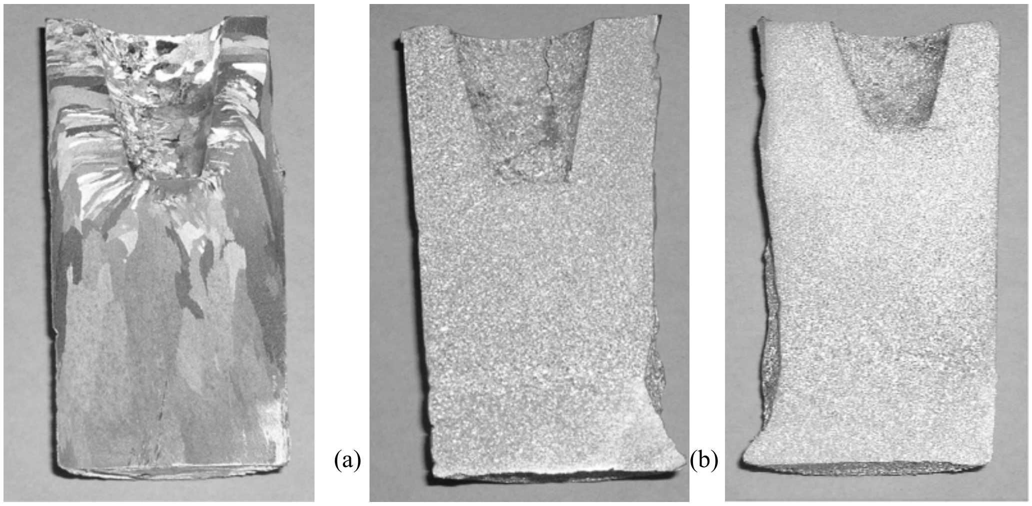

(a) (b) (c)

Fig. 3. The macrostructure of the alloy 6063 solidified through ALCOA test:
(a) unrefined sample; (b) refined sample (0.02% Ti); (c) refined sample (0.03% Ti, 0.006% B)

The non-refined alloys have columnar grains developed from the unidirectional cooling cone and from the mould walls, while at the samples refined using Al-Ti and those using Al5Ti1B or Al3Ti0.15C, the sizes decrease, smaller sized equiaxed grains being formed.

The grain refining effect on the parts cast with the Al-Si alloys can be studied by observing the structure of the ssα (Al) dendrites and by calculating the average indices of the dendrite fineness (Fig. 5).

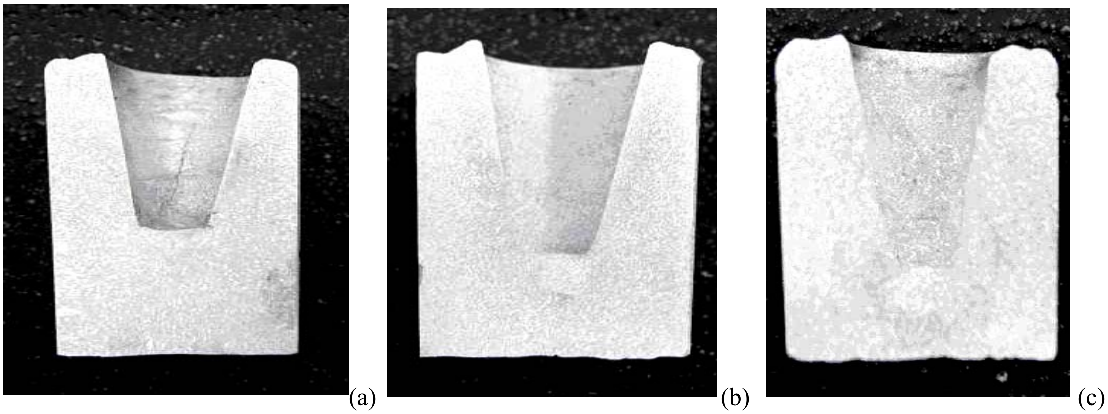
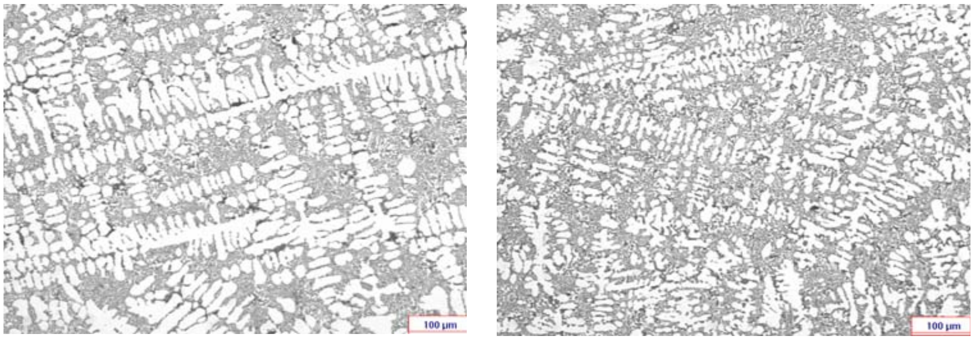
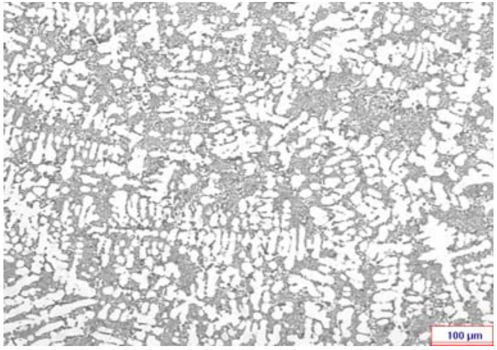


Fig. 4. The macrostructure of the alloy Al12Si solidified through ALCOA test:
(a) unrefined sample; (b) grain refined sample (0.02% Ti);
(c) grain refined sample (0.03% Ti, 0.002% C)

The dendrites, characterized through the average index of the grain refined dendrites, become smaller, more compact while the refined master alloy are introduced in the alloys. Unlike the unmodified Al12Si alloy, the average index of the dendrites decrease when the Al10Ti refined master alloy is introduced, so that this tendency could increase when the refined Al3Ti0.15C master alloy is introduced in the cast alloy (Fig. 6).



(a) IM = 18.05 µm (c) IM = 10.39µm



(b) IM = 12.67µm

Fig. 5. Optical micrographs indicating the average indices of dendrite fineness for the Al12Si alloy solidified in copper mould:
(a) Al12Si unrefined; (b) grain refined with Al10Ti (0.02% Ti) master alloy;
(c) grain refined with Al10Ti and Al3Ti0.15C (0.03% Ti and 0.002% C) master alloy

For the 6063 alloy, the grain size variation was highlighted through quantitative microscopic analysis (Fig. 7). Thus, a decrease of the grain average size from 269 µm for the unrefined sample to 143 µm for the refined sample, with 0.02% Ti and to 118 µm for the refined sample, with 0.03% Ti and 0.006% B was noticed.

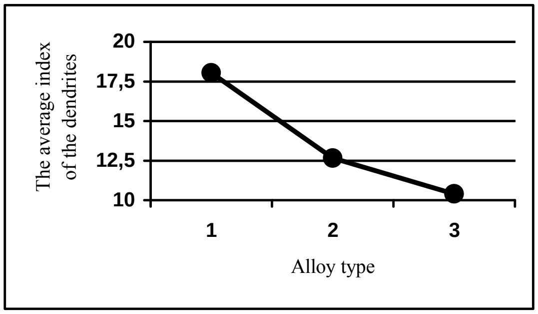
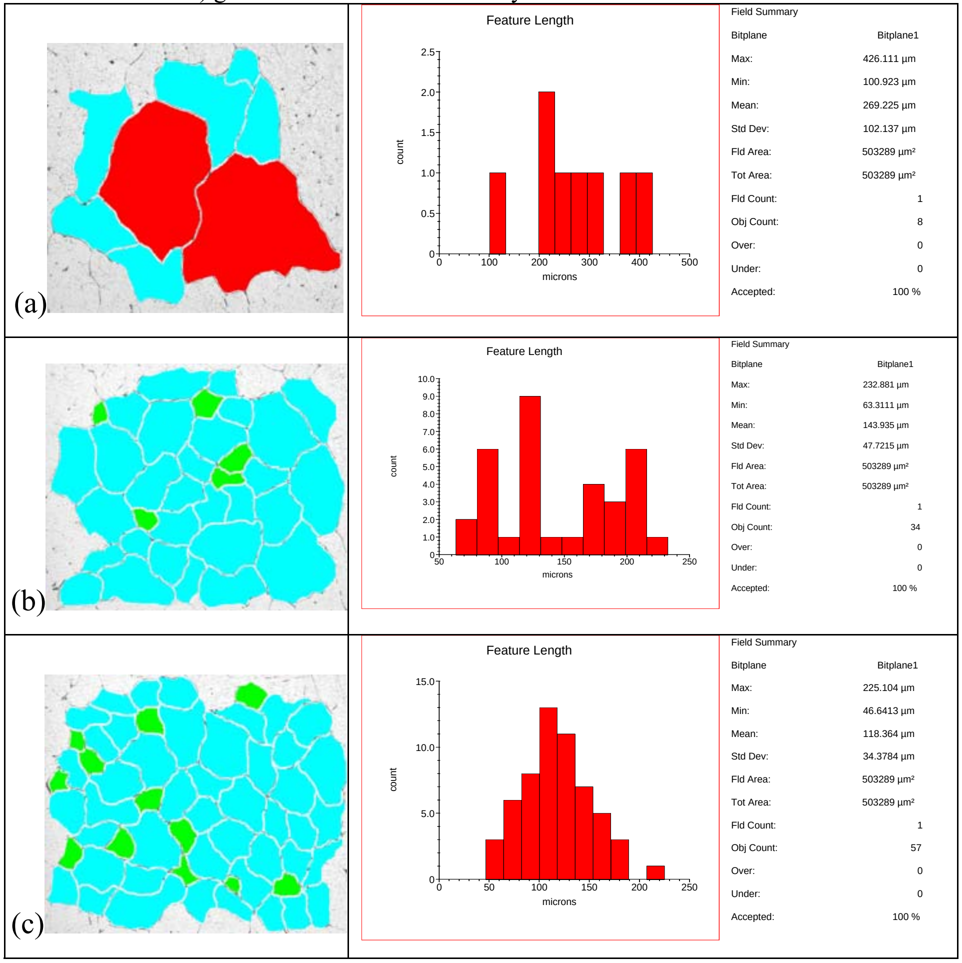


Fig. 6. The evolution of the average index of the refined dendrites (μm) for Al12Si alloy solidified in copper crucible: 1) unrefined; 2) grain refined with master alloy Al10Ti; 3) grain refined with master alloys Al10Ti and Al3Ti0.15C



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Field Summary
Bitplane        Bitplane1
Max:            426.111 µm
Min:            100.923 µm
Mean:           269.225 µm
Std Dev:        102.137 µm
Fld Area:       503289 µm²
Tot Area:       503289 µm²
Fld Count:      1
Obj Count:      8
Over:           0
Under:          0
Accepted:       100 %
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Field Summary
Bitplane        Bitplane1
Max:            232.881 µm
Min:            63.3111 µm
Mean:           143.935 µm
Std Dev:        47.7215 µm
Fld Area:       503289 µm²
Tot Area:       503289 µm²
Fld Count:      1
Obj Count:      34
Over:           0
Under:          0
Accepted:       100 %
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Field Summary
Bitplane        Bitplane1
Max:            225.104 µm
Min:            46.6413 µm
Mean:           118.364 µm
Std Dev:        34.3784 µm
Fld Area:       503289 µm²
Tot Area:       503289 µm²
Fld Count:      1
Obj Count:      57
Over:           0
Under:          0
Accepted:       100 %
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Fig. 7. Microscopic quantitative analysis of the 6063 alloy:
(a) unrefined; (b) grain refined with master alloy Al5Ti (0.02% Ti);
(c) grain refined with master alloy Al5Ti and Al5Ti1B (0.03% Ti + 0.006% B)

4. Conclusions

1. The mechanism for the nucleation and grain size modification processes occurring when refining the grain of the aluminum alloys from the Al-Si and Al-Mg-Si systems, using both elements restricting the grain growth (Ti) as well as carbides and borides for the increase of the number of nucleation centers, has been ascertained by our experiments;
2. We noticed a decrease of over 40% of the grain sizes determined on the 6063 alloy samples refined with binary master alloy Al5Ti;
3. In the case of the alloy Al12Si, we have noticed a decrease of the dendrite fineness average indices by 30% at a 0.02%Ti content;
4. After the second refining stage, using ternary master alloys with boride and carbide content, the values of the grain sizes in the 6063 alloy and of the dendrite fineness average indices in the Al12Si have decreased by approximately 20%;
5. The choice of the grain refiners, particularly in the case of the alloys with a content of over 3% Si, is quite unclear at the moment; it is therefore necessary to conduct experiments, especially for the industrial segment.

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