THE INFLUENCE OF COOLING RATE ON GRAIN REFINING OF 6063 ALLOY WITH ALTIC

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În lucrare se analizează influența vitezei de răcire asupra gradului de finisare a granulației aliajului 6063 finisat cu prealiajul binar AlTi10 şi prealiajul ternar AlTi3C0,15. Sunt prezentate microstructuri şi macrostructuri care pun în evidență influența elementului dizolvat (Ti - 0,03%) şi influența nucleantului (TiC). Testarea gradului de finisare s-a efectuat cu ajutorul unei instalații tip ALCOA, utilizându-se cochile din materiale cu diferite conductibilități termice.

In the paper the influence of cooling speed on the finishing granulate degree of 6063 alloy finished with binary master alloy AlTi10 and ternary master alloy Al3Ti0.15C is presented. Microstructures and macrostructures that evidentiated the influence for the dissolved element (Ti - 0,03%) and for the nucleant (TiC) are presented. Testing on the finishing degree were performed on an ALCOA device by using three different types of molds with different thermal conductivities.

Keywords: AlTiC Master Alloy, Cooling Speed, Grain Refining, ALCOA Modified Test

Introduction

The potential benefits of TiCAl grain refiner have been recognized from some time, but its introduction in industrial processes seems to be not as spread as it supposed to be. The lesser tendency to agglomeration in the melt and the absence of poisoning should be enough reasons for consistent investigations. Grain refining performance assessed by ALCOA Cold Finger testing [1] showed that AlTi5C0.25 to behave similarly to AlTi5C0.2 at equivalent addition rates which means that to achieve a given grain size a smaller volume fraction of TiC is effective compared with TiB_2. Also it was shown that the use of ternary master alloy instead of binary master alloy or salt mixture adding is more efficient [2], suggesting that the grain refinement is achieved not only by the addition of titanium and boron to aluminum melt but by heterogeneous nucleation.

Adding grain refiners master alloy results in formation of a fine equiaxed structure by deliberately brakeing the columnar grains growth. The brake of columnar grains growth can be achieved increasing the number of nucleation

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centers, thus the grain growth being limited by the growth of its own grain neighbors, or influencing the solid-liquid interface proprieties.

In the first case the industrial practice imposed as refiners the ternary master alloys from the Al-Ti-B system, the nucleation taking place on the boride surfaces from the master alloys, or, according to phase diagram theory, on the properitectic phase Al_3Ti.

It is a well known fact that segregation elements act directly at the solid-liquid interface. The most important element that influences strongly the interface's phenomena is titanium. Initially it was believed that borides are strongly nucleants but recently it was demonstrated that the borides [3] are pushed to the liquid-solid separation limit and in the lake of titanium granulation was not refined. Thus it was concluded that Al_3Ti is a much stronger nucleant than TiB_2.

Other theory [4, 5] stated that the nucleation takes place on the existing borides and carbides. Technical difficulties related to the TiC stability in the liquid alloy were overcame imposing special work condition, AlTiC master alloys being obtained at industrial and not expensive scale [6].

In the case of dendrites growth the system can easily be described establishing the connection between tip of dendrite parameters (growth speed, tip diameter) and the growth conditions (thermal gradients and solidification rate). Thus, we can discuss about the heat diffusion in/through solution taking care of interface's curvature for two distinct cases: equiaxed dendrites growth and columnar dendrites growth.

Solving the thermal and dissolution problems can give us a wide image. The dendrite point speed is given by the slowest phenomenon [7], the diffusion of the dissolved element. Difference between the growing temperature and liquidus temperature defines the diffusion driving force.

According to this, a relation between the speed and the radius of dendrite tip and the known system parameters was obtained. For a better system characterization we have to take into account capillary phenomena in the system [8]. The diffusive effects appear and the radius of the dendrite tip is small enough to efficient reject the solvent obtaining a maxim for the growth. After that another concept was developed [9]. According to this (edge's stability) the dendrite expands having a radius equal to the limit wave length of a perturbation which can disturb a front plan.

Examination of the variables that can characterize the growth shows that the parameter easier to be manipulated is the thermal parameter. Cells grow systematically in opposite direction with heat flux and, in the case of columnar growth, main trunks lines to the nearest direction to heat flux.

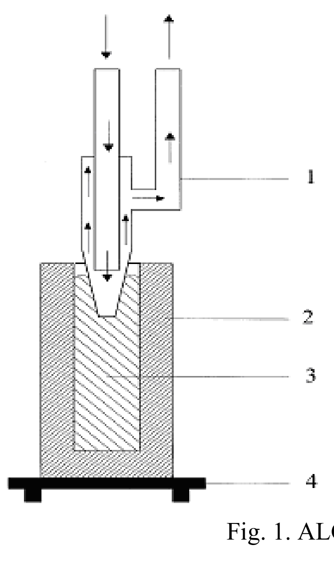
The second important variable that strongly influences the growth is the surface tension. Inhibition of growth processes, respectively atoms attachment, has as a result the decreasing of grain size. These two ways, controlling the thermal gradient and the influence of solid-liquid interface proprieties, will be the objectives of this work, by using the combination of two different types of master alloys.

The ALCOA grain refining test was initially developed bearing DC casting in mind. In order to evaluate the influence of the secondary cooling for the refining process for 6063 alloy with AlTiC master alloy, the ALCOA grain refining test was modified. The modification consists in the use of different types of molds kept not in a preheated coated steel mould but in the air. Thus, the influence of secondary cooling can be similar to a DC process.

2. Experimental method

The alloy studied is 6063 melted in a graphite mold with 1.1l capacity into an electric furnace. Three samples were poured for each type of mold, the first one was unrefined, the second one was refined with the AlTi10 master alloy and the third one was refined with Al10Ti and Al3T0.15C master alloys. Grain refining additions were made 5 minutes before casting at a temperature around 700^{o}C. The molds were preheated and the melt pouring temperature was 700^{o}C. The molds were made from Al_2O_3, graphite and copper. In order to better evaluate the influence of the secondary cooling, the molds dimensions were similar.

The test used to evaluate the grain refining grade was similar to ALCOA type and the cooling water flow rate was 2L/min. (Fig. 1). The equivalent quantities for titanium were 0.02%, in the case of using Al10Ti master alloy, and 0.03% Ti for the case of using Al10Ti + Al3Ti0.15C master alloys.



1 – copper pipe for cooling water;
2 – mold
3 – sample;
4 – mould support

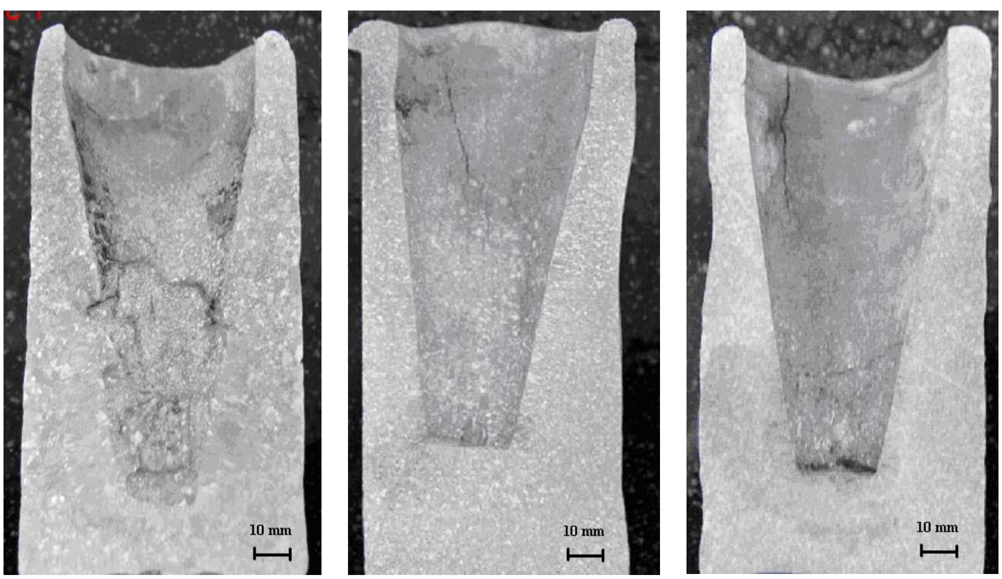
Fig. 1. ALCOA installation

After casting, cylindrical ingots were sectioned longitudinally through central axis and macroetching with 45mL. concentrated HCl, 15 mL. HF 48%, 15 mL. concentrated HNO_3 and 25 mL. H_2O to reveal grain structure.

Grain size measurements were made on samples wich were cut out of the bottom corner of the casting. The samples were treated for 5-10 seconds with an etching solution containing 100 mL. H_2O, 11 mL. 48% HF and 16 mL. concentrated HCl. Quantitative microscopic analysis was performed on a BX60M microscope, and the microstructural analysis were realized using XL30 ESEM electron microscope equipped with EDS system.

3. Results and discussions

Comparatively analyzing the macrostructures, an important grain refining degree can be seen at the use of titanium induced in the melt as Al10Ti or Al3Ti0.15C master alloys. Also, for different cooling speeds, related to the thermal conductivity for each mold used, an increase of grain refining degree is obtained for the materials with bigger values for heat transfer. (Fig. 2, Fig. 3 and Fig. 4).



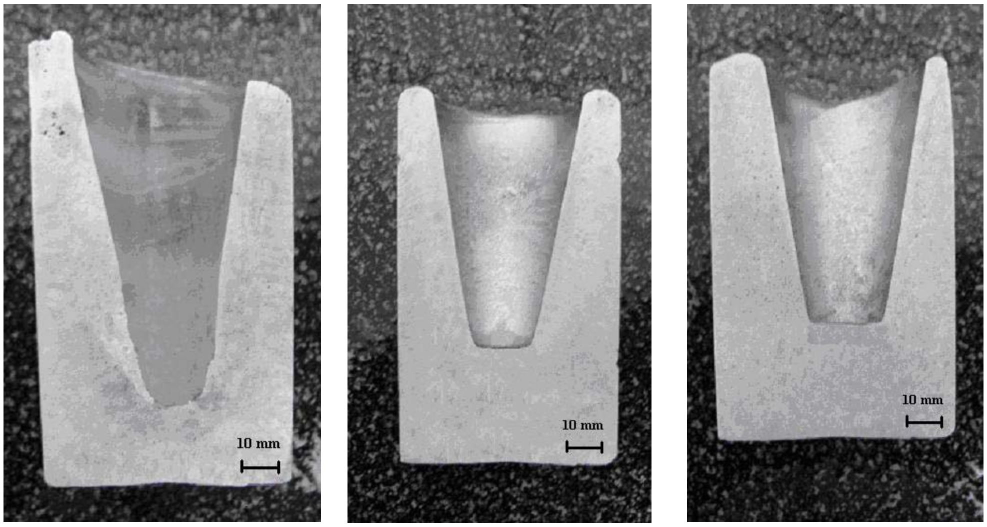
a b c

Fig. 2. Grain structure of 6063 alloy poured with alumina mold

a) unrefined

b) refined with Al10Ti master alloy (0.02% Ti)

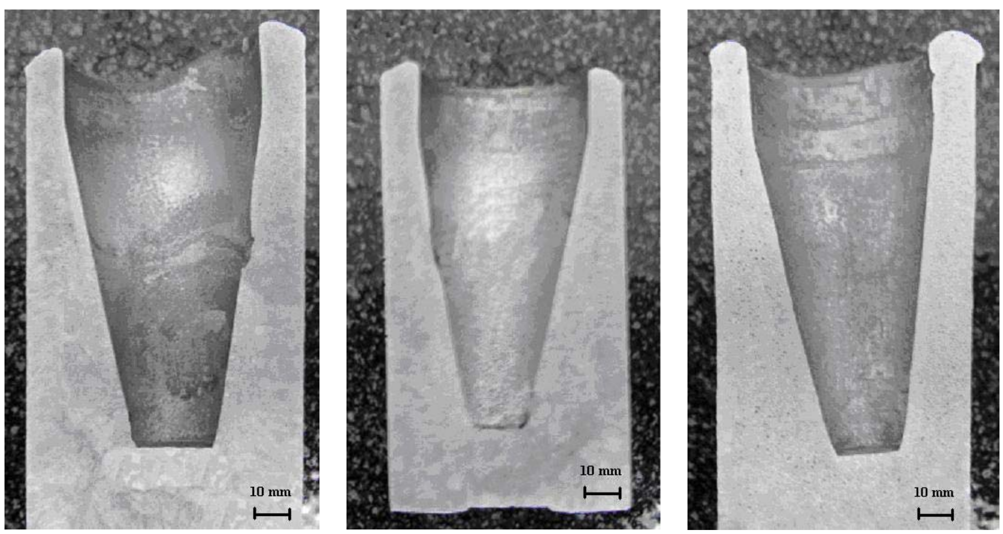
c) refined with Al10Ti + Al3Ti0.15C (0.03% Ti)



a b c

Fig. 3. Grain structure of 6063 alloy poured with graphite mold

a) unrefined
b) refined with Al10Ti master alloy (0.02% Ti)
c) refined with Al10Ti + Al3Ti0.15C (0.03% Ti)



a b c

Fig. 4. Grain structure of 6063 alloy poured with copper mold

a) unrefined
b) refined with Al10Ti master alloy (0.02% Ti)
c) refined with Al10Ti + Al3Ti0.15C (0.03% Ti)

This tendency is more obvious with microstructural analysis. With binary master alloy AlTi10 grain refiner the columnar grain growth was inhibited because at the free liquid-solid interface free titanium acts as a thermal shield. Also, uniform repartition of the grains suggests that Al_3Ti's nucleant effect is strong enough, probably because the retaining time was small enough to impede the appearance of the dissolution effect for Al_3Ti in the molten alloy due to the absorbed heat excess.

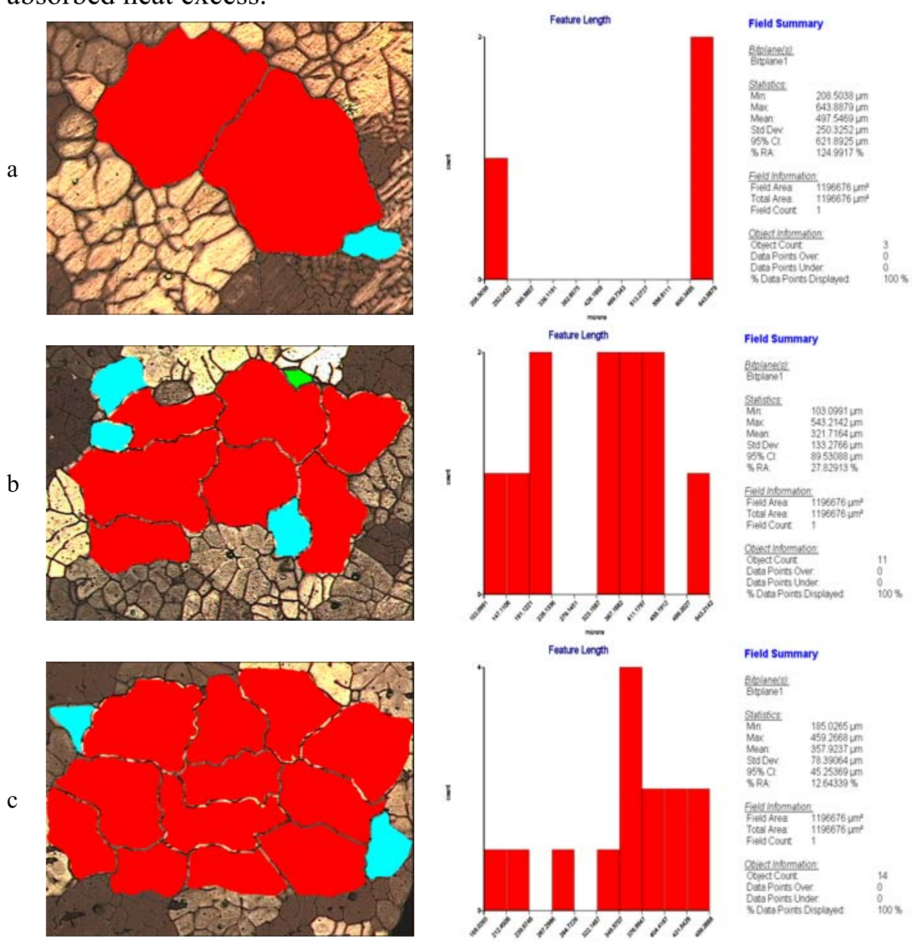


Fig.5. Microscopic quantitative analysis for 6063 alloy solidified in Al_2O_3 mold
a) unrefined
b) refined with Al10Ti (0.02% Ti)
c) refined with Al10Ti + Al3Ti0.15C (0.03% Ti)

In the third case, Al-Ti and Al-Ti-C additions, the structure is almost fully equiaxed, medium grain size being similar to that in the case of precedent inoculation with master alloy Al10Ti. Thus, added to the presence of Ti that acts as a segregator and favours Al_3Ti forming, TiC particles start the process of grain growing as nucleants. Nucleation in this case is starting on both TiC particles and properitectic Al_3Ti phase.

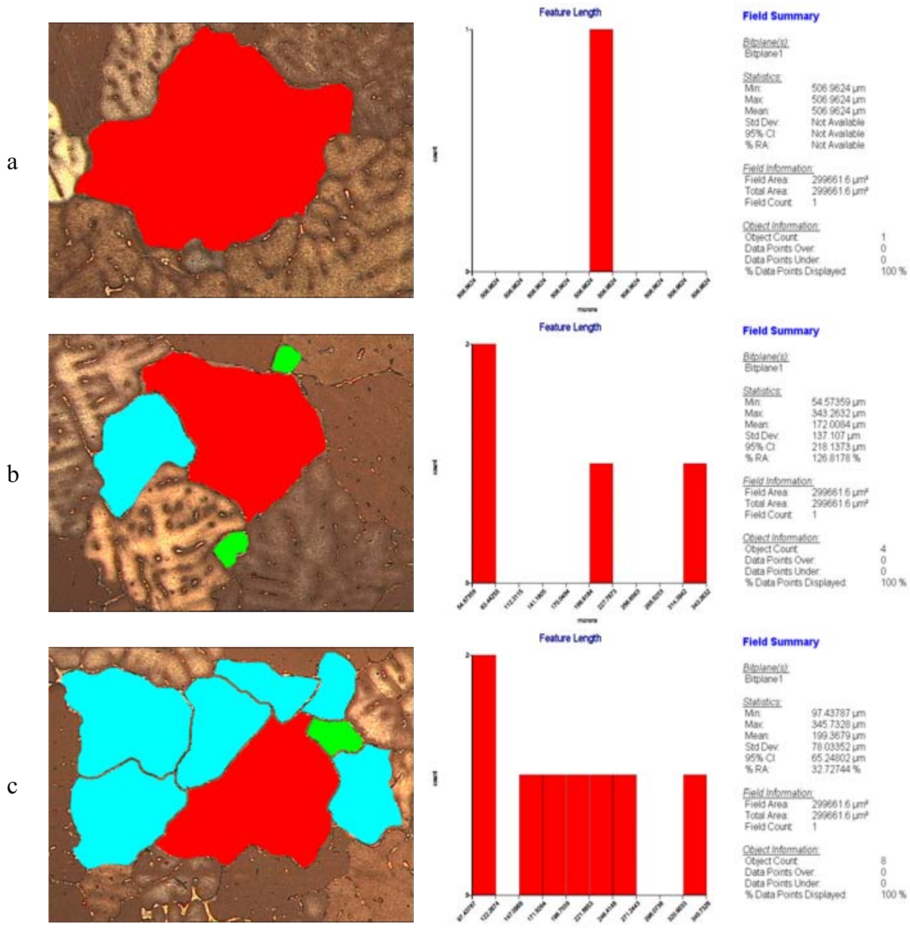


Fig.6. Microscopic quantitative analysis for 6063 alloy solidified in graphite mold
a) unrefined
b) refined with Al10Ti (0.02% Ti)
c) refined with Al10Ti + Al3Ti0.15C (0.03% Ti)

In Fig. 5, Fig. 6 and Fig. 7 the microscopic quantitative analysis for 6063 alloy solidified in different types of molds are presented. From comparison of medium grain size for the same type of refining, it can be seen that for the mold with the biggest thermodynamic conductivity the smallest medium grain size is obtained. The medium grain sizes for 6063 alloy refined with Al10Ti master alloy solidified in Al_2O_3, graphite and copper molds are smaller then the medium grain sizes for 6063 alloy refined with Al10Ti + Al3Ti0.15C master alloys solidified in the same molds. Thus, it is useful to compare the data also from another point of view, the difference between the maximal and minimal values for every microscopic analysis. The smaller this difference, the more uniform microstructure is.

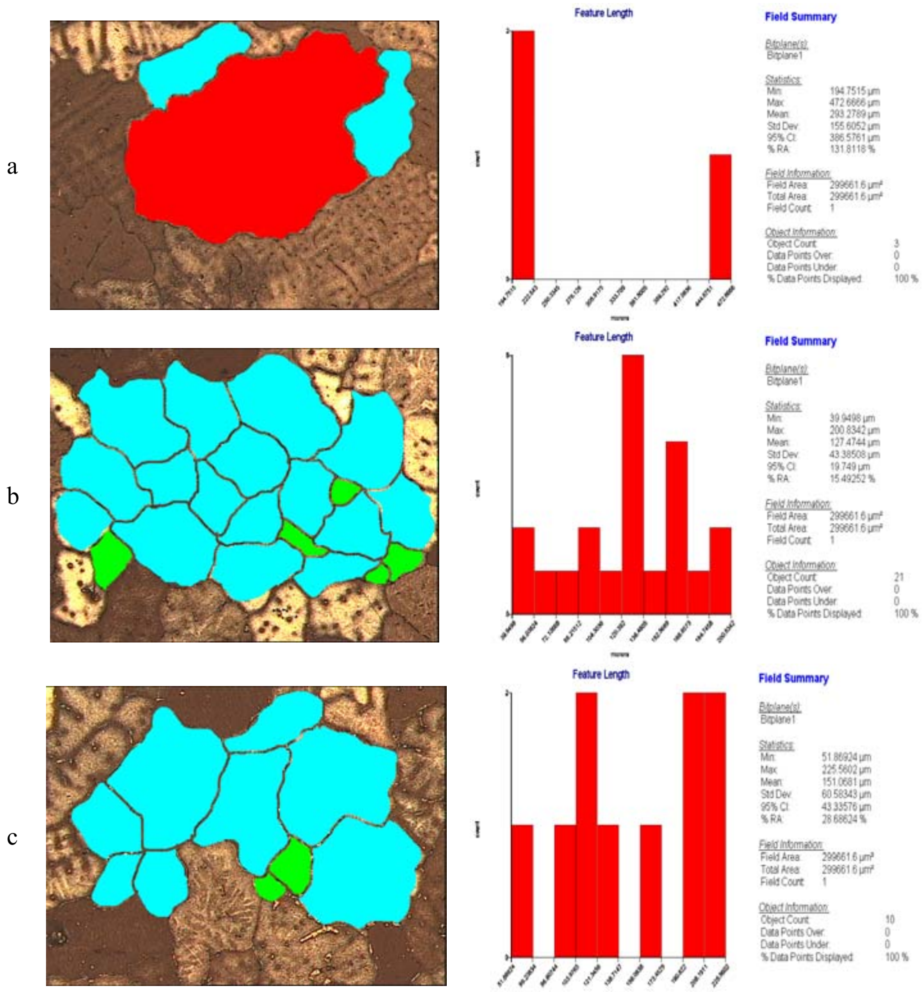
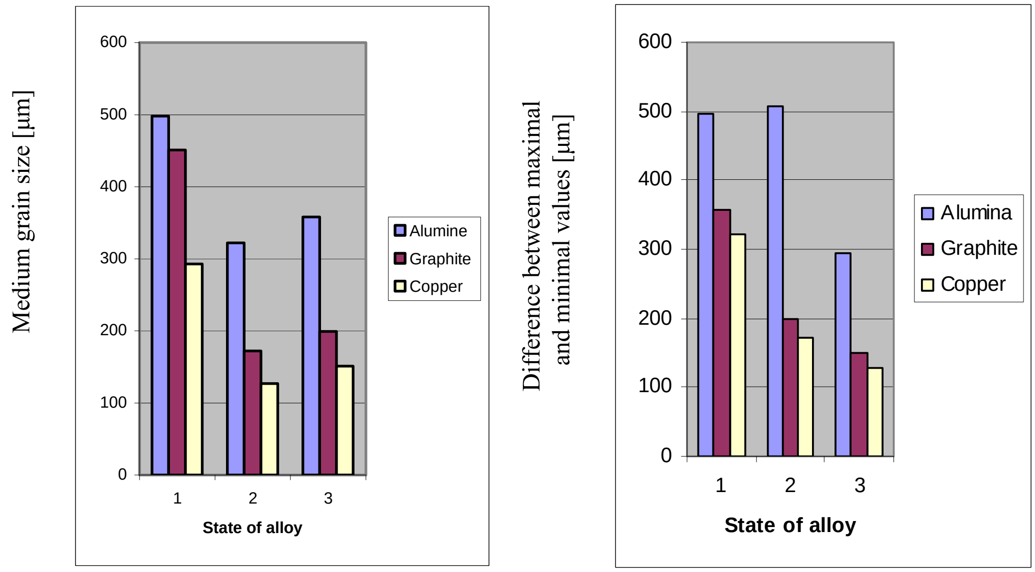


Fig.7. Microscopic quantitative analysis for 6063 alloy solidified in copper mold

a) unrefined
b) refined with Al10Ti (0.02% Ti)
c) refined with Al10Ti + Al3Ti0.15C (0.03% Ti)



a) b)

Fig. 8. Values obtained by microscopic quantitative analysis

a) Medium grain size for every type of mold versus stage of refining

b) Difference between maximal and minimal values obtained by microscopic quantitative analysis for every type of mold versus stage of refining.

Conclusions

The new TiCAl grain refiner is shown to be a considerable promise for critical applications and could be used in near future at industrial scale with minimal or additional costs.

The use of two master alloys, Al10Ti and Al3Ti0.15C, proved to be more efficient. The structure is more uniform with grains very similar in size.

Titanium, as solute element, seems to play an important role in growth restriction of dendrites acting as an inhibitor at solid-liquid interface. Also, combined in $TiAl_3$ phase it provides nucleation centers.

The TiC phase acts as a nucleation centre very similar with TiB_2 nuclei.

R E F E R E N C E S

1. J. Pearson, M.A. Kearns, Optimization of grain refiners in the cast house based on recent development programs, 5th Australasian-Asian Pacific Conference, 1997.

2. *G. P. Jones, J. Pearson,* Metall. Trans. B. 1976 7B, pp 223.

3. P.S. Mohanty, J.E. Gruzleski, Mechanism of grain refinement in aluminium, Acta Metall. mater., vol. 43, no. 5, 1995.

4. A. Cibula, , Journal of Inst. Met. Vol. 76, pp. 321-360.

5. P. Moldovan, Gabriela Popescu, The grain refinement of 6063 Aluminium Using Al-5Ti-1B and Al-3Ti-0.15C Grain Refiners, JOM November 2004 pp 59-61.

5. P. Hoefs, W. Reif, A.H. Green, Development of an improved AlTiC master alloy for the grain refinement of aluminium, Light Metals, 1997.

6. J.W. Cahn, Acta Metall. 1960/8, pp. 554.

7. C.Y. Wang, C. Beckerman, Metall. Mater. Trans. 1996/27A, pp. 2754.

8. W.W. Mullins, R.F. Sekerka, J. Applied Phys. 1964/34, pp. 444.