

Optimum grain refining with a high performance master alloy

A new grain refiner called *OptiFine* has proved at least twice as effective as existing refiners due to its more restricted size range of boride particles so enabling cost savings by reducing the amount required to be added to the melt and enhancing quality by improving melt cleanliness. **By R Vainik*, J Courtenay** & M Bryant****

OptiFine, is a newly developed type of 3% Titanium, 1% Boron master alloy grain refining rod which, for the first time in a low titanium grain refiner, combines potent grain refining effectiveness with good metal cleanliness. If this master alloy is used in combination with optimisation of the composition of the melt to restrict grain growth – eg, depending on the alloy, as little as 30ppm of titanium added to the melt – enable much reduced additions of *OptiFine* to give the same grain size as additions of much larger quantities of other commercial grain refiners present on the market today. Quantities can typically be more than halved.

The enhanced grain refining efficiency of *OptiFine* is explained by a uniquely narrow range of boride particle sizes in the master alloy, which allows simultaneous nucleation of a large number of aluminium crystals during the solidification process. Furthermore, the growth restriction of crystals, as determined by the alloy composition and aided by minute additions of titanium if needed, will allow a substantial proportion of these crystals to grow to full impingement size.

This use of a highly efficient grain refiner at very low addition rates not only brings significant cost savings but also has a beneficial impact on billet and ingot quality since the amount of hard boride particles required to produce a grain size resistant to ingot cracking is much less than with other grain refiners.

This paper deals with the theoretical background to growth restriction and grain

refining practice using the Opticast optimisation system. It then summarises experimental work carried out on a laboratory scale in a number of industrial casthouses around the world to compare *OptiFine* with other commercially available master alloy grain refiners.

Background

The sole purpose of grain refinement is to obtain a grain size in the final slab or billet that prevents the cast from cracking during solidification and subsequent rolling or extrusion. The grain refining addition should be as little as possible for two reasons: – decrease the cost for grain refinement; – minimise the amount of impurities added to the melt.

With regard to the amount of impurities it is known that the boride particles added via the grain refiner rod degrade the quality of the final product regarding surface finish, especially in

bright trim alloys and other products where the demand on surface quality is high. Other defects that can arise are pinholes in foils and can stock alloys. Impurities can also have a negative impact on the mechanical properties, since those boride particles not active in the nucleation process will end up in the grain boundaries of the solidified material.

Experience from grain refining optimisation work using the *Opticast*^[1,2,3,4] system has shown that in order to minimise grain refining addition it is essential to: – Improve the growth restriction conditions in the melt; – Choose the most efficient grain refiner; – Choose the optimum position to add the refiner.

Theory and practice

Growth restriction, which decides how fast nucleated crystals will grow, has a very large impact on the final grain size^[5] and is essentially a function of the melt composition. In principle the higher the concentration of alloying elements, the greater the restriction in grain growth. However, the growth restriction imposed by different alloying elements varies considerably. **Table 1** shows the growth restriction factor $(k-1)m$ for some common elements in aluminium alloys.

It is evident that Ti has a much higher growth restriction effect than any other element. Most grain refining agents therefore contain an excess of Ti, which goes into solution in the melt. It follows that growth restriction can be controlled in an easy way by

Element	k	m	(k-1)m
Ti	9	30.7	245
Si	0.14	-7.1	6.1
Mg	0.5	-6.2	3.0
Fe	0.02	-3	2.9
Cu	0.17	-3.4	2.8
Zn	0.4	-1.6	1.0
Mn	0.94	-1.6	0.1

Table 1 Growth restriction factor $(k-1)m$ for some common elements in aluminium alloys

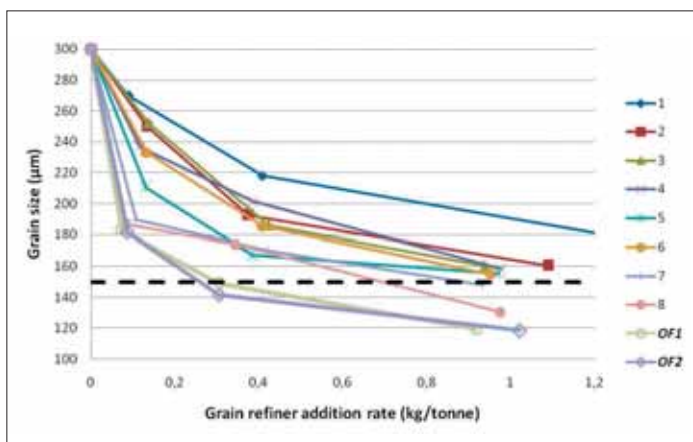


Fig 1 Opticast crucible tests during production cast of alloy AA1050, master alloys 1 to 8 are 5Ti/1B master alloys OF1 and OF2 are *OptiFine* alloys

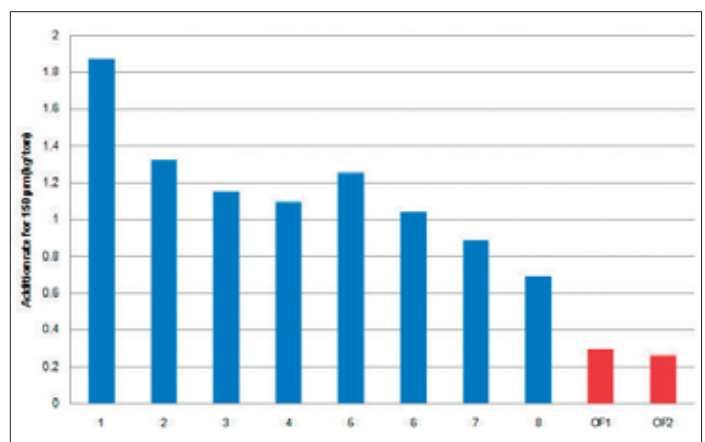


Fig 2 Master alloy addition rates necessary to obtain 150µm grain sizes, based on grain refinement curves in figure 1 (red bars are for *OptiFine*)

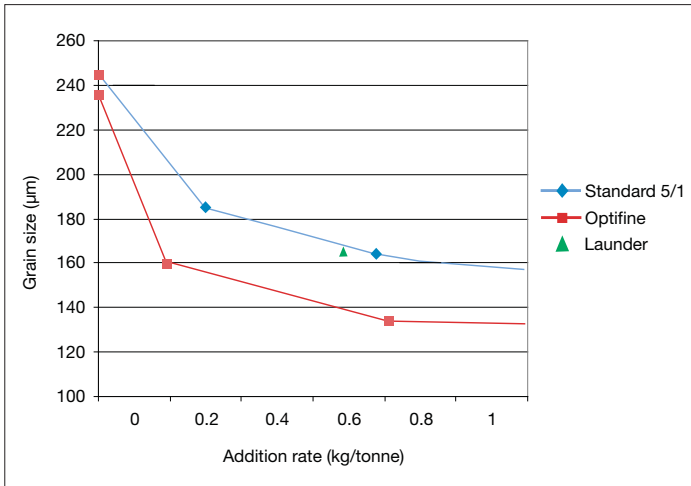


Fig 3 Opticast crucible tests and Opticast samples taken during production cast of alloy AA1050. Standard 5/1 grain refiner was used during cast

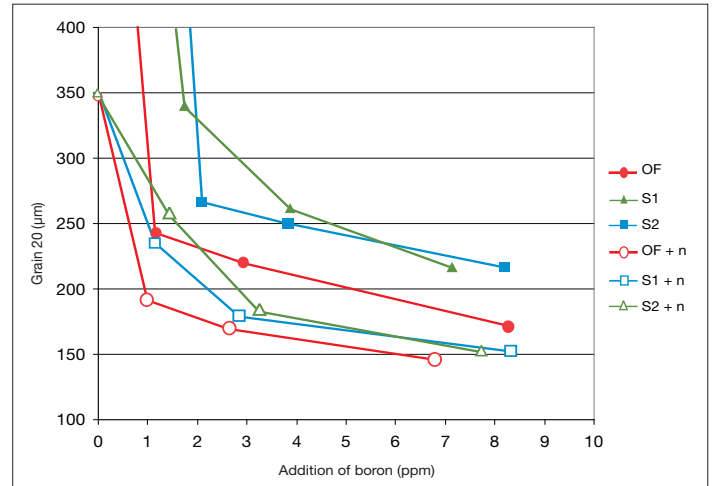


Fig 4 Crucible test in two charges of AA6060. OF = OptiFine, S1 and S2 are standard 5/1 master alloys. 45ppm Ti was added to the furnace in the second cast

adding the required amount of Ti in the melt. The necessary Ti level to achieve optimum growth conditions depends on the composition of the actual alloy. AA1000 and AA3000 series require more Ti in solution than AA6000 series alloy, whereas AA2000 and AA7000 series alloys require no extra addition of Ti at all.

An Al-Ti-B master alloy contains two forms of crystals in an aluminium matrix, Al_3Ti (aluminides) and TiB_2 (borides). When added to an aluminium melt, using a standard addition rate of 0.1 to 2kg per metric tonne (kg/t), the aluminide crystals are rapidly dissolved and the borides crystals are dispersed into the melt. According to present day theory, as presented by Greer and co-workers^[6, 7], there will be thin residues of Al_3Ti left on the borides, actively taking part in the nucleation process when the melt solidifies.

Two of the most important factors that determine the efficiency of a master alloy are the frequency of agglomerates and the boride particle distribution as a whole. Greer et al^[6] suggest that a highly efficient grain refiner can be produced if the borides are confined to a very narrow size range.

A grain refiner can be added at several alternative places in a casting system – before or after a degasser or before or after a filter. Each casting line has to be evaluated to find an optimum place for the addition.

Ahead of the degasser can be a favourable place since the intensive stirring in the degasser promotes a good dispersion of the boride particles. However, there are examples of degassing equipment that enhance the agglomeration of borides and so negates their grain refining potential.

The problem associated with additions before filters is the capturing of borides which decrease recovery and also can cause 'showers' of boride particles when these particles suddenly break loose from the filter.

The ideal solution would be a rapidly dissolving, clean grain refiner which allows a fast dispersion of equally sized boride particles. This could then be added after the

filter, and so eliminate the potential risks with pre gasser or pre filter additions.

Experimental programme

An experimental programme was designed to compare the grain refining efficiency of commercial master alloy grain refiners from all major producers with a newly produced type of 3/1 master alloy grain refiner. The new alloy, OptiFine, is made via a special production route, which optimises nucleation efficiency by creating a narrow range of boride particle sizes in the master alloy.

Grain refiners from all major producers and OptiFine have been tested by two methods. Firstly by taking Opticast samples during casting, before and after the rod addition point, so that the efficiency of the actual grain refiner is evaluated. Secondly using the crucible method^[5], where melt samples are taken in Opticast crucibles (100g) and master alloy pieces are added, in amounts simulating the normal addition rates for the alloys in question.

The Opticast sampling technique has been designed to give a slow solidification rate in order to exaggerate grain size differences, ie as compared to rapidly cooled samples. The crucibles have a wall thickness of 4mm and are made of stainless steel. Before sampling, the crucible is preheated to the same temperature as the melt. After the melt is collected, the crucible is placed on an insulating refractory material. The melt is then allowed to solidify and the crucible must not be disturbed during this time.

The grain sizes obtained in Opticast samples corresponds well to the grain sizes obtained near to the centre in commercially cast slabs. Thus, for the AA1050 tests to be discussed below, the grain sizes will correlate fairly well to those typically found in slabs. For the tests on AA6060, a billet alloy, there will however be a large difference in grain size between the samples and an actual billet. The grain size in an Opticast sample will typically be 80 to 100µm larger compared to the grain size in the corresponding billet.

When the method is implemented at a cast house, the method is calibrated against the actual casting conditions. Even the cooling rate for the Opticast samples can be changed, adapting to the casting operation. In this programme, all samples have been cooled similarly, since it is the comparison between the grain refiners which is in focus. The relative efficiencies of the grain refiners will, however, be the same regardless of casting operation and cooling.

All grain size measurements reported were performed using the line intercept method and at least 150 grain boundaries measured for each sample.

Experimental results

Three sets of results are presented:

- Laboratory grain refinement tests on a AA1050 alloy
- Casthouse grain refinement tests on a AA1050 alloy
- Casthouse grain refinement and optimisation of growth restriction tests on an AA6060 alloy

Laboratory grain refinement on AA1050

A number of 5Ti/1B master alloys from two commercial producers were obtained from an industrial cast house and compared with two 3Ti/1B OptiFine grain refiners. The results are shown in Fig 1. Alloy number 1 is from one producer while master alloys numbers 2 to 8 are from a different producer. It is evident that the efficiency of the two OptiFine grain refiners is markedly higher.

If it is assumed that the required grain size in the final ingots corresponds to a 150µm grain diameter in the Opticast samples, as shown by the horizontal broken line in Fig 1, then it is possible to calculate how much is needed of each master alloy. The point where the horizontal line hits the grain refinement curve gives the amount of grain refiner necessary, which is shown in Fig 2 for each refining master alloy.

About 0.3kg/t of the Optifine grain refiners will create a grain size of 150µm in AA1050.

At least twice that much is needed of the most efficient of the other grain refiners. In the worst case, alloy number 1, at least six times more master alloy is required to obtain the same grain size in this alloy.

Casthouse grain refinement tests on AA1050

To establish the required grain size and the necessary amount of OptiFine to obtain that grain size a normal production cast using a standard 5/1 master alloy as grain refiner during the whole cast, was monitored by using the crucible test and samples were also taken in the furnace and launder. The standard addition rate was 0.66kg/t, which corresponds to an addition of 6.6ppm boron when a master alloy containing 1% boron is used. The launder sample was taken at the casting table and had a grain size of 165µm, and a B concentration of 6ppm. This corresponds to 0.6kg/t, which means that the recovery rate was 90%.

Small pieces of the same standard 5/1 master alloy, as used during the cast were also used for crucible tests (blue curve in Fig 3). The launder sample (green triangle in Fig 3) fits very closely to this line, as expected. Crucible test samples were also taken for the OptiFine master alloy (red line Fig 3) which indicates that much less of this master alloy is needed to obtain the same grain size of 165µm ie about 0.13kg/t. This indicated a possibility to markedly reduce the addition of OptiFine, and still get the same grain size.

In a subsequent production trial it was found that an OptiFine addition of only 0.33kg/t gave a launder sample with a grain size of 138µm at a B concentration of 3ppm.

Casthouse grain refinement tests on AA6060

Preliminary work with Opticast, at a cast house where AA6060 alloy is produced indicated the need for an increase of the growth restriction conditions in this alloy. This was achieved by increasing the titanium level of the melt in the furnace. Crucible tests were then performed on one charge without extra Ti and one charge with an extra 45ppm Ti added in the furnace. The base Ti levels were practically the same in the two casts, 36 and 38ppm. The same grain refining alloys were used in both casts, two standard commercial 5/1 alloys from the cast house, and an OptiFine master alloy.

As can be seen from Fig 4, the addition of Ti has a strong enhancing effect on the nucleation efficiency for all three alloys, but the relative differences between the master alloys are retained.

From these results it seems that there is a possibility to reduce the grain refiner addition rate by about 70% if OptiFine is used in conjunction with an increase of the Ti level in the base melt.

To verify this, two tests were run at different refiner addition rates (Standard and Reduced) on two different casting units. The furnace melt Ti level was increased by approximately 30ppm for both. The results are shown in Table 2.

In both cases it is evident that the addition

Casting unit	Addition rate (kg/t)		Lowering of addition (%)	Final grain size (µm)
	Standard	Reduced		
1 (Standard)	0.6	0.45	25	172
2 (OptiFine)	1.15	0.55	52	153

Table 2 Comparison of master alloy additions and final grain sizes when Ti level of melt in furnace is increased by 30 ppm

rate can be reduced markedly, even if only a very moderate increase of Ti base level is made. Furthermore, the grain sizes in the launder samples are smaller than the smallest obtained in normal production casts.

Applicable alloys

In this paper, the high potency of OptiFine has been demonstrated in two alloys, AA1050 and AA6060. However, the same results have been obtained in various other alloys, eg AA6063, AA7075, AA5053, AA3003 etc. Even in 'difficult' alloys such as those containing high concentrations of Zr, the same relative difference in efficiency between OptiFine and standard grain refiners has been observed.

The production of OptiFine leads to a more homogeneous distribution of boride particle sizes. When nucleation starts, it will do so on a larger number of nuclei than for standard grain refiners. This means that a greater number of aluminium crystals are formed, which in turn leads to a finer grain size.

Calculations for the tests carried out on AA1050 alloy indicate that the number of grains per mm³ formed when using OptiFine is 70% higher than when formed using other master alloys, even although the addition rate of boron in OptiFine is 50% lower.

Thus we can conclude that OptiFine is a very potent grain refiner master alloy which can be used for all types of aluminium alloys. It contains a narrow range of boride particle sizes which allows nucleation of many aluminium crystals simultaneously.

Compared to other grain refiners OptiFine is at least twice as effective as the most potent of the other commercially available master alloy grain refiners. Grain refiner additions can thus be reduced by at least 50% using OptiFine. Thus the much lower addition rates when using OptiFine bring substantial cost savings and quality benefits in terms of far fewer impurities added in the form of boride particles. ■

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Salt-free melting environmentally sound

A recent patent, registered in Europe, should prove to be of keen interest to the secondary aluminium recycling sector.

Designed for use in tilting rotary furnaces, the product promotes salt-free melting of aluminium scrap and dross.

Tilting Rotary furnaces are without a doubt one of the most efficient scrap melting tools presently available offering; energy savings, high yields and fast melt rates.

However, most rotary furnaces require the use of salt (KCl + NaCl blends) to reduce any exothermic reaction during casting when the furnace door is open and air can ingress. The use of salt is environmentally unsound, costly to purchase and to reprocess and renders any waste slag toxic.

The newly salt-free patented process offers a number of advantages:

- Significant reduction of any exothermic reaction;
- Improved metal yields;

- Reduction in iron enrichment;
- Reduction in cleaning time; and
- Residual ash may be exploited.

The product will be in production on an industrial scale in the second half of 2009.

The owner of the patent is examining the possibility of granting local fabrication licences to reduce transport costs. The product is used as an additive in quantities of between 10 to 15% of charge weight.

Contact: mineraltechnology@orange.fr



Casting from a rotary furnace (left) without salt-free addition (right with addition).