RESULTS IN PRODUCTION OF AN IMPROVED GRAIN REFINEMENT PRACTISE FOR 6XXX EXTRUSION BILLETS

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Abstract

A project has been undertaken at Trimet Aluminium SE to improve grain refinement of extrusion alloys involving three stages. The first stage comprised making small scale tests using the Opticast method on 4kg melts at the MQP laboratory in Stockholm. In stage two testing was carried out on the R&D casting pit at Trimet in Essen where single billets were cast from a 1.2t furnace. The small scale tests predicted that grain refiner addition rate could be reduced by 88% and the results of the subsequent R&D and production tests confirmed that this was possible however the actual addition rate used for the trial was 0.16kg/t representing a reduction of 68%. The results of the third stage of the project, involving making full casts of 6060 billets on a production casting pit then evaluating these in terms of grain size, metallography and subsequent performance during extrusion, are reported.

Introduction

Nearly all aluminium produced today is grain refined by addition of master alloys containing titanium and boron, so called grain refiners. The most common grain refiners contain 3-5 % Ti and 1 % B, balance aluminium.

When aluminium solidifies, aluminium crystals are formed on nuclei present in the melt. Normally, the numbers of nuclei are very few, meaning that only a few aluminium crystals are formed, which leads to a coarse grain structure with weak mechanical properties. In the case of wrought alloys, cast in the form of slabs or billets, this frequently leads to cracks in the finals casts and this material cannot be further treated in rolling or extrusion processes, but must be re-melted. This means extra costs and in order to avoid this, grain refiners are added to supply nucleation sites for aluminium which results in a grain size fine enough to avoid cracks.

The grain refiners contain boride particles, TiB2, which are hard intermetallic particles. Apart from their ability to nucleate aluminium they have a detrimental effect on the quality of the final cast as they could give rise to pin holes and surface defects; negative effects that become more pronounced the more particles are added.

From the reasoning above it can be concluded that grain refiners are necessary for successful casts but if they are added excessively the material quality suffers. There is thus a large incentive to optimize the additions of grain refiners, ensuring crack-free casts with a low inclusion level. An additional argument to avoid over-additions is that a grain refiner is an expensive commodity in the cast house and large savings may be expected if the additions are optimized to the lowest possible level.

More than ten years ago a grain refinement optimization tool was presented, the Opticast method. The method has been successfully applied as a production tool at AMAG (Austria) since 2002 and at Hulamin (South Africa) since 2005 and is a means to decrease master alloy additions in a controlled way. The technique has been discussed in a number of papers [1-7] and its primary goal is to optimize each cast by adjusting the master alloy addition rate so that a minimum amount of grain refiner is added without risk for cracking.

During Opticast optimization work in laboratory scale and numerous tests at cast houses around the world the requirement for a very potent and consistent grain refiner was identified. Following significant development such grain refiner is now marketed under the brand name Optifine. It is of the Ti/B=3/1 type and seen on a relative scale, the efficiency is between twice, sometimes up to thirty times more efficient than the standard refiners normally used. It is produced via a special production route, which optimizes the nucleation efficiency.

An extensive experimental program with Optifine was conducted at Hulamin cast house and the outcome from these tests was that Optifine now is used to grain refine the annual production at this cast house.

Optifine is currently under testing or in production use at many cast houses around the world. This paper concerns the test results at one of these cast houses, a large smelter, Trimet Aluminium in Essen, Germany.

Theory

Growth restriction (Q), which decides how fast nucleated crystals will grow, has a very large impact on the final grain size [7]. This parameter is essentially a function of the melt composition and in principle it can be stated that the higher the concentration of alloying elements, the larger the growth restriction. However, the growth restriction imposed by the alloying elements varies in a large range. GRF or the Q factor is expressed in the following way:

$$GRF = Q = \Sigma(k_i-1)m_iC_i$$

Where C_i refers to the concentration of each individual element present in the melt and k_i represents the distribution coefficient for each element in the binary Al-i system and m_i is the slope of the liquidus line. Table 1 shows the growth restriction effect for some common elements in aluminium alloys.

Table 1. Phase diagram data.

Element	k	m	(k-1)m
Ti	9	30.7	245
Si	0.14	-7.1	6.1
Mg	0.51	-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

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. As a conclusion, the Q-factor can be controlled in an easy way by 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.

An Al-Ti-B master alloy contains two forms of crystals in an aluminium matrix, Al_3Ti (aluminides) and TiB_2 (borides). The relative proportions of these crystals depend on the Ti and B concentrations, which normally vary in the following intervals: Ti:1.5-10% and B:0.2-1%. The by far most common master alloys used in are of the 5/1 and 3/1(%Ti/%B) type.

When added to an aluminium melt, using a standard addition rate of 0.1 to 2 kg/ton, 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 [8] there will be thin residues of Al_3Ti left on the borides, actively taking part in the nucleation process when the melt solidifies.

There are a number of factors that determine the efficiency of a master alloy. Two of the most important are the frequency of agglomerates and the boride particle distribution as a whole. This was discussed in a previous paper [3], where it was stated that a narrow particle distribution was necessary in order to obtain optimum grain refining properties for a grain refiner. The background for this is that the size of a particle decides at what undercooling nucleation will start. When aluminium crystals are formed on the large boride particles, the solidification heat will mask off the possibility for the smaller crystals to nucleate aluminium. Greer et al [9] suggest that a highly efficient grain refiner can be produced, if the borides are confined to a very narrow size range.

The most likely explanation for the enhanced nucleation potency in Optifine is thus that the production route allows formation of a very narrow size fraction of small borides and prevents the formation of larges boride particles and agglomerates. The correlation between boride particle size distribution and grain refinement efficiency was investigated by Shu et al [11], who used ultrasound in order to finely divide the boride particles in a master alloy melt. They could show that the boride particle distribution was narrowed and the grain refinement efficiency increased accordingly. A larger investigation of the boride particle size distribution in Optifine is under way.

Results of small scale laboratory tests and casting trials at the Trimet R&D casting pit

In the first phase of this programme, reported at the 2014 TMS [12] data was presented from three methods of assessment; starting with Opticast tests performed on 4kg laboratory melts under closely controlled conditions, followed by an extensive number of ring tests and culminating with the casting of a 347 mm diameter 4 m long billet in alloy 6060 on the research casting pit.

The trend observed from each test sequence was consistent throughout confirming that Optifine is a potent and consistent grain refiner.

Overall it was concluded, based on the results obtained, that it should be possible to achieve the target grain size of ASTM Grade 2.5 for alloy 6060 in production using an application rate of 0.16 kg/t of Optifine. This would enable a reduction of 68% to be achieved from the current standard addition rate of 0.5 kg/t which would give significant benefits in terms of both overall economy and quality.

Results with Optifine grain refiner in comparison with normal grain refiner tested on a 6060 alloy in continuous casting in production

In this paper we report the results of the second phase of the programme in which testing in full scale production is undertaken in the Trimet casthouse.

Production casting conditions

Table 2 Details of production casting conditions

Batch number	**109-1640 Optifine**	**109-1642 Standard**
Number of billets	9	9
Diameter of billets	346mm	346mm
Billet length	7300mm	7300mm
Total Weight of billets	16,670kg	16,670kg
Waterflow	1500lt/min	1500lt/min
Temp. in launder	720°C	720°C
Cast time	118min	118min
Filter	40ppi CFF	40ppi CFF
Metal treatment	Chlorine+argon	Chlorine+argon
Pre grain refine in furnace	none	none
Grain refiner addition rate	**0.2kg/t**	**0.5kg/t**

Table 3 Analysis of production batches

Production batch 109-1640 with Optifine rod (0,2kg/t)		
	before rod addition (in furnace)	after rod addition (in launder)
Si:	0,457	0,448
Fe:	0,173	0,169
Cu:	0,008	0,008
Mn:	0,017	0,017
Mg:	0,437	0,426
Ti:	0,0093	0,0095
B:	0,00059	0,00073

Production batch 109-1642 with normal rod (0,5kg/t)		
	before rod addition (in furnace)	after rod addition (in launder)
Si:	0,446	0,433
Fe:	0,173	0,167
Cu:	0,014	0,014
Mn:	0,018	0,18
Mg:	0,426	0,416
Ti:	0,0081	0,0094
B:	0,00062	0,00070

After casting billet slices where taken from the head and bottom of the as cast billet and discs cut as shown below:

Figure 1. Position of sample discs cut from billets

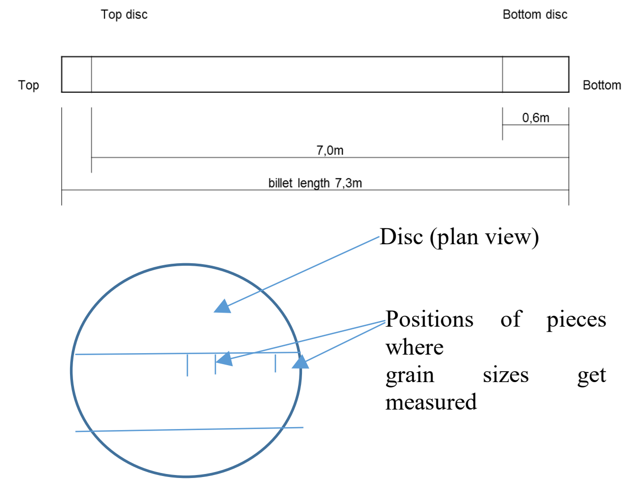


Figure 2. Grain size measurement - standard grain refiner

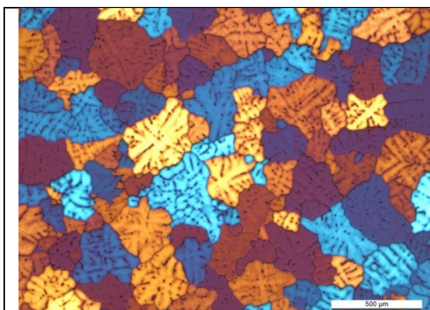
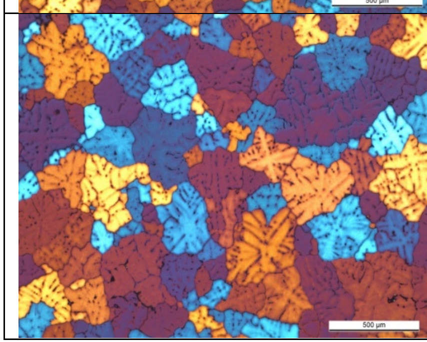
	Probe: 109-1642 Position: Head 2cm from shell zone ASTM: 2,36 Grain size: 158,7µm
	Probe: 109-1642 Position: Bottom 2cm from shell zone ASTM : 2,08 Grain size:175,0µm

Figure 3. Grain size measurement - Optifine grain refiner

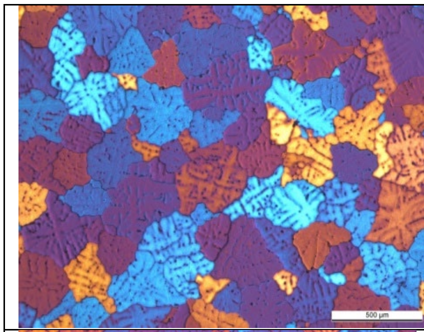
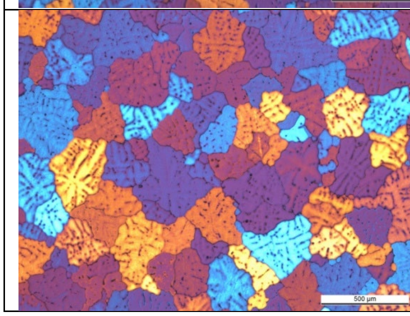
	Probe: 109-1640 Position: Head 2cm from shell zone ASTM: 2,15 Grain size: 170,6µm
	Probe: 109-1640 Position:Bottom 2cm from shell zone ASTM: 2,15 Grain size: 170,6µm

Table 4. Summary of grain size measurement results

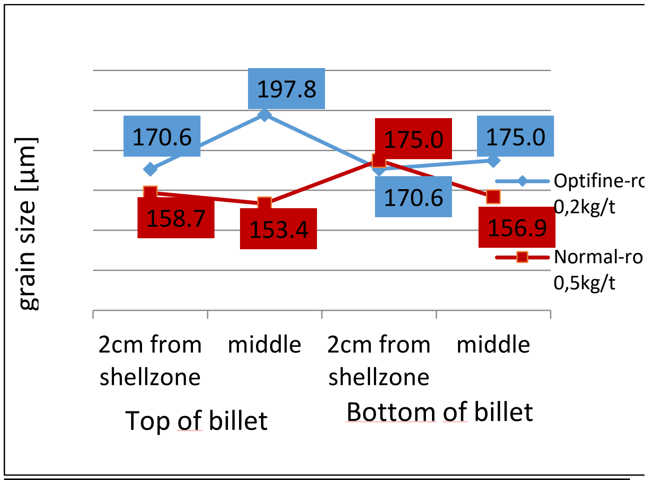
Production-batch with 0,5kg/t normal grain refiner

Number of batch	Billet-position	Piece-Position	Grain size [µm]
109-1642	Top	2cm from shellzone	158,7
109-1642	Top	middle	153,4
109-1642	Bottom	2cm from shellzone	175,0
109-1642	Bottom	middle	156,9

Production-batch with 0,2kg/t Optifine grain refiner

Number of batch	Billet-position	Piece-position	Grain size [µm]
109-1640	Top	2cm from shellzone	170,6
109-1640	Top	middle	197,8
109-1640	Bottom	2cm from shellzone	170,6
109-1640	Bottom	middle	175,0

Figure 5. Chart of grain size measurements of disks



Results of samples taken using Opticast sampler during production cast batch 109-1640 of 6060 billet (cast with 0.2kg/t Optifine)

Samples were taken using the Opticast sampling method at the beginning, middle and the end of the cast. The sample pairs were collected, one sample from furnace exit, i.e. no grain refiner added, and the other sample was taken close to the casting table, when grain refiner had been added. The 6 samples were sent to the MQP lab in Sweden.

The grain sizes measured in the samples are shown below:

Table 5. Grain size measurements of Opticast samples taken during casting of batch 109-1640

Furnace samples		Casting table samples	
	Grain size (µm)		Grain size (µm)
Start	421	Start	188
Middle	454	Middle	183
End	433	End	184

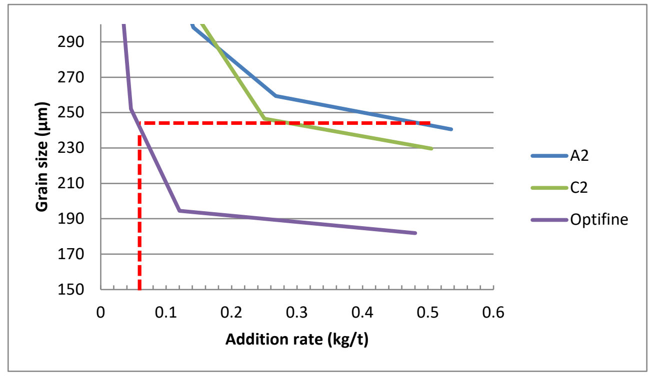
In order to see how Optifine performs against two grain refiner samples used in the tests reported in the 2014 TMS paper [12], the furnace samples were re-melted and grain refiner pieces were added to the melt according to the crucible test procedure described earlier[4] . Two of the least efficient grain refiners were used in the tests.

Table 6. Grain size measurements of crucible test samples

Supplier	Batch	Addn rate (kg/t)	Grain size (µm)
MQP	OF800	0.19	189
A	2	0.49	260
C	2	0.50	230

The Optifine addition gave about the same grain size as in the casting table samples above but the two other grain refiner samples behaved very poorly.
It is interesting to compare these results with the tests made last year, see figure below.

Figure 6. Results of crucible tests of different grain refiner samples in batch 109 - 1640



The master alloys A2 and C2 are identical with the grain refiners used in the current test. The A2 master alloy grain size is reproduced while the C2 master alloy grain size was somewhat larger in the present test, 260 versus 245 µm.
Regarding the Optifine used at this first round of tests, which was another batch than we use as a reference now and which is different from the Optifine used during the recent trials at Trimet, the grain size was measured to be around 190 µm, which is very close to what we have measured now. To conclude, three different batches of Optifine gave the same grain size at an addition level of 0.2 kg/t. i.e. between 183 to 190 µm confirming that a consistent and reproducible grain size is achieved.

Discussion

The further important observation is that Optifine can give the same grain size at an addition rate of 0.06 kg/t as the least efficient of the "normal" master alloys that had been used at Trimet at an addition rate of 0.5 kg/t. It is then important to establish what the largest acceptable grain size is. If all casts of AA6060 have been successful during the last year, then we can with certainty state that the addition rate can be reduced from 0.5 to 0.06 kg/t, i.e. a reduction of 88 %. However, if problems had occurred during this time period, it probably means that the use of "bad" grain refiners have given these problems. And then a maximum grain size has to be defined.
Finally, the grain size in the Opticast samples are close to the grain size found in the billets, about 180-190 µm as compared with 170-190 µm in the billets. This means that when the two rather inefficient standard alloys discussed above were used in production, the grain size must have been well over 200 µm in the billets.

Conclusion

A production cast of 6060 was successfully carried out using Optifine with an addition rate of 0.2kg/t compared to the addition rate of 0.5kg/t used for one of the standard grain refiners used at the cast house – representing a reduction of 60%.

Analysis of the variation observed with standard grain refiners indicates that an addition rate as little as 0.06kg/t could be used, provided the grain size obtained with the least efficient one is acceptable and do not result in cracks or create problems downstream. On balance an addition rate of 0.16kg/t can be used in production with a good safety margin giving a reduction in grain refiner addition of 68%.

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