Light Metals 2014
Edited by: John Grandfield
TMS (The Minerals, Metals & Materials Society), 2014

IMPROVED GRAIN REFINEMENT OF AA6060 EXTRUSION BILLETS

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Keywords: Grain refinement

Abstract

Improving grain refinement practice in production is time consuming although the benefits of reduced costs, improved quality and better control of finished product grain size are highly desirable. 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 4 kg melts at the Opticast 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 5t furnace. Finally production casts of AA6060 billets will be made using Optifine grain refiner. The small scale tests predicted that grain refiner addition rate could be reduced by 88% and the results of the subsequent R&D casting pit production tests to confirm this 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 a number of 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:

$$\mathbf{GRF = Q = \Sigma(k_i\text{-}1)m_iC_i}$$

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 of 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.

Small scale trials at Opticast Laboratory in Stockholm

A batch of 8 kg of alloy AA6060, taken from a production cast, was sent for laboratory trials, see table 2 for composition.

Table 2. Composition of AA6060, used for laboratory trials at Opticast Laboratory.

Element	Concentration (wt-%)
Si	0.45
Mg	0.44
Fe	0.18
Ti	0.009
B	0.00003

12 master alloy samples from three different suppliers were supplied at the same time, all of the Ti/B = 3/1 type. These were samples of grain refiners from three suppliers that had been used or were going to be used in production, see table 3.

Table 3. Ti/B = 3/1 master alloys received for testing.

Supplier	Master alloy
A	A1
	A2
	A3
B	B1
	B2
	B3
C	C1
	C2
	C3
	C4
	C5
	C6

In order to decrease the number of tests it was decided to make a selection of the master alloys above, i.e. to choose the least efficient master alloy from each producer. This decision is based on a long time experience of master alloy testing, which has shown that the grain refinement practice in almost all cast houses is adjusted according to a "worst scenario case", where the master alloy addition rate is set to a level that can take care of the spread in master alloy efficiencies.

The screening test, which involves the comparison of Optifine with the supplied master alloys, was performed according to a procedure described earlier [4], the so called crucible test. In short, this test is performed using 99.7 % Al, in which the growth restriction condition is set to a constant level by addition of 0.016 % Ti. The temperature of the melt was kept at 730°C throughout the test. Portions of 100 g samples of this melt were collected in stainless steel crucibles and master alloy pieces were added to give addition rates corresponding to 0.4 and 0.8 kg/t. This means that the master alloy pieces weighed approximately 0.04 and 0.08 g respectively.

The outcome from the screening test is shown in figure 1.

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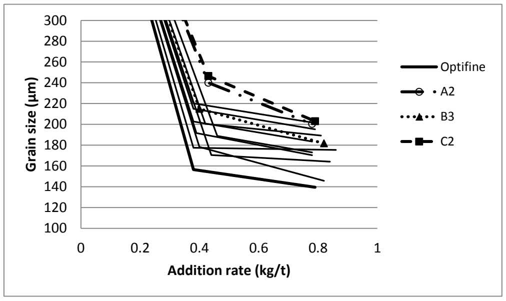

Figure 1. Grain refiner comparison test in 99.7 Al + 0.016 % Ti. All master alloys from table 3 are included in the figure. The four alloys selected for tests in AA6060 are pointed out.

The figure above shows that Optifine, which is the bottom curve, was a more efficient or much more efficient master alloy than the production grain refiners. Three of these refiners, indicated by hatched lines, were picked out for specific testing in alloy AA6060. One refiner was chosen from each producer.

For the AA6060 test, 4 kg of the received metal was melted and kept at 710°C, the nominal production casting temperature for this alloy. The crucible tests were performed the same way as for the screening test and the grain refiner addition rates and the resulting grain sizes are shown in table 4. Figure 2 summarizes the results in a graph.

The production addition rate is 0.5 kg/t for this alloy and therefore the following selection was made for the test: 25, 50 and 100 % of nominal addition rate. This corresponds to sample weights of 0.0125, 0.025 and 0.05 g in a 100 g melt sample. However, the initial test in 99.7 % Al indicated that Optifine was much more efficient and therefore a 12.5 % addition rate was also included, corresponding to a master alloy sample weight of 0.006 g.

Table 4. Addition rates and grain sizes for crucible tests in alloy AA6060

Sample	Addn. rate (kg/t)	Grain size (µm)
Furnace	0.00	452
A2:1	0.14	298
A2:2	0.27	259
A2:3	0.54	241
B3:1	0.12	271
B3:2	0.23	244
B3:3	0.45	204
C2:1	0.13	317
C2:2	0.25	247
C2:3	0.51	230
Optifine:1	0.05	252
Optifine:2	0.12	195
Optifine:3	0.23	182
Optifine:4	0.48	171

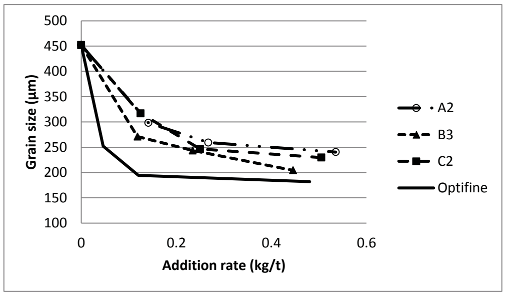

Figure 2. Grain refinement tests in alloy AA6060.

As in 99.7 Al, figure 2 shows that the grain refinement efficiency of Optifine is markedly better than for the selected three alloys even in alloy AA6060.

The crucial point with these tests is to show how much Optifine is needed to obtain the same grain refinement as with the production grain refiners. Assuming that these master alloys has been used in production and that no problems, i.e. no cracks, were encountered during production, this means that the grain size obtained with the least efficient grain refiner was acceptable.

Figure 3 shows how much Optifine is be needed to obtain the same grain refinement as when the least efficient grain refiner is used, refiner A2. At the addition rate of 0.54 kg/t, the grain size was measured to 241 µm. At the addition rate of 0.5 kg/t the grain size can be extrapolated to 245 µm. Optifine is able to take down the grain size to 252 µm at the very low addition rate of 0.05 kg/t, see table 4. Thus, slightly more than this amount of Optifine, about 0.06 kg/t is needed to give 245 µm in the final sample. This discussion is summarized by figure 3, where the horizontal dotted line shown the grain size level and the vertical dotted line shows at which concentration Optifine can give the same result.

In summary, by using Optifine, the current results indicate that the addition rate can be decreased from 0.5 to 0.06 kg/t, which means a reduction of 88 %.

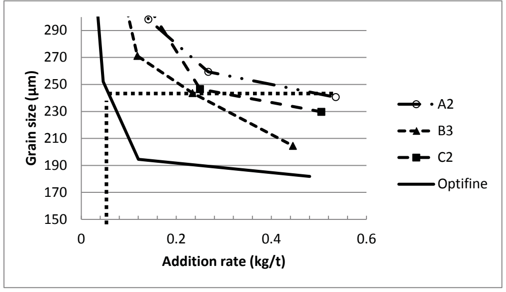

Figure 3. Blow-up of figure 2 and illustration of Optifine amount needed to obtain a certain grain size.

Results of small scale laboratory tests at Trimet R&D

A procedure based on a ring test [10] was used to give a preliminary assessment of the efficiency of Optifine grain refiner compared to one of the standard grain refiners used supplied by producer A. The test procedure comprises casting of 99.7% aluminium melt into 20 mm deep 40 mm diameter steel rings placed on a room temperature steel plate. After addition of respective grain refiner, samples were cast at 20 second intervals up till 2 minutes and then at 200 second intervals up till 20 minutes had elapsed. The melt temperature was maintained between 750ºC - 760 ºC. The test results are tabulated in table 5 and the bar charts in figures 4 and 5 shows the values in the table graphically.

Table 5. Addition rates and grain sizes for ring tests in alloy AA6060. All grain sizes are in µm.

	Producer A		Optifine	
Addn. rate (kg/t)	0.5	1.5	0.5	1.5
Time (s)				
20	272	265	257	204
40	197	341	175	180
60	476	326	220	212
80	445	304	297	221
100	409	-	228	257
120	388	-	268	256
200	306	316	232	236
400	329	334	295	266
600	241	257	238	197
1200	252	221	190	195

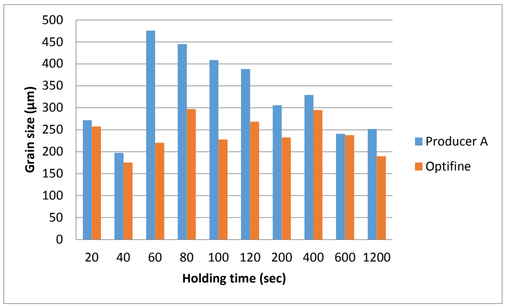

Figure 4. Grain size test values from table 5, for 0.5 kg/t addition rates.

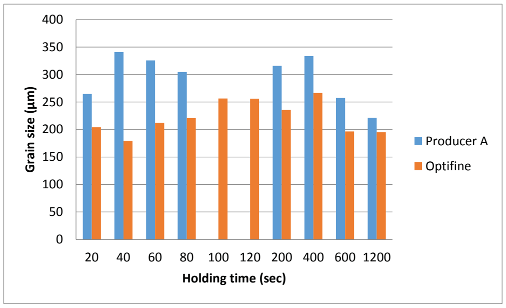

Figure 5. Grain size test values from table 5, for 1.5 kg/t addition rates.

From the table and the figures above it is clear that Optifine was more effective than the production master at every holding time.

Casting of experimental billets on the R&D casting machine

Trimet has invested heavily in developing a full scale casting pit dedicated to research and development that can produce 7.5 m long billets up to 600 mm in diameter, see figures 6 and 7.

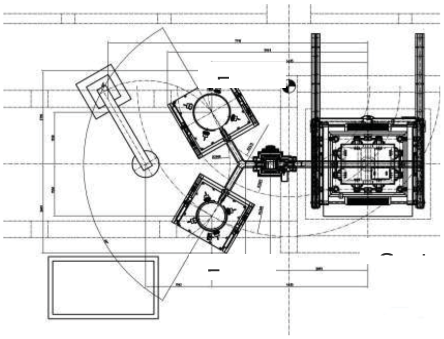

Figure 6. Layout of R&D casting pit.

Figure 7. Photograph of R&D casting pit taken from the casting table side.

The features of the facility can be described as follows:

Details of R&D casting pit

- 2 Casting furnaces (2.5 mt)
- 7.5 m casting length
- 600 mm max. casting diameter
- Fully automated casting process
- Stop/Go casting
- 1.8t and 5t furnace
- Pulsation water cooling
- Inline ultra sonic check (option)
- Impeller cleaning (argon/nitrogen /chlorine)
- CF-filter with pore burner

Following the promising results obtained from the small scale laboratory tests carried out in the Opticast laboratory in Stockholm and the ring tests in the R&D laboratory it was decided to cast a single 7.5 m long, 346 mm diameter billet on the R&D casting pit using Optifine grain refiner. The procedure adopted was to start the cast with the standard addition rate for this grade and then reduce it in steps.
Casting conditions for experimental billet:

- Diameter: 346 mm
- Same metal input (percentage of scrap, primary metal etc.) for standard and Optifine casting
- Same casting parameters for standard and Optifine casting
- Alloy: AA6060 (identical composition in standard and Optifine casting)
- AA6060 alloy in production with 0,5 kg/t grain refiner (standard)
- 6060 alloy on R&D pit with Optifine:
    - 0,5 kg/t the first 1000 mm cast length
    - 0,3 kg/t between 1000 mm and 2600 mm cast length
    - 0,16 kg/t between 2600 mm and end of casting (4100 mm)

Table 7. Composition of AA6060, used in R&D-casting trials at Trimet.

Element	Concentration (wt-%)
Si	0.44
Mg	0.43
Fe	0.2
Ti	0.01
B	0.0005

The casting parameters were identical to those used in production casts:

Casting speed:	62 mm/min
Water:	125 l/min (1 mould)
Melt temperature (launder):	720°C

Four different master alloys were used in the trials, see table 8.

Table 8. Master alloys used at R&D-casting trials at Trimet.

Master alloy	Comment
A4	New master alloy from supplier A
B2	Used in lab test, figure 1.
B4	New master alloy from supplier B
Optifine	Used in lab test, figure 1.

The potency of alloys A4 and B4 was tested in lab scale. In figure 8 their performance is shown against the master alloys used in the lab tests in alloy AA6060, see figure 1.

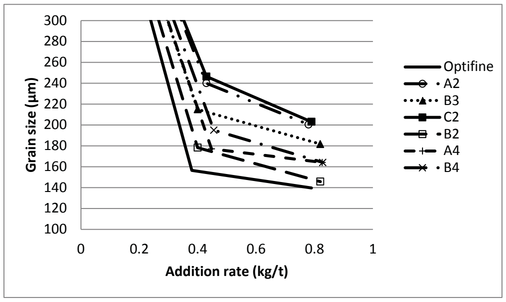

Figure 8. Lab test in 99.7 Al + 0.016 Ti on master alloys used in lab tests and R&D pit trials in AA6060.

Figure 8 shows that the R&D test master alloys lie relatively close to Optifine in performance and a therefore a small grain size would be expected in these tests.

Results of the casting trials on the R&D casting pit

The results of the casting trials on the R&D casting pit are summarised in table 9.

Table 9. Results of casting trials on the R&D casting pit

Test no.	Rod addition [kg/t]	Grain size [µm]	ASTM grain size (E112)	Master alloys
111-6449	0.5	151	3.10	B4 + A4
111-6470	0.5	152	3.01	B2 + A4
111-6442	0.5	172	2.70	B2 + A4
114-0304	0.5	146	3.14	Optifine
114-0304	0.3	182	2.50	Optifine
114-0304	0.16	187	2.40	Optifine

The standard grain refinement practise at the R&D pit is to feed from two coils simultaneously; this is necessary to achieve the required addition rate and achieve a good dissolution and distribution of the grain refiner rod. As can be concluded from table 9, the three production master alloy batches used in the tests managed to give a relatively fine grain size compared to Optfine. However it should be noted that on some occasions when grain refiner batches with low efficiency are used, e.g. the ones used in lab scale tests in AA6060, the expected grain size would be > 200 µm.

The target grain size in the billets in production of alloy AA6060 at Trimet is between Grade 2 and Grade 3 on the ASTM scale with a value of 2.5 being ideal. Production casts do arise with gain sizes of Grade 2 and these are not rejected. This practise is consistent with the observation of the spread in performance of standard grain refiners seen in the Opticast tests.

The Optifine trial billet showed a grain size of ASTM 2.5 at a 40% reduction in addition rate and ASTM of 2.4 at 68% reduction, that is, an addition rate of 0.16kg/t.

Detailed examination of the billet slices confirms the trend shown in the overall summary results given in table 7. The billet slices of the Optifine trial casts at the lowest rate of addition, 0.16 kg/t from either of the shell zone, middle or for alloy AA6060, 2 cm from the shell, have equivalent grain sizes and structures to those of the production casts. Micrographs of the middle grain sizes are shown in figures 9 and 10.

In light of the spread of grain sizes seen in normal production, and observed on the R&D pit charges of alloy 6060, this was considered to be a highly satisfactory result.

The next step will be to carry out full scale casting trials on a production casting pit and the results of these will be presented at the TMS conference in February 2014.

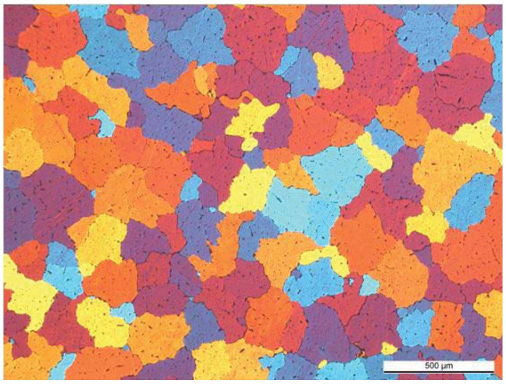

Figure 9. Production charge 111-6470, standard grain refiners, middle, 0.5 kg/t. ASTM = 3

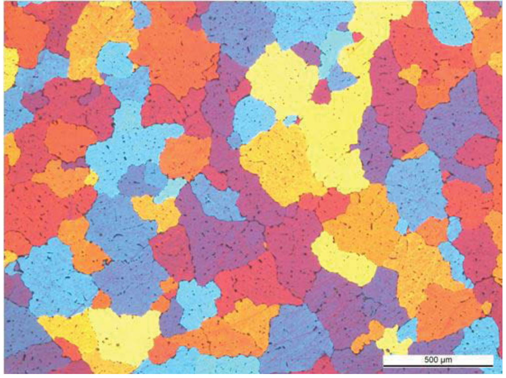

Figure 10. Trial charge 114-0304, Optifine, middle, 0.16 kg/t. ASTM = 2.4

Discussion

All Optifine batches are quality control checked against a full grain refinement curve template, therefore performance is always reproducible and addition rates can be set at the casting pit with full confidence that the ASTM grade can be kept within very narrow limits. Thus in this case an addition rate of 0.16 kg/t could be set to reach the standard target of ASTM Grade 2.5. However the range required is between Grade 2 and Grade 3 and in fact Grade 2 casts are not necessarily rejected. Therefore, depending on the criticality of application, for standard qualities, where Grade 2 is acceptable a lower application rate for Optifine could be used, because, since there is no variation in the efficiency of the Optifine grain refiner, this grade would be achieved with certainty. Referring to the results of the Opticast crucible tests shown in table 3 it can be seen that that with an addition rate of 0.48 kg/t of Optifine a grain size of 171 µm was achieved. The grain size given by the crucible test is equivalent to that achieved in a 400 mm thick slab but in the case of casting billets, because the cooling rate is higher, grain size needs to be adjusted downwards by a factor determined in practise by checking the billet slices. In the case of these tests a grain size of 148 µm was reached in the cast billet and therefore the factor is approximately 20µ. Thus where a grain size of 200 µm is acceptable in the billet the equivalent grain size in the crucible test would be 220 µm, corresponding to an addition rate of 0.08kg/t or a reduction of 84%, which is very close to original prediction from the crucible tests that a reduction of 88% was possible.

On the other hand, higher addition rates could be used when ASTM Grade of 3 or higher is required for special orders.

Nonetheless, the focus of the next phase of the programme will be to verify that an addition rate of 0.16

kg/t can be applied in production of 6060 billets to consistently produce an ASTM Grade 2.5 grain size.

Conclusions

At the time of writing, data is available 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 7.5 m long billet in alloy 6060 on the research casting pit.

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

Overall it was concluded, based on the results so far, 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.

The next step will be to carry out full scale production casts and the results of these will be presented at the forthcoming TMS conference in San Diego in February.

Acknowledgements

The authors would like to thank the management of Trimet Aluminium SE for their support in undertaking this project and giving permission for publication.

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