The effect of grain refiner on aluminium filtration

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Abstract

Grain refinement of aluminium and its alloys is a common industrial practice. Fine equiaxed, grain structure leads improved castability, strength, machinability, formability, and good surface finish. Filtration is one of the widely used technologies to remove inclusions from the melt. Ceramic Foam Filters (CFFs) are commonly used to clean the aluminium melt before the casting process. However, at a high inclusion load and with grain refiner addition, reduced filtration efficiency is well known to occur.
In the current work, the filtration behaviour of CFFs with three different levels of inclusions and grain refiner has been systematically studied in plant scale pilot trials at Hydro's reference centre in Sunndalsøra, Norway.

The results show that oxide films capture grain refiner particles. Grain refiners tends to agglomerate heavily with inclusions at higher inclusion content. These heavy and compacted small clusters are more likely to be released from the CFF during the filtration process. Little effect from grain refiner addition on filtration efficiency is observed when the level of grain refiner and chips addition is relatively low or when both are high, but not with high grain refiner addition with middle level of inclusion load. The threshold of the grain refiner addition effect is further discussed in this paper.

Introduction

Grain refinement by adding master alloys is a routine industrial process for the casting of aluminium. It has been investigated over the past 50 years [1-3]. It has been confirmed that increasing the number of nuclei by heterogeneous nucleation informs fine equiaxed grain structure upon solidification. This fine equiaxed grain structure results in high yield strength, high toughness, good Hot- and cold- formability, and uniform distribution of second phase and microporosity on a fine scale resulting in improved machinability, good surface finish, resistance to hot tearing, and various other desirable properties.

In the past few decades, CFF is known as one of the most common ways to remove inclusion from aluminium before casting [4-13]. However, introduction of grain refiners leads to a reduced filtration efficiency, significantly at high inclusions loading [14, 15]. Retention of inclusions in the filter voids by built up inclusion ``bridge`` [16] is associated with high filtration efficiencies. While the presence of the grain refiner would seem to prevent the occurrence of bridges within the filter structure. Still, few direct evidences prove this hypothesis.

It was found in our previous work that grain refiner master alloys, Al-3Ti-1B, Al-5Ti-1B, Al-3Ti-0.15C, wet alumina substrate better than pure aluminium in sessile drop method [17]. Heavy grain refiner particles not only settle at the metal - substrate interface, but also adhere to the surface oxide skin regardless of gravity, as indicated in Figure 1. Furthermore, it was observed that TiB_2 chains and clusters adhered to aluminium oxide films floating at the top of the melt with chips contamination [18].

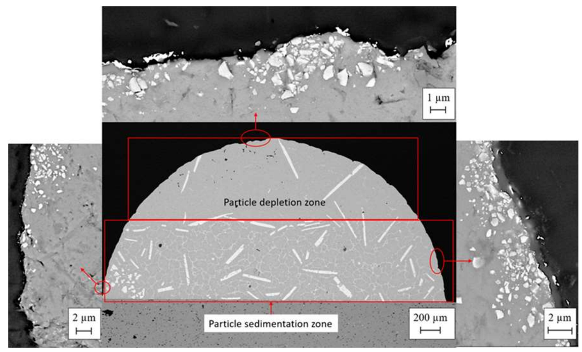

Figure 1 Microstructure of Al-5Ti-1B master alloy on alumina substrate after 1h holding at 1000°C in high vacuum [19]

In the current work, industrial scale pilot filtration test with different level of grain refiner and inclusion load has been carried out. The effect of grain refiner on filtration will be discussed.

Experimental Procedure

The industrial scale pilot test has been carried out in a DFF [20] the reference center casthouse, Hydro Aluminium Sunndalsøra, Norway. The DFF is a new drain free CFF technology developed by Hydro where the metal is lifted by under pressure and the priming of the filter is done in the reverse direction. The center has a loop with a melting furnace containing 9 tons of aluminium alloy, launder system, and a metal pump. A schematic of this loop is shown in Figure 2. The chips were added right into the furnace, and metal circulated in the loop at 150 RPM pump speed, leading to a flow rate of 6.9 ton/h. Al-3Ti-1B grain refiner master alloy was fed upstream from the filter, but after LiMCA position 1, when requested. New metal was charged into the furnace for each test. Three tests were carried out as listed in Table **I**. Two liquid metal cleanliness analyzers (LiMCA II), which give online information of the inclusion size and level, were positioned before and after the filter. Sampling with Porous Disk Filtration Apparatus (PoDFA) were also taken.

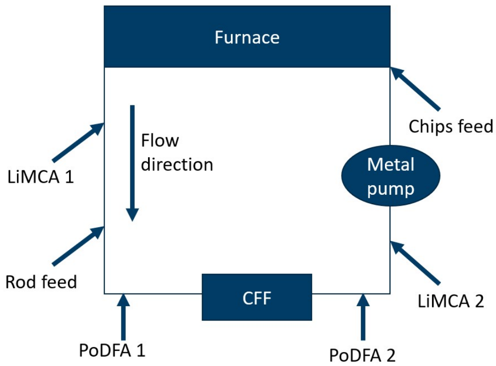

Figure 2 The schematic top view of the filtration loop

The 6060 aluminium alloy contained about 0.4 Si, 0.17 Fe, 0.01 Cu, 0.11 Mn, 0.4 Mg in wt%. The CFF filters used in the experiments were a low phosphor 80 PPI (pores per inch) SIVEX filter produced by Pyrotek, with dimensions of 584 × 584 × 50 mm (23 inch). The composition of the filters were > 70 wt.% aluminum oxide, 0.5-1.5 wt.% bentonite, and 1-10 wt.% aluminum phosphate.

Table I Experimental overview

Test nr.	Filter [PPI]	Grain refiner addition [g/kg]	Chips addition [kg]	Max* incl. > 20 µm [K/kg]	Avg.* incl. > 20 µm [K/kg]
1	80	0.5	50	16.2	4.5
2	80	2.0	50	40.0	9.9
3	80	2.0	80	54.1	22.3

*It is the inclusion number before the filer.

Result and Discussion

Inclusion measurement by LiMCA II

Figure 3 shows the N20 (number density of inclusions larger than 20 µm in the unit of 1000 per kg of melt) in Test 1. During approximately 90min filtration, the increase in inclusion level before the filter (In as indicated in Figure 3) is a response to the chips addition at 17.9 min and the maximum amount of 16.2 K/kg is achieved at 31.4 min. This gives average N20 of 4.5 K/kg as summarized in the last two columns of Table **I**. It is worth mentioning that the first LiMCA was positioned before the rod feeding. Thus, it cannot sense the effect of grain refiner, which was added at 49.9 min. But grain refiners run through the filter would flow into the melting furnace and meet the first LiMCA at their next round. It is observed that metal quality improves during filtration as inclusions are removed.

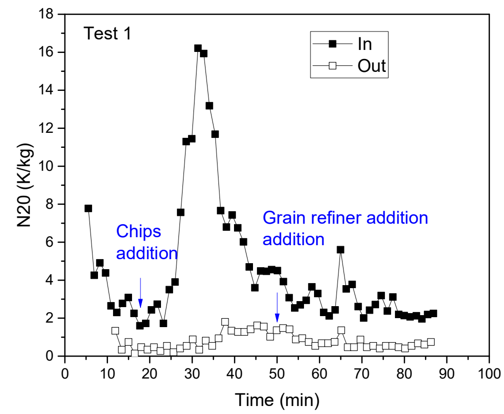

Figure 3 The inclusion level N20 in Test 1. N20 means number density of inclusions larger than 20 µm. In refers before the filter and out refers after the filter.

Similarly, Figure 4 and Figure 5 show the N20 in Test 2 and 3 with the indication of chips addition and grain refiner addition. Increase of inclusion number at filter outlet (Out) at around 58 min in Test 3 is the disturbance due to PoDFA sampling. As expected, the maximum N20 has increased from 40.0 K/kg with 50 kg of chips addition to 54.1 K/kg with 80 kg of chips addition with the same grain refiner addition of 2.0 g/kg. Average inclusion number before the filter doubles (from 4.5 to 9.9 K/kg in Table I) when increases the grain refiner addition for 4 times (0.5 to 2.0 g/kg in Table I). This might be due to a portion of the grain refiner escapes from the filter and runs into the loop after the holding furnace.

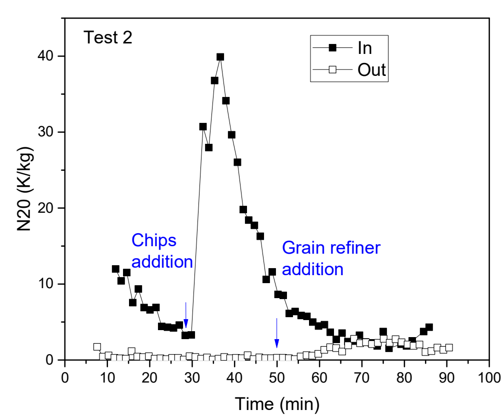

Figure 4 The inclusion level N20 in Test 2

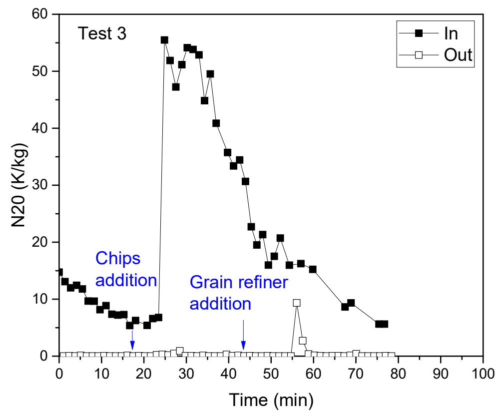

Figure 5 The inclusion level N20 in Test 3

As shown in Figure 6, more than 98% of the inclusions are smaller than 35 μm in Test 1, 87-95% in Test 2, and 90- 92% in Test 3. Chips and grain refiner increase the possibility of the formation of large inclusions. Inclusions up to 60 μm, 80 μm, and 100 μm have been randomly detected in Test 1, 2, and 3 even after filtration, respectively. After the grain refiner addition, the N15-25 after the filter increases 3.3% in Test 1 and 15.9% in Test 2, while N15-25 drops 10.4% and N25-50 increases 12.8% in Test 3. This means that grain refiner, generally in size of 0.1-2.0 μm, only increases the number of inclusions in size of 15-25 μm with 50kg chips. However, with further increase of chips up to 80kg, the number of larger inclusions in size of 25-50 μm rises. This confirms the agglomeration of oxide films and grain refiner particles. Authors in previous work [21] verified that chips addition increases the amount of oxide film in the melt. Cake filtration has stopped part of the inclusions and grain refiners, especially when they tangled with each other. One part of particles that did not halt by cake filtration at the filter inlet has further escaped from the depth filtration in the filter. They might have agglomerated again as early as in the filter. The result also indicates that metal cleanliness after the filter depends on that at the inlet. As fine as 80 PPI filter still cannot guarantee the same metal quality for different level of dirty metal.

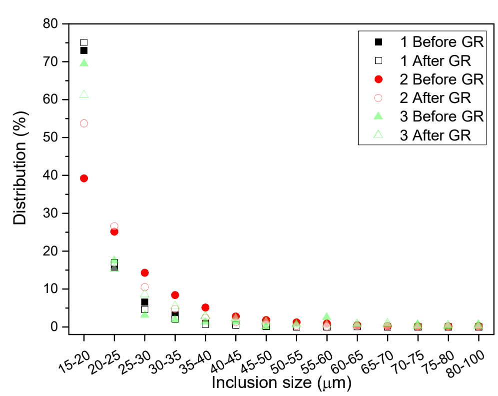

Figure 6 The inclusion size distribution after the filter. The inclusions after chips addition were summarized.

As shown in Table **II**, the inclusion number after the grain refiner addition decreases in test 1 with low chips and low grain refiner addition. But more inclusions after the grain refiner addition is detected in Test 2 with low chips and high grain refiner addition. While inclusion number slightly increases with further increase of chips addition at high grain refiner load. This is often described as that grain refiner spoils the filtration. Here, we will discuss the tolerance of CFF regarding metal dirt combined with grain refiner.

With average N20 of 9.9 K/kg, grain refiner addition reduces the filtration effect, but not with half of this inclusion load. This effect has been released partly when further decrease the metal cleanliness, but inclusion size slightly raises.

Table II Average inclusion number (K/kg) detected after the filter: Before = average inclusion number measured before the grain refiner addition and after chips addition, After= average inclusion number measured after the grain refiner addition until the filtration end, and difference Δ = Before-After

Test nr.		N20	N15-20	N20-25	N25-30	N30-35
1	Before	0.90	2.43	0.52	0.22	0.11
	After	0.72	2.15	0.49	0.13	0.06
	Δ	0.18	0.28	0.04	0.09	0.05
2	Before	0.29	0.93	0.22	0.04	0.00
	After	1.56	3.46	1.05	0.29	0.13
	Δ	-1.28	-2.52	-0.83	-0.26	-0.13
3	Before	0.14	0.32	0.08	0.01	0.01
	After*	0.54	0.85	0.21	0.12	0.08
	Δ	-0.39	-0.52	-0.13	-0.10	-0.07

* The spike due to PoDFA sampling is included.

Dynamic filtration efficiency

There are several ways to calculate filtration efficiency from the LiMCA data [22]. Generally, the filtration efficiency or inclusion removal efficiency is calculated as

$$F.E. = \frac{N_{in} - N_{out}}{N_{in}} \quad (1)$$

where N_{in} refers to the inclusion number into the filter, and N_{out} refers that out from the filter.

To investigate the influence of inclusion size, which affected by grain refiner and chips addition, on filtration efficiency, the dynamic filtration efficiency as a function of times will be presented. The decreasing trend in the inclusion number during all three 3 tests (Figure 3 to Figure 5) is likely a result of settling in the melting furnace, as well as inclusion consumption in the CFF. It is also possible that the inclusions interact with each other and agglomerate, which will lead to the decrease of inclusion number. With the analytical method verified by authors in earlier work [22] considering settling, Figure 7 shows that filtration efficiency reduces with time, where inclusion number after chips addition has been curve-fitted with a model assuming exponential decay.

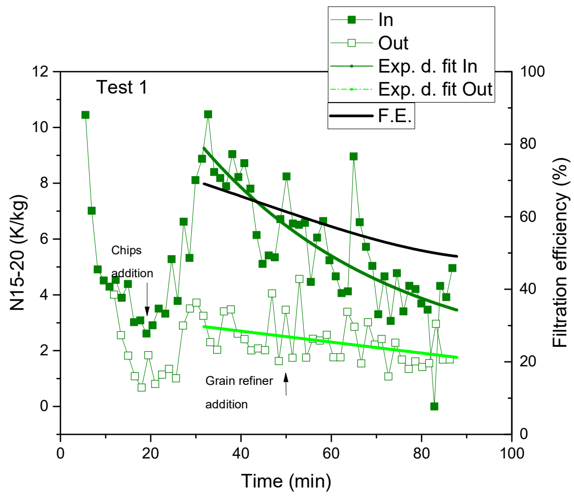

Figure 7 The inclusion level N15-20 and its dynamic filtration efficiency

With the same calculation, filtration efficiency of various particle sizes has been summarized in Figure 8 to Figure 10 using the same scale. Filtration efficiency less than zero means that metal gains inclusions during filtration, most probably that inclusions and grain refiners agglomerate and becomes noticeable. Adjusted R^2 [1]is up to 0.98 for inclusions before the filter (In), and up to 0.58 for after the filter (Out). Apparently, this model describes best the dirty metal. Generally, higher filtration efficiency is observed for larger inclusions. Filtration efficiency reduces with time, especially after grain refiner addition. However, filtration efficiency for N20-30 rises again following grain refiner addition in Test 1. The inlet fitted N20-25 equals to 2.0 K/kg and N25-30 0.7 K/kg at trough. Authors had qualified that CFF filtration without grain refiner addition still can practice reducing filtration efficiency, for example 20% in 1 h for N20-25 [22]. But, sliding from 100% to between 28% and -71% has been observed for Test 2 (Figure 9). Thus, grain refiner addition accelerates this harmful effect.

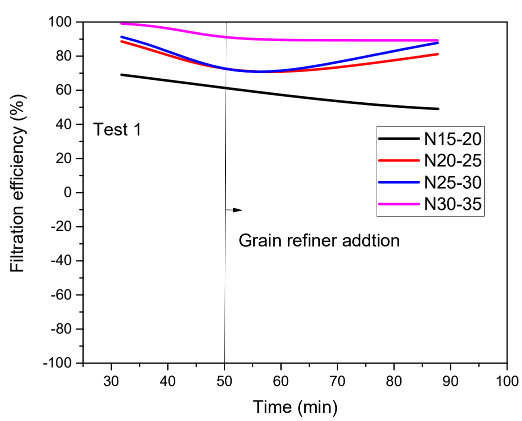

Figure 8 Filtration efficiency in Test 1

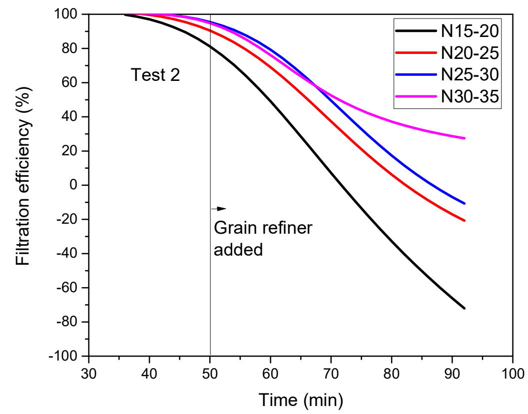

Figure 9 Filtration efficiency in Test 2

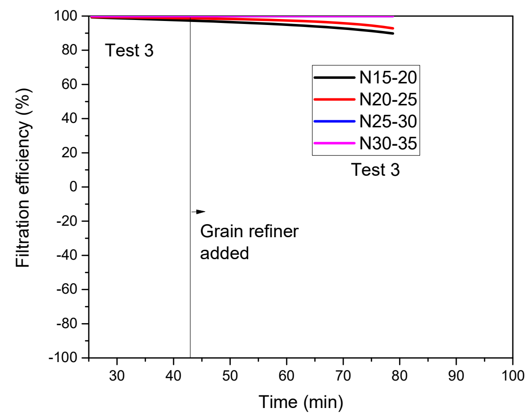

Figure 10 Filtration efficiency in Test 3

The tolerance of grain refiner in CFF

Then, does grain refiner always destroy filtration effect? If not, where is the threshold? It seems that grain refiner addition at inclusions N20-25 > 2.0 K/kg and N25-30 > 0.7 K/kg at average N20 of 4.5K/kg speeds up filtration efficiency drop in Test 1 (Figure 8). This effect is more evident at average N20 of 9.9 K/kg in Test 2 (Figure 9), and slows down again when average N20 is increased to 22.3 K/kg in Test 3 (Figure 10). Thus, generally we can conclude for 80 PPI CFF that

it has no detrimental effect when

- small amount of grain refiner addition (0.5 g/kg) in low inclusion load (N20 of 4.5K/kg) or
- high amount of grain refiner addition (2.0 g/kg) in high inclusion load (N20 of 22.3K/kg),

but it has detrimental effect when

- high amount of grain refiner addition (2.0 g/kg) in medium inclusion load (N20 of 9.9K/kg)

As discussed, grain refiner increases the inclusion number in size of 15-25 µm, but can increase the number of 25-50 µm inclusions as well when more inclusion are added. This indicates that grain

[1] R^2 is a statistical measure that represents the proportion of the variance for a dependent variable that is explained by an independent variable or variables in a regression model. An R^2 of 1 indicates that the regression predictions perfectly fit the data.

refiners particles mainly do not agglomerate with each other but would like to attach onto inclusions. Yang et. al [18] confirmed the likelihood of oxide film and TiB_2 particles. They have more chance to meet each other in the wandering passages in CFF. These heavy clusters are easier to escape from the CFF when compared to lighter inclusions without grain refiner addition. It seems that grain refiner has most detrimental effect on small inclusions as in size of 15-20 µm (Figure 9). If we look at N20-30 in Figure 11, filtration efficacy after 60min increases again for low inclusion load case in Test 1. This might trace the tolerance of CFF. But more tests should be carried out.

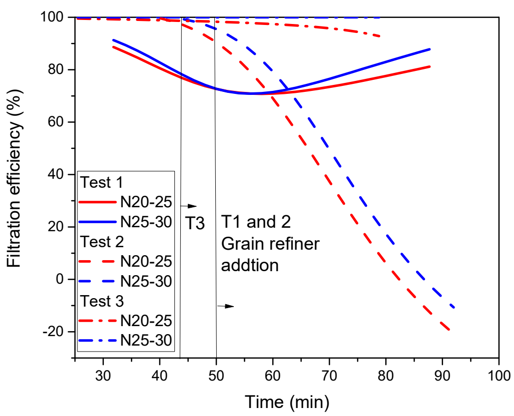

Figure 11 Filtration efficiency for inclusions N20-30

Why wouldn't high inclusion load with high grain refiner spoil the filtration efficiency? Our hypothesis is that this detrimental effect enhances when the ratio between grain refiner amount and inclusion load rises. Even greater amount of grain refiner is requested to destroy the inclusion removal by filter with high inclusion load. Larger cake formation on top of the filter would halt more inclusions, while less grain refiners escape into the filter and agglomerate with inclusions. Of course, this also indicates the more loss of grain refiner.

Then, how we can prevent this detrimental effect? Adding grain refiner after filtration promotes the agglomeration. Probably reduced grain refiner addition at certain inclusion level would be a choice. But grain refinement will be reduced. This need to be compensated by some other way.

The particles less than 15 µm is under the detection limit of LiMCA II. Thus, we cannot see the agglomeration of smaller inclusions. PoDFA should give a clue in next publication. We will only focus on LiMCA data in the present work. It is also hard to estimate the loss of grain refiner in filtration. This would be an interesting topic to study regarding economic benefit.

Conclusions

In this work, plant scale filtration tests were carried out with three different level of inclusion load and grain refiner. LiMCA II was used for inclusion counting. The dynamic performance of filters has been studied.

A statistical analysis of LiMCA data confirms that grain refiner ruins the filtration efficiency, but not always. With lower ratio of grain refiner at certain level of inclusion, no matter high or low load, filtration efficiency still can be kept at a proper value. Grain refiner seems affect largely the small inclusions in size 15-20 µm, sometimes up to 50 µm depending on the inclusion load. For 80 PPI filter in the current work, 2.0 g/kg grain refiner addition with average N20 of 9.9K/kg inclusion load destroys the filtration effect, but not much when increasing the inclusion load or decreasing the grain refiner addition. This threshold would change depending on parameters, such as flow rate, filter type, and inclusion type etc. Descending filtration efficiency during filtration can be seen in any CFF with or without grain refiner addition. Filtration efficiency rises with inclusion size. CFF still cannot guarantee the same metal quality for different level of dirty metal.

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