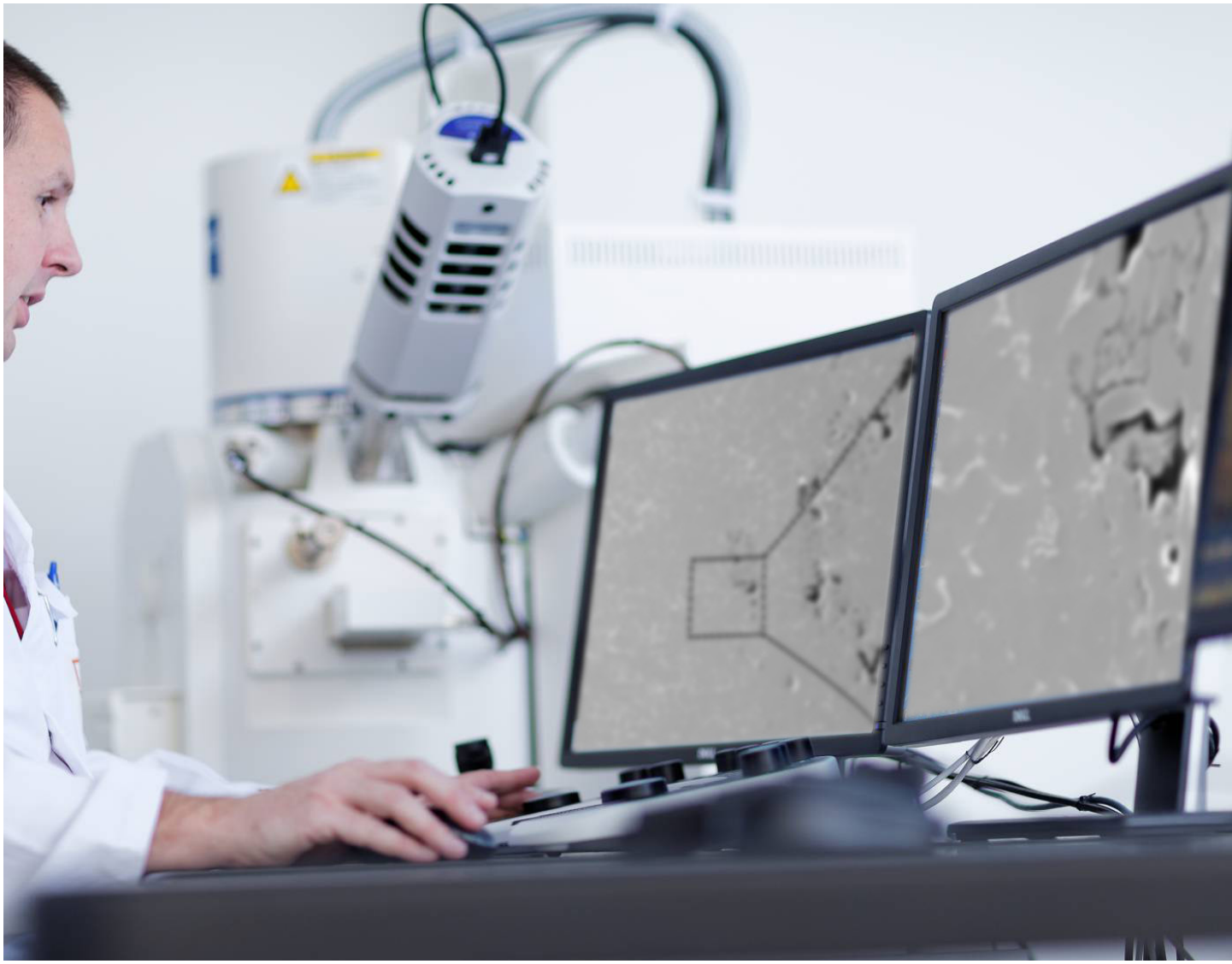

A COMPARISON OF DIFFERENT SAMPLING TECHNIQUES FOR ALUMINIUM METAL CLEANLINESS INVESTIGATIONS

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K-mold sampling is the traditional method of choice for obtaining aluminium samples for Vmet melt cleanliness investigations. The K-mold sampling process involves pouring liquid metal into a mould; however, this can negatively impact melt quality of the sample by introducing shrinkage pores into the cast metal. To overcome this, different sampling methods for Vmet were tested and compared, including K-mold, copper mould and immersion sampling. It was found that by using new sampling methods, such as copper mould and immersion sampling, with different geometries and mould materials, the shrinkage pore volume fraction and density can be lowered, without observing any clear negative impacts on the melt quality. The results suggest that, with the correct sampling technique, Vmet remains a viable method for determining the cleanliness of an aluminium melt in high detail.

INTRODUCTION

Aluminium alloys account for an increasing proportion of global castings production, notably in the casting of high-quality automotive parts (Kordas, Soiński and Skurka 2016). Melt treatment is a critical part of such casting processes in order to ensure appropriate standards of metal quality and cleanliness. This in turn requires a precise understanding of the melt properties.

There are essentially three main approaches to measuring metal cleanliness: chemical analysis, metallographic evaluation, and techniques based on physical principles (Doutre 2016). One example of chemical analysis is Vmet. Developed by Vesuvius, this technique uses a specialized scanning electron microscope (SEM) that is set-up to record the size, morphology, and composition of defects (pores and oxides) using energy-dispersive x-ray spectroscopy (EDXS), together with pre-defined rules and image processing algorithms adjusted for aluminium (Shi 2018).

To achieve representative monitoring of metal cleanliness with Vmet, a reliable melt sampling technique is required. Sampling should highlight any defects for image processing and reflect the effects of melt treatment on the casting quality. In the past, K-mold sampling has been widely used to obtain Vmet samples (Shi 2018). Although the geometry of the K-mold is quite simple, there are some drawbacks in the design that influence the efficacy of the mould to produce clear and repeatable results. For example, the K-mold design introduces shrinkage pores in the cast metal because certain sections solidify more slowly than surrounding areas, and so do not have enough metal flow or feed to fill completely.

To address this problem, this study tested and compared three sampling methods in range of conditions: K-mold sampling, copper mold sampling, and immersion sampling (Figure 1). It is shown that, by using the new sampling methods, the shrinkage pore volume fraction and density can be lowered, without observing any clear negative impact on the melt quality. With the correct sampling technique employed, Vmet therefore remains as a viable technique for measuring the cleanliness of the aluminium melt in high detail.

Figure 1. Three different sampling techniques were compared in this study: a) K-mold, b) copper mould (left: with coating and right: without coating), and c) an immersion sampler.

EXPERIMENTAL PROCEDURES

A number of samples were taken using each of the three techniques across three distinct trials as summarized in Table 1. It should be noted that the copper mould was specifically designed for this study to overcome the challenges observed during K-mold sampling. For this, a simple design was chosen that allows easy sampling and safe removal of the sample upon solidification (see Fig. 1-b). The immersion sampler used in this study was provided by Vesuvius Sensors and Probes division under the article code "HSF-DDX-NK-SCA-HO-12MM-EXP/TB39,5" (see Fig. 1-c).

The first trial aimed to observe the effect of different moulds or sampling techniques (K-mold vs copper mould vs immersion sampler) on the metal cleanliness results in a straightforward manner. Four samples were collected from each mould: one before degassing, and three after degassing, with samples collected at five minute intervals. This understandably caused a temperature increase at the surface of the K-mold and copper mould, which was recorded via a pyrometer so the effect on the Vmet results could be seen. The usual practice when using a K-mold includes the application of a coating on its pattern surface (HeBoCoat 401E Boron Nitrite Spray coating from Henze was used in this study), and this coating was applied on the K-mold for the first trial. Since the use of a copper mould is new for this study, no coating was applied to see if there were any detrimental effects when molten aluminium interacts with the copper surface. The immersion sampler does not require a coating due to its design and use.

The second trial focused specifically on the copper mould to establish whether mould temperature had any effects on the Vmet results. In this trial, three samples were collected using the copper mould and two with the immersion sampler before degassing. After degassing, four samples were collected using only the copper mould. All samples were collected at five minute intervals. As in the previous trial, this caused a temperature increase at the mould surface, which was again recorded with a pyrometer so the effect on the Vmet results could be seen. HeBoCoat 401E Boron Nitrite Spray coating from Henze was used to coat the copper mould, since it was observed during the previous trial that the interaction between the molten aluminium and mould surface had resulted in etching of the mould surface, which caused difficulties when removing the sample.

Trial	Before Degassing			After Degassing			Total Number of Samples			Coating applied		
	K-Mold	Copper Mold	Immersion Sampler	K-Mold	Copper Mold	Immersion Sampler	K-Mold	Copper Mold	Immersion Sampler	K-Mold	Copper Mold	Immersion Sampler
1st	1	1	1	3	3	3	4	4	4	Yes	No	N.A
2nd	N.A	3	2	N.A	4	0	N.A	7	2	No	Yes	N.A
3rd	N.A	12	1	N.A	12	1	N.A	24	2	No	Yes	N.A

Table 1.
Summary of trials that were conducted with different conditions. The number of samples collected is also indicated (N.A. = not applied)

The last trial was conducted to confirm the findings on the first two trials. 12 samples were collected using the copper mould, and one with the immersion sampler, before and after degassing. All samples were collected at five minute intervals. As previously, the temperature increase at the surface of the copper mould was recorded with a pyrometer, and HeBoCoat 401E Boron Nitrite Spray coating from Henze was used to coat the copper mould.

During the trials, several K-mold, copper mould and immersion samples were cast with AlSi7Mg0.3 alloy, before and after rotary degassing, using Foseco's XSR rotor. A Stotek 500 kg aluminium furnace was used at the following temperatures:

+ 1st trial: 745°C
+ 2nd trial: 742°C
+ 3rd trial: 747°C

Degassing with the XSR rotor was done for 600sec at 450rpm, with argon at 10 l/min. The sampling sequence was as follows:

+ Measure the temperature at bottom of the K-mold or copper mould.
+ Pour the sample.
+ Wait 1 min for its solidification.
+ Remove the sample.
+ Wait five minutes and then measure temperature at bottom sample holder again.

After considering all the parameters involved, as explained above, Table 2 summarizes the sample IDs.

All samples were sectioned using a diamond saw with continuous water cooling. Sectioned samples were mounted into 32mm sample stubs with a heat set resin, and mounted samples were polished using a Buehler auto-polisher.

Optical microscopy (Zeiss Axiocam) was used to determine the morphology of the samples upon polishing. SEM (at 20kV) was then performed on the samples to determine metal cleanliness. An area of 10x10 mm2 per sample was investigated using Vmet parameters. In summary, this involves the selection of a feature for EDXS quantitative analysis by the microscope's software, due to its contrast level being under the threshold set. Such a feature then appears darker on the image than the rest of the sample (the aluminium matrix in this case). A spectrum is collected from the centre of that feature for a short amount of time (within seconds). The centre of the feature is chosen as the contrast centre. Further details on Vmet experimental procedures can be found in earlier reports (Shi 2018).

Sample ID						
1st Trial		2nd Trial		3rd Trial		
K-Mold		Copper Mold		Copper Mold		
1-1KM-B		2-1CuM-B		3-1CuM-B	3-13CuM-A	
1-2KM-A		2-2CuM-B		3-2CuM-B	3-14CuM-A	
1-3KM-A		2-3CuM-B		3-3CuM-B	3-15CuM-A	
1-4KM-A		2-4CuM-A		3-4CuM-B	3-16CuM-A	
Copper Mold		2-5CuM-A		3-5CuM-B	3-17CuM-A	
1-1CuM-B		2-6CuM-A		3-6CuM-B	3-18CuM-A	
1-2CuM-A		2-7CuM-A		3-7CuM-B	3-19CuM-A	
1-3CuM-A		Immersion Sampler		3-8CuM-B	3-20CuM-A	
1-4CuM-A		2-1ImS-B		3-9CuM-B	3-21CuM-A	
Immersion Sampler		2-2ImS-B		3-10CuM-B	3-22CuM-A	
1-1ImS-B				3-11CuM-B	3-23CuM-A	
1-2ImS-A				3-12CuM-B	3-24CuM-A	
1-3ImS-A				Immersion Sampler		
1-4ImS-A				3-1ImS-B	3-2ImS-A	

Table 2. Summary of all sample IDs referred in this study. Letters B and A indicate before (B) and after (A) degassing/treatment.

RESULTS & DISCUSSIONS

3.1 FIRST TRIAL

Figure 2 shows SEM images taken of samples from the first trial, before Vmet analysis, for each sampling method. In addition, images of the samples before and after degassing are given for each sampling method (A indicates after and B indicates before degassing).

From the images of the K-mold samples (Figures 2a-d), pores with different sizes can be seen as dark spots against the grey aluminium matrix background. In general, porosity is attributed to either hydrogen or shrinkage in aluminium castings (Sabau 2002): the former usually results in rounded, isolated, and well-distributed pores, whereas the latter results in interconnected or clustered pores of an irregular shape, corresponding to the shape of the interdendritic region. The occurrence of microporosity in aluminium alloys is usually assigned to the combined effects of solidification shrinkage and gas precipitation (Sabau 2002). From a detailed observation of the morphology of the pores hereby detected (Figures 2a-d), it is likely they are a result of shrinkage.

A K-mold is made from cold rolled steel. Additionally, the pattern is designed almost like a thin slab. Upon pouring, solidification takes place rapidly. As noted earlier, the K-mold design damages sampling quality by introducing shrinkage pores into the cast metal. This is due to sections that solidify more slowly than surrounding areas upon pouring. For such sections, there is not enough metal flow – or the chance to feed more into them – for them to be filled completely. As Vmet is based on recognising features that appear with darker contrast in comparison to the background matrix, all these shrinkage pores are recorded and contribute to the results. Since, microporosity in aluminium alloys can be related to both solidification shrinkage and gas precipitation, this makes it difficult to consider K-mold sampling further in such investigations.

When the images of samples obtained using the copper mould or immersion sampler are considered, however, it is clearly seen that there are no such areas of highly-concentrated porosity, as are caused by shrinkage and are present in the K-mold samples.

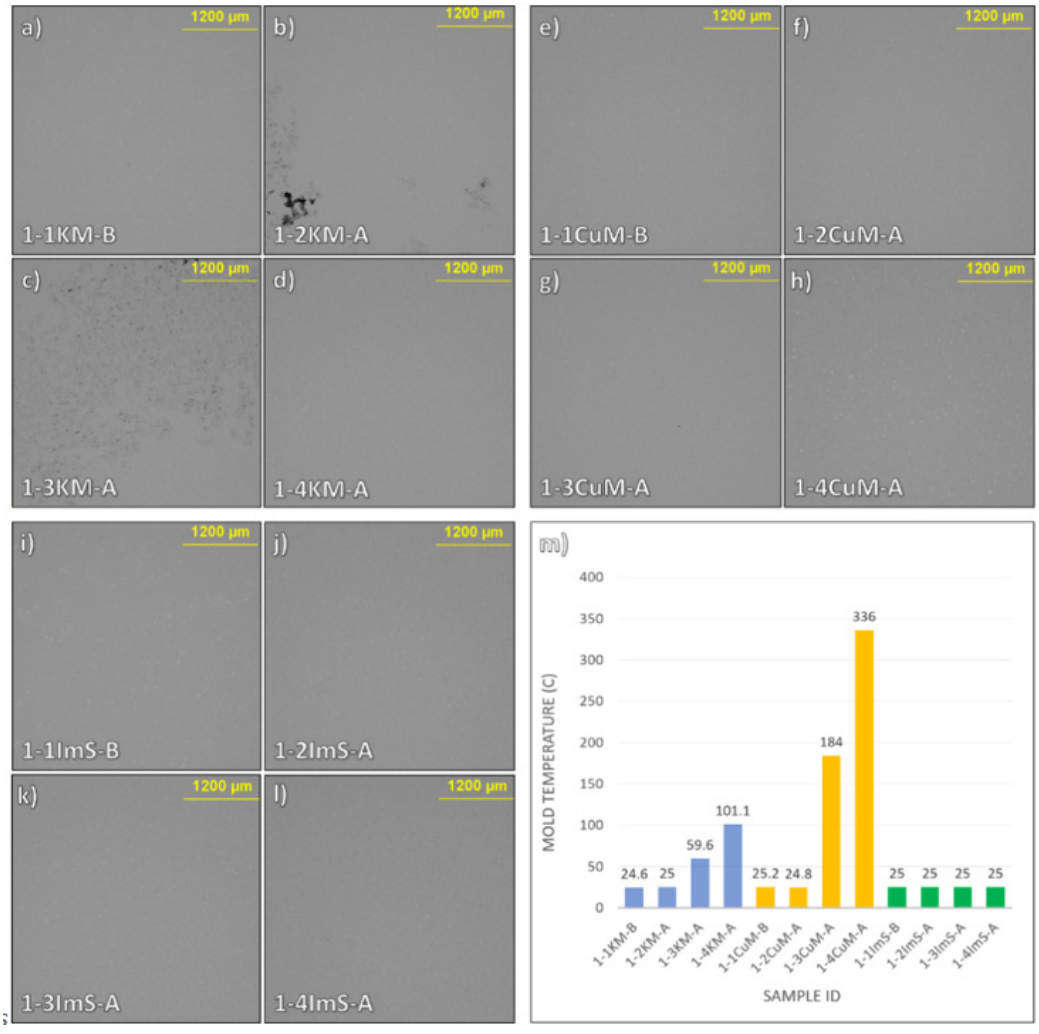

Figure 2.
SEM images taken from samples of the 1st trial, before Vmet analysis, for each sample method: a-d) K-mold, e-h) copper mould, and i-l) immersion sampler. Sample IDs given on images indicate before and after degassing with MTS 1500 (A indicates after and B indicates before degassing). The graph (m) shows sample IDs vs mould temperature (°C) taken by a pyrometer.

To demonstrate the effects of mould design on metal cleanliness, a Vmet analysis was conducted. The results of this experiment are shown in Table 3. One of the key parameters when measuring metal cleanliness via Vmet is to calculate the number of total number of features analysed, which includes all pores, aluminium oxides, and other inclusion types. As can be seen in Table 3, the K-mold samples showed a significantly higher number of total features than the samples taken with both the copper mould and immersion sampler. When the total number of pores, aluminium oxide and other type of inclusions are considered individually, similar conclusions can be drawn.

Inclusions with large dimensions (>15 micrometre) are of particular concern for the mechanical properties of the castings. In the K-mold samples, the number of such inclusions fluctuates significantly before and after degassing. This runs contrary to normal expectations of degassing to lower the number of inclusions and thus result in a cleaner metal. However, such an interpretation cannot be easily made on the basis of K-mold samples. On the other hand, the results from copper mould and immersion sampling indicate that degassing has a clear positive effect on the melt quality. Using any of these two samples methods therefore makes it easier to show that rotary degassing can improve melt cleanliness.

Sample IDs	1-1KM-B	1-2KM-A	1-3KM-A	1-4KM-A	1-1CuM-B	1-2CuM-A	1-3CuM-A	1-4CuM-A	1-1ImS-B	1-2ImS-A	1-3ImS-A	1-4ImS-A
Mold Type	K-Mold				Copper Mold				Immersion Sampler			
Mold Temperature C	24.6	25	59.6	101.1	25.2	24.8	184	336	25	25	25	25
Treatment	untreated Al	degassing	degassing	degassing	untreated Al	degassing	degassing	degassing	untreated Al	degassing	degassing	degassing
Total Features	8793	7754	7583	1800	323	161	19	318	412	248	62	72
Area Analyzed (mm^2)	100.44	100.44	100.44	100.4	100.44	100.44	100.44	100.44	100.44	100.44	100.44	100.44
Total Pores	7649	6535	6662	1121	267	111	17	194	93	169	51	56
0.5 – 15 µm	7646	6480	6662	1121	262	111	17	194	93	169	51	56
15 – 30 µm	3	34	0	0	4	0	0	0	0	0	0	0
30 – 75 µm	0	18	0	0	1	0	0	0	0	0	0	0
> 75 µm	0	3	0	0	0	0	0	0	0	0	0	0
Total Aluminum Oxides	453	392	339	62	14	16	0	15	62	28	2	3
0.5 – 15 µm	453	378	339	62	14	16	0	15	62	28	2	3
15 – 30 µm	0	6	0	0	0	0	0	0	0	0	0	0
30 – 75 µm	0	7	0	0	0	0	0	0	0	0	0	0
> 75 µm	0	1	0	0	0	0	0	0	0	0	0	0
Total Other Inclusions	691	827	582	617	42	34	2	109	257	51	9	13
0.5 – 15 µm	681	816	581	616	42	34	2	108	255	49	6	13
15 – 30 µm	9	11	1	1	0	0	0	1	2	2	3	0
30 – 75 µm	1	0	0	0	0	0	0	0	0	0	0	0
> 75 µm	0	0	0	0	0	0	0	0	0	0	0	0

Table 3. Vmet analysis of samples taken during the 1st trial, before and after treatment with MTS 1500 degassing.

If the designs of K-mold and copper mould are compared to each other, some similarities are observed (Figure 3). For instance, sample volume in both cases is significantly higher than that of immersion samples. In the case of the K-mold, however, a thinner slab-like geometry is prominent; whereas for the copper mould, a thick cup shape is to be seen. It is this geometry that causes the above mentioned issues with inconsistent solidification and shrinkage, which is illustrated well in Figure 3a.

To discover whether shrinkage pores also exist in the copper mould sample due to its bulky geometry, a cross section was cut directly from the middle of the sample. It was further polished by hand until a scratch free surface was obtained for microscopic imaging. Figure 4 was acquired using the tiles and stitching module of the optical microscope, which enables the user to zoom in and observe the microstructure and defects of a sample in detail. As can be seen, the top part of the copper mould sample contains a large area where shrinkage pores are present. For the rest of the sample, round-shaped pores are seen, which indicates pores with a gas origin. Since the Vmet section is cut from the bottom of the sample, this makes the area that is to be characterized through Vmet falls far away from the region where a high density of shrinkage pores is seen.

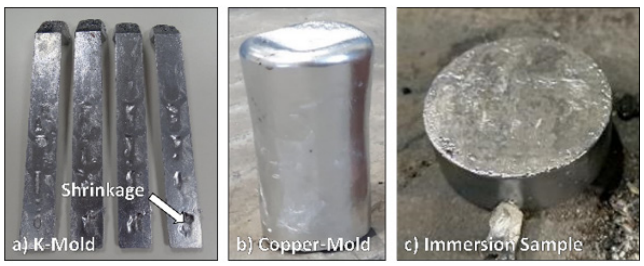

Figure 3. Samples taken using a) a K-mold (back side), b) a copper mould, and c) an immersion sampler. Note the shrinkage prominent defects in the K-mold sample.

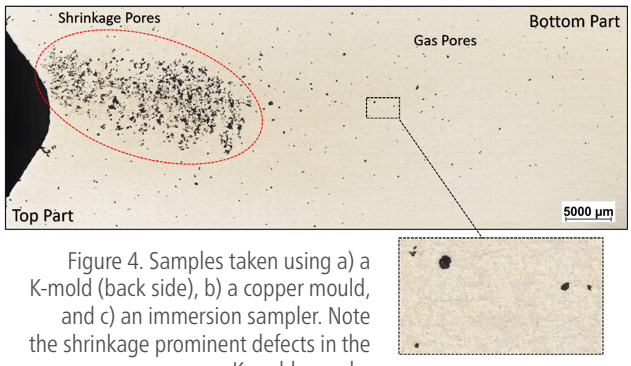

Figure 4. Samples taken using a) a K-mold (back side), b) a copper mould, and c) an immersion sampler. Note the shrinkage prominent defects in the K-mold sample.

3.2 SECOND TRIAL

The details of the second trial are given in Tables 1 and 2, and Vmet results can be seen in Table 4.

This trial aimed to investigate the effect of an increase in copper mould temperature during sampling. As can be seen, taking consecutive samples increased the copper mould temperature from room temperature to a value slightly higher than 300 °C, at which it stabilised. During the trial, it was observed that such temperatures are usually reached with a sample interval time of five minutes; whereas decreasing the sample interval time increased the mould temperature to a maximum of 450 °C. The trials therefore showed that it is not advisable to decrease sample interval times to less than five minutes, as this results in overheating of the mould and negatively impacts the mould coating, causing the aluminium sample to stick onto the walls of the mould.

When Vmet results of this trial are considered, the first collected sample has a high number of pores (751 in total); this number decreases and mostly stabilises when a temperature above 150 °C is reached. Although this number is considered high, it is significantly lower than those obtained with K-mold samples (see Table 3 for comparison). When sampling continued after degassing, there is a further decrease in the number of pores, as expected. A decrease in the number of aluminium oxide and other types of inclusion was also observed after degassing. Although the melt was not treated with a flux – and keeping in mind that the initial status of the melt was already rather clean, with almost no inclusions larger than 30 micrometres – the positive impact of degassing on melt quality is still clearly seen.

If the results from the copper mould and immersion sampler are compared, the immersion sampler shows a clear advantage over the copper mould, as both immersion samples give similar results, with very low number of pores, aluminium oxide, and other type of inclusions. This is probably due to the negative impact of mould geometry on the sample. Although a much better candidate than the K-mold, there are negative impacts of a bulky sampling mould on sample quality due to the introduction of shrinkage pores into the cast metal.

Sample IDs	2-1CuM-B	2-2CuM-B	2-3CuM-B	2-4CuM-A	2-5CuM-A	2-6CuM-A	2-7CuM-A	2-1ImS-B	2-2ImS-B
Mold Type	Copper Mold							Immersion Sampler	
Mold Temperature C	28	167	201	288	314	315	317	28	28
Treatment	untreated Al	untreated Al	untreated Al	degassing	degassing	degassing	degassing	untreated Al	untreated Al
Total Features	854	346	620	363	326	278	232	193	125
Area Analyzed (mm^2)	100	100	100	100	100	100	100	100	100
Total Pores	**751**	**313**	**348**	**325**	**285**	**258**	**192**	**154**	**111**
0.5 – 15 µm	749	313	344	323	274	248	189	153	108
15 – 30 µm	2	0	4	0	9	6	2	1	2
30 – 75 µm	0	0	0	2	2	4	1	0	1
> 75 µm	0	0	0	0	0	0	0	0	0
Total Aluminum Oxides	**41**	**14**	**37**	**18**	**24**	**13**	**22**	**24**	**8**
0.5 – 15 µm	41	14	37	18	22	12	22	24	8
15 – 30 µm	0	0	0	0	2	1	0	0	0
30 – 75 µm	0	0	0	0	0	0	0	0	0
> 75 µm	0	0	0	0	0	0	0	0	0
Total Other Inclusions	**62**	**19**	**235**	**20**	**17**	**7**	**18**	**15**	**6**
0.5 – 15 µm	58	19	232	20	17	3	15	12	4
15 – 30 µm	3	0	3	0	0	4	3	3	1
30 – 75 µm	1	0	0	0	0	0	0	0	1
> 75 µm	0	0	0	0	0	0	0	0	0

Table 4. Vmet analysis from the second trial, before and after degassing of the melt with MTS 1500.

The third trial aimed to compare the copper mould vs. immersion sampler. In particular, the goal was to confirm the effect of copper mould temperature on the observed total number of pores. Vmet results for this trial can be observed in Table 5. As can be seen again, taking consecutive samples increased the copper mould temperature from around room temperature to slightly higher than 300°C. One of the main common findings in each of these trials is that sampling tends to increase the copper mould temperature to a value between 300-350°C, before it stabilizes within that temperature range.

If the number of total pores is observed, it is clear that the collected samples before degassing have a high number of pores (171 min. and 731 max.), while this number decreases after the degassing to a minimum of 110 and maximum of 652. When the total number of aluminium oxide and other inclusions are observed, the positive impact of degassing on these values is much more obvious. It is observed that, in most of the cases, both numbers are significantly lower after degassing than before treatment.

In addition, when the results from the copper mould and immersion sampler are compared, the immersion sampler gives similar results across samples, with very low number of pores, aluminium oxide and other type of inclusions. The samples taken with immersion sampler show the lowest number of pores (untreated or degassed at room temperature) when compared to copper mould. This is due to the fact that the immersion samples are much smaller than the copper mould samples and so solidify more rapidly. Because shrinkage is caused by inconsistent solidification through the mould, the rapid solidification of a small volume of metal explains the lack of shrinkage pores within.

Sample IDs	3-1CuM-B	3-2CuM-B	3-3CuM-B	3-4CuM-B	3-5CuM-B	3-6CuM-B	3-7CuM-B	3-8CuM-B	3-9CuM-B	3-10CuM-B	3-11CuM-B	3-12CuM-B	3-1ImS-B
Mold Type	Copper Mold												Immersion Sampler
Mold Temperature C	21.5	246	266	275	293	299	308	305	310	309	316	319	21
Treatment	untreated Al												untreated Al
Total Features	804	752	243	359	248	288	186	274	223	215	627	214	171
Area Analyzed (mm^2)	100	100	100	100	100	100	100	100	100	100	100	100	100
Total Pores	**731**	**668**	**219**	**294**	**223**	**262**	**171**	**237**	**189**	**190**	**575**	**194**	**148**
0.5 – 15 µm	729	659	219	292	222	260	168	236	188	190	575	194	147
15 – 30 µm	2	8	0	2	1	2	2	1	1	0	0	0	0
30 – 75 µm	0	1	0	0	0	0	1	0	0	0	0	0	1
> 75 µm	0	0	0	0	0	0	0	0	0	0	0	0	0
Total Aluminum Oxides	**66**	**68**	**23**	**34**	**22**	**25**	**11**	**19**	**26**	**23**	**47**	**19**	**9**
0.5 – 15 µm	66	67	23	34	22	24	11	19	26	23	47	19	9
15 – 30 µm	0	1	0	0	0	1	0	0	0	0	0	0	0
30 – 75 µm	0	0	0	0	0	0	0	0	0	0	0	0	0
> 75 µm	0	0	0	0	0	0	0	0	0	0	0	0	0
Total Other Inclusions	**7**	**16**	**1**	**31**	**3**	**1**	**4**	**18**	**8**	**2**	**5**	**1**	**14**
0.5 – 15 µm	7	16	1	29	3	1	4	15	6	0	4	1	10
15 – 30 µm	0	0	0	2	0	0	0	3	2	2	1	0	3
30 – 75 µm	0	0	0	0	0	0	0	0	0	0	0	0	1
> 75 µm	0	0	0	0	0	0	0	0	0	0	0	0	0

Sample IDs	3-13CuM-A	3-14CuM-A	3-15CuM-A	3-16CuM-A	3-17CuM-A	3-18CuM-A	3-19CuM-A	3-20CuM-A	3-21CuM-A	3-22CuM-A	3-23CuM-A	3-24CuM-A	3-2ImS-A
Mold Type	Copper Mold												Immersion Sampler
Mold Temperature C	27.5	206	300	312	325	306	322	326	320	329	319	320	21
Treatment	degassing												degassing
Total Features	475	180	204	115	214	207	746	134	148	150	224	144	163
Area Analyzed (mm^2)	100	100	100	100	100	100	100	100	100	100	100	100	100
Total Pores	**336**	**167**	**182**	**110**	**181**	**186**	**652**	**118**	**135**	**139**	**206**	**132**	**149**
0.5 – 15 µm	336	167	181	110	178	186	649	118	134	139	204	131	149
15 – 30 µm	0	0	1	0	3	0	3	0	1	0	2	1	0
30 – 75 µm	0	0	0	0	0	0	0	0	0	0	0	0	0
> 75 µm	0	0	0	0	0	0	0	0	0	0	0	0	0
Total Aluminum Oxides	**42**	**8**	**20**	**5**	**24**	**15**	**73**	**12**	**11**	**11**	**12**	**8**	**9**
0.5 – 15 µm	42	8	19	5	24	15	69	12	11	11	12	8	8
15 – 30 µm	0	0	1	0	0	0	4	0	0	0	0	0	0
30 – 75 µm	0	0	0	0	0	0	0	0	0	0	0	0	1
> 75 µm	0	0	0	0	0	0	0	0	0	0	0	0	0
Total Other Inclusions	**97**	**5**	**2**	**0**	**9**	**6**	**21**	**4**	**2**	**0**	**6**	**4**	**5**
0.5 – 15 µm	94	5	0	0	4	6	21	2	2	0	5	4	4
15 – 30 µm	3	0	2	0	0	0	0	2	0	0	1	0	0
30 – 75 µm	0	0	0	0	5	0	0	0	0	0	0	0	1
> 75 µm	0	0	0	0	0	0	0	0	0	0	0	0	0

Table 5. Vmet analysis from the third trial before and after degassing with MTS 1500

CONCLUSIONS

Metal cleanliness is an important parameter that plays a vital role in determining the final quality of aluminium castings. In this study, it is demonstrated that metal cleanliness can be determined with high precision (i.e., at the micrometre scale) using the Vmet technique. Vmet enables one to optimise melt quality by adjusting melt treatment procedures, which in return will ensure castings with good physical properties.

For Vmet to reflect the precise condition of the melt, different sampling methods for Vmet were investigated. Three sets of trials, before and after rotary degassing, with different sampling techniques (K-mold, copper mould and immersion sampling), were undertaken. Based on the results of these trials, it can be concluded that copper mould and immersion sampling techniques are superior to the traditional K-mold sampling method, both in terms of replicability and the elimination of shrinkage in the final samples. Specifically, the results demonstrate immersion sampling to be slightly more reliable than the copper mould sampling; however, both were shown to be trustworthy techniques for investigating samples through Vmet.

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