Effect of the sampling method on the results of melt quality assessment of aluminum alloys with computed tomography

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Abstract. Liquid metal quality is one of the key factors which determine the soundness of cast parts. This is the reason why the assessment of the melt quality is of critical importance prior to casting. The most common and most deleterious defects of liquid aluminum alloys are the so-called bifilms whose quantity can be characterized by the computed tomographic porosity analysis of reduced pressure test pieces. During the sampling of these specimens, however, generally pouring is involved, which is known to damage melt quality and introduce bifilms into the liquid metal. For this reason, a new sampling method was tested and compared with conventional pouring. It was found, that by using the new sampling method, the pore volume fraction of the test pieces can be lowered, however, regarding the pore number density, no clear difference could be observed. The results also suggest that fluxes have a remarkable effect on the structure of double oxide films, and in this way, on the susceptibility to pore formation.

1. Introduction
Regardless of the type of the casting process used for making cast components, in order to ensure decent mechanical properties and avoid casting defects like porosity, inclusions, and leakage, ensuring proper melt quality is essential [1-4]. The most frequently occurring, and in fact, the most harmful inclusions of aluminum casting alloys are the double oxide films (also known as bifilms), which have a remarkable effect on the microstructure and thus, the properties of cast metals [5-7]. Bifilms can easily form during melt handling and processing, as the smallest amount of disturbance of the surface oxide layer of the melt can cause its incorporation into the bulk liquid [8]. During this so-called entrainment process, the surface oxide layer folds over itself, and a doubled-over film is created, which has a central unbounded interface, thus bifilms constitute cracks in the microstructure of the cast metals [9]. During the solidification of the alloy, bifilms can initiate pore formation, as the hydrogen precipitation between their layers, and the local pressure drop in the interdendritic regions (caused by the solidification shrinkage) leads to the unfurling and inflation of the double oxide films [5, 10, 11]. This phenomenon is schematically illustrated in Figure 1. In fact, it was proposed by Campbell [5], that in the case of aluminum alloys, microporosity can only form if bifilms are present in the liquid metal. This theory is supported by extensive theoretical calculations [11-15] and experimental findings [16-23].

The quantification of the bifilm content of liquid alloys is particularly difficult, as double oxide films are usually small-sized and the thickness of their layers is often only a few nanometers [5, 6]. However, the ability of bifilms to initiate pore formation can be utilized for the characterization of bifilm quantity. For this purpose, the so-called reduced pressure test (RPT) can be a valuable tool, as it was revealed by Fox and Campbell [24, 25].
Figure 1. Bifilm initiated pore formation during the solidification of the alloy

Under reduced pressure, pore formation is enhanced during the solidification of the alloy, as the solubility of hydrogen is lowered and the entrapped air between the layers of the double oxide films is being expanded due to the lower pressure [10, 26]. In this way, small-sized bifilms in the liquid alloy can be also detected, as they expand into relatively large-sized pores during the solidification of the metal. The detection of the pores inside the RPT samples can be realized with the quantitative image analysis of the cross-section of the specimens or with non-destructive techniques such as computed tomography (CT) [23, 27, 28].

It was revealed by the authors [23], that volumetric pore number density, which can be determined with the CT-aided porosity analysis of RPT samples, can be used for the characterization of bifilm quantity. However, during the casting of the samples, the selection of pouring height could have a significant effect on the bifilm content of the specimens [17]. According to Campbell [5, 29], a fall of only 12.7 mm is enough to reach the critical melt velocity of aluminum alloys, which is 0.5 m/s. At velocities higher than this, surface turbulence occurs, and the entrainment of the surface oxide layer is unavoidable. On the other hand, the height of typical cups used for the casting of RPT samples is about 40 mm. This means that bifilm formation should be expected during the sampling process, which can significantly alter the results of the melt quality assessment. For this reason, the aim of this study was to investigate the effect of the sampling method on the results of CT-analysis of RPT specimens and to develop a casting technique, which minimizes the chance of bifilm formation during the sampling process.

2. Experimental

To perform the experiments, 2968 g of an AlSi7MgCu alloy was melted in a clay-graphite crucible in an electric resistance-heated furnace. The exact chemical composition of the alloy was evaluated with optical emission spectroscopic analysis. The results are shown in Table 1.

| Table 1. Chemical composition of the alloy used for the experiments (wt. %) |
|-----------------|-------|-----|------|------|-----|-----|
| Si              | 6.760 | Fe   | 0.105 | Cu   | 0.522 | Mn  | 0.064 |
|                 |       |      |       |      |      |     |       |
| Mg              | 0.390 | Ti   | 0.124 | Sr   | 0.014 |

For the casting of RPT specimens, two sampling methods were compared:
- in the first case, conventional gravity casting conditions were used, in which the melt was poured into coated steel cups with the aid of a ladle (Figure 2a). In order to minimize the falling height, the pouring was realized directly from the top of the steel cups.
- in the second case, the steel cups were immersed into the liquid alloy and were immediately taken into the vacuum chamber of the RPT apparatus (Figure 2b). In this way, no pouring was necessary for the sampling of the test pieces.
Before the casting of the RPT specimens, the steel cups used for the sampling process were preheated to 200 °C. The samples stayed in the vacuum chamber of the RPT machine at 80 mbar pressure for 6 minutes.

![Figure 2. The sampling of reduced pressure test specimens by (a) pouring with a ladle and (b) immersing the cups into the melt](image)

Following the melting of the charge material, 3 RPT samples were taken with both sampling methods. The temperature of the melt was 750 ± 15 °C, which was checked in each sampling step with a K-type thermocouple. After the casting of the samples, 3 g of a commercially available granular cleaning flux was manually stirred into the alloy. The chemical composition of the flux was previously characterized by X-ray powder diffraction measurements. The identified components of the flux are KCl, NaCl, CaF₂, and Na₂SO₄. Following the application of the flux, and a 10 minutes long resting period, the dross layer was removed from the top of the melt, and another series of RPT samples were taken.

Using the Archimedes principle the densities of the RPT specimens were evaluated. For the characterization of the porosity inside the RPT samples, computed tomography (CT) aided porosity analysis was conducted on the test pieces. The CT scanning of the specimens was executed with a GE Seifert X-Cube Compact 225kV apparatus with a tube current of 0.8 mA and an acceleration voltage of 135 kV. 900 projections were acquired during each rotation of the test pieces. For the image reconstruction and processing, VGSTUDIO MAX 3.2 software was used. The segmentation of the voids inside the samples was performed with the VGDefX algorithm, which is included in the porosity analysis module of the software. Based on the local contrast in the grey level of the voxels, a probability value was calculated by the software for each pore. Pores with a volume smaller than 0.05 mm³ and those which had a probability value lower than 0.9 were excluded. Following the CT analysis, the RPT specimens were cut in half and were ground. For this purpose, SiC grinding papers were used with a grit size up to 1200. The inner surface of the pores found in the cross-section of the RPT samples was studied with a Zeiss EVO MA 10 scanning electron microscope (SEM) equipped with an energy-dispersive X-ray spectroscopic (EDS) detector.
3. Results and discussion

The results of the density evaluation of RPT samples are shown in Figure 3. As can be seen, the pieces which were taken by the immersion of the sampling cup into the melt had a slightly higher density. This can be attributed to the quantity of air being entrapped into the melt during the sampling process. As Figure 3 shows, the liquid metal has a falling height of approximately 4 cm, significantly more air can be entrained into the metal than in the case of immersion of the sampling cup. This suggests that the melt flow is more laminar when no pouring is involved during the sampling process. Based on the results, it is also evident, that due to the fluxing treatment, the average densities of the specimens are remarkably higher, and the deviation of the density values are notably lower. During the experiments, no degassing was conducted, and no bubble formation could be observed during the fluxing treatment which indicates that the flux did not have any degassing effect on the melt. Therefore, the reason for the increment of the density values is either the effect of the flux on the bifilm quantity or on the ability of the double oxide films to grow into pores during the solidification of the alloy.

Figure 4. shows the pore number density and volume fraction results evaluated by CT-aided porosity analysis of reduced pressure test pieces. Regarding pore number density, no clear relationship could be observed between the method of sampling and the results, as after melting, the specimens taken by immersion had higher pore number density, while after the fluxing treatment, the values of the poured samples were significantly higher. According to the results of Tiryakioğlu et al. [17], the pore number density of the RPT samples increases with the falling height of the liquid metal. This is reasonable because increased melt velocity causes more turbulent flow, which in turn, results in the entrainment of more oxide films into the melt. However, this phenomenon could not be evinced during the experiments of this study. After fluxing, in the case of poured samples, the pore number density values had substantially increased, which indicates higher bifilm content. In practice, fluxing treatments should reduce the number of double oxide films present in the melt, however, this could not be achieved in this case. This can be attributed to the method of flux addition, as manual stirring was applied, which could lead to the entrainment of numerous oxide films into the melt. On the other hand, the results of the pieces taken by immersion were nearly the same as after melting.
Based on the results shown in Figure 4b, the average pore volume fraction was remarkably higher when the samples were taken by pouring. This is in agreement with the density results and can be attributed to the air being entrapped into the liquid metal during the pouring process. After fluxing, significantly lower pore volume fraction values were determined, and as the average pore number density had not been decreased, it is evident, that the fluxing treatment resulted in smaller sized pores. This is clearly visible on the three-dimensional reconstructed CT images of the pores found in the samples (Figure 5).

![Figure 5](image)

**Figure 5.** Reconstructed CT images of the pores found in RPT samples: after melting: (a),(b); after fluxing: (c),(d); taken by pouring: (a),(c); and by the immersion of the sampling cup: (b),(d)

During the investigations of the pores found in the RPT samples with SEM, in the case of specimens taken after melting, thin, wrinkled films were found which were covering the inner surface of the pores (as it is demonstrated in Figure 6). These films could be found in each pore, which supports the pore formation mechanism presented in Figure 1. In some cases, they could be easily detected (Figure 6a), while in other cases, only vanishingly thin creases indicated their presence (Figure 6b). However, these films were so thin, that no significant amount of oxygen could be detected during their EDS analysis.

On the other hand, after fluxing, the morphology and the size, as well as the chemical composition of the surface of the pores had changed. Figure 7 shows the SEM image of the microstructure of a sample cast following the fluxing treatment. As can be seen, the pores mostly have interdendritic or tear-like morphology, and darker grey regions could be found linking them together. As the contrast of the BSD SEM images is dependent on the atomic number of the sample, these darker areas have a different chemical composition as the alloy.
Figure 6. SEM images of the inner surface of pores found in RPT samples with vanishingly thin oxide films covering the \(\alpha\)-Al dendritic and eutectic phases.

Figure 7. SEM image of pores found in the RPT samples after the fluxing treatment.

Figure 8. The results of the EDS analysis of a thick oxide inclusion observed in an RPT specimen after fluxing.
However, the degree of thickening of the oxide films cannot be explained by only the holding of the melt, as the formation of thick oxide inclusions is only expected after a resting period of several hours [5]. On the other hand, the application of fluxes can remarkably change the rate of oxidation of the alloy. The applied flux contains Na₂SO₄, which is known as an oxidizing compound, which reacts with molten aluminum in a strongly exothermic reaction [31-33]. Based on this, the flux could cause accelerated oxidation of the alloy, which resulted in the formation of thick oxide films whose layers bond together. These oxide films could not open and inflate into pores during the solidification of the alloy, which resulted in lower pore volume fraction and smaller pore size.

4. Conclusion
In this study, two different sampling techniques were compared for the melt quality evaluation of an aluminum casting alloy by the computed tomographic porosity analysis of reduced pressure test pieces. The effect of fluxing treatment on the tendency to pore formation was also investigated. It was found that the type of sampling has a significant effect on the volumetric porosity of the specimens, as by pouring of the liquid metal, a considerable amount of air can be entrapped into the melt, which results in higher pore volume fraction. Fluxing has a notable effect on the structure of oxide films, and in this way on the tendency to pore formation, as the layers of the bifilm defects can bond together due to the application of fluxes.

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