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MEASUREMENT OF NON-METALLIC INCLUSIONS IN THE SIZE RANGE OF 10-20µm BY LiMCA

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Abstract

Regular monitoring of the melt quality is employed in Aluminum cast house production where optimized processes and high quality are required. A well established method for the quantitative measurement of non-metallic inclusions is the LiMCA system. In this method, inclusions flowing together with the liquid Aluminium through a 300µm orifice of a submerged glass tube are detected due to their high electrical resistance. The LiMCA system can identify the size, typically in the range between 20µm and 300µm and number of particles in the liquid Aluminium.
Increasing product quality standards have resulted in demands to monitor particles even smaller than 20µm. This paper reports results of a parametric study to assess the capability of the LiMCA system to monitor non-metallic inclusions in the particle size range of 10-20µm through changing the orifice hole size and by adjustment of the basic measurement parameters.

Introduction

Non-metallic inclusions are one of the main quality aspects in the production process of rolling ingots and several measurement techniques like K-Mold, PoDFA or MetalVision have been developed to monitor the concentration of particles in the melt and in the product [1].
Well established in the Aluminium Industry for rolling ingots is the LiMCA system, operating on the principle of the resistive pulse / electric sensing zone technique (ESZ). This system is capable of monitoring of the concentration and size of particles in the range of 20-300µm in the absence of microbubbles in the melt. The Hydro Aluminium research centre in Bonn, Germany has operated LiMCA II for many years for optimization of the cast house processes and development of filtration technology [2],[3],[4].
Today's excellent production standard with very low particle concentration and the increasing demand for high quality products like 6µm foil for liquid packaging or lithographic sheet brought fine particles, which currently cannot be monitored online into focus.
The current paper reports on a development program performed at Hydro Aluminium to extend the measurement capabilities of LiMCA towards finer particles in the size range of 10-20µm by reducing of the orifice diameter in the glass tube and adjustment of operational parameters.

LiMCA's resistive pulse / electric sensing zone (ESZ) principle

The LiMCA technique is based on the principle of the resistive pulse / electric sensing zone technique (ESZ) [5]. A non-conducting glass tube with a 300µm orifice and pairs of tungsten electrodes, one inside the tube and one outside, are submerged in the molten aluminium during measurement, see Figure 1.

One of the electrode pairs supplies a constant current. The electric circuit is completed by the molten metal and the voltage measured is determined by the metal inside and around the probe orifice in the ESZ. During the measurement the metal level inside the glass tube is periodically exchanged by an applied over- and underpressure. A particle of different conductivity entering the glass tube during the transient filling process causes a voltage peak, which is used to identify the inclusion. The primary voltage signal is pre-amplified in two steps and background noise is reduced by a band-pass-filter before the signal is analyzed in a multi-channel analyzer for particle concentration and size.
The inclusion identification is here disturbed in the presence of microbubbles due to their non-conducting behaviour. Much effort was spent to separate non-metallic inclusions and gas bubbles [4], but no final solution is available to measure downstream degasser units. Anyhow, this topic is out of scope for the current evaluation and will not be considered.

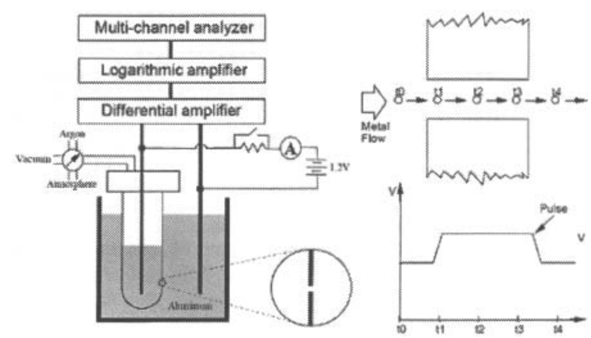

Figure 1: LiMCA II operation schematic and resistive pulse principle of particle measurement [5]

The particle size detection range of LiMCA is defined by the orifice size of 300µm and by the ratio of the voltage peak and the noise reduction filter. Smaller voltage peaks are erased by this filtering process. The magnitude of the primary voltage peak ΔV in the ESZ is related to the particle diameter [6], which allows the particle size discretisation and can be described by:

$$\Delta V = \frac{4 \cdot \rho \cdot I \cdot d^3}{\pi \cdot D^4} \tag{1}$$

with ρ = resistivity of metal (Al = 25 x 10^{-8} Ωm), I = current (typically 60A), d = diameter of the equivalent spherical particle, D= diameter of the cylindrical sensing zone (and the probe orifice diameter)

Taking the description of the primary signal in (1) and the signal processing into account, the following operational parameters might be adjusted to reduce the lower particle detection limit:

- The **cut-off frequency** of the band-pass filter might be increased to pass smaller voltage peaks
- The applied **current I** has a linear relation to the voltage peak and therefore a cubic root relation to the particle size. Doubling the current theoretically reduces the lower detection limit by ~20%.
- The diameter of the ESZ and therefore the **glass tube orifice size** has a relation of power 4 to the voltage peak and a relation of power 4/3 to the particle size. Halving the orifice opening could theoretically reduce the lower detection limit by ~60%.

Procedure

The investigation of the influence of the operational parameter on the LiMCA particle monitoring was performed in laboratory tests using one LiMCA unit and in on-site tests using two LiMCA units in the following three steps:

1. <u>Laboratory tests using standard 300µm LiMCA probes</u>
   In the first step the particle size detection limit of a LiMCA with 300µm probe was analyzed for standard settings of the current and cut-off frequency and for settings within the possible range provided by the LiMCA software (Table I).

2. <u>Laboratory tests using 200µm LiMCA probes</u>
   In the second step the probe orifice size was reduced to 200µm and the settings of current and cut-off frequency were varied to move the particle detection limit as much as possible towards finer particles but ensuring stable LiMCA operation.

3. <u>On-site tests with 2 LiMCA units</u>
   In the final step the measurement accuracy of the identified LiMCA setup for a 200µm orifice probe was evaluated by direct comparison to standard LiMCA monitoring in side-by-side tests.

Table I:

Parameter	Standard	Variations
Cut-off frequency [kHz]	10.67	12.80, 16.00, 21.33
Current [A]	60	10, 25, 40, 75
Orifice diameter [µm]	300 ± 10	200 ± 10

The laboratory tests were performed in a 60 kg resistance heated crucible furnace filled with high purity aluminium melt (99.99%) to avoid any alloy influence on the measurement data. In selected tests solid pieces of DURALCAN W6S.14A composite material were added to the melt to evaluate the LiMCA particle size identification under controlled conditions. The composite material comprises an aluminium matrix with 14% ± 2% of spherical Al_2O_3 particles in the size range of 10-50µm with an average size of 17µm.
The on-site tests were performed in a Hydro Aluminium cast house for rolling ingots during production charges of 1000 series alloys. The two LiMCA units were placed closed to each other at the launder near the furnace exit to analyze almost identical particle levels with both units.

Results and Discussion

The results of the two laboratory and the on-site evaluation will be reported and discussed in individual chapters reflecting the above mentioned evaluation steps "Laboratory tests using standard 300µm LiMCA probes", "Laboratory tests using 200µm probes" and "On-site tests with 2 LiMCA units".

<u>Laboratory tests using standard 300µm LiMCA probes</u>

Firstly, the LiMCA readings using standard operational parameters of current and cut-off frequency and standard 300µm probe were analyzed for the laboratory test conditions. Figure 2 shows a typical LiMCA result for tests with low particle concentrations. The top figure shows the standard LiMCA N15 run-chart, the measured particle concentration in the size range of 15 to 300µm for the 1 hour measurement period. An average N15 concentration of 1610 particles per kg melt (1.61 k/kg) and an average N20 value of 0.31 k/kg was recorded in this test.

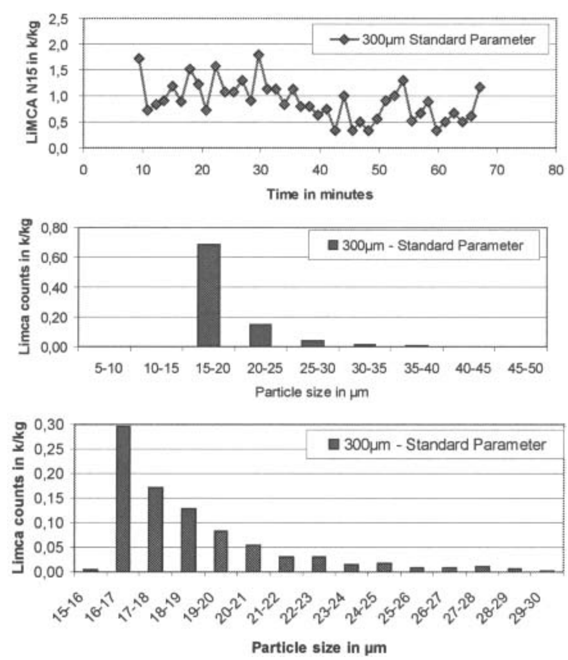

Figure 2: LiMCA results achieved using standard operating parameters and LiMCA probe with 300µm orifice in melt with low particle concentration; top) N15 run-chart; centre: particle size distribution in 5µm bins; bottom) particle size distribution in 1µm bins

The centre Figure shows the average particle size distribution within the measurement period subdivided in 5µm bins. Due to the set-up the system did not report particles smaller than 15µm. It shows the typical decreasing concentration with increasing particle size. The bottom figure shows the particle size distribution within the size range of 15 to 30µm in 1µm discrimination. For values higher than 17µm the steadily decreasing number of particles with growing size was also evident here, however, LiMCA reported almost no particle in the size range of 15-16µm. From this reading it seemed, that the smallest measurable particle using the standard parameter was around 16µm. This is caused by the influence of the tube orifice size as indicated by equation (1), which varied between 290µm and 310µm. LiMCA reported therefore only data for particles larger than 17µm, reporting of LiMCA data in 5µm bins might therefore be misleading.

Starting from the standard parameter set of LiMCA the operational parameters "electrical current" and "cut-off frequency" were varied within their limits to extend the measurement capabilities towards finer particles.

The simplest approach to influence the detection limit is lowering the cutting level of the band-pass filter. This can be done by increasing the pre-amplifier gain of the primary signal or increasing the cut-off frequency of the filter. Here, the second approach was used and figure 3 shows the influence of different cut-off frequencies on the particle size detection. As can be seen no influence on the particle size detection limit was monitored, but at higher cut-off frequencies the extremely high 15µm values show that background noise was measured within that area. The approach was therefore considered not suitable to enhance the LiMCA capabilities.

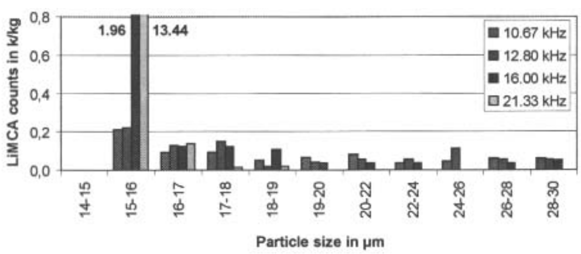

Figure 3: Variation of the cut-off frequency during a laboratory LiMCA test with 300µm orifice tube

The other option to influence the particle detection limit of LiMCA is changing the applied current during the measurement period. According to equation (1) the current has a linear correlation to the voltage peak and a cubic root correlation to particle size, so the influence on the particle size detection might be limited. However, doubling the applied current would decrease the limit by ~26%.

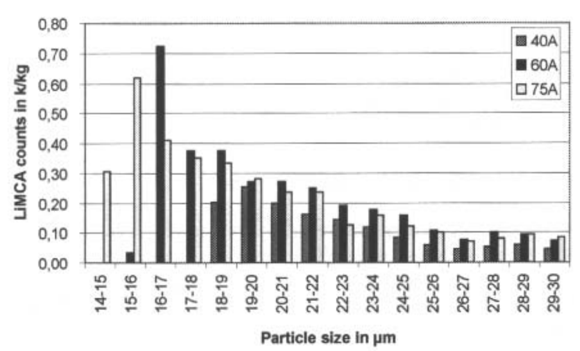

Figure 4: Variation of the electrical current during a laboratory LiMCA test with 300µm orifice tube

Figure 4 shows the particle size distribution of a laboratory test performed with varied applied currents; the electrical current was decreased from standard value of 60A to 40A and then increased to 75A. This process was repeated for several measurements. The particle size distribution showed in all cases the typical distribution mentioned above. Decreasing the current to 40A increased the detection limit to 19µm, while the detection limit of 15µm was reached using 75A. The results agreed with the theoretical correlation shown in equation (1) and consequently it might be concluded that "real" N15 values can be achieved using a current of 75A and tubes with an orifice size not larger than 300µm.

Laboratory tests using 200µm probes

Changing the orifice diameter of the LiMCA tube has a significant effect on the voltage peak of a particle travelling through the ESZ. The diameter of the ESZ is correlated to the amplitude of the voltage peak by exponent 4 and therefore correlated to the particle diameter by exponent (-4/3) as shown in equation (1). Changing the orifice diameter from 300µm to 200µm could change the particle detection limit by ~41% from about 16µm with 300µm probes to less than 10µm using an orifice of 200µm. The measurement of a real LiMCA N10 value could then theoretically be possible.

Figure 5 shows the particle size distribution in the size range of 8-30µm of two laboratory tests performed with LiMCA probes of 199µm and 210µm orifice size. LiMCA reported in both tests the typical particle size distribution and a N15 particle concentration of 2.0 k/kg (N20: 0.9 k/kg). The measurement using the 199µm orifice size close to the nominal size of 200µm shows as predicted particles down to 9 µm. The N10 value would be here 5.32 k/kg. The detection limit for the measurement using the 210µm tube seems to be within the 10-11 µm size range and a reported N10 value would include a small error because particles close to 10µm would not be measured by the system.

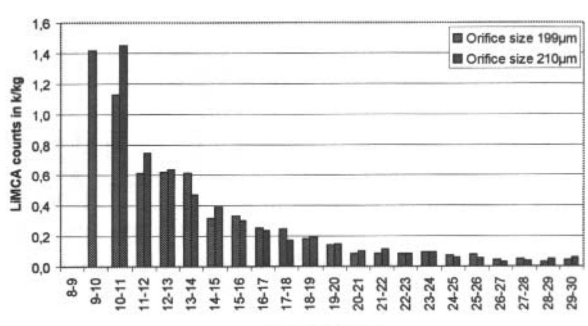

Figure 5. Particle size distribution of particles <30µm in laboratory test with LiMCA probes of 199µm and 210µm orifice

One option to overcome the small error in the reported LiMCA N10 in case of orifice sizes bigger than 200µm is an elevated electrical current during the measurement cycle as already discussed above.

Figure 6 shows the particle size distribution in the size range of 8-30µm obtained in another laboratory test using different electrical currents during the measurement cycles. The lower particle size detection limit reduces from 13µm at 30A to 11µm at 50A and 9µm at 70A. Based on the experience from the laboratory tests shown in figure 5 and 6 it might be concluded, that a LiMCA N10 value can be reported with all LiMCA tubes of 200±10µm orifice size, when using an electrical current of 70A.

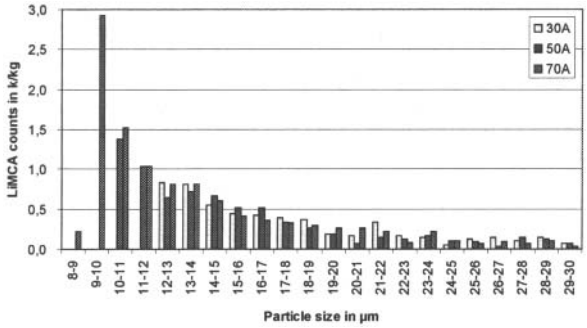

Figure 6. Particle size distribution of particles smaller than 30µm in laboratory test with LiMCA tube of 201µm and variation of electrical current

While these tests demonstrate that LiMCA using a 200µm probe might be generally capable to report N10 values, it has to be checked, whether the reported particle levels and sizes correspond with reality. This was tested in a first step by addition of solid pieces of DURALCAN W6S.14A to the melt in the region of the LiMCA tube orifice. A significant peak of particles of 10-20µm was expected due to the high fraction (14 ± 2%) of Al_2O_3 particles in this size range present in the composite material.

Figure 7 shows the LiMCA run-chart of a laboratory test with addition of the composite material for a LiMCA with a 300µm orifice. A solid piece of Al99.99 of similar size was added in the same manner for comparison.

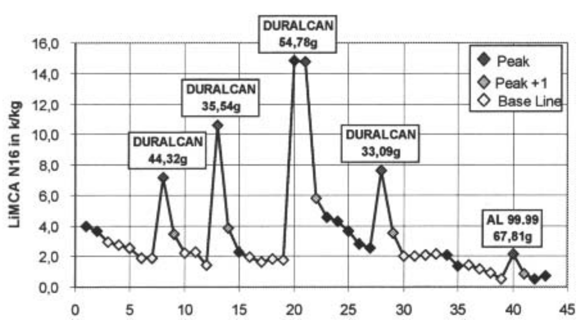

Figure 7. LiMCA run-chart of laboratory test with addition of composite material in front of the 300µm orifice

Significant particle peaks were measured during addition of the composite material, followed by a decreasing concentration during the following measurement cycles due to settling of the particles. The addition of the solid Al99.99 piece of similar size resulted in a smaller and rapidly vanishing particle peak.

The measured data was processed according particle peak shape and described by the following three levels:

- **Base level:** Describing the general particle level of the melt by averaging the data before composite addition and after settling (yellow points in Figure 7)
- **Peak level:** Describing the particle concentration during addition by averaging the peak data (red) point
- **Peak+1 level:** Describing the particle concentration during the settling (orange data)

Figure 8 shows the particle size distribution of the three levels described above. The peak level shows an increased particle concentration for particles up to 100µm compared to the base level, while the difference was especially large for particles up to 25µm. During the peak+1 data monitoring, about 1-2 minutes after the composite addition, most of the larger or agglomerated particles were already settled and the increased concentration within the 15-25µm range was clearly visible.

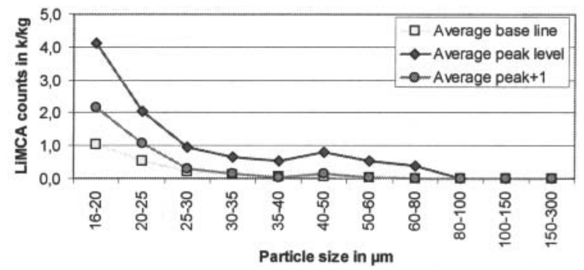

Figure 8. Particle size distribution during base level (yellow points in figure 7), peak level (red points) and peak+1 level (orange points) of the laboratory test shown in figure 7

The same particle distribution of the three levels (base level, peak level, peak+1 level) of a laboratory test with a LiMCA tube of 200µm is shown in figure 9 for the size range from 11µm to 25µm in 1µm size steps. The majority of particles reported by LiMCA here was in the size range of 11-18µm as visible by the peak+1 data. This was consistent with the size of the Al_2O_3 particles sizes of the composite material.

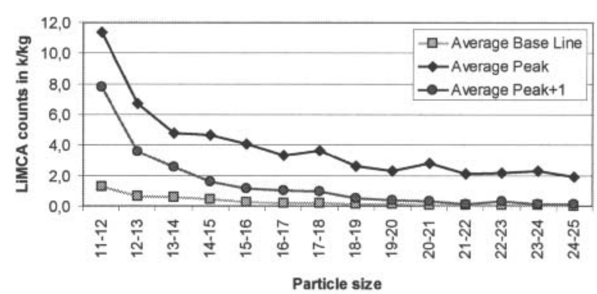

Figure 9. Particle size distribution during base level, peak level and peak+1 level of a laboratory LiMCA test with 200µm tube and addition of Al_2O_3 composite material

The laboratory test with one LiMCA using a glass tube of 200µm orifice size confirmed the general capabilities of the measurement system to identify particles as small as 10µm.

On-site tests with 2 LiMCA units

The on-site measurements were performed to investigate the measurement accuracy of LiMCA using a 200µm probe by direct comparison to measurements with 300µm probes. The measurements were performed at the furnace exit of a Hydro Aluminium DC cast house by placing two LiMCA units next to each other. Position and tube type were changed between the LiMCA units during the evaluation campaigns to exclude effects of unit and position. The correct particle monitoring was additionally checked by measuring periods with identical orifice sizes on both units.

The figures 10 and 11 show the LiMCA N16 run-chart and the particle size distribution for particles larger than 16 µm for two side-by-side measurements. A particle size of 16µm was identified as the detection limit for a standard LiMCA operation in the section "Laboratory tests using standard 300µm LiMCA probes". The test V1 in Figure 10 with low incoming inclusion level showed only a weak agreement between the N16 run-charts of the two units. Only the decreasing particle concentration with time due to settling of particles in the casting furnace was reliably detected by both units. The particle size distribution in the bottom graph of figure 10 showed that the mismatch was evident in all particle size ranges in this test.

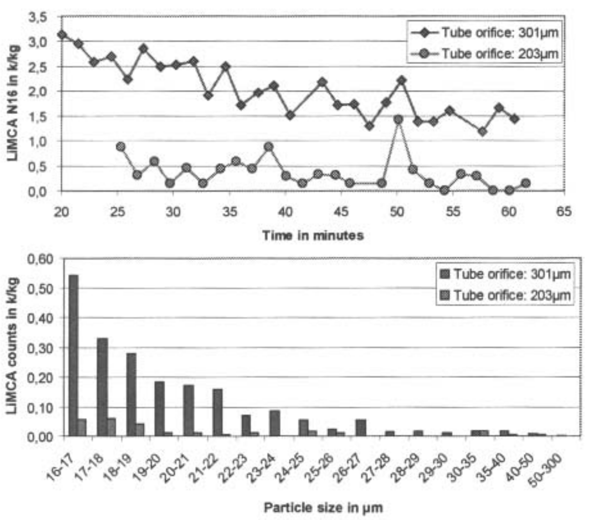

Figure 10. LiMCA N16 run-chart and particle size distribution of side-by-side test (**V1**) of one LiMCA unit with 203µm orifice tube and one LiMCA unit with 301µm orifice tube

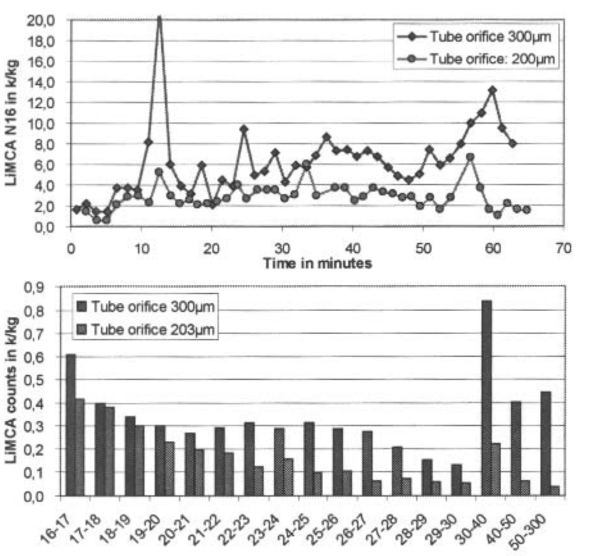

Figure 11. LiMCA N16 run-chart and particle size distribution of side-by-side test (**V2**) of one LiMCA unit with 203µm orifice tube and one LiMCA unit with 301µm orifice tube

Changes of the particle concentration were also well monitored by both units in the side-by-side test V2 in figure 11 at medium particle concentration. A much better agreement between the two units with the different probe orifice sizes was reached in this test, however lower values were still reported by the LiMCA with the 200µm orifice. Additionally, the agreement decreased with time during the measurement period. A fairly good agreement was evident for particles smaller than 20µm as visible by the particle size distribution in figure 11, while an increasing mismatch with larger particle sizes was monitored.

One operational parameter that differed in addition to the particle concentration in test V2 compared to test V1 was the resistance of the 200µm probe. An average resistance of 2900µΩ was evident during the first half of test V2, while 2500µΩ was monitored during the second half and only 2400µΩ during the complete test of V1.

The mismatch between the measurements using 200µm and 300µm tube orifices and in some cases the correlation to the orifice resistance was confirmed in further tests.

Aspects of the LiMCA particles measurement

Several aspects of the LiMCA particle detection principle were analyzed to understand the mismatch of the particle monitoring by LiMCA with 200µm and 300µm orifice probe. Three of these aspects will be discussed in the following chapter shortly.

Particle shadowing

LiMCA measures the particle concentration by counting voltage peaks during the movement of the particles through the ESZ. LiMCA is not ready to identify a new particle until the voltage peak of one particle has decreased under a certain threshold limit. In case two particles flow at the same time though the orifice only one particle is measured. This effect is called particle shadowing. The number of monitored particles and therefore the probability of the particle shadowing effect is increased by the reduction the tube orifice size.

For LiMCA monitoring of melts with low particle concentration the particle shadowing effect should be rather small. In the side-by-side test **V1** 2200 particles larger than 16µm were measured by the standard LiMCA unit and another 2000 particles per kg metal smaller than 16µm were monitored by 200µm LiMCA. This means, that the 200µm LiMCA has measured in average 14 particles smaller than 16µm and should measure 15 particles larger than 16µm within one measurement cycle of 30 seconds and measurement volume of 2.9 ml. The time of passage for a particle through the ESZ is about 0.1 ms [7] and it is therefore very unlikely, that significant particle shadowing occurred in the ESZ. Another contra-indication is the fact, that the much higher concentration of small particles in the test V2 (63,2 k/kg) did not increase the effect. In fact an even better agreement between the two LiMCA units was achieved in this test.

Electromagnetic effects

The influence of electromagnetic effects on the particle and melt movement in the ESZ has been extensively discussed in the literature by R.I.L. Guthrie at el. [8][9][10][11].

The measurement current produces a self-induced magnetic field in the probe orifice with the highest magnetic field density at the orifice wall and directed towards the centre. Non-conducting particles, like inclusions or gas bubbles are pushed by the electromagnetic field away from the hole centre, which might hinder their entering into the probe. Li calculated the pass-through fraction of Al_2O_3 particles and gas bubbles of different sizes in standard LiMCA operation (Figure 12) and concluded, that LiMCA can be used in the Aluminium industry with the limitation of microbubbles as the pas-trough fraction is high for both types in the relevant size range. The pass-through fraction is dependent on the so called blockage ratio k, the ratio of particle diameter and tube orifice diameter. The blockage ratio is of course increased by the reduced probe orifice size and the reported pass-through fractions might be valid for smaller particles, which may partly explain the decreased concentration for particles larger than 30μm shown in Figure 11.

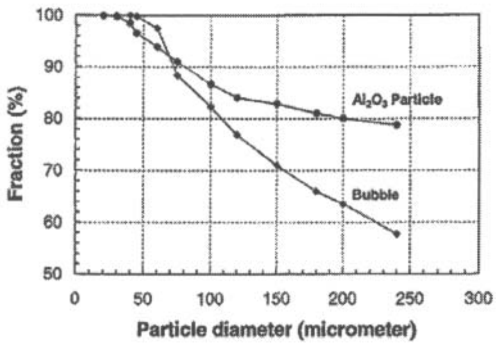

Figure 12. The predicted pass-through fraction of Al_2O_3 particles and gas bubbles of different sizes flowing into the ESZ of LiMCA in molten aluminum; taken from [9]

Another relevant electromagnetic effect is the occurrence of a circular melt flow in front of the probe orifice at high current densities, which is used in standard LiMCA operation to clean the orifice from particles by applying a high current of 300A for a short period, called "conditioning". This circulation is generated as a result of the high pressure established at the throat of the orifice by the interaction of the strong electric current density and its self-induced magnetic flux density [10][11]. The reduction of the orifice size increases the electric current density and might change the melt flow at much lower currents preventing some particles from entering the orifice.

Hole diameter effects

The orifice size has a significant influence on the particle size detection as previously discussed. Investigation using an optical microscope showed, that the real orifice size of the specially produces LiMCA probes was in all cases within ±10μm of the target size as promised by the supplier. One aspect related to the reduction of the orifice size, which is not taken into account here during the signal processing, is the change of the orifice shape and therefore the shape of ESZ and the voltage peak.
Another aspect is the stability of the orifice during LiMCA operation. Operating a LiMCA at the same electrical current (60A) with a smaller orifice increases the local current density. An increased number of 300A "conditioning" cycles was additionally necessary during the operation of this probe. The higher current density and conditioning frequency may have increased the orifice size during operation and resulted in the steadily decreasing orifice resistance. The mismatch between orifice size in the software (200μm) and the real diameter (larger) shifts the particle size detection towards smaller particles and a direct comparison of 300μm and 200μm probe would no longer be valid.

Summary and Conclusions

The possibility to enhance the monitoring capabilities for non-metallic inclusions of LiMCA towards finer inclusions in the size range of 10-20μm was evaluated by laboratory crucible tests using one LiMCA and on-site tests with two LiMCA units. LiMCA operates with the coulter-counter principle and produces voltage peaks by a forced flow of melt with particles through an electrical sensing zone of a small orifice established by an applied constant electrical current. These particle voltage peaks were either increased by adjustment of the operational parameters, such as the applied current and/or the probe orifice diameter or the signal-to-noise-ratio was changed by adjustment of the band-pass filter.

The evaluation might be summarized in following results:

- Influencing the signal processing by adjustment of the band-pass-filter was not considered effective as background noise was immediately visible at lower particle sizes.
- Manipulation of the LiMCA particle detection limit was possible via the applied electrical current. The detection limit with standard LiMCA probes of 300μm orifice was reduced from 16μm to 15μm by applying 75A instead of 60A.
- The LiMCA particle detection was severely influenced by the LiMCA probe orifice size. By changing the probe orifice diameter from 300±10μm to 200±10μm the detection limit was decreased from 17μm to 11μm. Combining these measures with an increased current of 70A reduced the limit down to 10μm.
- The particle detection accuracy of the adjusted LiMCA setup with 200μm probe orifice showed in on-site side-by-side tests acceptable agreement to the standard 300μm probe for particle sizes smaller than 20μm, but significantly lower reading for all larger particles. The mismatch seemed to be related to the resistance of the 200μm orifice.
- Several hypotheses to explain the mismatch were discussed. Electromagnetic separation of particles due to the higher current density in a 200μm orifice and influences of the orifice shape and stability were considered the most probable causes.

It might be concluded from this evaluation:

- LiMCA N20 reporting should be preferred against N15 reporting for LiMCA monitoring with standard 300μm probe and standard electrical current of 60A. The particle size detection limit for this setup varies with the actual orifice size within the 15-20μm size range. The reported LiMCA N15 value is therefore dependent on the actual probe orifice size.
- The detection limit of LiMCA can be enhanced by increasing the applied current to report real N15 values for standard probes and to report N10 monitoring with 200μm orifice probes
- LiMCA monitoring of the melt quality with 200μm probes provides useful information about the particle concentration within the 10-20μm size range, which can be beneficial for charges with low concentration of particles, while the monitoring of charges with higher inclusion levels should be currently performed by

LiMCA using standard operational parameters. Further evaluation of the situation in the ESZ and improved operational parameters would be necessary to fully utilize the LiMCA capabilities to measure the smallest particles.

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