Light Metals 2015
Edited by: Margaret Hyland
TMS (The Minerals, Metals & Materials Society), 2015

DEVELOPMENT OF A LiMCA METHODOLOGY FOR THE MEASUREMENT OF INCLUSIONS AT DIFFERENT DEPTHS IN MOLTEN ALUMINIUM

Pierre Le Brun[1], Fabio Taina[1]
[1] Constellium Technology Center, Parc Economique Centr'alp, CS10027, Voreppe 38341 cedex, France
Keywords: Inclusions, Characterisation, LiMCA, Molten aluminium

Abstract

LiMCA is the reference technique available today for on-line measurement of inclusions in molten aluminium. With LiMCA a glass sampling tube is immersed in molten aluminium and metal is alternatively pumped and rejected in this tube. By construction, LiMCA gives access to inclusions ranging from 20 to maximum 300 µm (which is the sampling hole size). Commercially available tubes have the sampling hole located 50 mm under the surface of molten metal. In this work elongated tubes have been assessed in order to measure inclusions deeper in the melt. The paper illustrates the work done to validate the elongated tubes, by measurements done in crucibles and launders. The need for elongated electrodes is also discussed. This work provides a methodology for the measurement of inclusions at different depths into the melt, or for the measurement in locations with difficult access.

Introduction

Aluminium alloys are widely used for applications in sectors as varied as automotive, aerospace, packaging, building and sports. The quality criteria of the aluminium products depend on the final application; however the inclusion content is always a significant parameter. The characterization of inclusions is a difficult task: off-line (mainly PoDFA and Prefil) and inline techniques (LiMCA and ultrasonics) exist. All have advantages and limitations, in terms of size of inclusions that can be detected, volume of an individual analysis, frequency of sampling and capacity to provide information on dynamic processes [1]. Since the mid '90s LiMCA has become the reference technique for in line inclusion analysis in molten aluminum [2].

The distribution of inclusions in molten aluminium is a topic of theoretical interest [3]. It is known that inclusions settle and/or are entrained by convection currents or other forces applied on molten aluminium. Inclusions also interact and potentially agglomerate. Work has been done to understand and describe the movement of inclusions inside furnaces [4]. With regard to launders there has been no focus on inclusion distribution. Although turbulence resulting from the metal flow may favor inclusion homogeneity [5] there are situations where the inclusion distribution is not homogeneous.

There are almost no experimental means to characterize the distribution of inclusions in the melt. Most of the above mentioned techniques either have a constant position in the melt (LiMCA, ultrasonic) or require a sampling spoon to be used that results in the loss of any positioning information. Only recently a technique has been proposed for the in-depth sampling in a furnace which allows PoDFA/Prefil data to be connected to the sampling position [6].

By construction LiMCA samples are taken at 5 cm under the surface. The position is constant and LiMCA users never mention this information. Only limited information suggests measurements have been done deeper in the melt, however the measurement practice has not been described [5].

Given the need to know inclusion distributions at different positions in the melt [7], it has been decided to develop a measurement methodology that allows LiMCA to provide accurate information from samples taken at different depths in molten aluminium. This was done by using longer sampling tubes and adapting the other equipment features.

Experimental conditions

The tests have been performed with LiMCA II mobile units (supplier ABB). The work reported in this paper used sampling tubes of two different lengths: conventional LiMCA tube with sampling hole at 180 mm from the top of the tube, and long tube with sampling hole at 280 mm from the top of the tube (figure 1), both in aluminosilicate glass.

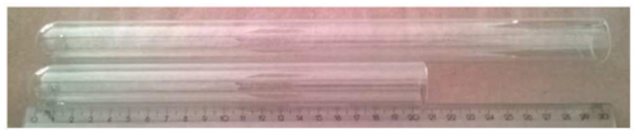


Figure 1: Conventional LiMCA sampling tube (bottom) and long sampling tube (top)

In LiMCA operation electrodes are immersed in the melt. In the conventional configuration the tip of the measuring electrode is located very close to the sampling hole (figure 2). When a long sampling tube is used, the tip of the conventional measuring electrode is located about 100 mm above the sampling hole. The impact of the electrode length when using a long tube has been studied by installing electrodes 100 mm longer, with which the electrode tip is very close to the sampling hole. Example of short and long electrodes is shown in figure 3.

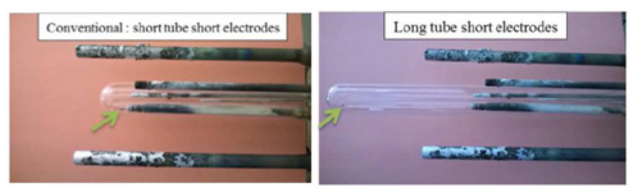


Figure 2: Illustration of both tubes combined with short electrodes (sampling hole is indicated by an arrow)

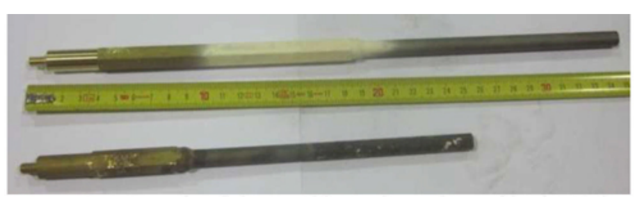
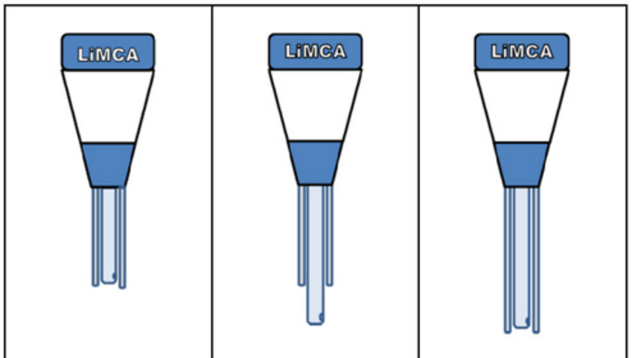


Figure 3: Example of short and long electrode used in the trials

The thermocouple used by the LiMCA to regulate the sampling cycle was set at different positions from the LiMCA heat deflector depending on the objective of the trial: 80 mm (elongated tube and low depth measurement) or 180 mm (all other situations).

Three configurations will be referred to in the following paragraphs (figure 4):



ST/SE	LT/SE	LT/LE
Short tube / short electrodes	Long tube / short electrodes	Long tube / Long electrodes

Figure 4: Schematics of the three configurations under study

The initial feasibility test was done in a 25 kg lab scale crucible with an Al-5% Mg melt. The other tests were done in a 100 kg laboratory scale crucible with resistance heating (alloy 3104) or on an industrial casting line (alloy 3104). The test temperature was in the range of 710°C to 730°C during the trials.
In order to make a decision on the validity of LiMCA results the following parameters have been used in combination:

- Visual observation of the LiMCA data: does the trend correspond to known or expected behaviors?
- Stability of the signal: is the electronic signal stable?
- Value of the current: how do the amperages compare in different configurations?
- Up/Down ratio: is the tube filling correctly (assessed by the mean Up/Down ratio in a given configuration)?
- Resistance: is the electric resistance stable (assessed by the mean and the standard deviation of the electric resistance)?

Results

1. Feasibility

The initial test aimed at confirming the feasibility of a LiMCA measurement with a long tube. In this trial LiMCA measurements were done with a standard tube (ST/SE). After a while the LiMCA head was removed and the short tube was replaced by a long tube (LT/SE). The long tube was preheated longer than usual, and progressively immersed in the melt. The filling time was long (about 6 minutes) however there was no apparent issue in the process. Once the tube was filled the LiMCA was set in automatic mode and measurements were done without any issue. Figure 5 shows the results obtained during this trial.
It can be seen that the frequency of data acquisition is similar with the long tube and the short tube, suggesting the operation is similar although the immersion depth was different. In terms of LiMCA counts, the data are stable when the metal is not stirred (Periods 1 and 2). Once the metal is stirred the usual settling behavior is observed regardless the kind of tube used (Periods 3 and 4). There was a slight difference in LiMCA counts between Period 1 and Period 2. When this difference is significant it may be due to either homogeneity of the inclusion distribution in the melt or to the impact of the configuration (short electrodes used throughout the test even with long electrodes).

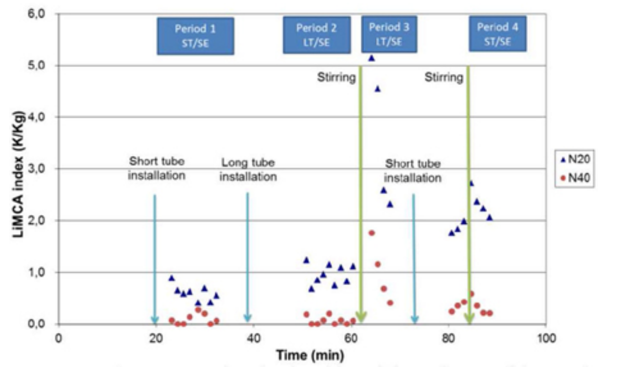


Figure 5: LiMCA results obtained in a lab scale crucible (20 kg) – Successive configurations: ST/SE and LT/SE

The next step is to define and validate a practice for LiMCA measurement deeper in the melt.

2. Definition of a measuring practice

In order to define a practice for LiMCA measurements deeper in the melt a three steps approach has been used:

- In the first one, the measurements obtained 50 mm under the metal surface with the reference system (ST/SE) are compared to measurements obtained with a long tube long electrode configuration measuring at the same depth (LT/LE).
- In the second one, data obtained with a long tube long electrode configuration (LT/LE) are analyzed while the tube is fully immersed (150 mm under the melt).
- In the third step, the possibility to measure with a long tube and short electrode is considered. This would be a preferred option as short electrodes are available to the LiMCA users for measurement with the short tubes.

Several trials have been made to answer these questions. During these trials two LiMCA have been used in parallel in a 120 kg laboratory furnace (figure 6). This procedure allows the direct comparison of different test set-up with the same melt. One trial will be used as an illustration. In figure 7 all the data obtained with the 2 LiMCA are shown. These data are reworked in the next paragraphs to compare the configurations.

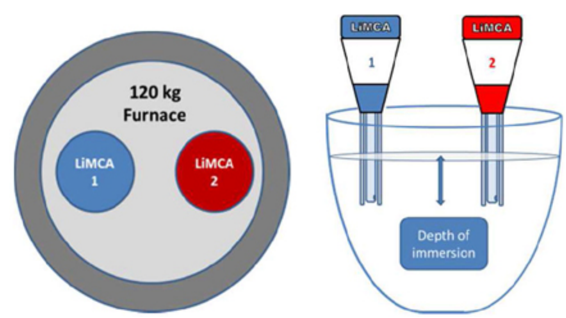


Figure 6: Set up for comparative measurements

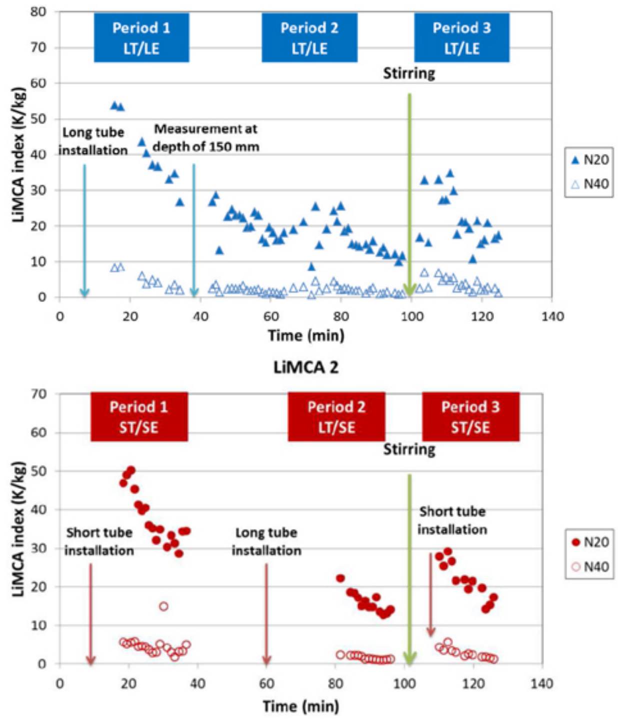


Figure 7: LiMCA results obtained in a lab scale trial with 2 LiMCA in various configurations

2.1. Measurements at a depth of 50 mm (ST/SE and LT/LE)

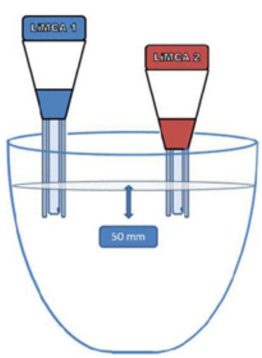


Figure 8: Configuration ST/SE vs LT/LE (-50 mm)

During the period 1, LiMCA 1 was in the LT/LE configuration while LiMCA 2 was in the ST/SE configuration (Figure 8). The aim of this trial is to validate the operation of the LiMCA with different configurations at the usual measuring depth of 50 mm under the surface.

Figure 9 shows the data obtained during this period. It can be seen that both LiMCA measured very similar N20 and N40 inclusion counts. Both showed the expected exponential shape.

The operating parameters of the LiMCA equipments are summarized in table 1 and are very similar as well.

Unit	Configuration	Current (Amp)	H. Resis. Average [E-6]	H. Resis. Std Dev. [E-6]	Up/Down ratio
LiMCA 1	LT/LE Measurement at 50 mm	63,4	2201,3	16,8	1,00
LiMCA 2	ST/SE Measurement at 50 mm	60,8	2078,6	16,9	1,07

Table 1: Operating parameters of both configurations at 50 mm

The results obtained in this configuration validate the operation of the LiMCA with a long sampling tube and long electrodes as similar results are obtained for the measurement of inclusions at the usual depth of 50 mm under the surface.

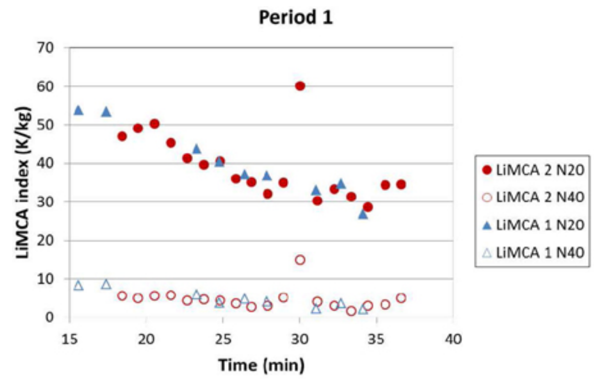


Figure 9: Period 1 – Measurement at 50 mm depth with LiMCA 1 (LT/LE) and LiMCA 2 (ST/SE)

2.2. Measurements at a depth of 150 mm (LT/LE)

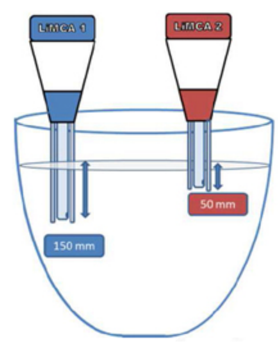


Figure 10: Configuration ST/SE (-50 mm) vs LT/LE (-150 mm)

During the period 3, LiMCA 1 was measuring deeper in the melt (150 mm) with LT/LE configuration while LiMCA 2 was measuring at the usual depth (50 mm) in the ST/SE configuration (figure 10).

Figure 11 shows the data obtained during this period: exponential shape resulting from the intense manual stirring done at the start of the measurement (also explaining the spread observed with both LiMCA. Table 2 shows very similar operating parameters.

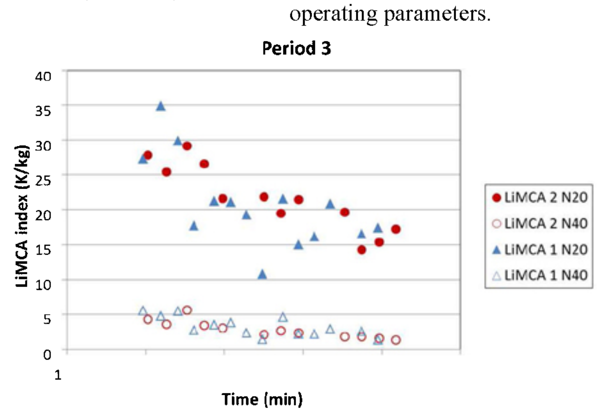


Figure 11: Period 3 – Measurement of LiMCA 1 at 150 mm (LT/LE) and LiMCA 2 at -50 mm (ST/SE)

Unit	Configuration	Current (Amp)	H. Resis. Average [E-6]	H. Resis. Std Dev. [E-6]	Up/Down ratio
LiMCA 1	LT/LE Measurement at 150 mm	60,4	2189,6	29,8	1,11
LiMCA 2	ST/SE Measurement at 50 mm	59,3	2247,0	19,7	1,14

Table 2: Operating parameters of LiMCA 1 at 150 mm (LT/LE) and LiMCA 2 at -50 mm (ST/SE)

These results confirm the feasibility of in-depth measurement with elongated LiMCA tubes. It may be seen that the some data points were not valid when the measurement was done at 150 mm from the surface.

2.3. Validation of a LT/SE

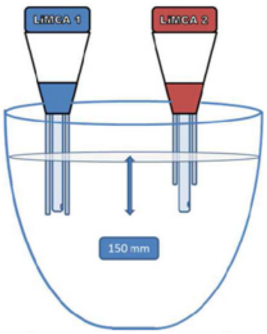


Figure 12: Configuration LT/LE (-150 mm) vs LT/SE (-150 mm)

During the period 2, both LiMCA were equipped with long tubes. LiMCA 1 had long electrodes and LiMCA 2 short electrodes (figure 12). All LiMCA users have short electrodes as standard attachment to the LiMCA, so that it is worth checking if short electrodes can be used without impact on the measured data. The curve shown in figure 13 reveal similar results in both configurations (N20 and N40). Table 3 also shows the operating parameters are very similar.

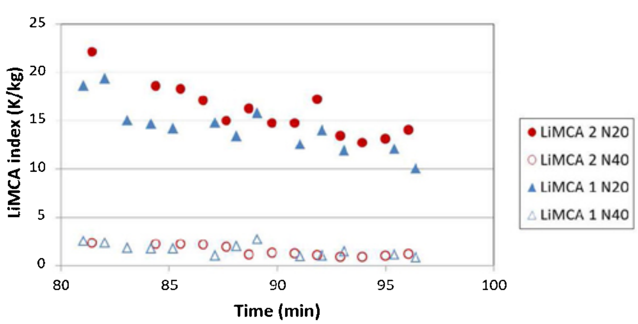


Figure 13: Period 2 – Measurement 150 mm under the surface (LiMCA LT/LE and LiMCA 2 LT/SE)

Unit	Configuration	Current (Amp)	H. Resis. Average [E-6]	H. Resis. Std Dev. [E-6]	Up/Down ratio
LiMCA 1	LT/LE Measurement at 150 mm	59,3	2189,0	7,3	1,02
LiMCA 2	LT/SE Measurement at 150 mm	59,5	2277,9	9,7	1,03

Table 3: Operating parameters of LiMCA 1 (LT/LE) and LiMCA 2 (LT/SE) both 150 mm under the surface

In addition figure 14 shows the histograms obtained in both configurations. It can be seen that the distributions of inclusions are very similar at high inclusion counts (small inclusion size) and show a scattered distribution at low inclusion counts (large inclusion sizes).

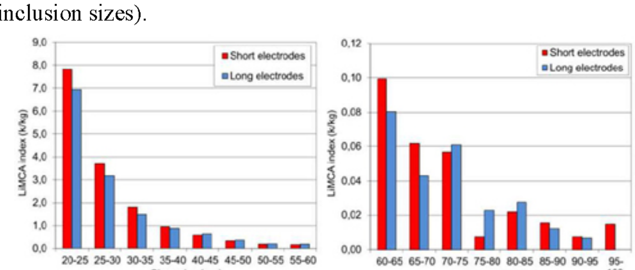


Figure 14: Period 2 – Comparison of the histograms obtained when measuring 150 mm under the surface (LiMCA LT/LE and LiMCA 2 LT/SE)

While short electrodes are very reliable it should be noted that the operation of long electrodes has led to failures. Figure 15 illustrates an example of electrode failure that led to an increase of the inclusion count that was recorded only by the LiMCA equipped with long electrodes. In this occurrence the LiMCA display indicated an unstable electric signal associated to a severe increase of the inclusion count. The behavior was easily noted as another LiMCA equipped with short electrodes was used aside of the LiMCA with long electrodes.

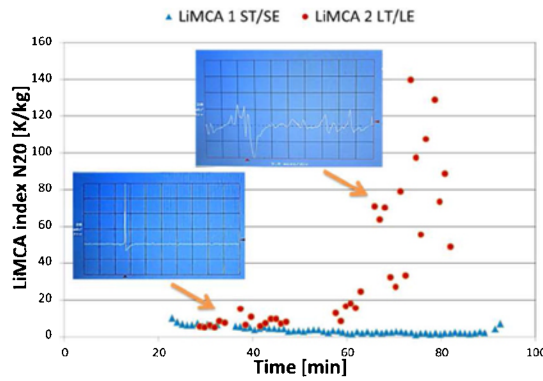


Figure 15: Illustration of a LiMCA measurement performed with long electrodes failing after about 30 min of use

3. Industrial test

Additional trials were performed on an industrial casting line to validate the feasibility of in-depth LiMCA measuring in flowing metal. Trials were performed at the exit of a holding furnace using two LiMCA set side by side (figure 16). Both units were equipped with short electrodes and maintained at a distance of 80 cm. An elongated tube was installed on LiMCA 2, while LiMCA 1 was equipped of a standard tube (reference).

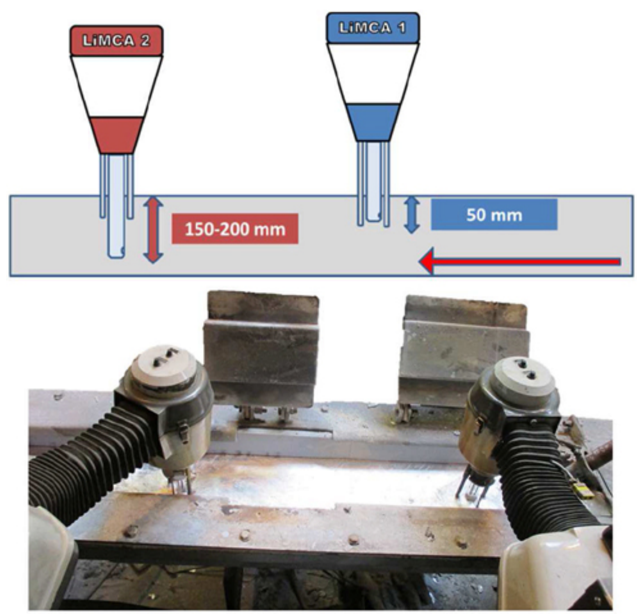
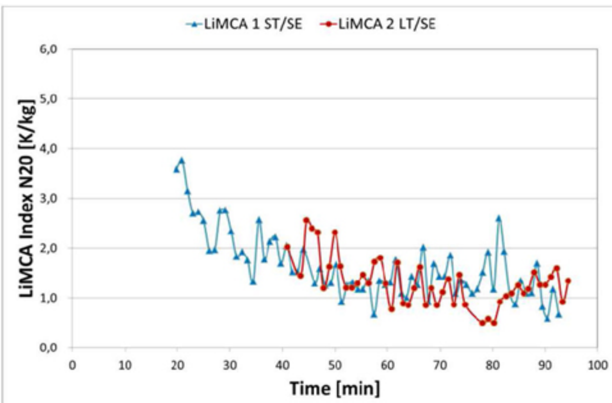


Figure 16: Evaluation of in-depth LiMCA measurement in flowing melt

Figure 17 shows the N20 and N60 LiMCA measurements. It can be seen that the inclusion counts are very similar and repeatable all over the cast, even at low inclusion counts (actually 0 inclusions were counted for the N60 index in both configurations). During the industrial measurements the LiMCA with long tube was started once the other LiMCA was in operation. This explains the delay seen on the graph, together with the fact the long tube required an extensive filling time before measurements can be performed. These promising results were confirmed over a large number of casts and confirm the good operation of the measurement with long tube in troughs.



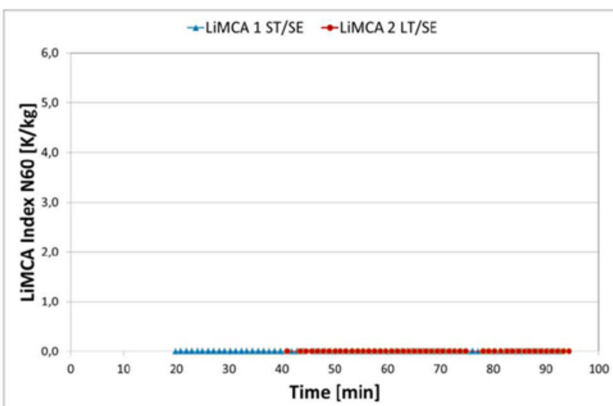


Figure 17: Comparison of LiMCA N20 measurements in a trough (casting furnace exit) with short electrodes and long or short tube.

Discussion

The distribution of inclusions in the depth of a furnace or a trough has not been experimentally studied in detail so far. This is mainly due to the absence of a quantitative characterization method. LiMCA is seen as the most efficient in-line inclusion measurement tool for molten aluminium, measuring 5 cm under the surface, and that is the reason why expanding the depth range was explored.

The lack of experimental data for inclusion distribution in molten aluminium has forced to carry out a progressive approach. Clear conclusions can be drawn when comparisons are done at a depth of 5 cm under the surface (period 1, figure 9) which is the reference for the LiMCA. The accuracy of a LiMCA measurement is considered to be ±10% at high inclusion counts and will reduce at low inclusion counts [8]. With this in mind the data shown in figure 9 can be considered as similar and validate the LT/LE configuration at 50 mm immersion.

Any other configuration is hard to assess in terms of accuracy. Differences observed with a reference configuration may be due either to a change in the inclusion distribution or to an issue in measuring. Indeed in the 120 kg laboratory scale furnace the metal depth is about 33 cm. This means that a LiMCA measuring in a reference configuration (5 cm under the surface) samples metal at about 15% of the bath depth while a LiMCA with long tube (15 cm under the surface) samples close to half the depth of the metal bath (15 cm). Differences in metal distributions are possible as convective currents will be very different, as a consequence direct conclusions can hardly be drawn. The inclusion distribution in a crucible furnace is under study which will provide guidance on this topic [7]. As a consequence it was decided to use the LiMCA parameters to assess the good operation of the equipment in each configuration. It appeared that each configuration studied was satisfactory with this respect.

From a user standpoint the most interesting configuration would be to use a long tube in combination with short electrodes. This would allow only one electrode size to be used. Also the risk of long electrode failure would be cancelled. Although no deep investigation has been done it is thought that long electrodes suffered from thermal stress that affected the electrical connection, leading to signal degradation. However using short electrodes may have an impact on the measurement as the electric path was completely changed. Based on the trials done it is thought that there is no major impact and short electrodes can be efficiently used with long tubes.

The operation of a LiMCA with a long tube has been shown possible. This configuration however impacts the performance of the equipment:

- LiMCA offers the opportunity for the monitoring of dynamic phenomena. This is still valid with long tube. However the long initial filling time cancels the ability to detect early dynamic phenomena. It can be assessed that there is an additional 6 to 8 minutes before a measurement can be done, as compared to the reference configuration.
- Although tests have been done with a variety of melt qualities it is believed that measuring with a long tube may be difficult when a melt contains a high inclusion concentration and in particular when oxide skins are numerous. The risk of sampling hole clogging would be high and boosting is more difficult or even impossible unless the electrodes are immersed (a few minutes with short electrodes).

Conclusions

- A methodology has been developed and validated that allows LiMCA measurements to be performed at depths different than the pre-set value of about 5 cm. The work presented in this paper has shown the validation of measurements done 15 cm under the surface.
- It has been shown that the combination of a long tube and short electrodes was convenient for the valid measurement of inclusions by LiMCA.
- Results presented cover a wide range of inclusion content. It may be expected that measuring inclusions in highly loaded inclusion melts with the developed procedure proves to be more difficult as boosts cannot be applied during the initial and critical filling phase.

- The methodology that has been developed has its main application for measurements at different depths. Using long tubes may also be beneficial for measurements in areas with limited accessibility, for instance in narrow troughs. In this configuration long electrodes will be needed for the measurements.

Acknowledgment

The contribution of Yves Pouyet, from Constellium Technology Center, is gratefully acknowledged. The research leading to these results has been carried out within the framework of the AMAP (**A**dvanced **M**etals **A**nd **P**rocesses) research cluster at RWTH Aachen University, Germany.

References

[1] J.W. Fergus, "Sensors for measuring the quality of molten aluminum during casting", *J. of Materials Engineering and Performance, Vol 14 (2), April 2005,* 267-275.

[2] P. Le Brun, "Melt Treatment – Evolution and perspectives", *Light Metals 2008, TMS,* 621-626.

[3] C. Sztur, F. Balestreri, JL. Meyer, B. Hannart., "Settling of Inclusions in Holding Furnaces: Modeling and Experimental Results", *Light Metals 1990*, 709-716

[4] S. Instone, A. Buchholz, G.-U. Gruen, "Inclusion Transport Phenomena in Casting Furnaces", *Light Metals 2008, TMS,* 811-816.

[5] J.P. Martin, G. Dubé, D. Frayce, R. Guthrie, "Settling phenomena in casting furnaces – An fundamental and experimental investigation", *Light Metals 1988, TMS,* 115-125.

[6] S. Instone, M. Badowski, D. Krings, "Sampling Tool for In-Depth Study of Furnace Processes", *Light Metals 2014, TMS,* 1003-1008.

[7] M. Badowski, M. Goekelma, J. Morscheiser, T. Dang, P. Le Brun, S. Tewes, "Study of particle settling and sedimentation in a crucible furnace", *Light Metals 2015, TMS.*

[8] ABB, LiMCA CM brochure, 2013.