# Discussion of Bi-film index and LiMCA data in industrial aluminum remelting trials

Anne Kvithyld (<u>anne.kvithyld@sintef.no</u>, +4793033104)<sup>1</sup>, Jan Anders Sæter<sup>2</sup>, Martin Syvertsen<sup>1</sup>, Harry Fossheim<sup>2</sup>, Arne Nordmark<sup>1</sup>, Ronny Sottar<sup>2</sup>, Thorvald Abel Engh<sup>3</sup>,

Corresponding author: Anne Kvithyld (anne.kvithyld@sintef.no)

## **Abstract**

The trend in the aluminum high end market is to set unrealistic quality requirements e.g. inclusion free metal. The importance of quality and the ability to measure is growing. LiMCA is a unique, well-established tool for monitoring inclusions in molten aluminum. However, the cost of LiMCA and/or availability sometimes becomes a problem. The "old" vacuum test has long been a simple tool for qualitative analysis of the quality. This vacuum method has been developed further into a quantitative method for estimating metal quality in terms of a bi-film index.

In this paper, industrial remelting trials are presented where both bi-film index and LiMCA measurements were performed. The two measurement techniques are compared. The conclusion is that the bi-film index and LiMCA demonstrate a similar behavior with time. Thus, the bi-film index may become an additional tool also for quantitative analysis of aluminum quality.

## 1. Introduction

Metal quality includes many aspects, from chemical specifications to lack of complaints from the customer. The problem with molten metal quality is mainly inclusions. Inclusions can vary in size ( $\mu m$  to mm) and composition with borides (TiB<sub>2</sub>), carbides (Al<sub>4</sub>C<sub>3</sub>), oxides (Al<sub>2</sub>O<sub>3</sub>, MgO), salts (chlorides/fluorides), refractory, intermetallic particles (TiAl<sub>3</sub>), nitrides (AlN) and others.

The two techniques compared in this paper are the electrical method LiMCA, and bi-film index vacuum method. Techniques used to evaluate inclusions, that will not be discussed here are: chemical, filtration and metallography, and ultrasonic. However, parallel hydrogen measurements with AISCAN is taken into account.

## 2. Characterization methods

<sup>&</sup>lt;sup>1</sup>SINTEFMaterials and Chemistry, Trondheim, Norway

<sup>&</sup>lt;sup>2</sup>Alcoa Norway ANS, Mosjøen, Norway

<sup>&</sup>lt;sup>3</sup>Norwegian University of Science and Technology, Trondheim, Norway

## 2.1. Bi-film index

The bi-film index, or Reduced Pressure Technique (RPT), is a vacuum method where a metal sample solidifies under reduced pressure. This expands the bi-films/oxides with pores in the metal so that they are more easily measured. Samples are machined to the center, and image analysis is performed on the samples. The sum of the maximum length of the expanded pores gives a measure in mm of metal quality. This method was developed and refined by John Campbell, University of Birmingham UK and Derya Dispinar [1,2]. If the sum of lengths is low, giving a low bi-film index, the metal quality is good. On the other hand, if the sum of lengths is high, the metal quality is bad.

The other technique that are used is the well-known LiMCA-method (Liquid Metal Cleanliness Analyzer). The patent by Doutre, D. and Guthrie, R. I. L from 1985 [3] has been a foundation for process developments in furnace practices and metal treatment since the 90's. LiMCA counts and measures individual inclusions, by measuring the resistance change in an orifice when the inclusion passes through.

# 3. Laboratory study of bi-film index

#### 3.1. Bi-film index robustness

The bi-film index involves inspection of the cross section of the metal sample with optical microscopy. Figure 1 shows the results from the evaluation of the same set of samples done by two different persons. The samples were taken from an experiment which involved three different crucibles where scrap was added four different times (Add. B to E) to the initial melt (A). After each addition, ten bi-film samples were collected from each crucible and are represented by one data point as presented in Figure 1, giving a bi-film index. Thus, every person analyzed 150 samples. As seen from Figure 1 the bi-film index results follow the same trends for the two persons.

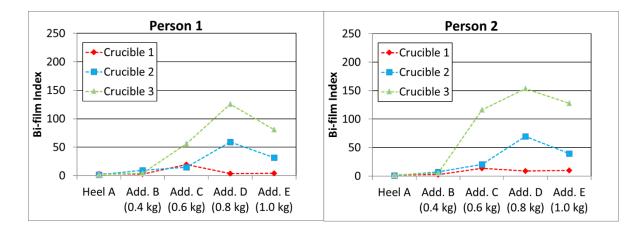


Figure 1 Bi-film index measured by two independent persons, showing same trends

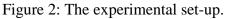
## 3.2. Gas purging and salt additions effect on Bi-film index in small scale

## 3.2.1 Procedure of lab scale trials

In addition to the lab-test with addition of scrap (Figure 1), another experiment was conducted where the bi-film index characterization technique was used to study the effect of gas fluxing on melt cleanliness. The metal (alloy 5083) was melted in a Morgen resistance furnace and held at 750 °C.

Again, three crucibles, as shown in Figure 2, with 15 kg metal in each were used to compare effect of stirring (without gas), two types of gas purging, and the addition of salt on bi-film index. The two gas purging tests was (i) Ar + 0.5 %  $Cl_2$  and (ii) pure Ar. The gas treatment was performed twice. After the gas purging, salt was added to the crucibles. Ten melt samples for bi-film index measurements were taken after each melt treatment from all three crucibles.







## 3.2.2 Results of lab scale trials

Before first stirring, ten bi-film samples were taken from the melt and analyzed to 0.1 mm, which means a clean melt. Figure 3 shows that stirring with the impeller increased the bi-film index, while the use of Cl<sub>2</sub> reduced the index (first data point of 0.1 mm is not included in the graph). Addition of salt increased the index again. Hydrogen tended to follow the bi-film index.

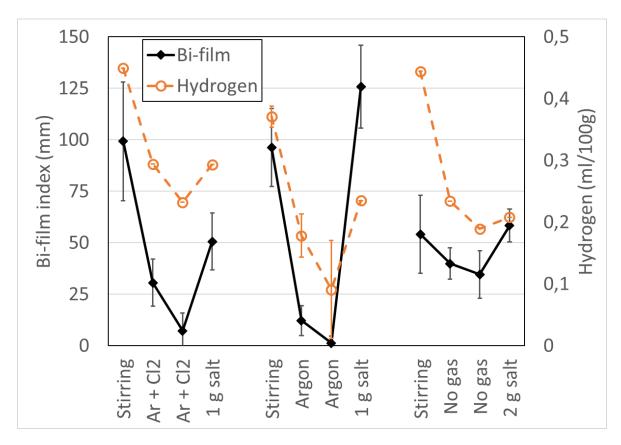


Figure 3 Influence of various melt treatments on hydrogen and bi-film index. Each measurement series correspond to one of the three crucibles in the parallel setup. The three first hydrogen measurements in the second series (with error bars) are average value of two analysis. The end points of these error bars show the value of these two analysis

The hydrogen level in Figure 3 was measured on solid samples with a Galieo-G8 from Bruker Corporation [4]. The three first samples of the second series in Figure 3 were measured two times. The end points of the corresponding error bars shows the two values of measured hydrogen in the samples.

# 4. Test material and procedure for the industrial trials

Five remelting trials were conducted in a 50-tonnes industrial gas-fired furnace. The material was melted and cast in accordance with normal practice, i.e. gas fluxing, holding, degassing and filtering. Charge No. 1-4 was alloy AA5042 while charge No. 5 was a AA5083-alloy. Below in Table 1 the alloy compositions are given. The Mg content is highest in charge No. 5.

Table 1 Composition of alloys AA5042 and AA5083 in wt% [5]

| Alloy   | Si   | Fe   | Cu   | Mn    | Mg   | Cr    | Zn   | Ti   | Others |
|---------|------|------|------|-------|------|-------|------|------|--------|
| AA 5042 | 0.20 | 0.35 | 0.15 | 0.20- | 3.0- | 0.10  | 0.25 | 0.10 | 0.15   |
|         |      |      |      | 0.50  | 4.0  |       |      |      |        |
| AA 5083 | 0.40 | 0.40 | 0.10 | 0.40- | 4.0- | 0.05- | 0.25 | 0.15 | 0.15   |
|         |      |      |      | 1.0   | 4.9  | 0.25  |      |      |        |

The raw material was a blend of liquid metal from the electrolysis cell and scrap metal. The re-melted scrap consisted of 1-3 mm thick compacted sheets with 4-5 % Mg, shown in Figure 4. Table 2 gives the scrap ratios for charges No. 1-5. Gas fluxing and addition of salt bags were done in the furnace before the holding time started. Both LiMCA, bi-film samples and hydrogen measurement were done in the launder by the furnace spout, before any launder melt treatment.





Figure 4 The compacted sheets used in the remelting trials

Table 2 The cold metal ratios for charge No. 1-5, together with hydrogen content and treatment of the melt.

| Charge No           | 1      | 2      | 3      | 4      | 5     |
|---------------------|--------|--------|--------|--------|-------|
| Scrap metal (%)     | 7.5    | 7.5    | 10     | 0      | 30    |
| Hydrogen (ml/100 g) | 0.40   | 0.42   | 0.40   | 0.45   | 0.55  |
| Gas fluxing         | Yes    | Yes    | Yes    | Yes    | No    |
| Salt addition       | Normal | Normal | Normal | Normal | Twice |
| Alloy               | 5042   | 5042   | 5042   | 5042   | 5083  |

## 5. Results from industrial trials

## 5.1. Bi-film index and LiMCA vs casting time

The bi-film index results are presented as the median in each group of ten measurements. Figure 5 shows an example of such a set. In Figure 6 the bi-film index and LiMCA results are plotted for all 5 charges verses casting time. All LiMCA results presented for particles larger than 20µm (N20) are normalized by the same value for all the charges. (In order to anonymize the true N20-value, all N20 values for all charges are divided by the same constant.)

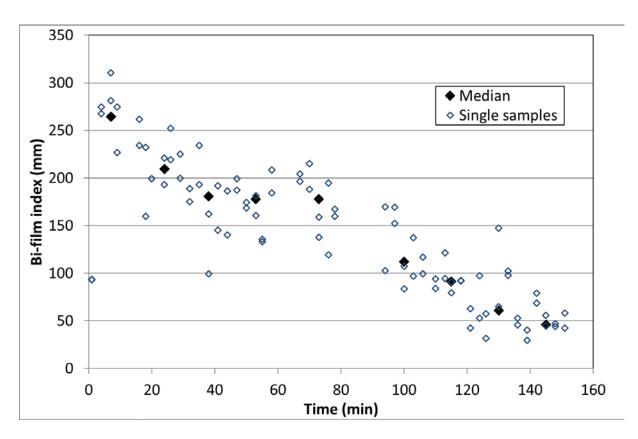
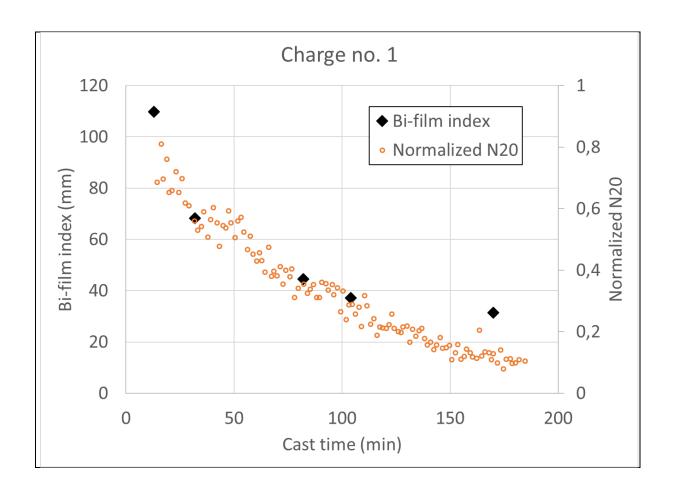
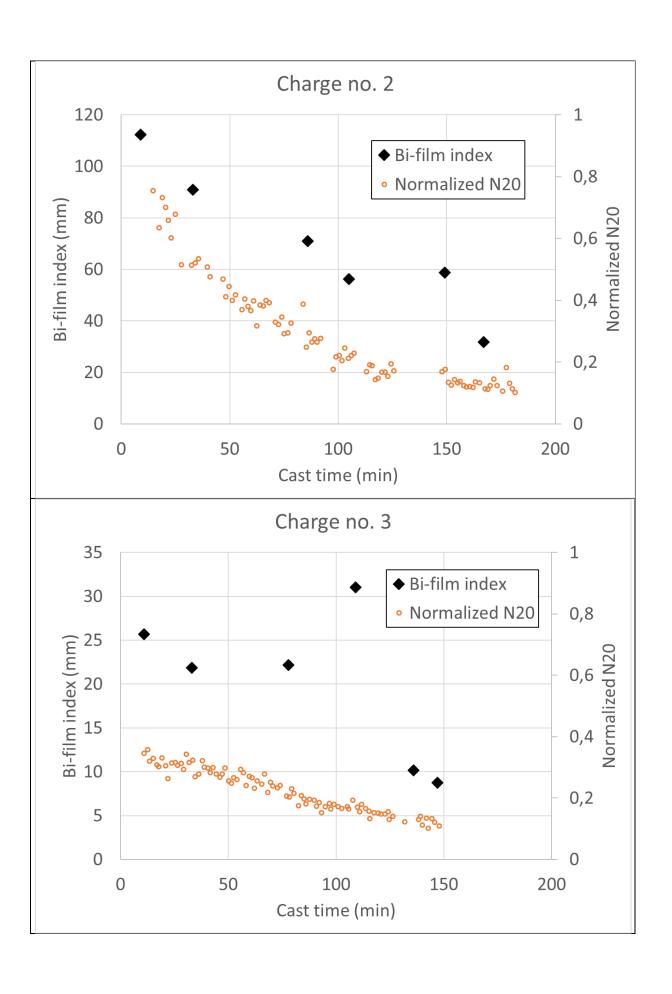


Figure 5 All bi-film index results from one charge together with the median bi-film index of each group of ten samples taken





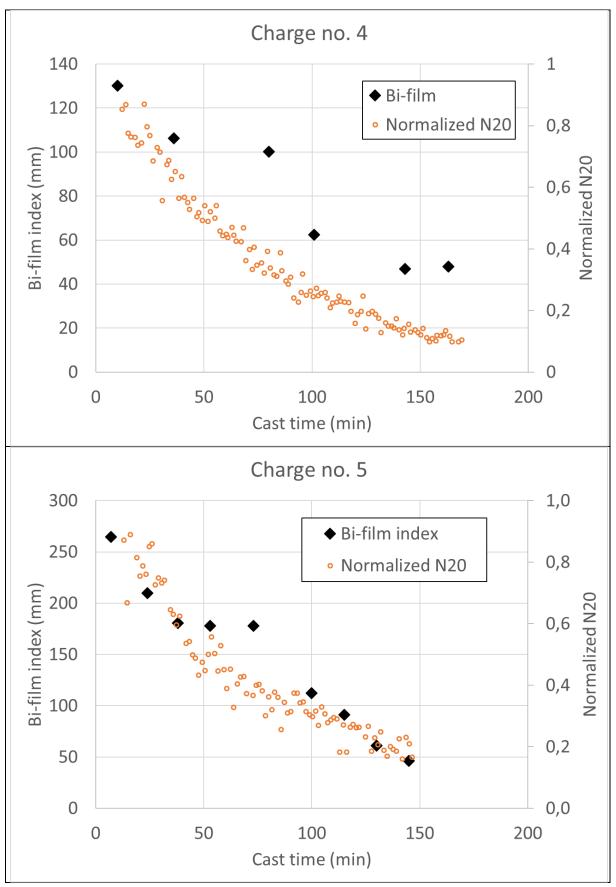


Figure 6 The bi-film index and LiMCA verses casting time is plotted for 5 charges

# 6. Summary of comparison between LiMCA and bi-film results

Figure 6 shows that the time behavior of the bi-film measurement and LiMCA are similar. Plotting the LiMCA values together in Figure 7 shows that the LiMCA curves overlap. Please note that the x-axis is plotted from start of the holding time of the furnace, as settling effects the LiMCA values. As seen in the figure charge No. 3 has a long holding time and a slightly higher LiMCA values. In Figure 8 the bi-film index is plotted from start holding of the furnace. Compared to LiMCA results in Figure 8 the bi-film index has a much larger variation between the different charges.

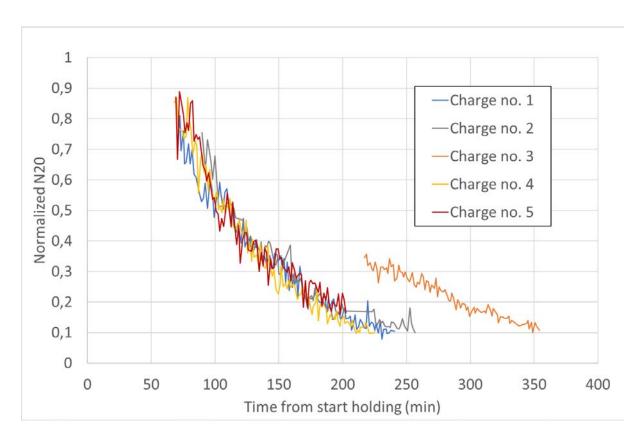


Figure 7 LiMCA plotted from start of the holding for the 5 charges, showing that that the normalized are similar. Charge No. 3 had a very long holding time

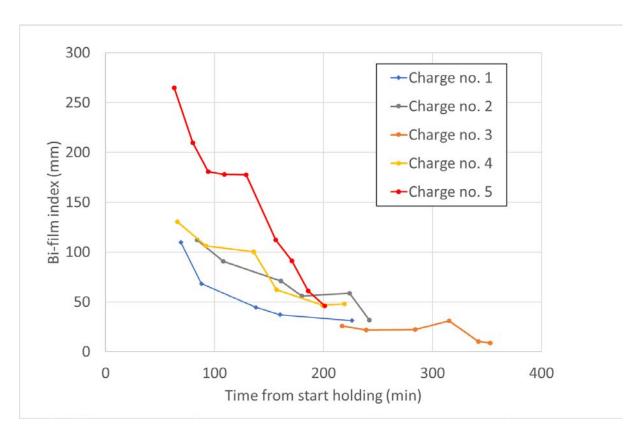


Figure 8 Bi-film index plotted from start of the holding for the 5 charges, showing variation in bi-film index values

# 7. Relation between bi-film index and hydrogen

The variation in bi-film index is in Figure 9 plotted as a function of measured hydrogen using AlSCAN (see Table 2). The bi-film index number used here is the second (median) value after start casting in the plots of Figures 6. Figure 9 indicates that hydrogen and the presence of films are correlated.

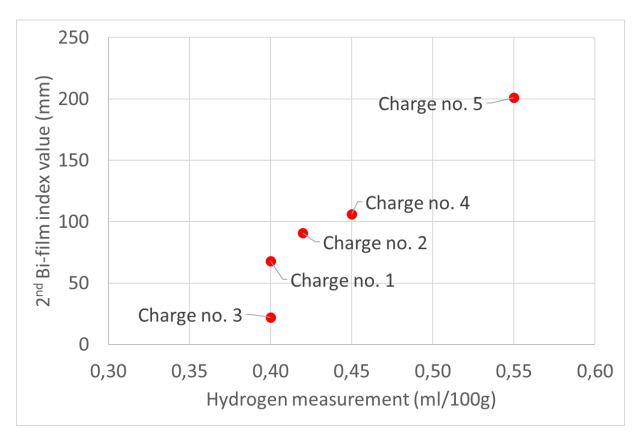


Figure 9 Bi-film index versus measured hydrogen for all the 5 charges. Hydrogen is measured with AlSCAN at the same point as samples for the bi-film index were taken

## 8. Discussion

An explanation for the large bi-film index variation between charge No. 1-4 and charge No. 5 could be the higher Mg content, gas fluxing and use of salt. Charge No. 5 did not have any gas fluxing, but employed twice the amount of salt.

Care must be taken comparing the LiMCA and bi-film index values as they are not measuring the same property. With a LiMCA you will not be able to measure a large oxide film since the orifice is only 300 µm in diameter. The bi-film index is by far more sensitive for oxide films.

The bi-film index measures the length of films. The number includes information about the oxide (possibly from scrap or Mg alloying additions), metal treatment (fluxing and salt use), and some effect of hydrogen. Based on this, it is sometimes possible to discover batches with low quality already during casting.

The plots in Figure 6 shows that both the N20 value and bi-film index follow decreasing trends. In addition, Figure 9 also shows a positive correlation between hydrogen and the bi-film index. This indicates that a bi-film index measurement can be used as a melt quality indicator; a low bi-film index means high quality and high bi-film index means bad quality. Additional measurements might be needed to evaluate the source.

## 9. Conclusions

Measurements of inclusions is essential for understanding and process control of metal quality. LiMCA is a standard method for on-line detection. In this work, it is shown that the bi-film index can be a very useful tool to evaluate quality, and an alternative in cases where LiMCA equipment is not available or too expensive.

## Acknowledgements

This research was carried out as part of the Norwegian Research Council (NRC)-funded BIP Project No. 219940/O30 Quality Aluminum Cast House (QuAlity). The project includes the partners: Alcoa Norway ANS, Gränges Sweden AB, Hydro Aluminium AS, and SINTEF. Funding by the industrial partners and NCR is gratefully acknowledged. Gratefully acknowledged is also Professor Derya Dispinar and Ozen Gursoy for help with the bi-film index. Don Doutre is gratefully acknowledges as discussion partner.

## References

- 1. Dispinar D, Campbell J (2004) Critical assessment of reduced pressure test. Part 2: Quantification. International Journal of Cast Metals Research, 17(5): 287 294
- 2. Dispinar D, Campbell J (2014) Reduced Pressure Test (RPT) of bifilm assessment, Shape Casting: 5<sup>th</sup> Internaional Symposium 2014, 243 251
- 3. Doutre D, Guthrie RIL (1985) Method and apparatus for the detection and measurement of particulates in molten metal. U.S. Patent 4,555,662. November 26, 1985
- 4. Bruker (2017) High Performance Oxygen, Nitrogen and Hydrogen Analyzer. <a href="https://www.bruker.com/products/x-ray-diffraction-and-elemental-analysis/csonh-analysis/g8-galileo-onh/overview.html">https://www.bruker.com/products/x-ray-diffraction-and-elemental-analysis/csonh-analysis/g8-galileo-onh/overview.html</a>. Accessed September 27, 2017
- 5. The Aluminum Association Inc 2015, International Alloy Designations and Chemical Composition Limits for Wrought Aluminum and Wrought Aluminum Alloys, <a href="http://www.aluminum.org/sites/default/files/TEAL\_1\_OL\_2015.pdf">http://www.aluminum.org/sites/default/files/TEAL\_1\_OL\_2015.pdf</a>