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Relationship between the Permeability of the Porous Disk Filter and the Filtrate Weight – Time Curves Generated with the PoDFA / Prefil® Footprinter Method

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Abstract

Current methods to quantify metal quality suffer from disadvantages in complexity, expense or response time. One method suggested to provide a real-time quantitative assessment of metal quality is the use of filtrate weight-time curves produced by filtering the metal through a porous disk filter. The slope and overall shape of this curve should indicate the level of inclusions present in the metal.

Hydro Aluminium increasingly employs such methods to assess the quality of both Aluminium und Magnesium alloys. Several different types of crucible and porous disk filter are used. Due to the increasing importance of this assessment method a better understanding of the importance of variations in the permeability of the filter was considered necessary. This paper describes a device to measure the permeability of the filter discs. It also gives details of trials conducted to quantify the effect of variations in filter permeability on the filtrate weight – time curves.

Introduction

Despite many efforts over several years the difficulty of obtaining a reliable real-time indicator for Aluminium melt quality persists. State of the art methods such as LiMCA and PoDFA are either complex, expensive or too slow. Recently the development of the Prefil®-Footprinter[1] for molten metal quality promised a breakthrough in obtaining a simple, quick and inexpensive method of quantifying the quality of Aluminium melts. This method utilizes the filtrate weight-time curves produced by filtering the metal through a porous disk filter. The actual methodology is similar in nature to the PoDFA method, the difference being the (almost real time) generation of the weight time curves. The slope and overall shape of this curve should indicate the level of inclusions present in the metal.

This paper attempts to examine the significance of the filter characteristics on the development of such filtrate weight-time curves generated when filtering aluminium melts. The filter disk itself has become of interest as several different types of crucible and porous disk filter can now be used to test aluminium and magnesium melts.

The flow of liquid through a tortuous media such as a filter is influenced by the permeability of the filter and build up and compression of any filter cake on the filter itself. The laminar flow of a fluid medium through a filter can be described by Darcy's Law:

$$v = \frac{K \cdot \Delta P}{\mu \cdot \Delta x}$$

Where v is the fluid velocity in filter in cm/s
K is the filter permeability (Darcy)
ΔP is the pressure gradient across the filter in atmospheres
μ is the dynamic viscosity in centipoise
Δx is the filter thickness in cm

This can be rewritten for the permeability in Darcy:

$$K = \frac{v.\mu.\Delta x}{\Delta P}$$

$1\ \text{Darcy} = 9.86923 \times 10^{-13}\ m^2 \approx 10^{-12}\ m^2$

The permeability of the filter is therefore an important parameter to characterize the filter performance in terms of the filtrate weight-time curves. For this reason the supplier of filters for the Prefil®-Footprinter measures the permeability of each filter and delivers only those filters with permeabilities within the defined specification limits. **The question this paper attempts to answer is: are there measurable differences in the filtrate weight-time curves for filters with different permeabilities <u>within</u> the supplier specifications and are these differences significant in the evaluation of the filtration weight – time curves.** Specifically can a correlation between the filtration weight – time curves and the resulting PoDFA evaluation results be established?

Typical Prefil® filtrate weight- time curves for foil alloy 8006 is shown in Figure 1. The cleanliness of the melt is reported to be related to the gradient or slope of the weight-time curve. For the initial linear component of the curve, the cleanliness of the melt should be proportional to the slope of the curve. Non-linear curves are attributed to the build up of filter cake on the filter and a corresponding reduction in the effective permeability of the filter.

Equipment

<u>Test Equipment to Measure Filter Permeability</u>

To determine the permeability of the filter it is necessary to measure both pressure and flow rate of the fluid, which in this case is air. Test equipment for this purpose was designed and built at the Hydro Research and Development Centre Bonn, Germany. The equipment was designed for semi-automatic operation and documentation of results. The equipment comprised a multi-function holder capable of supporting all current PoDFA and Prefil® filter and crucible types and the associated flow and pressure regulation hardware and software. The equipment and function is shown schematically in Figure 2.

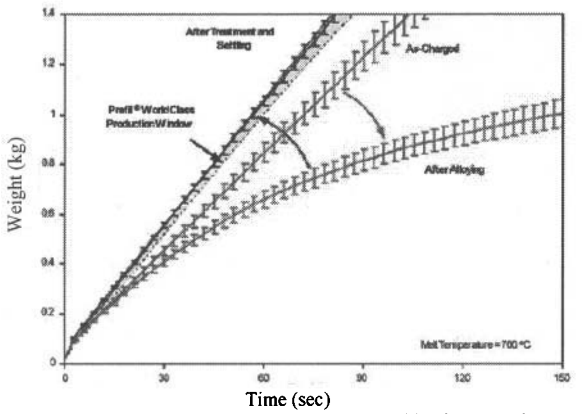


Figure 1. Prefil® curve for typical AA8006 Alloy (source ABB Bomem Prefil® Brochure Prefil_Spec B4277 2005-11[2]).

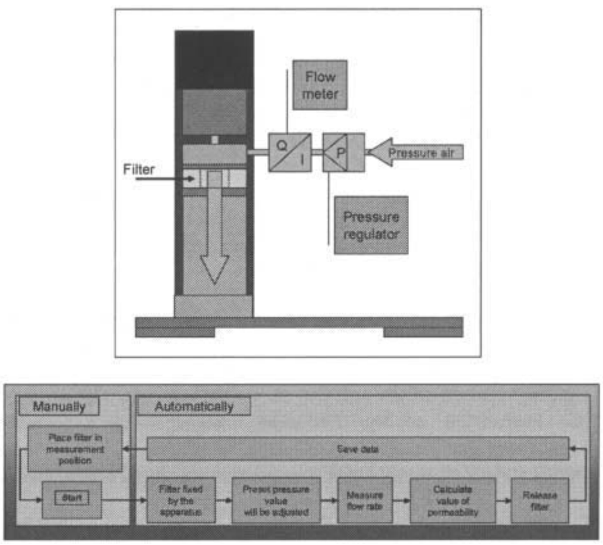


Figure 2. Test Equipment for measuring filter permeability: schematic diagram of construction and algorithm of test procedure showing high degree of automation.

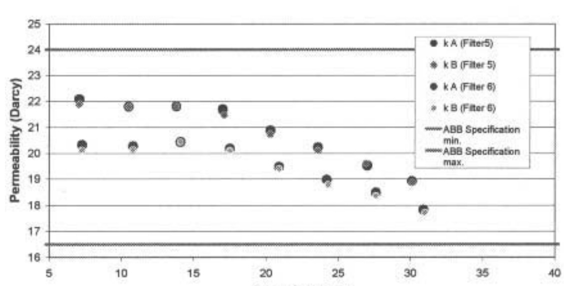


Figure 3. Repeatability testing results for filter permeability tester.

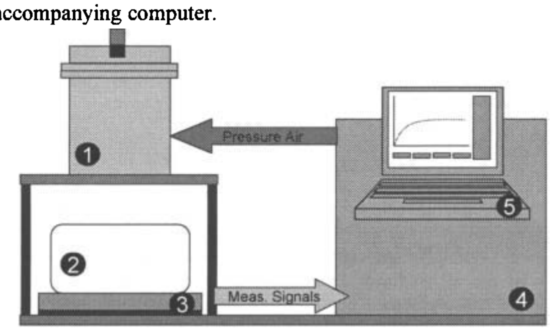
The functionality and repeatability of the equipment was first proven before the current experimental trials were run. Reference filters of known permeability were obtained from the supplier and tested to verify the accuracy of the equipment. The repeatability of the equipment was also assessed as shown in Figure 3.

Hot Tests – Filtration Weight – Time Curves

To obtain accurate and reproducible filtrate weight – time curves similar pressure and mass flow controllers as were employed in the test equipment for filter testing were combined with a pressure chamber and a precision scale with digital output. The equipment built is shown in Figure 4. Although more suited to laboratory investigations the equipment is similar in operation to the Prefil®-Footprinter and provides:

- full control and automation of all parameters
- high accuracy of all components
- high frequency sampling and logging of all process parameters
- automated report generation using customized software applications

The PoDFA or Prefil filter to be tested is filled with the liquid metal and placed in the pressure chamber (1). Once the correct temperature is attained the procedure begins and a preset pressure is built up in the pressure chamber. This forces the metal through the filter in the base of the crucible. The scale measures the weight of the filtered metal which is then logged on the accompanying computer.



1- Pressure chamber
2- Collecting crucible
3- Precision scales
4- System Control (Signal interface, Pressure regulation etc.
5- PC for operation, dataprocessing and visualization

Figure 4. Equipment used to generate filtrate weight - time curves.

In this way it was possible to accurately measure each filter before use and with comparable equipment generate filtrate weight – time curves using melts taken from both laboratory equipment and under production conditions from industrial casting lines.

Experimental

To assess the significance of small differences in filter permeability, filtrate weight – time curves were generated using filters within very narrow tolerance window. Five filters with permeability of 55 Darcy (low) and five filters with 60 Darcy (high) were selected from one batch of Prefil filters supplied by the manufacturer. An alloy of 99.99% pure Aluminium was used for these tests to ensure a low level of inclusions and eliminate any alloy effects related to surface tension, fluidity or even rate of oxidation.

Approximately 2kg of Aluminium was placed in each of ten clay graphite crucibles which were heated to approximately 740°C in two electric resistance (Muffel) furnaces. After the crucibles had reached temperature equilibrium they were removed singly and the contents decanted into the standard reusable Prefil crucibles. These crucibles had been preheated for at least 60 minutes prior to use. The filled Prefil crucible was then placed in the pressure chamber shown in Figure 4 and the filtration test commenced once the metal temperature had reached a temperature of 730°C. The time and filtration weight were logged on a notebook PC. Sampling frequency was 100 Hz. Each test was terminated automatically once the filtrate weight had reached 1 kg. Five repeat measurements were made for each permeability level according to the procedure described above giving a total of ten trials. The details of these laboratory trials are given in Table 1 and Figure 5.

Table 1. Details of experimental conditions

Filter type	Prefil filter for reusable crucibles
Nominal filter permeability	55 Darcy (low) 60 Darcy (high)
Alloy	99.99% Aluminium
Metal Temperature in melting furnace	740°C
Metal Temperature at start of filtration test	730°C
Pressure in chamber (1)	0.8 Bar (gauge, 1.8 bar absolute)

Furthermore the results of several measurements taken during casting of one charge of AlMg3 alloy under industrial production conditions were also evaluated for comparative purposes.

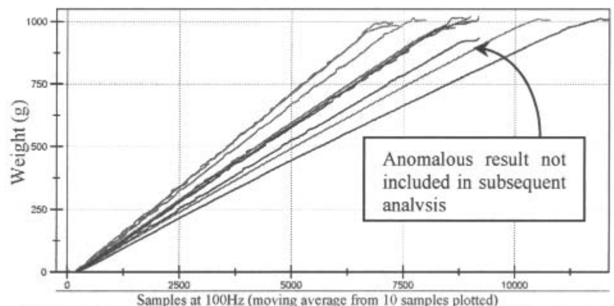
The Prefil filters were subsequently sectioned and prepared using standard metallographic techniques and evaluated using the standard PoDFA grid method for total inclusion content as expressed in mm^2/kg.

Results and Discussion

Laboratory Trials

The resulting filtrate weight – time curves and PoDFA results in mm^2/kg for the ten individual filtration tests are shown in Figure 5. As far as was possible the experimental conditions and procedures were kept constant. The actual temperature of the filter is difficult to determine as once the crucible containing the filter is removed from the preheating device a relatively rapid temperature decrease is expected. One curve, 1237-1 shows a low slope of the filtrate weight – time curve although the filter had a high permeability. As this was the first test performed in this campaign there is some suspicion that the crucible was, due to processing delays, allowed to cool too much before the metal was added and this resulted in a slower filtration rate. The PoDFA result for this test at $0.00032 mm^2/kg$ was very low. This test was therefore considered to be an outlier and has been omitted from the subsequent analysis. The form of the curves obtained is essentially linear indicating no significant change in the permeability of the filter (no filter cake build-up) during the filtering operation. The absence of filter cake build-up was confirmed in the subsequent metallurgical (PoDFA) investigations (example shown in Figure 6).

Figure 7 shows the narrow band for the filter permeability used in each of the two groups. Relative to this narrow band the difference in the mean values of permeability of the two groups is quite large. Figure 8 is a box plot of the slopes of the 9 curves shown in Figure 5. Specific test output for the statistical significance of the difference between the mean values of the slopes of these curves is given in Figure 9. At a α level of 0.05 this difference was considered to be statistically significant. This indicates that the differences in the slope of the filtrate weight – time curves can be explained by differences in the permeability of the filters used. The alternative argument that the differences in slope are due to differences in the amounts of inclusions present in the melt will be discussed below.



	Sample	RegCoeffB	PoDFA [mm²/Kg]	Perm. [Darcy]
———	1237-1	9.89	0.00032	60.22
———	1237-2	8.8	0.00099	54.36
———	1237-3	14.65	0.00207	59.76
———	1237-4	11.96	0.00989	55.25
———	1237-5	13.55	0.06434	60.1
———	1237-7	12.05	0.00288	59.99
———	1237-8	10.68	0.00347	55.27
———	1237-9	14.62	0.00219	60.31
———	1237-10	11.97	0.00127	55.46

Figure 5. Filtrate weight–time curves and PoDFA results for the ten filters tested. The form of the curves is essentially linear and the slope of the curves is shown (under RegCoeffB).

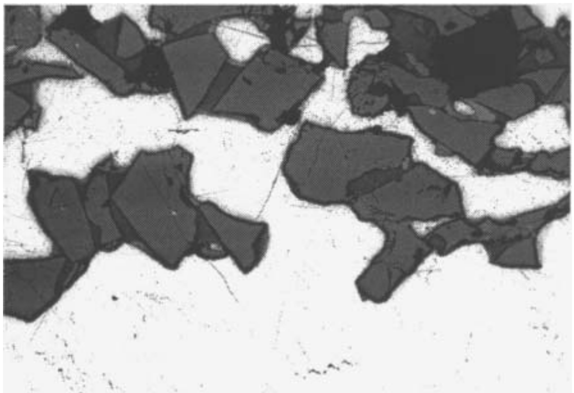


Figure 6. Micrograph of filter from test 1237-1 showing absence of filter cake build up or higher level of inclusions which may have explained the unusually slow filtration rate observed with this filter.

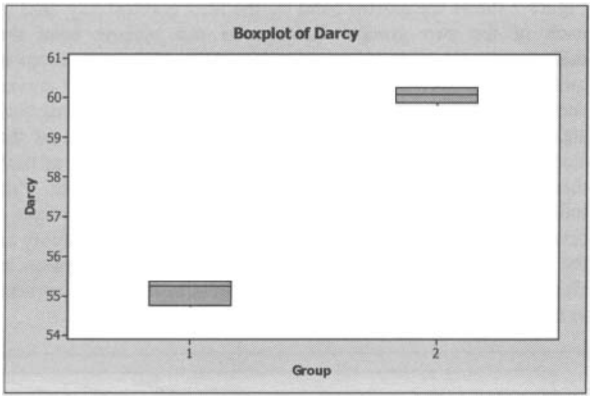


Figure 7. Box diagram of filter permeability showing clear differentiation into the two experimental groups.

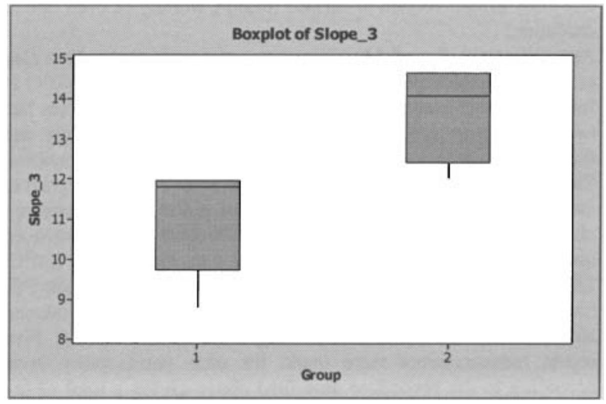


Figure 8. Box diagram of the slopes of the nine filtrate weight – time curves divided into two groups based on the underlying filter permeability. The single anomalous result indicated in Figure 5 is omitted here.

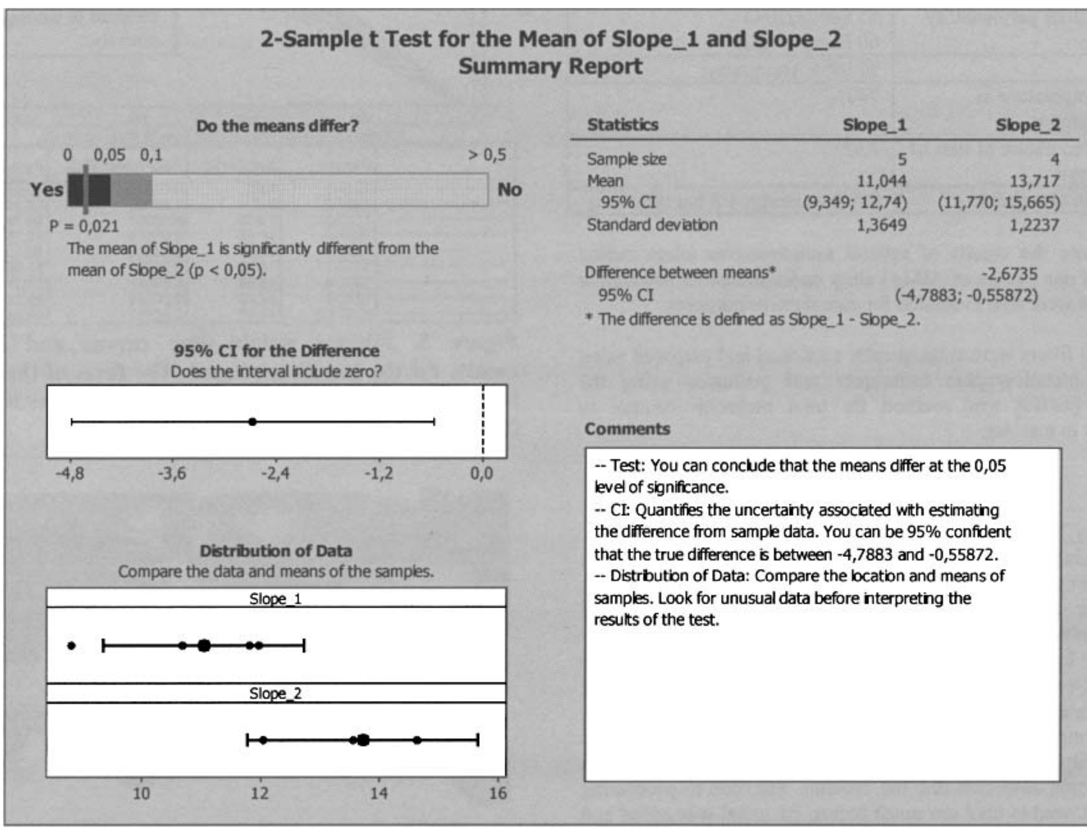


Figure 9. Statistical significance of differences between mean values of the two slopes of the filtrate weight – time curves. Excluding test results for sample 1237-1.

To assess to what degree the differences in the slopes of the filtrate weight time curves are due to variations in the amounts of inclusions present in the melt the filters were prepared and analysed according to the standard PoDFA grid method. The PoDFA results for total inclusion content given in mm2/kg are tabulated in Figure 5 and plotted below in terms of the slope of the individual filtrate weight –time curves. The results from the two groups of filters are indicated separately. No correlation is immediately obvious. The results therefore do not suggest differences between the slopes of the filtrate weight time curves were due to variations in the amounts of inclusions present in the melt in these trials.

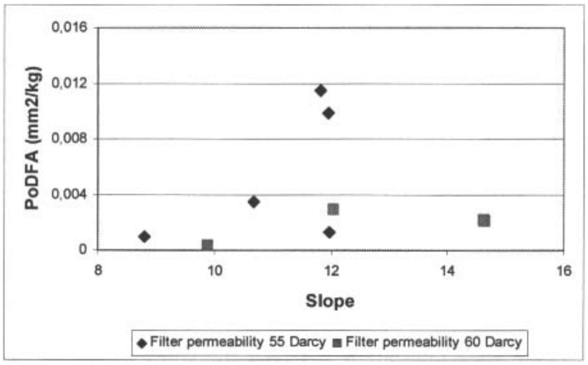


Figure 10. Comparison of the relationship between the slope of the filtrate weight – time curves and the PoDFA results for the same samples.

Comparison of current results with published results
To assess the relevance of the current results a comparison with similar curves obtained from the literature was made as shown in Figure 11. Here lines calculated using the average slopes of the filtrate weight – time curves for the two filter permeability groups are included in the graph mentioned. The difference in slope between the two lines appears to be of a comparable order of magnitude as a measured change in metal quality from for example alloying or holding of the melt[3]. In this context it appears that the differences in the slopes of the curves obtained by using filters of varying permeability is indeed significant and should be considered when interpreting the results of such tests. In the current example the difference between the two calculated curves is larger than the width of the "Prefil world class production window" indicating that the permeability of the filter used for such a comparison could significantly influence whether this melt quality is reached.

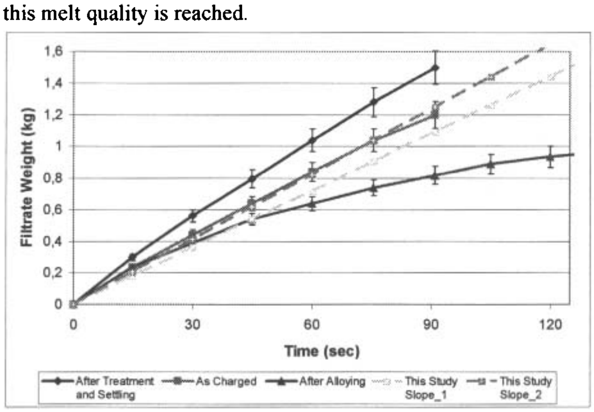


Figure 11. Linear plot of the current results using the average slopes for the two groups of filter permeability superimposed on the graph of published data from Figure 1.

Production Trials
As a further example a single charge of approximately 45 tons of AlMg3 alloy was measured under normal production conditions at various stages of the production process: Melter – Holder – before CFF (after degasser) and after CFF filtration. Figure 12 gives details of the measurement results for this charge including the resulting PoDFA results. The corresponding filtrate weight – time curves are shown in Figure 13.

At each position two samples were taken and evaluated for filter permeability and slope of the filtrate weight – time curve. The PoDFA evaluation was conducted on the same sample as was used to generate the filtrate weight – time curves. A steady decrease in the overall inclusion content of the metal was observed along the various stages of the processing chain. This is in line with expectations and extensive production experience. The filters used for each measurement were selected at random so that the permeability values varied randomly along the process chain.

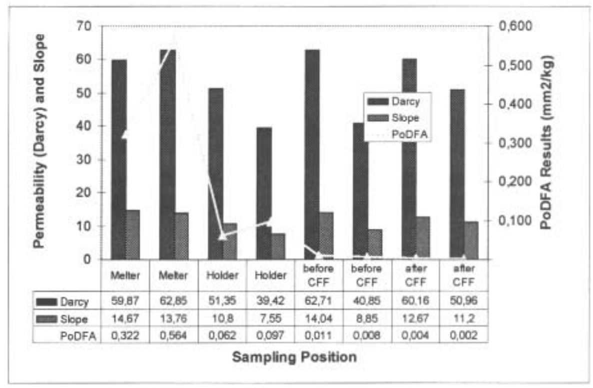
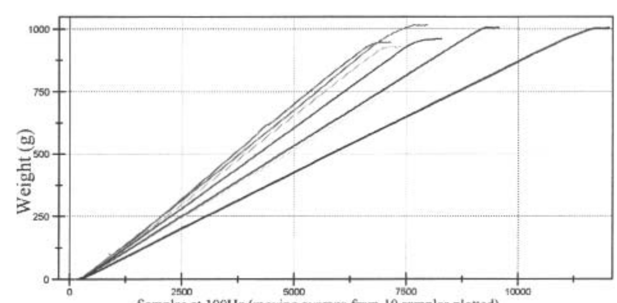


Figure 12. Details of production charge of AlMg3 alloy.



	Sample	RegCoeffB	PoDFA [mm²/Kg]	Perm. [Darcy]
	1236-1	14.67	0.322	59.87
	1236-2	13.76	0.564	62.85
	1236-3	12.67	0.004	60.16
	1236-4	14.04	0.011	62.71
	1236-5	10.8	0.062	52.35
	1236-6	11.2	0.002	50.96
	1236-7	8.85	0.008	40.85
	1236-8	7.55	0.097	39.42

Figure 13. Filtrate weight - time curves for the above charge of alloy AlMg3. Again the form of the curves is essentially linear and the slope of the curves is shown (under RegCoeffB).

Despite the random selection of the filters and hence the filter permeability used for these measurements a strong correlation between the filter permeability and the slope of the filtrate weight – time curve was again observed as shown in Figure 14. A correlation between the slope of the filtrate weight – time curves and the PoDFA results could not however be substantiated as evidenced in Figure 15.

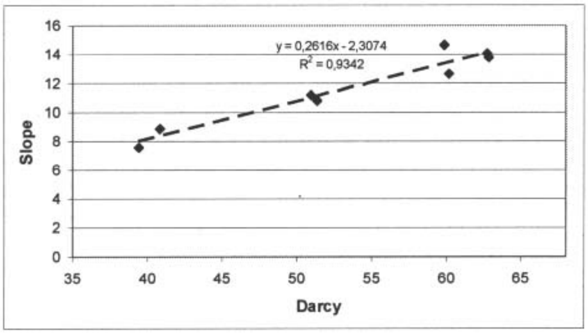


Figure 14. Correlation between filter permeability and the slope of the filtrate weight – time curves.

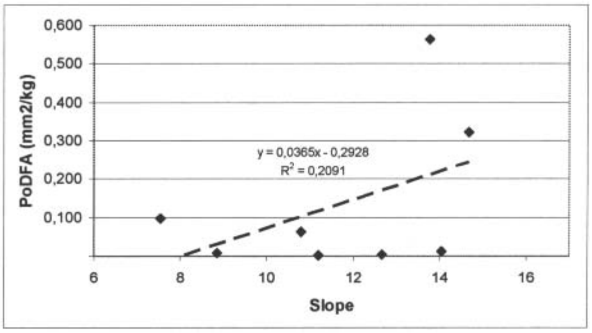


Figure 15. Correlation between the slope of the filtrate weight – time curves and the PoDFA results.

Conclusions

The permeability of several filters has been measured with a purpose built test apparatus. Hot tests with these same filters were conducted and filtrate weight – time curves generated. The significance of the relative roles of the differences in the permeability of the filters used and the amounts of inclusions present in the melt was investigated and the following conclusions made:

- A good correlation between filter permeability and the slope of the filtrate weight – time curve was established under both laboratory and production conditions
- No significant correlation between the slope of the filtrate weight – time curves and their corresponding PoDFA values was established in this study.
- No improving trend in the slope of the filtrate weight – time curve was observed along the process chain melting furnace – casting furnace – in-line treatment for the industrial trials conducted although this was indicated in the PoDFA results
- Care should be exercised in deciding whether to apply such a melt quality evaluation method to melts characterized by very low numbers of inclusions and characteristically linear filtrate weight – time curves

Closing Remarks – Future Work

An improvement to this test for clean melts and linear filtrate weight – time curves may be the introduction of a permeability correction factor to normalize the slope of the filtrate weight – time curves for differences in filter permeability

This method may be well suited to characterizing melts with significantly higher levels of inclusions and filter cake build-up. This type of evaluation would certainly be valuable but was outside the scope of the present work.

References

[1] P. G. Enright, I.R. Hughes, J. Pickering, A. Simard, and J. Proulx, "Characterisation of Molten Metal Quality Using the Pressure Filtration Technique," in *Transactions of the American Foundry Society and the One Hundred Seventh Annual Castings Congress*, Milwaukee, WI; USA, 2003, pp. 193-203.

[2] ABB Analytical, "Pressure Filtration Melt Cleanliness Analyzer," ABB Analytical, Quebec, Product Specification Brochure B4277 2005-11, Nov. 2005.

[3] Alain A. Simard, François Dallaire, Jasmin Proulx, and Paul Rochette, "Cleanliness Measurement Benchmarks of Aluminum AlloysObtained Directly At-Line Using the Prefil-Footprinter Instrument," in *Light Metals 2000*, Nashville, Tennessee, USA, 2000, pp. 739-745.

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