Paul Evans of Technology Strategy Consultants, tsc, and Michael Bryant of MQP, explain and provide insight into the capabilities of web based process analysis service Premetz, newly developed by tsc and distributed by MQP Limited.

Melt quality control now in real time

Premetz brings a totally fresh approach to melt quality. Aluminium casthouse customers can now access, in real time, meaningful and informative information on their melt cleanliness and quality by uploading their Prefil data onto the Premetz website.

We all like an excursion. Whether to our favourite city, or to our favourite beach, or to our favourite restaurant, it’s an enjoyable experience.

However if you are a producer of aluminium products for quality critical applications, such as in the automotive or aerospace industries, the experience of so called “quality excursions” can be anything but enjoyable.

What is a quality excursion?

A quality excursion is when a manufacturing process, which is being closely monitored for quality, is found to have experienced a temporary period where the parameter being measured moved significantly, and unexpectedly, out of specification. An example of this from casthouse aluminium production is as follows:

Metal / melt cleanliness (MC) level was being tracked in an aluminium casthouse over time at a single location, i.e. after the ceramic foam filter (CFF), in the melting process. Figure 1 shows data measured, all for the same alloy, and indicates a generally consistent level of MC, suggesting that the melt quality was also consistent. Typical quality levels were running at an acceptable level of around 10 MC. However, something happened to MC and metal quality for three casts at the end of April 2011 and the beginning of May 2011 which caused MC to increase to well above the acceptable level for the process. This event is known as a quality excursion.

Melt cleanliness and quality

In the example of a quality excursion in Fig. 1, the MC is taken as being a measure of the melt quality. Melt cleanliness relates to the level of non-metallic inclusions present. Even though the concentration of inclusions is typically < 1 ppm, many products are very sensitive to their presence and the low concentration makes it difficult to quantify and identify them. The PoDFA test developed in the 1960s is still widely used to do this. In the PoDFA test a volume of melt is pressurised and passed through a very fine filter. Although 1.4 kg of sample is pumped, the amount of metal effectively being ‘seen’ by the metallographic technique is still only around 5 g.

PoDFA measurements are characterised by large error bars, which increase relatively as the metal gets cleaner. Very dirty metal with a PoDFA level around 1.0 mm²/kg might have a measurement error of ±10%. In contrast, cleaner metal with a level of 0.01 - 0.001 mm²/kg could exhibit a measurement error of ±100% or more. Given the time required for a single analysis (2 hours), very few PoDFA users undertake repeat measurements, and thus often do not appreciate the size of the error.

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As well as predicting the form of a Prefil curve, the software provides a metric describing the melt inclusion content in ‘MC’ units. MC (metal cleanliness) is a parameter arising from PoDFA measurements are characterised by large error bars, which increase relatively as the metal gets cleaner. Very dirty metal with a PoDFA level around 1.0 mm²/kg might have a measurement error of ±10%. In contrast, cleaner metal with a level of 0.01 - 0.001 mm²/kg could exhibit a measurement error of ±100% or more. Given the time required for a single analysis (2 hours), very few PoDFA users undertake repeat measurements, and thus often do not appreciate the size of the error.

Web based service

The Premetz analysis is the basis of a web based service developed by tsc where customers can securely upload Prefil data files for immediate analysis, singly or in batches. Ten companies have tested the service, with more than 1,000 Prefil tests analysed. Over 95% of the uploaded tests were successfully analysed and goodness of fit recorded for all these tests was R² > 0.9997. Essentially all the information available in the Prefil curve is being quantified by these analyses.

Interestingly, the analysis makes no assumption as to the intrinsic permeability of the filter, which is determined by the analysis itself. When this approach was applied to more than 1,000 Prefil tests, it was found that the distribution of permeabilities arising in the commercially available filters was surprisingly very large, typically falling in the range 6 to 20 darcy units, as shown in Fig. 5, where 1 darcy unit is 10-12 m². The average darcy value of filters was 11, with 66% of filters tested having permeabilities in the range 9-13 darcy.

In this testing phase, we have deliberately focussed on wrought aluminium, i.e. metal destined for rolling or extrusion. Most of the analysed tests were found to be at the clean end of the spectrum, with two thirds of recorded results with MC values less than 10. The deliberate intention was to determine whether Prefil and Premetz together had sufficient sensitivity to analyse relatively clean metal, specifically in the regime where PoDFA measurements become seriously inaccurate and unreliable.
This range of measured MC levels in wrought metal, combined with the intrinsic variation in filter permeability, together represent a very significant finding. Fig. 6 shows the weight versus time curves expected for perfectly clean metal (MC<10) passed through filters with permeabilities of 9, 11 and 13 darcy. Also shown is the curve you would see with a metal cleanliness MC=10 passed through an 11 darcy filter.

Clearly attempts to interpret raw Prefil curves in clean wrought aluminium (MC<10) are going to be compromised by the intrinsic variations in the filter properties. Premetz solves this problem by determining both the filter permeability and the metal cleanliness in each analysis. In this regime, a visually observed change in the Prefil curve is as likely to be due to variation in the filter characteristics as it is to be due to changes in metal quality. Premetz solves this problem by determining both the filter permeability and the metal cleanliness in each analysis. It is probably fair to say that without application of an analysis such as Premetz, the basic Prefil test is inappropriate for testing relatively clean aluminium destined for rolling or extrusion.

**Normalisation**

A powerful tool provided on the Premetz web site is normalisation of the data. The test is re-plotted using the measured MC value, but assuming a constant 11 darcy filter as in Fig. 4. The user can then visualise the effects of metal quality alone on the flow curve, and not be confused by the additional variation arising from variation in filter permeability.

A selection of Prefil tests taken over a period of time at one point in a process route is shown in Fig. 7. In this example the raw data (left) shows no discernible pattern in the tests. The normalised data (right) shows clearly that the majority of the tests were indicating very similar quality, and a very consistent MC value, except for two very obvious outliers.

This helps to explain criticisms of the Prefil test when at times the results have seemed to be incongruous. It seems that in these instances correlation between the slope of the Prefil test and the measured filter permeability are being observed, rather than any correlation with metal quality. The Premetz analysis confirms that the basic Prefil slope is dominated by filter permeability, and that whilst the effect of inclusions causes a deviation from that slope, this can be quite small and not easily identified, particularly in clean metal.

Clearly this has a major implication for the use of ‘footprints’ to define an acceptable range of metal quality. The inescapable conclusion about the variability in the filter used in the Prefil test is that it is about the variation in metal quality.

**Interpreting results**

The Prefil test was developed as a means to reduce the amount of effort involved in PoDFA analysis. It was originally thought the flow characteristics of a melt through a filter could be used as a visual indicator of melt quality, and in particular, highlight when a significant quality excursion might have occurred. The intention was that such excursions from a normal flow characteristic would trigger a PoDFA investigation, whereas a Prefil curve falling in the footprint of normal production would avoid the need for such an analysis.

Clearly the preceding results have suggested that such an approach will not work using the basic Prefil test by itself for relatively clean metal, due to the inherent variability of filter permeabilities. However it has also been shown that the use of Prefil to analyse a Prefil test allows information to be extracted from the Prefil curve, successfully separating out the effects of both the filter and metal quality, even in relatively clean metal (MC<10).

The Premetz web site also allows the user to append PoDFA total inclusion counts when available, and will plot the analysed MC (metal cleanliness) value as a function of PoDFA inclusion level.

In general it should be stressed that there is not a universal correlation between the analysed Prefil MC value and PoDFA total inclusion count, nor should we expect there to be. It should be recalled that the MC parameter depends on a number of factors, including both the concentration (volume fraction) of inclusions in the melt, and the permeability of the cake which is produced on the surface of the Prefil filter.

It should not be a surprise that different populations of inclusions will exhibit different permeabilities, and hence different MC values, even for the same volume fraction of inclusions in the melt. A well-known example of this pertains to the ease with which excessive fine inclusions (borides and carbides) can block filters in commercial production. The volume fraction of fines can be small in absolute terms, but they produce a far greater effect on metal flow than might have been expected based purely on concentration. It may be concluded that different types of inclusions, and different combinations of inclusions in a population, may well exhibit different permeabilities, presumably depending whether they can pack down densely in the cake, or whether they maintain an open structure, allowing the melt to continue to flow more freely.

These differences in behaviour are intrinsic to the Prefil test. Prefil and Premetz together provide a robust measurement of the effect of an inclusion population in the melt on the flow characteristics through a filter. A PoDFA test provides a direct measure, however imperfect, of the volume fraction of inclusions present in the melt. The two tests are not measuring the same things, although the flow characteristics should be related to the types and quantities of inclusions in the melt.

In cases where there is generally a consistent type or types of inclusions in the melt, it would be expected that a better correlation might be found between Premetz MC and PoDFA. For example, a series of samples tested at the same location (e.g. exiting a furnace, or after a CFF) might be expected to differ over time in volume fraction of inclusions, but present essentially inclusions of the same type, and hence exhibit a relatively narrow correlation. Figure 8 shows such an example. Fig. 8 also shows best estimates of typical error bars which might be expected in a PoDFA test and which are seen to increase as the metal gets cleaner. It should be stressed that there are very limited data published on errors in the PoDFA test, so the PoDFA errors may be even greater than this.

Evidence suggests that Prefil errors, when analysed by Premetz, are probably around ±1 MC for measurements in the range 1-10 MC, and probably closer to ±10 MC for measurements close to 100 MC, although again controlled repeat test results have not generally been published.

**Casthouse customers**

When first developed, the Prefil test was seen as a means of reducing the amount of time and cost involved in PoDFA analysis. It was thought that the flow characteristics of a melt through a filter, the footprint, could be used as an indicator of melt quality, and in particular, highlight when a significant problem such as a quality excursion might have occurred. As discussed here, results have indicated that such an approach will not work using the basic Prefil test by itself.
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On the other hand it has now been shown that the use of Premetz to analyse a Prefil test allows information to be extracted from the Prefil curve, successfully separating out the effects of both the filter and metal quality, even in relatively clean metal.

Uploading Prefil data files onto the Premetz website provides immediate analysis and enables casthouses to obtain meaningful information on their melt cleanliness and quality in real time. It is possible to obtain a full analysis of a Prefil test within 5 minutes of taking the sample. This means that molten melt cleanliness can be known before the onset of casting, and in particular a quality excursion can be detected, and action taken in the casthouse, to avoid it rather than recording it as an historical event at a later date (see Fig. 1 earlier).

Access to the tsc Premetz web service can be arranged through the company MQP. Customers will have the facility to upload Prefil data singly or in batches for immediate analysis, and MQP is able to offer a data interpretation service.

www.mqpltd.com

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