Advanced Analytical Methodology for Rapid Assessment of Cleanliness

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Summary

The mechanical properties of alloys, and, in particular creep performance, are heavily dependent upon the cleanliness of the alloy where deleterious inclusions can lead to premature failure. In alloys for fusion reactors cleanliness is even more critical where potential in-service activation of the steels may lead to the rejection of the steel prior to installation. The Materials Processing Institute in collaboration with the University of Sheffield, European Technology Development Ltd. and the University of Warwick, are evaluating potential alloys that have higher performance at the temperatures above the limits of the current fusion alloys. The alloys were made at the Materials Processing Institute using vacuum induction melting (VIM). In addition to the chemical composition, the cleanliness was initially evaluated using an in-house method for scanning electron microscopy (SEM). Furthermore, a new analytical method was also created for optical emission spectroscopy (OES) offering a rapid assessment of inclusion distribution. This method was verified against the SEM evaluation. The scope was determined for the future assessment. This study offers a method development approach to streamline the chemical analysis and inclusion assessment of all alloys including those for fusion. In addition to a rapid and cost-effective evaluation, and minimal sample preparation, the method provides an analysis on a larger surface area that can be potentially used for rapid quality assessment.

Background

The application of arc/spark optical emission spectroscopy (OES) is common in steel industry. Although it is a relatively conventional analytical tool, it has gained some developments in recent years such as pulse distribution analysis (OES-PDA) for inclusion analysis [1]. In this technique, single discharge intensities that belong to non-metallic inclusions are identified and analysed. Higher intensities indicate higher concentration of a particular element [2, 3]. In this research, a semiconductor detector technology (CMOS) combined with time resolved capability in a SpectroLAB S system was used to assess non-metallic inclusions in two martensitic boron nitride steel alloys (MarBN alloys). The technique, so-called single spark evaluation (SSE), involves time resolved determination of spectral line intensities (Fig. 1).

Fig. 1. Single spark evaluation (SSE) of alumina inclusions in steels.

❖ Rapid inclusion analysis using OES-SSE technique:

- The SSE chart: Intensity against sparks (in this case, Figure 1, 1600 single sparks were evaluated)
- Determining the discriminator: RSD limits (yellow lines)
- Determining the outlying intensities (red marks showing the outlying sparks)
- Countable inclusions: Outlying intensities at the same spark for all the elements

Objectives

- Materials manufacturing using vacuum induction melting (MarBN alloys)
- Developing an SEM/EDX feature detection method to analyse inclusion distribution (control method)
- Developing a method using SSE technique for rapid inclusion analysis
- Assessment of the SSE method against SEM/EDX large area mapping (control method)

Methodology

Two MarBN alloys were made using vacuum induction melting (VIM). To validate the technique, a designated area (48 fields) was mapped using an in-house feature detection SEM/EDX method prior to the SSE analysis. The SEM/EDX mapping identified inclusion features using the crystallographic parameters and chemical compositions.

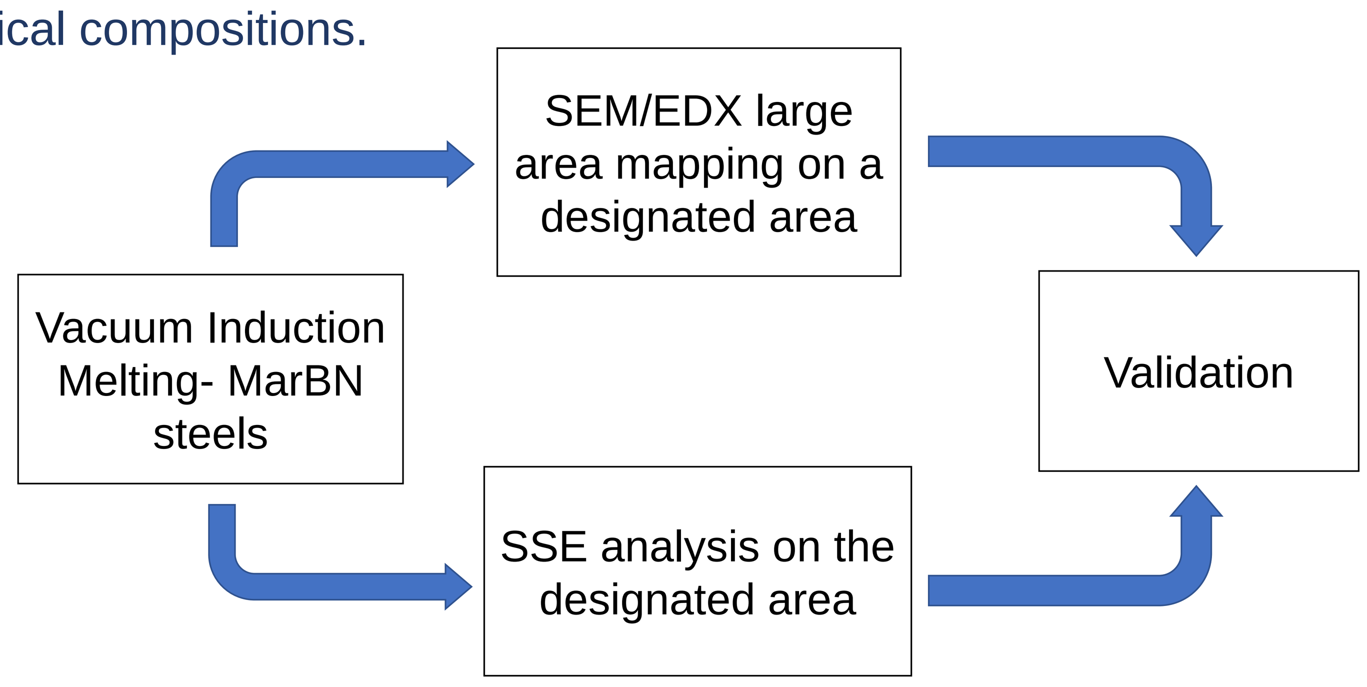

Fig. 2. Methodology chart.

Cleanliness Assessment

To validate the developed method for cleanliness assessment, the number of inclusions from both developed and control methods was compared. For some inclusions in a certain size range, a strong agreement between the SEM/EDX large area mapping and SSE analysis was observed. Figure 3 displays an analysis of sulfides inclusions (MnS) in IBN1 steel sample. Table 1 shows the agreement between the developed SSE method and the SEM/EDX map in counting sulfides inclusions in the size range of 5-20 µm².

- Scanned and spark area: Ø =5 mm
- Inclusion type : MnS (Sulfides)
- Delay start to stabilise the atmosphere: 35 sparks
- MnS inclusions can be detected in the size range between 5 µm² to 20 µm²

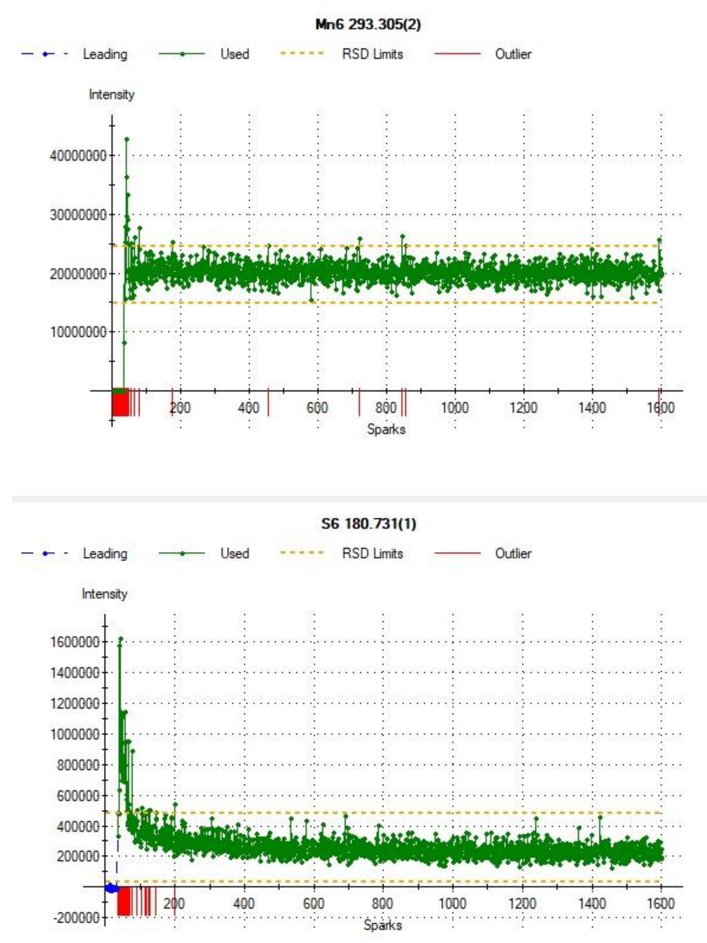

Figure 3. Outlying intensities for the scanned area (IBN1).

Table 1. Agreement between the SEM/EDX data (5-20 µm²)and SSE analysis on the same area.

Alloy	Filtered Area	Sulfides Count (SEM/EDX)
IBN1	5 µm² < and < 20 µm²	11
IRA1		1

Alloy	Sulfides Count (SSE)
IBN1	11
IRA1	1

Conclusion

SSE technique is an alternative method for the conventional inclusion analysis like scanning electron microscopy (SEM/EDX) and optical microscopy (OM). There are some limitations on the dimensions of inclusions due to the limited size of the spark. SSE technique is a rapid tool for cleanliness assessment of alloys. No additional sample preparation is required beyond that already used for OES and the total turnaround time of the analysis is significantly quicker. Although the detection is limited to a size range depending on the inclusion type, a larger area and/or multiple samples of a cast/wrought process can be analysed rapidly. SEM/EDX large area mapping provides highly detailed information on the surface; however, each scan area can be analysed in several hours while SSE analysis takes a couple of minutes.