INCLUSIONS CHARACTERIZATION IN METALS AND ALLOYS BY MEANS OF AUTOMATED ELECTRON MICROSCOPY

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Abstract

The characterization of non-metallic inclusions is of great importance for the production of metals and alloys. The precise determination of their size, abundance, shape and chemistry is of crucial interest to control and to improve production and quality of steel or other alloys. For two years, a combined research team including ERAMET RESEARCH and AUBERT & DUVAL has developed a new characterization method based on automated SEM measurements. This method allows analyzing centimeter-sized samples within a few hours. Each inclusion with a diameter larger than two microns is detected and analyzed. A fast BSE measurement coupled with EDS analyzes, calibrated with a microprobe, enables to establish precisely, for each inclusion, its size, morphology, chemistry and mineral composition. The main advantage of this method, in comparison with the other commercially available applications, is that with increasing inclusion size, the amount of chemical analyses can be increased. It is even possible to determine the compositional variations within large inclusions. In particular, these data are used to plot inclusion compositions on thermodynamically derived phase diagrams. It is also possible to establish the evolution based on variations in inclusion chemistries during a process. This method is efficient, both on samples originating from liquid metal sampling and on forged samples.

Keywords: Material characterization, non-metallic inclusions, automated SEM

1. INTRODUCTION

Non-metallic inclusions in steel and alloys are well known to be directly linked to their physical properties [1, 2]. They can appear during the elaboration process or during the use of materials. As function of their abundance, chemistry, size and shape, these inclusions can be critical for mechanical properties, especially under fatigue solicitations [3]. Thereby, for many years, numerous direct or indirect methods were developed to characterize these non-metallic inclusions with increasing precision. These methods are still mainly operator-assisted and almost entirely based on optical detection [2]. Nowadays, new systems are almost completely automated and allow most of the time to have information on chemical composition [4, 5]. New computational systems can simulate inclusion dispersions in order to improve 3D standard deviation knowledge [6]. The method described here is a novel and fully automated scanning electron microscope (SEM) technique.

2. SAMPLE PREPARATION

In order to be analyzed, samples are prepared as follows: the pieces of metal are cut to be inserted in molds of 30 mm diameter. They are then embedded in an appropriate resin. These mold samples are then polished, first on silicon carbide papers and afterward with diamond suspensions on polishing cloths. Finally, the samples are coated with a 10 to 20 nm carbon layer. This carbon layer is especially useful to analyze big inclusions which are not electron-conducting. This preparation is used for liquid state (lollypop type) as well as forged samples.

3. SEM EQUIPMENT

The SEM device used is a FEI Quanta 650F equipped with a field emission gun (FEG), two Bruker energy dispersive spectrometers (EDS) of 30 mm² each and a Qemscan® (Quantitative Evaluation of Minerals by SCANning electron microscopy) software.

Used sample holders allow analyzing up to 14 samples prepared as described above (**Figure 1**). In the case of bigger samples which cannot be cut, another sampler holder enables to characterize large surfaces of up to 10 cm x 10 cm (**Figure 1**). Both sample holders are equipped with a Faraday cup and standards allowing calibrating BSE measurements.

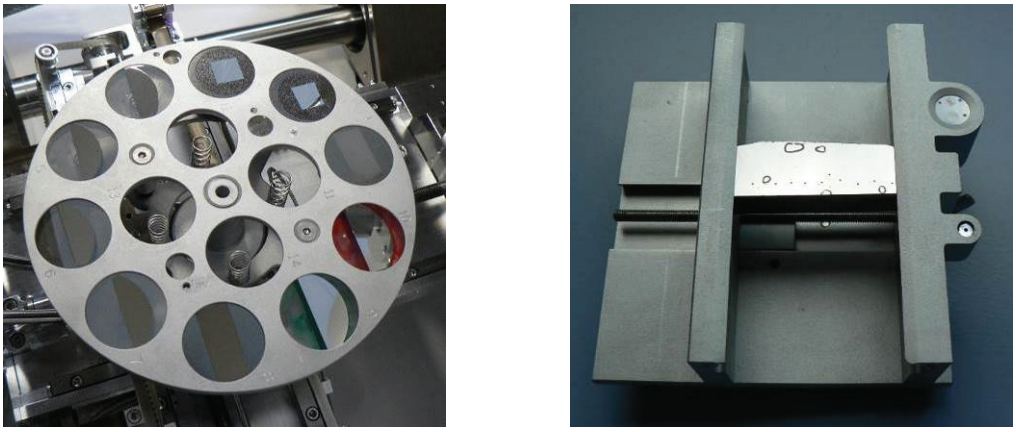

Figure 1 Sample holders used for series analysis (left) or larger blocks characterizations (right)

4. INCLUSIONS CHARACTERIZATION METHODOLOGY

This new automated SEM technique was developed on the basis of the SEM / Qemscan® application. Usually, this software is dedicated to analyze geological samples in the mine industry, coming from drilling cuttings for example. The measurement consists of BSE and EDS automated analyzes on the surface of polished samples. The values measured on each point of the analyzed surface (the step size between two points is adjustable) are compared to data bases which allow to identify the mineral components.

This method was adapted to perform non-metallic inclusion analyzes within a short time (few hours per regular sample), with a high resolution. To reach a good compromise between these two points, the following methodology was used. The sample surface was divided in fields of 1 mm². The number of fields can be adjusted as function of sample sizes. Usually, a surface of 160 or 200 mm² is preferred. Analyses are performed field by field, by using x and y mechanical displacements inside the SEM. Each field is then analyzed with a defined step size (scare grid) which can be also adjusted by using electron beam shift. A step size of 1 µm was used and enables to detect inclusions larger than 2 µm.

As a first step, a fast backscattered electrons (BSE) scan of the entire surface allows to detect the non-metallic inclusions. In most cases these inclusions have a lower BSE level than the metallic matrix. Subsequently, EDS measurements were performed on the detected non-metallic inclusions, with the same step size as defined above.

Unlike most natural minerals, inclusions in metals can have very small crystal sizes. They occur frequently in the form of solid solutions and, in the case of liquid state sampling, they can even be amorphous (**Figure 2**). Thus, an exhaustive database to analyze precisely the inclusion chemistries would be highly complex. In order to facilitate the analyses, it has been decided to calibrate the EDS SEM measurements by microprobe analyses. To this point, well identified inclusions were analyzed both with the automated SEM system and a CAMECA SX100 microprobe.

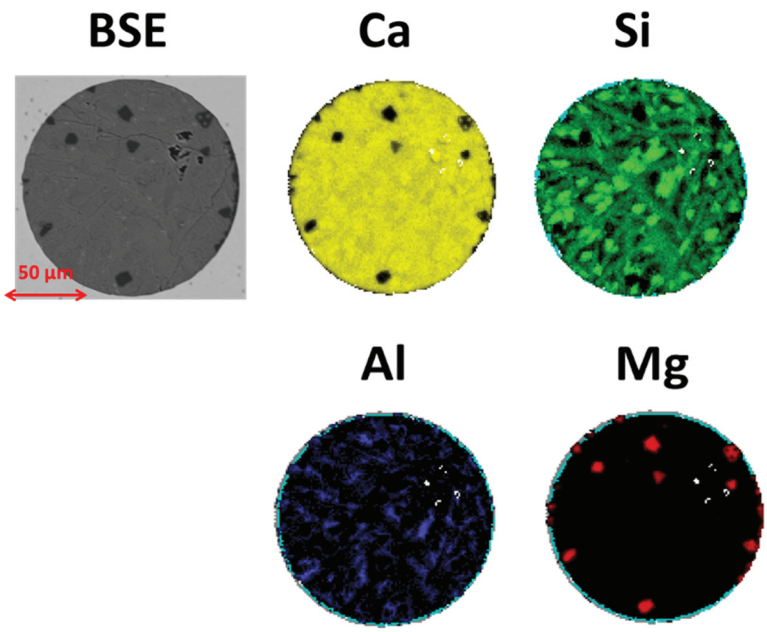

Figure 2 Example of a large inclusion detected in a solidified piece of steel sampled at liquid state

In comparison to other commercialized automated SEM techniques, by means of which only a few EDS measurements are performed on each inclusion, the advantage of the presently applied technique is to obtain a number of analyses directly linked to the surface area of each inclusion. By using the example of the inclusion shown in **Figure 2**, it can be appreciated that only a complete mapping of the entire inclusion surface provide information on the precise variation of each chemical element. Note the rather homogeneous BSE area shown in **Figure 2**, which alone does not allow to differentiate chemistry variations. By using only BSE image for chemistry differentiations and recording only one EDS spectrum for this large area, critical information on element concentrations could be lost.

5. RESULTS

5.1. Size and morphology data

By using the method described above, it is possible to obtain data and information about size and shape of inclusions. On the representation of the non-metallic detected and fully analyzed inclusions (**Figure 3**), each color corresponds to a certain crystalline phase, with a specific composition, defined in the database. For example, in **Figure 3**, the red areas inside the spherical inclusions correspond to magnesium oxide crystallites.

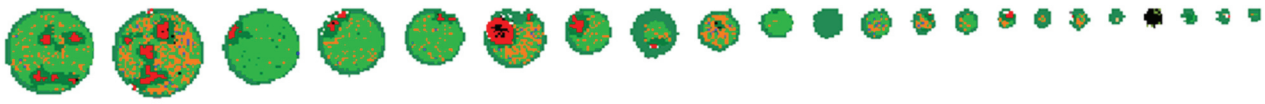

Figure 3 Example of an inventory of non-metallic inclusions detected in a piece of steel sampled at liquid state

The size and shape information can also be numerically extracted and regarded in multi-variant diagrams as shown in **Figure 4**. It is for instance possible to discriminate the influence of large or elongated inclusions. By means of this approach, multi-variant properties can be used to compare in more detail inclusions or inclusion populations.

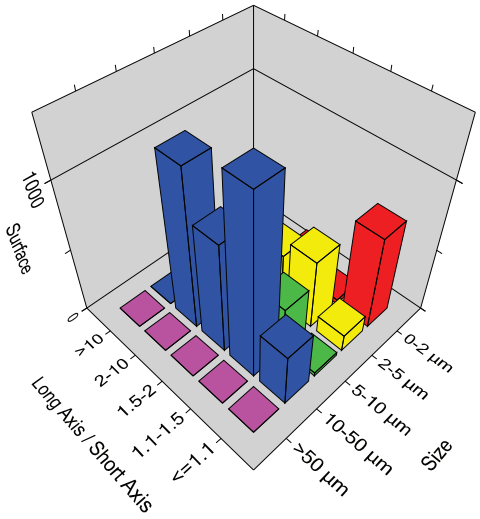

Figure 4 Example of 3D diagram obtained from size and morphology data

5.2. Chemistry and mineral composition

As shown in **Figure 3**, the color code of phases in inclusions can already give an idea of the overall composition. One of the strong points of this method is that all the information recorded for each point of the analysis (*i.e.* each pixel of each non-metallic inclusion) is stored and can be reprocess at any time and as required.

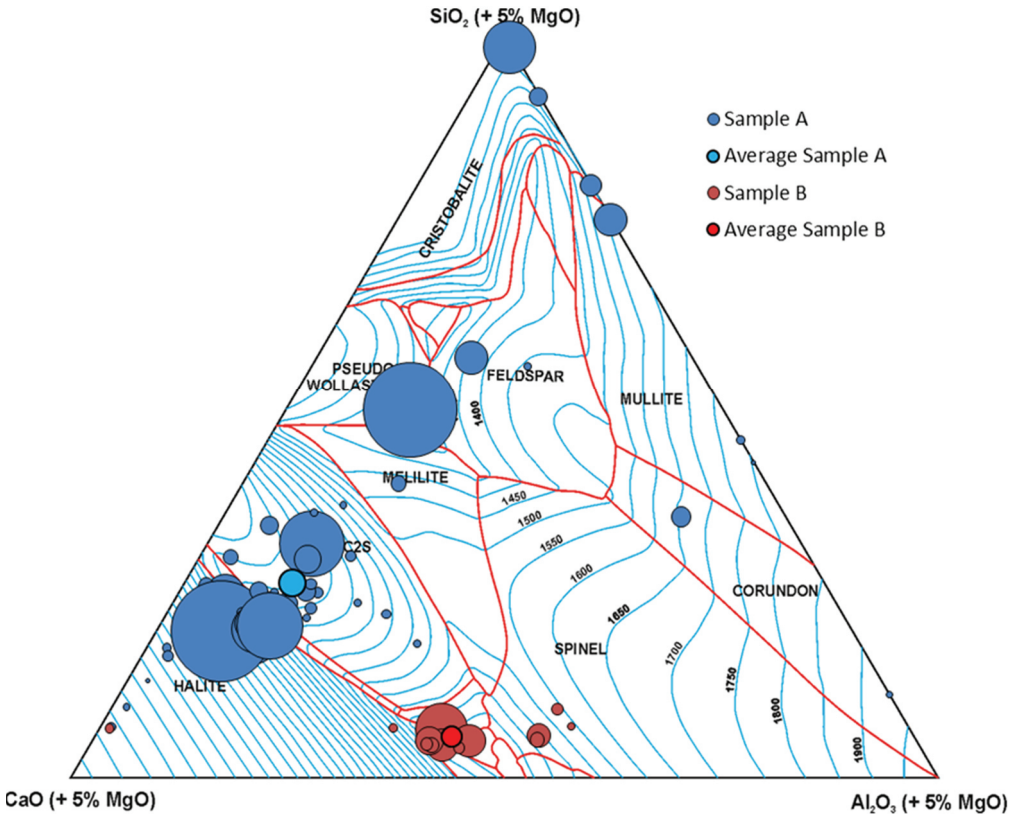

Figure 5 Example of ternary diagram drawn with chemical information obtained for two samples analyzed with this automated technique

As described above, to reach a better precision of the EDS measurements, microprobe calibrations were used. In **Figure 5** an example of a ternary diagram reporting all the non-metallic inclusion chemistries of two samples is presented. The size of each circle corresponds to the size of the inclusion. Such a diagram enables to follow the composition changes of the non-metallic inclusions during a metallurgical process, and therefore to understand their formation mechanisms.

6. CONCLUSIONS

On the basis of a device originally dedicated to geological samples analysis (Qemscan®), a new automated SEM characterization technique was developed to analyze non-metallic inclusions in steels and alloys. This method is based on a fast BSE measurement (non-metallic inclusion detection) coupled with numerous EDS analyzes, calibrated with microprobe analyses. This enables to establish precisely, for each inclusion, its size, morphology, chemistry and crystalline composition. All these pertinent information can be used to control and adjust a metallurgical process by identifying pollution mechanisms or to estimate the physical properties of samples, resulting in production and quality improvements of steel or other alloys.

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