Method of Automatic Characterization of Inclusion Population by a SEM-FEG/EDS/Image Analysis System

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This text describes the system developed to automatically characterize the inclusions observed in polished surfaces of steel samples. A coupling was carried out between a field-emission scanning electron microscope (JEOL, JSM-6500F), an energy-dispersive spectrometer (EDS) (PGT, detector SDD SAHARA) and image analysis software (APHELION). The communication between these elements made it possible to develop software for inclusion measurement. The control functions allow automatic measurements over long periods according to various conditions of observation and analysis. It becomes possible to collect a very large amount of data thus enabling accurate statistical results to be obtained (thousands of analyzed inclusions per day in a clean steel sample). An example is given with improved machinability steels. The knowledge of the distributions and the characteristics of their various inclusion populations permit us to estimate the metal machining behavior.

Introduction

In 2001, ASCOMETAL research centre purchased a field-emission scanning electron microscope. In the list of requirements transmitted to the various suppliers of microscopes, we included the capacity of developing methods of inclusion characterization in an automatic way.

Indeed, until now, no microscope has made possible a complete control of the essential functions (displacement, magnification...) we need for our measurements. However, this demand is essential to obtain precise and statistically exploitable data.

If they are carried out in a manual way, these measurements are often too long and too tedious. For example, it is necessary to measure 300 inclusions to get results with an error lower than 10%, in the case of a homogeneous inclusion distribution. Nevertheless these results are essential for many studies about steels in order to know their properties or to improve the elaboration processes.

Thus, an apparatus running automatically 24 hours per day, and 7 days a week is required.

To achieve this goal, JEOL (microscope), SYNERGIE 4 (EDS system, PGT) and ADCIS (image analysis system APHELION) have collaborated in the development of the control functions necessary for the application to inclusion characterization.

The expected functions, defined in the specifications, were added to the APHELION software as ActiveX components.

The application of inclusion characterization was then developed in VISUAL BASIC.NET based upon the APHELION ActiveX components.

The system **(Figure 1**) is more thoroughly described below. The next section is devoted to the benefits provided by the use of a scanning electron microscope compared to the use of an optical microscope. The second section introduces the list of the functions developed for the SEM-FEG/EDS/Image analysis system. Lastly, an example is provided to highlight one among the many possibilities of the system.

Description of the System

Advantages of a field-emission scanning electron microscope

The main limits of the inclusion characterization made by optical microscopy are:
- not very sensitive to the type of inclusion (the morphological criteria are often insufficient),
- the problems involved in the artifacts (dust, scar...),
- the problems involved in the poor depth of field.

Fig. 1 SEM-FEG/EDS/IA system.

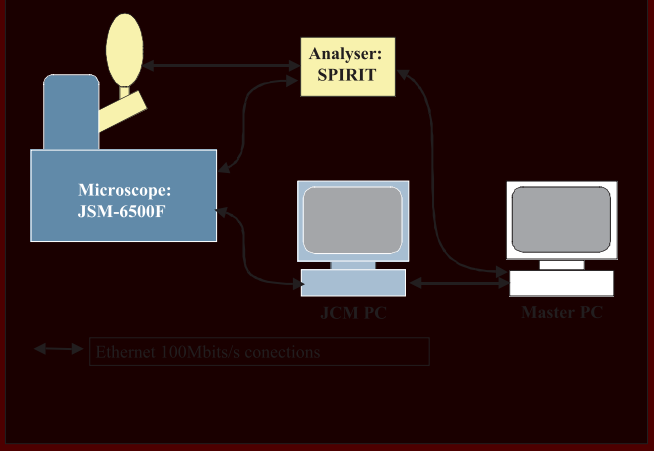

Fig. 2 General diagram of the system.

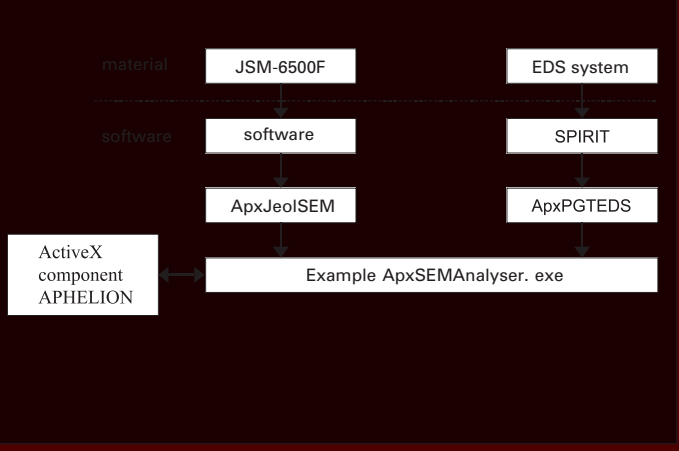

Fig. 3 General structure of the system.

From this non exhaustive list, the specifications of the ideal system for inclusion characterization are:
- signal stability to obtain comparable images,
- stage control to be able to scan a sufficient area in order to obtain statistically representative results,
- magnification control to obtain accurate inclusion sizes whatever the size of the inclusion,
- chemical contrast to discriminate the various inclusion phases,
- chemical composition to discriminate the various inclusion populations,
- resolution suited to detect inclusions smaller than a micrometer,
- improved depth of the field to maintain the image clarity.

We observe that the JEOL JSM-6500F field-emission scanning electron microscope provides these specifications. In particular, its beam can operate for hours without any signal variation. The accuracy of the motorized stage motion allows to detect and to observe the inclusions at various magnifications.

The control functions of the field-emission scanning electron microscope

The general structure of the application is illustrated in **Figure 2**.

The equipment contains two PCs. One is dedicated to the control of the electron microscope. The second, on which SPIRIT software (analysis) and APHELION software (image analysis) are installed, controls the totality of the system. The inclusion characterization application is installed on this PC. The images acquired by SPIRIT are used in the application. The PCs and the various apparatus intercommunicate via Ethernet 100 Mbit/s connections.

Figure 3 illustrates in a simplified way the general structure of the application. It uses the functions of the microscope software, the analysis system (SPIRIT) and the ActiveX components of the image analysis software (APHELION).

The totality of the functions intervening in the application is listed in the following paragraphs a, b and c. The parameters manipulated by these functions are accessible in reading and writing.

a) Control functions of the JSM-6500F microscope:

- X, Y, Z axes movements,
- Rotation of the stage,
- Beam current value,
- Accelerating voltage,
- Magnification,
- Brightness,
- Contrast,
- Autofocus,
- Choice of the detector,
- Data backup of the initial conditions.

b) Control functions of the SPIRIT analyser:

- Positions (x,y) of the measured spectrum,
- Acquisition time of the spectrum,
- Acquisition mode of the spectrum,
- Data backup of the spectrum,
- Choice of the elements to be measured,
- Measurements on the selected elements,
- Automatic search of the elements on the spectrum,
- Size of an image (512, 1024, etc),
- Position and zoom factor,
- Time by pixel,
- Image mode,
- Number of frames to be added,
- Data backup of the image.

c) Image analysis functions:

- Measurement of grey levels values,
- Operators on the images,
- Possibility to use several images,
- Multiple thresholds,
- Morphological functions,
- Segmentations,
- Operators on the binary images,
- Measurements of the parameters.

These functions are necessary for the development of the general application of the characterization of inclusion populations.

General algorithm

The chain of the following principal actions constitutes the algorithm of the method.

Input data:
1 - Choice of the method,
2 - Identification of the samples,
3 - Positions of the samples on the stage,
4 - Positions of the zones of measurements,
5 - Method of focus,
6 - Method of scanning,
7 - Choices of the measurement options,
8 - Method of threshold,
9 - Criteria of chemical analysis,
10 - Backup files.

Measurements:
1 - Acquisition of an image,
2 - Image processing,
3 - Measurements of the parameters,
4 - Saving of the results.

Data processing:
1 - Treatment of the results,
2 - Final documents.

Performance of the system

The application allows to optimize the use of the electron microscope. Some studies require long analyses. They can last several tens of hours. They can be carried out without human intervention thanks to the system set up.

More than 100 inclusions are characterized per hour on a sample with a low inclusion density. Morphological and analytical measurements of each inclusion are saved in a results file. The data are then post-treated according to the nature of the results, adapted to the needs.

We distinguish measurements and treatments to allow multiple exploitations of the results files.

An example of application is proposed in the following paragraph.

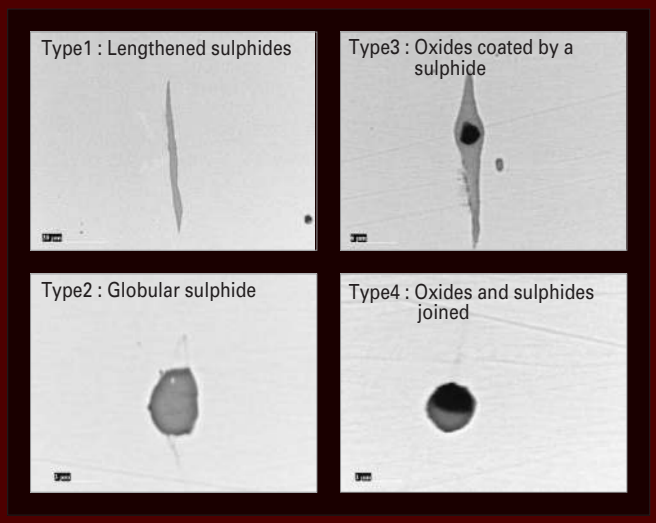

Fig. 4 Inclusion populations.

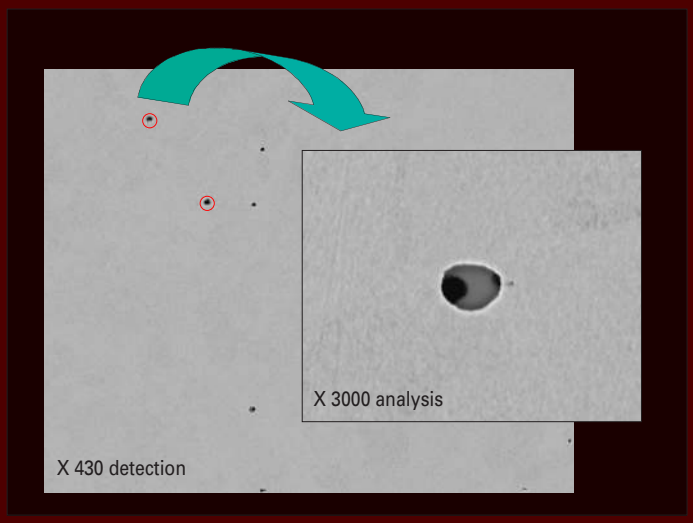

Fig. 5 Inclusion detection.

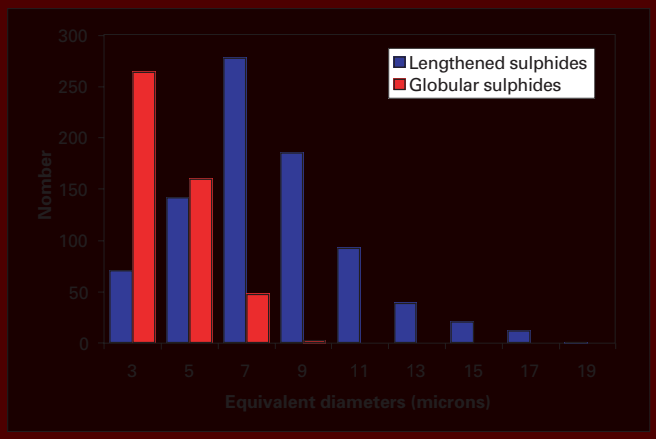

Fig. 6 Histogram of equivalent diameter of sulphides.

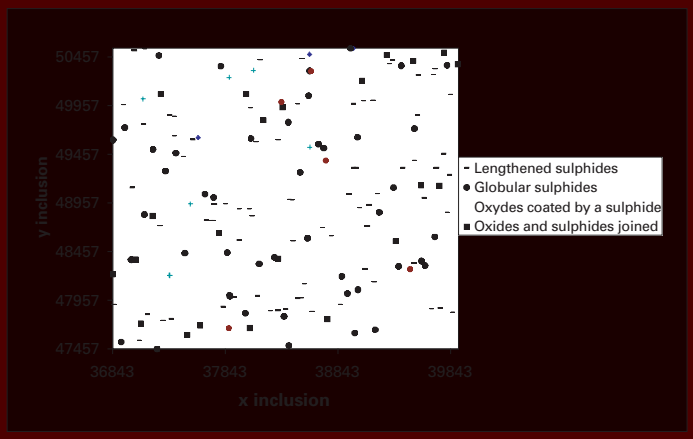

Fig. 7 Inclusion positions.

Application: Characterization of VITAC 3000 Steels

VITAC 3000 steels are steel grades subjected to a specific treatment during casting. The goal is to obtain inclusions whose chemical nature, density, size and distribution improve the operations of machinability by limiting the wear on the tools. If one wishes to qualify castings, we can define a method of measurements adapted to the characterization of the various types of inclusions.

The various types included in these grades of steels are (**Figure 4**):

- lengthened sulphides,
- globular sulphides,
- oxides coated by a sulphide,
- oxides and sulphides joined,
- oxides.

The method must allow to distinguish precisely oxide and sulphide phases. It is also necessary to obtain data on the chemical nature of oxides and sulphides.

So, we define a method using the scanning electron microscope and its chemical analysis system. The sample is observed with a scanning magnification which makes it possible to detect and to measure inclusions larger than 3 micrometers in size (**Figure 5**). This magnification is chosen so that enough surface is observed in a reasonable time. In the method used, this magnification is equal to 430, which corresponds to 11 images per mm_.

The defined threshold permits to segment the image. The sizes of the objects are measured on the binary image obtained.

The objects whose size is sufficient (small diameter larger than 3 micrometers) are analyzed, automatically (Figure 5). Under these conditions of observation, the measurement accuracy is not sufficient. So, each selected object is observed with a higher magnification. This magnification of analysis is higher than 2000 (defined during the setting in data). The stage moves so that each selected inclusion is placed in the centre of the image acquired.

The morphological inclusion parameters and the various phase compositions are then measured. A threshold with two levels permits to isolate oxide and sulphide phases. If the inclusion width is larger than 3 micrometers (the greatest measurement accuracy allows to determine it), a chemical analysis of the different phases is made. The analysis time is equal to a few seconds. These measurements are made for each phase either in the geodetic center of the objects or by a scanning on the area.

Measurements are obtained from a sufficient area of the surface to reach the quality of results required from the statistical point of view.

The data file about inclusion parameters is then treated in an automatic way in order to determine the characteristic values of the various inclusion types. For examples, **Figure 6** represents the histograms of the equivalent diameters of lengthened sulphides (type 1) and of globular sulphides (type 2). In **Figure 7**, one can observe the positions of inclusions which were detected on the observed surface.

Conclusions

The inclusion characterizations of steels are essential to classify the products or for the improvement of the processes of the steel plant.

When precise data about small size inclusion populations are expected, it is then necessary to analyze a significant area of a polished sample to attain a satisfactory precision in the results.

To achieve this goal, the automation of the scanning electron microscope coupled to an EDS (energy-dispersive spectrometer) system and a very complete software of measurements were developed. This software permits fast and precise measurements of a very significant number of inclusions considering the measurement conditions defined by the operator beforehand.

Indeed, having thorough input data makes it possible to describe all the methods that can be used in our domain. The automated application gives an optimal use of the microscope and its analysis system.