

Ultra-Fast Inclusion Analysis with Spark-OES

– Thermo Scientific ARL iSpark, the All-in-One Steel Analyzer

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Introduction

The inclusion analysis methods extend the capability of the Thermo Scientific ARL iSpark OES spectrometer for the analysis of solid steel samples much beyond usual elemental concentration. It allows performing various types of inclusion related determinations in the time of a standard OES analysis.

This offers unequalled perspectives for controlling the steel production on-line.

Benefits

The main benefits of using the Spark-DAT inclusion analysis methods of the ARL iSpark Metals Analyzer are the following:

- **Inclusion information is available during the production of steel.** The inclusion analysis which is performed in combination with the classical spectrochemical analysis does not extend the time needed for the traditional OES analysis.
- **Drastic reduction of investment costs.** The inclusion analysis is performed with the ARL iSpark Metals Analyzer, used also for production process control
- **Extremely short time for inclusion analysis.** A two-runs combined analysis takes about 1min and is therefore possible for more than 30 samples per hour
- **No additional cost or time for operations.** Sample and its preparation, maintenance and service operations are the same as for a standard OES instrument

Principles

Spark-DAT inclusion analysis methods are analytical methods based on specialized algorithms included in the analytical software OXSAS. With Spark-DAT the single intensity values that are generated by the single sparks are acquired separately on the channels of the inclusion elements. Fast algorithms are used to calculate the values corresponding to the information of interest.

The intensity of a Spark-DAT signal depends on the composition of the sample at the position struck by the corresponding single spark. If the spark hits a sample area containing an Al based inclusion (e.g. Al_2O_3), the outcome is an intensity peak, because the Al concentration is much higher than in the metal matrix (FIGURE 1).

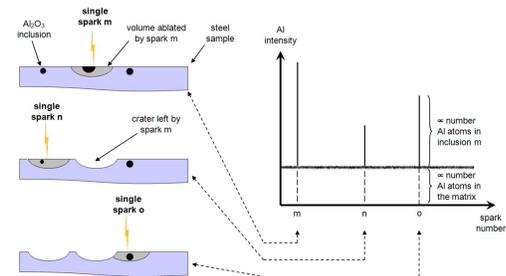


Figure 1. Principle of Spark-DAT illustrated for a steel sample containing Al_2O_3 inclusions of different sizes.

The intensity of the baseline signal is proportional to the number of Al atoms dissolved in the metallic matrix and the intensity of a peak depends on the amount of Al atoms contained in the inclusion(s) ablated by a single spark.

Consequently, the number of peaks is related to the number of such inclusions and their intensity to such factors as the inclusion size and the concentration of Al in the inclusion.

Practical details

Sample preparation

The standard OES sample preparation can be used for Spark-DAT inclusion analysis. However, with paper grinding, the paper should be chosen in order to avoid any influence on the analysis of the inclusions of interest (e.g. using SiC paper when Al_2O_3 inclusions have to be analyzed). For advanced, quantitative Spark-DAT applications, milling is advisable.

Analysis time

Combined inclusion and elemental concentration analysis are performed in slightly more than 20s for a single measurement, i.e. the time needed for the standard OES analysis. This makes inclusion analysis extremely attractive in the context of production, where costs significantly depend on analysis time.

Examples of inclusions analyzed

Various types of endogenous and exogenous inclusions may be observed in steel, e.g. oxides (Al_2O_3 , MgO , CaO , MnO , TiO_2 , SiO_2 ...), spinels (Al_2O_3 - CaO , Al_2O_3 - MgO ...), sulfides (CaS , MnS , AlS ...) and many others.

Counting inclusions

The algorithm **Peaks** allows the evaluation of the number of inclusions by counting intensity peaks on the channels of elements present in inclusions. A peak is normally defined as an intensity signal $I_{peak} > m + 3 \cdot SD$, where m is the mean intensity of the element dissolved in the matrix and SD its standard deviation.

With Peaks, steel samples can easily be classified as clean or dirty according to the number of peaks counted on the channels of the inclusion elements.

The algorithm **Composition** allows counting peak coincidences, i.e. peaks appearing simultaneously on the channels of several elements consecutively to a single spark. Coincidence of peak on Ca and S channels means that the two elements are part of the same inclusion, typically a CaS inclusion (FIGURE 2).

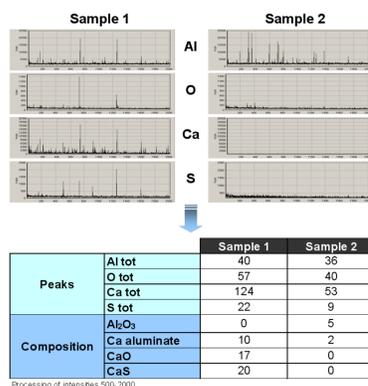


Figure 2. Analysis of two low alloy steel samples with the algorithms Peaks and Composition.

Qualitative inclusion size analysis

Knowing the size of the inclusions is important, since large inclusions are normally more detrimental to steel quality. The algorithms Peaks and Composition can be used in order to count inclusions (peaks) belonging to different size (intensity) classes. Setting the threshold at 3-SD allows counting all the inclusions that are large enough to be detected. Setting it higher, for example at 9 or 15-SD, allows counting inclusions with larger size. Calculating the inclusions between consecutive threshold values provides the number of inclusions in the size class that they delimit. (FIGURE 3).

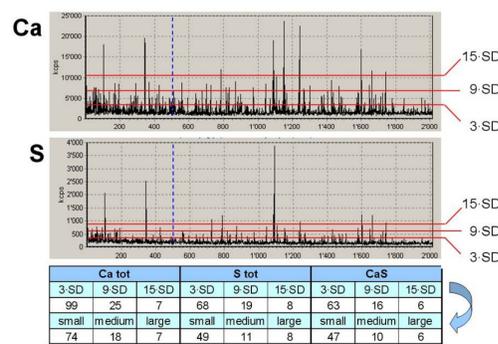


Figure 3. Peaks (Ca tot and S tot) and peaks coincidences (CaS) counted at three different threshold values and corresponding numbers in classes small, medium and large.

Quantitative inclusion size analysis

Traditional methods to determine inclusion sizes (e.g. SEM/EDX) are extremely time-consuming, taking typically 2-4h per analysis in a very competent laboratory.

The algorithm **QuIC** (Quantification of Inclusion Content) enables quantitative analysis of inclusions size and size distribution. With QuIC, the average equivalent spherical diameters (ESD) of different inclusion types and size classes can be calculated. FIGURE 4 presents an example of size distribution diagram of Al_2O_3 , based on the application of the QuIC algorithm and size calculation method for several production samples. Note the horizontal axis unit in μm and the vertical one in inclusions per cubic mm.

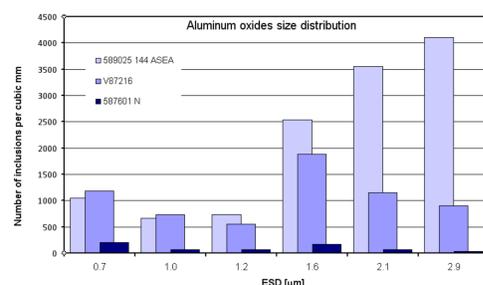


Figure 4. Size distribution diagram of Al_2O_3 in three low alloy steel samples built up on results from QuIC. Horizontal axis: average ESD. Vertical axis: number of inclusions per cubic mm.

Quantitative determination of total oxygen content in killed steels

Oxygen at very low concentration in steel is normally analyzed using dedicated combustion analyzers, due to the relatively low sensitivity of its OES analytical line.

In killed steels most of the oxygen is insoluble, i.e. present in the form of inclusions. The total oxygen content can therefore be calculated from the oxygen contained in the inclusions measured with QuIC, i.e. without using the oxygen optical channel (indirect oxygen determination).

This method is extremely quantitative below 50ppm, as demonstrated in FIGURES 5 and 6. For higher oxygen concentrations, the standard (direct) analysis with the oxygen channel may be the preferred method.

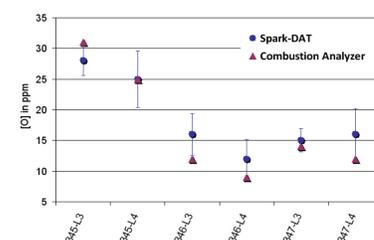


Figure 5. Oxygen concentration in low alloy steel samples taken in the continuous casting mould obtained with QuIC and by combustion analysis (samples and combustion results with permission of R. Dumarey and F. Medina, from ArcelorMittal, Gent).

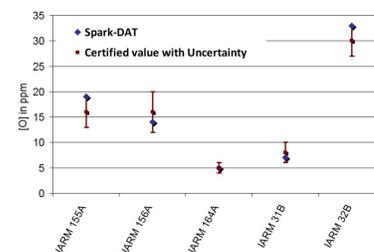


Figure 6. Oxygen concentrations obtained with the algorithm QuIC in certified low alloy steel reference materials.

Combined analysis

As mentioned above, elemental concentration and inclusion analyses are normally performed and their results displayed simultaneously (FIGURE 7).

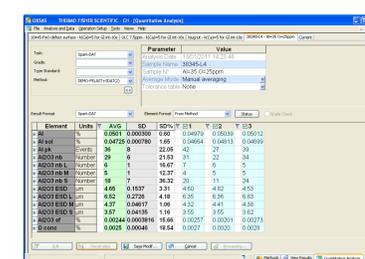


Figure 7. Results of a three runs analysis of a low alloy steel sample in OXSAS analytical software. Results in concentration and from Spark-DAT algorithms Peak, Composition and QuIC are mixed.

References

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Acknowledgements

We are indebted to Rolf Didriksson from SSAB (Oxelösund) for enlightening discussions. We acknowledge Ronny Dumarey and Frank Medina from ArcelorMittal (Gent) for extremely valuable help and discussions, and for giving permission to publish the results obtained on their samples.

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