Ultra-Fast Inclusion Analysis with Spark-OES
Thermo Scientific ARL iSpark, the All-in-One Steel Analyzer
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INTRODUCTION
Analysis of non-metallic micro-inclusions extends 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 unequaled perspectives for controlling the steel production on-line.

BENEFITS
The main benefits of using the Spark-DAT inclusion analysis methods of the ARL iSpark 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.

• Low investment costs. The inclusion analysis is performed with the ARL iSpark OES spectrometer used for process control in steel production.

• Extremely short time for inclusion analysis. Analysis results combined analysis takes about 1 minute and is therefore possible for more than 30 samples per hour.

• No additional cost and time of operations. Sample and sample 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 OXAS. 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 on-line 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 single spark. If the spark hits a sample area containing an inclusion (e.g. Al2O3), 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 Al2O3 inclusions of different sizes.

The intensity of the baseline signal is proportional to the number of Al atoms dissolved in the 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 factors like inclusion size and 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 contamination that can influence the analysis of the inclusions of interest (e.g. using SiC paper when Al2O3 inclusions have to be analyzed). For advanced, quantitative Spark-DAT applications, milling is advisable.

Analysis time
Counting inclusion and elemental concentration analysis are performed in slightly more than 20 s 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 (Al2O3, MgO, CaO, MnO, TiO2, SiO2...), spinels (Al2O3•CaO•Al2O3•MgO•...), sulfides (CaS, MnS, AlS...) and many others.

COUNTING INCLUSIONS
Our algorithms allow 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 higher than m + 3·SD, where m is the mean intensity of the element dissolved in the matrix and SD its standard deviation.

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

Our algorithms also allow counting peak coincidences, i.e. peaks appearing simultaneously on the channels of several elements consecutively to a single spark. Coincidence of peaks 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

QUANTITATIVE INCLUSION SIZE ANALYSIS
Knowing the size of the inclusions is important, since large inclusions are normally the most detrimental to steel quality. The algorithms 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 intensities 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 define. (FIGURE 3).

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

QUALITATIVE 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 advanced algorithms, 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 advanced algorithms and to combustion analysis (samples and combustion results with permission of R. Dumary and F. Medina, from ArcelorMittal, Gent).

REFERENCES