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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 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 are the following:

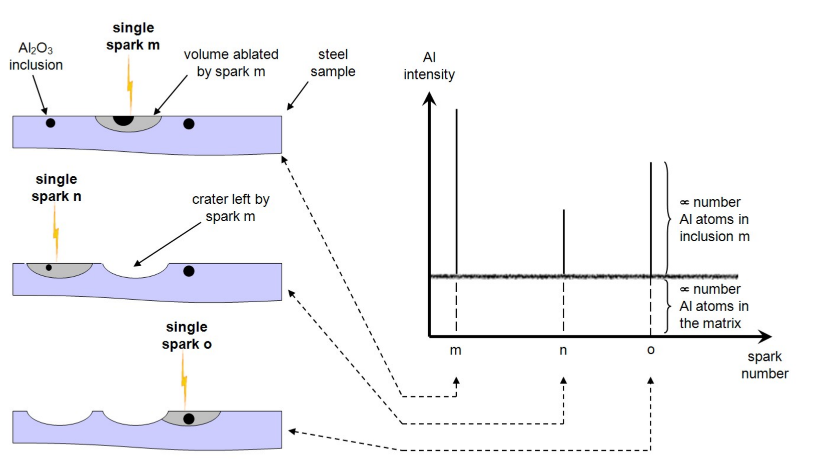
- Inclusion information is available during the production of steel. The inclusion analysis which is performed in combination with the classical spectrochemical analysis does not extend the time needed for the traditional OES analysis.
- Low investment costs. The inclusion analysis is performed with the ARL iSpark OES spectrometer used for process control in steel production
- Extremely short time for inclusion analysis. A two-runs 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 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 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 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 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 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 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 (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.

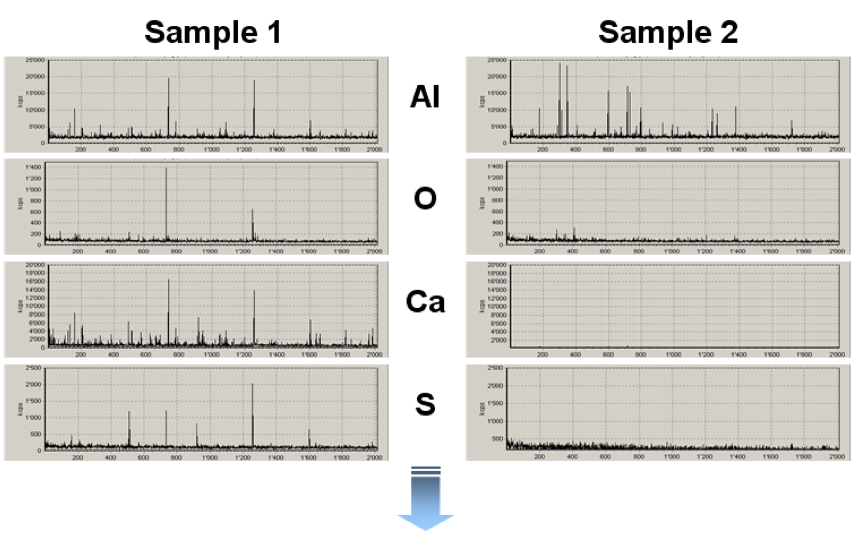
COUNTING INCLUSIONS

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 $I_{peak} > m + 3 \cdot SD$, 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



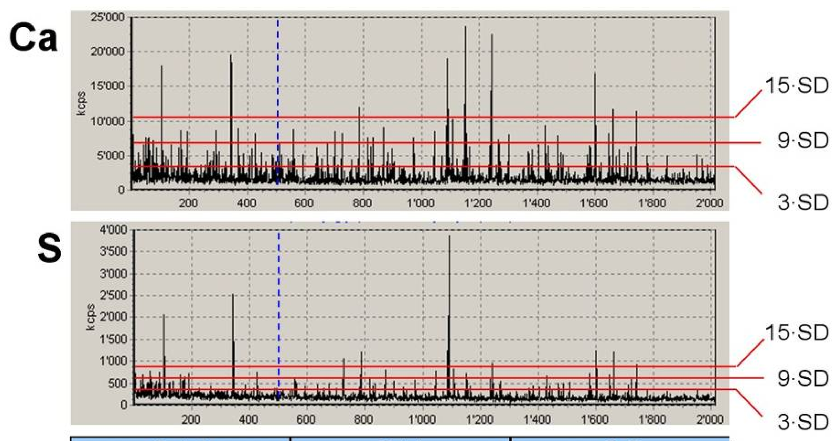
		Sample 1	Sample 2
Peaks	Al tot	40	36
	O tot	57	40
	Ca tot	124	53
	S tot	22	9
Composition	Al_2O_3	0	5
	Ca aluminate	10	2
	CaO	17	0
	CaS	20	0

Processing of intensities 500-2000

QUALITATIVE 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 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 peak coincidences (CaS) counted at 3 different threshold values and corres- ponding numbers in classes small, medium and large



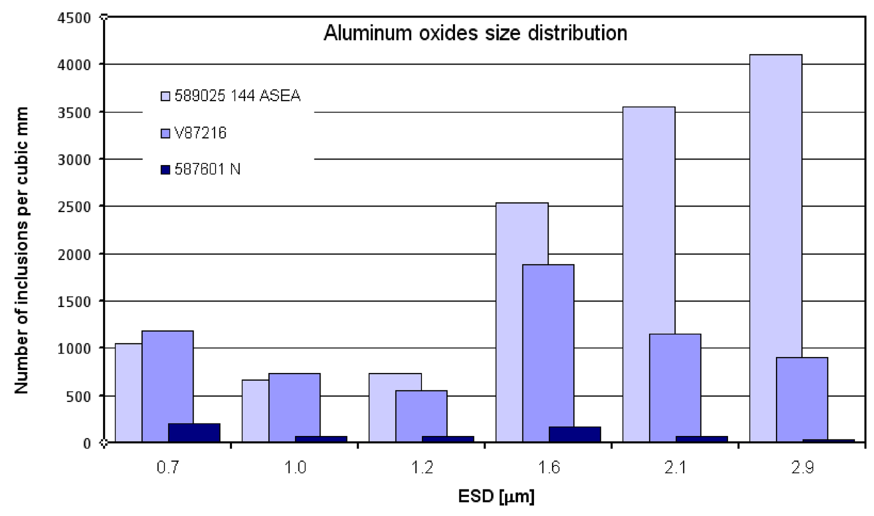
Ca tot			S tot			CaS		
3·SD	9·SD	15·SD	3·SD	9·SD	15·SD	3·SD	9·SD	15·SD
99	25	7	68	19	8	63	16	6
small	medium	large	small	medium	large	small	medium	large
74	18	7	49	11	8	47	10	6

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.

Furthermore, our advanced algorithms enable quantitative analysis of inclusions size and size distribution. 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 such 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 advanced algorithms. 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 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 by combustion analysis (samples and combustion results with permission of R. Dumarey and F. Medina, from ArcelorMittal, Gent)

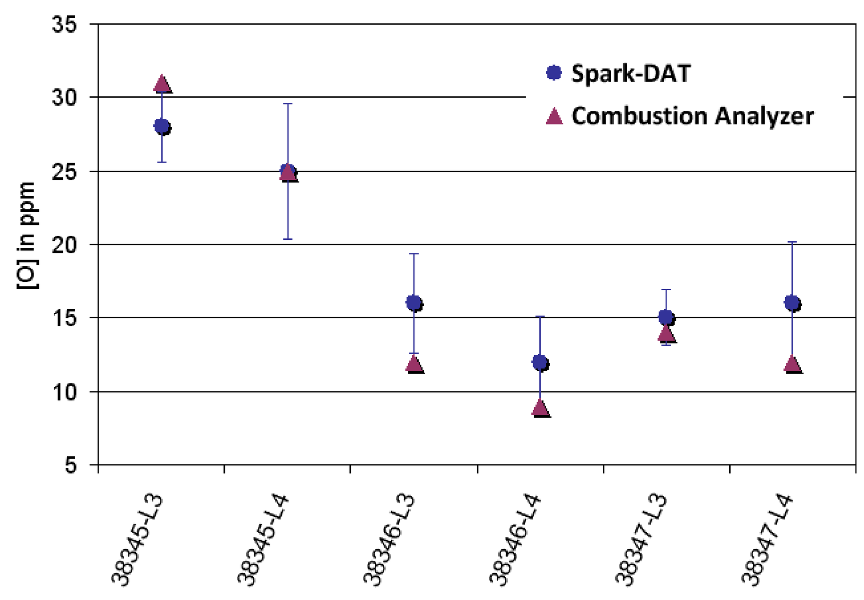
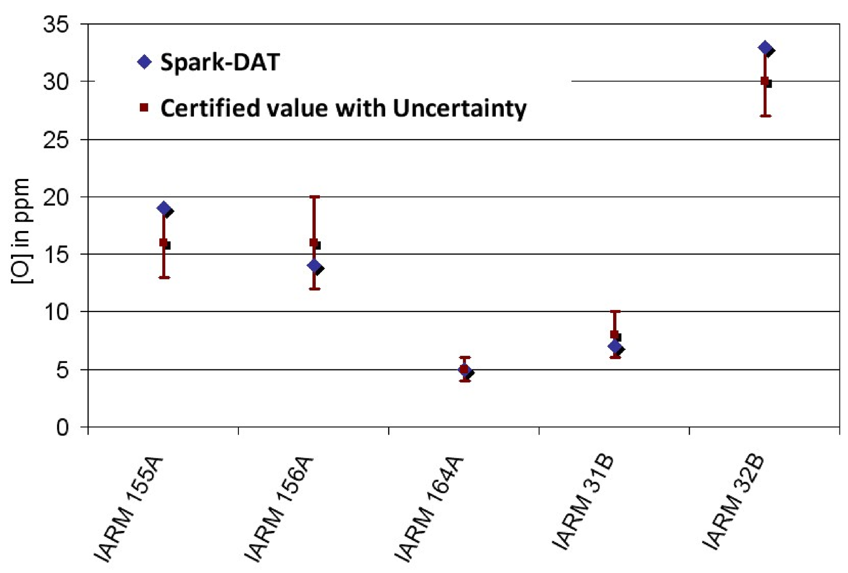


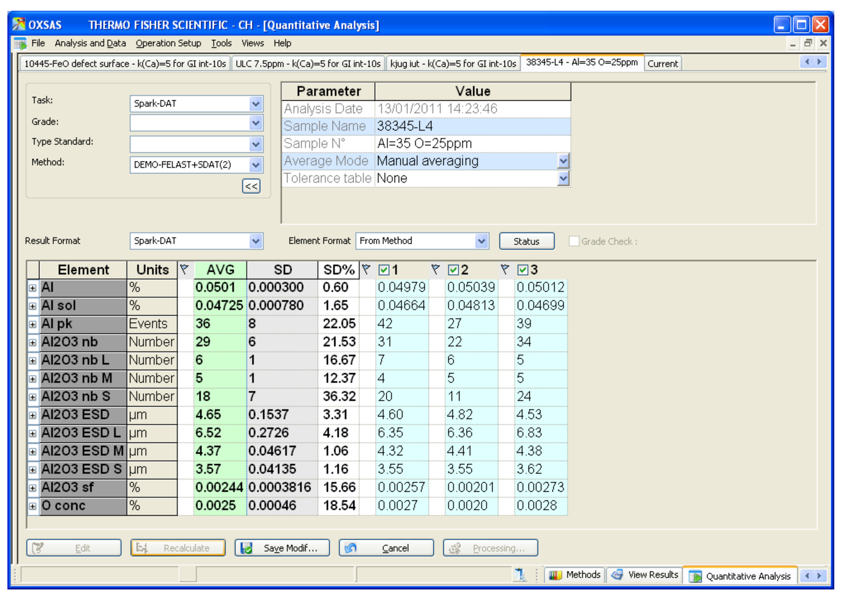
Figure 6. Oxygen concentrations in certified low alloy steel reference materials



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 are mixed.



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