Ultra-fast analysis of micro inclusions in steel with Thermo Scientific ARL iSpark Metal Analyzers – Advanced Inclusion Analysis

Dr Jean-Marc Böhlen, Thermo Fisher Scientific, Ecublens, Switzerland

Key Words

ARL iSpark, Micro inclusions in steel, Process control, Quantitative size distribution, Steel cleanness, Total oxygen

Goal

Perform ultra-fast analysis of micro inclusions in steel including quantitative determination

Introduction

Optical emission spectrometry (OES) is a fast, easy-to-use and cost-effective analytical technique used for elemental analysis of solid iron and steel samples in various contexts, from production to recycling and from foundries to service laboratories. The Thermo Scientific™ ARL iSpark™ Series metals analyzer is a high-performance OES spectrometer platform delivering the ultimate precision and accuracy for the analysis of iron and steel from trace to alloying element levels.

The Spark-DAT (Spark Data Acquisition and Treatment) methods considerably extend its capability beyond the spectrochemical analysis by also enabling ultra-fast inclusion analysis. In the steel industry, these methods enjoy growing popularity, in particular for their ability to provide information about the inclusions during the steel elaboration process.

The principles of the Spark-DAT analysis and its basic applications are presented in another application note (AN41243) on the Standard Inclusion Analysis option. The Advanced Inclusion Analysis option includes the application of this option and the advanced applications presented here further extend its capability, making it able of performing quantitative determination of size or size distribution of inclusions and quantitative analysis of oxygen in killed steels at concentrations even lower than 30ppm.

Benefits

A single instrument, the ARL iSpark metals analyzer with optional Spark-DAT methods, is capable of performing inclusion analysis in addition to the analysis of elemental concentrations, allowing considerable reduction of the investment cost. Sample preparation, maintenance and service remain the same as for the standard ARL iSpark spectrometer, ensuring minimal operation cost and time.

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The combined analysis of elemental concentrations and inclusions is so fast that it can be performed in production control for typically 30 samples per hour (see AN41243). Besides the general benefits that can be brought by the standard inclusion analysis, the additional benefits provided by advanced Spark-DAT applications are the following:

- Quantitative inclusion size and size distribution determination, allowing extremely complete inclusion information to be available, in particular during the steel elaboration
- Quantitative analysis of oxygen at levels of 30 ppm and lower in killed steels, reducing the need for costly combustion analysis

Analysis time

The Spark-DAT analysis alone takes typically 7s for a single measurement (including 2s Ar flush). This mode is recommended only for quick counting and confirmation of inclusion types, and for obtaining raw data for off-line interpretation. However, the Spark-DAT analysis offers more possibilities when combined with the analysis in concentration. In this case, the analysis time taken between the start of the analysis and the display of the results is in average the following:

Application	Model	Time [s]
Low alloy steel (without N, O)	ARL iSpark 8880	20
	ARL iSpark 8860	16
Low alloy steel (with N, O)	ARL iSpark 8880	25
	ARL iSpark 8860	21

These analysis times (unchanged compared to standard elemental analysis) make inclusion analysis possible in many contexts, in particular during steel production, where analysis times are extremely critical.

Sample preparation

Milling is more and more used for steel samples surfacing, because it guarantees a clean, unpolluted surface, ideal for inclusion analysis. Milling is necessary to guarantee quantitative results with the advanced Spark-DAT applications.

Algorithm for quantitative applications

The algorithm *QuIC* delivers the insoluble fraction of an element due to its presence in a particular type of inclusion (e.g. the insoluble fraction of Al as Al_2O_3 inclusions), for several peak intensity classes, which then allows us calculating the average ESD (Equivalent Spherical Diameter) of inclusions for the corresponding size classes.

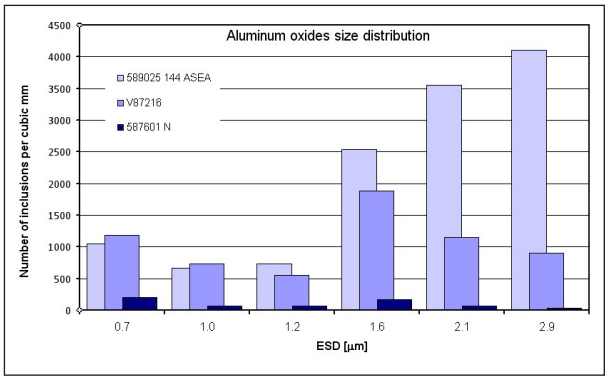
Quantitative size determination

Knowing the size of the inclusions or, better, their size distribution is important, since large inclusions are normally the most detrimental to the metal quality. The two algorithms *Peaks* and *Composition* can also be used in order to count signals belonging to different intensity classes (see AN41243).

These algorithms allow building up qualitative size distribution diagram that can for example be used in order to compare inclusion distributions in different samples of a given grade. The Spark-DAT algorithm *QuIC* (Quantification of Inclusion Content) enables quantitative analysis of inclusions in terms of size and size distribution.

Traditionally, measurements of inclusions size and size distribution are performed by techniques such as optical microscopy and scanning electron microscopy (SEM/EDX). However, the methods are time-consuming, taking typically 2-4h per analysis (0.5-1h for sample preparation and 2-3h for analysis and interpretation of results in case of SEM/EDX) in a very competent laboratory.

The next figure presents an example of a size distribution diagram, based on the application of the *QuIC* algorithm and size calculation method for several production samples.



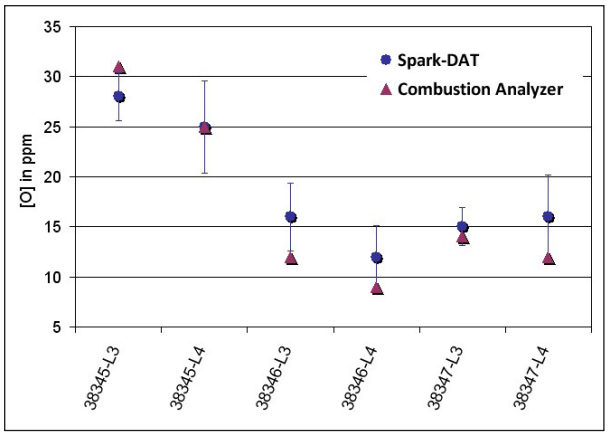
Quantitative analysis of oxygen at low concentration

The standard routine determination of total oxygen by OES is currently semi-quantitative below 60ppm and very difficult below 30ppm. In the steel industry, oxygen at very low concentration is therefore normally determined by dedicated combustion analyzers.

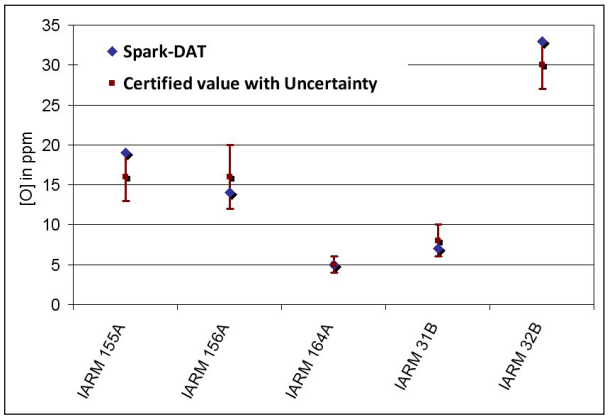
In killed steels treated with the typical deoxidizers (Al, FeSi, FeMn, etc.) most of the oxygen is in the form of insoluble oxygen, more precisely in the form of oxide inclusions.

With the Advanced Inclusions Analysis using Spark-DAT algorithm *QuIC* the total oxygen concentration can be calculated directly from the information obtained on the oxide inclusions (composition and concentration).

The method is quantitative and determines the oxygen concentration by OES even well below 30ppm, as demonstrated in the following chart which compares results obtained with a combustion analyzer for low alloy steel samples taken in the continuous casting mold (samples and combustion results with permission of R. Dumarey and F. Medina, from ArcelorMittal, Gent).



Another illustration of the method is given in the chart here after, where total oxygen concentrations obtained by measuring several CRMs is compared to their certified values.



Inclusions quantifiable with Spark-DAT methods

Various types of endogenous and exogenous inclusions may be observed directly or indirectly in steel with the ARL iSpark spectrometer with Spark-DAT methods, 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 others.

The detection of an inclusion is limited mainly by the sensitivity of the analytical lines used, by the size of the inclusion and by the concentration level of the inclusion elements as soluble elements in the matrix: higher line sensitivity and lower soluble content allow determining smaller inclusions. For example in a steel with 50ppm of Al, the smallest detectable Al_2O_3 inclusion is about 1µm diameter, while with 0.2% of Al it is about 4.5µm diameter.

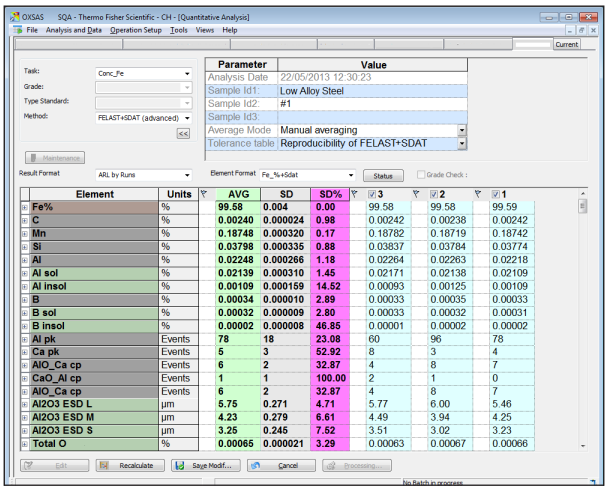
Although the relatively big micro inclusions can be detectable by Spark-DAT methods, the quantification on number or size is difficult due to the limitations on sparking condition. In practice, the quantification methods we discuss here work well for micro inclusions less than 20µm diameter typically.

Other applications of the algorithm *QuIC*

Some other important analytical parameters can be calculated from the results of the algorithm *QuIC*, e.g.:

- **Area fraction** (or surface fraction Sf), i.e. the fraction of the surface occupied by the inclusions of the given type
- **Insoluble concentration** (or fraction) of an element as a particular type of inclusion
- **Concentration of an inclusion type**

The following OXSAS screen shows partial results of an analysis including elemental determinations (total and soluble concentrations) and inclusion related information (peak counts, inclusion numbers and sizes – for inclusion size classes "Small", "Medium" and "Large" – total oxygen determination).



Conclusions

The optional Spark-DAT methods available with the ARL iSpark 8860 and ARL iSpark 8880 metals analyzer increase the versatility of the instrument. From routine use to research, Spark-DAT methods provide quick, simple and cost-effective solutions for inclusion analysis in the steel industry. Among all the inclusions analysis methods available today for the steel industry, the Spark-DAT methods are the fastest.

The Standard Inclusion Analysis Spark-DAT method allows ultra-fast on-line qualitative inclusion analysis e.g. counting of inclusions and identification of their type.

The Advanced Inclusion Analysis Spark-DAT method allows quantitative inclusion analysis e.g. determining their size, the total oxygen content and other parameters in a time ranging from several seconds to a couple of minutes, making it highly effective for controlling inclusions and steel cleanness during production. The instrument maintenance and consumables are equivalent compared to a standard OES instrument. This means extremely low operation costs compared to the case where a dedicated instrument is used for inclusion analysis.

In addition, the ability to obtain elemental analysis information and inclusion contents with a single OES instrument greatly reduces investment costs.

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