Summary

The new SPECTROLAB is the first real Hybrid system in the market and uses new technologies. This leads to significantly improved performance in terms of accuracy, reproducibility and short and long term stability for the analysis of iron and steel and iron alloys.

The instrument provides many more advantages: Lowest detection limits, reduced analysis times using the advanced read-out system in combination with the new Plasma Generator.

In addition to achieving the lowest detection limits, SAFT (Spark Analysis For Traces) is used to reduce interelement effects to a minimum. The variable plasma observation and, frequently, newer spectral line selections result in higher accuracy and precision of the measurements.

SSE (Single Spark Evaluation) features a fast detection of any possible, harmful inclusion populations in the sample.

1. Introduction

The SPECTROLAB represents a new class of metal analyzers distinguished by improved analytical performance, greater investigative flexibility and simpler operation. Almost all of its components have been redesigned and some of them are new inventions. The group of components that is most important for analysis – excitation source, optics and read-out system – is optimally adapted to each other; it is the unbeatable analytical core of the new generation of arc/spark analyzer that combines performance and flexibility.

This new analyzer allows the accurate analysis of all main components and trace elements in steel and iron and all possible alloys.
2. Instrumentation

2.1 SPECTROLAB Features

Optical systems:
- Hybrid optic is made of 2 independent segments; one equipped with CCD’s the other with PMT’s. Full wavelength coverage.
- Focal length for both segments is 750 mm.
- CCD segment is equipped with a holographic grating with 3600 gr./mm
- PMT segment requires 2924 gr./mm for the best performance.
- Argon filled
- Pressure and temperature controlled.

Plasma Generator (DDD)
The benefit of this full digitalization with high fidelity is the guarantee that the spark pulse created is the spark pulse desired, every time.
Precision is improved and so is the similarity of the sources (unit uniformity). With our off-line digital control, we ensure that each source is identical with a deviation of less than 1% between any two sources.
The Plasma generator has an almost free selectable output power waveform.

2.2 The Following Technologies Are Used where Applicable:

Time Resolved Spectroscopy:
A time delayed integration to reduce the background and ionic radiation observation:
- Improved detection limits
- Reduction of interelement effects

SSE:
Single spark evaluation:
- Improvement in precision
- Tool for fast detection of, inclusion-complexity dependent, soluble and insoluble components
- Detection of inclusions
- Correlation between elements in inclusions

Accuracy of Analysis:
The most important objective of analysis with OES Spark Spectrometers is the accuracy of the analysis. The DIN 55 350 Norm describes the commonly used and understood expression “accuracy” as “conventional true value”, which means values obtained with, e.g., Certified Reference Materials. Certified Reference Materials are however not in all cases a guarantee for true analysis. The compositions as well as the metallurgical structures of Reference Materials are often different from the specimens (e.g., production samples) to be analyzed. It is the aim of Modern OES Spectrometers to reduce the influence of the structure and composition (matrix) effects to a great extent. The SPECTROLAB combines the traditional technologies like High Energy Prespark (to reach the stationary phase by “remelting” a small portion of the sample, resulting in a reduced structure effect) with new technologies:
Variable Mask

Plasma Observation is a second cause for the lack of accuracy. Especially for carbon, but also for other alloying elements, the effects of specific plasma observation cannot be neglected. For carbon in both cast iron (2-4.5% C) and steel (0.2-1% C), the plasma observation has to be different compared to the requirements for elements like sulphur phosphorous arsenic, etc. The spectral lines selected for elements like S, P and As are typical atomic lines requiring a plasma observation near the electrode, whereas carbon, depending on the selected wavelength, requires an observation near the sample surface or between the electrode and the sample. To accommodate these different requirements, SPECTRO has developed the technology of two selectable plasma observation possibilities for the relevant spectral lines and analytical tasks. These two mask positions are used in one analytical measurement cycle. The optimum performance for ultra low carbon using the C 133 spectral line requires, due to the high excitation potential of the line, plasma observation near the sample surface.

Diagnostic System

The Diagnostic System shows the status of all of the relevant and important components inside the instrument. The major benefits are: The system indicates the correct position and the activated security circuit after the samples have been clamped. The system also assists in identifying and locating a faulty component in the case of a defect. This decreases the reaction time for the service organization as well as the down-time of the instrument.

3. Single Spark Evaluation (SSE)

Rapidly determined information about steel cleanness, enables steel producers to react quickly in controlling the properties of the final product. SSE can be an additional useful tool to achieve this. By characterizing inclusions using SSE, comparisons to conventional technologies can be made. In addition, it can also be a unique tool with its own features.
Single Spark evaluation (SSE) reports the intensities of each spark individually. These intensities are recorded for further data treatment.

The new SPECTROLAB Single Spark Evaluation now includes:

- Inclusion detection by multi-element outlier correlation.
- Simultaneous display of “normal chemical analysis” and inclusion detection.
- Combined, channel-individual, Time Resolved Spectroscopy and Single Spark evaluation.

**Soluble and Insoluble Components:**

Single Spark Evaluation can separate homogeneous and non-homogeneous elemental components by using dedicated statistical algorithms. The detection limit for inclusion sizes using OES is generally around 1 micrometer. The traditional acid sol/insol determination method includes all sizes of inclusions. Detecting larger (> 1 micrometer) inclusions gives better selective information as they have a greater influence on the metallurgical properties of the metal. In most cases, SSE, Optical microscope and SEM also detect inclusion sizes above 1 micrometer. For this reason, OES-SSE, OM and SEM result, in many cases, in a better correlation than the correlation with the acid sol/insol method.

CRM’s and RM’s certified for sol/insol elements have, in many cases, a relatively complex inclusion population. Production sample inclusion populations are often different from the artificially composed commercially available reference materials. For larger laboratories with an appropriate metallurgical department, it can be beneficial to create “production-like” samples for evaluation with a similarity in inclusion population.
Inclusion detection by multi-element outlier correlation:

Inclusion Detection for Nitrides: NIST SRM 1763 (low alloy steel)

Inclusion Detection for Sulphides:

Inclusion Detection for Oxides:

To obtain the highest sensitivity, the detection of oxides is often performed by using the oxide forming element as a single indicator.

Two examples for the detection of oxide inclusions:
Comparing the two samples, i.e., 4 and 47 ppm Al-insol., it can be observed that the number of inclusions per defined surface increases as the Al-insol. content increases. 2D studies have confirmed that not only the peak intensity, but also the number of inclusions increases when the two samples are compared.

The analysis of oxygen by inclusion counting and peak intensity determination:
Steelworks and research centers have performed Total Oxygen analysis by Al and Ca inclusion counting for a very limited scope of steel qualities. The achievable detection limit is lower than obtained with the “normal” elemental analysis. The influence of the usual contamination effects related to oxygen contamination in the argon and in the analytical stand is, in this case, highly reduced, due to the fact that the oxide forming element and not directly oxygen is reported. Appropriate reference material is, however, not sufficiently available. Applications have been done with samples produced by steelworks. An example with a defined steel grade is given below:

Repeatability is similar to that with conventional oxygen analyzers: RSD (4 measurements) = 10 à 20 %

Combined, channel individual, Time Resolved Spectroscopy and Single Spark Evaluation:

The example below shows the effect of the entire single spark duration evaluation of the element Si in high alloy steel compared to the response on “Time Resolved” evaluation of the single sparks:
Quantification of inclusion properties by Single Spark Evaluation:

In the steel industry and in larger research laboratories, studies have been made to quantify inclusion sizes and inclusionary elemental analysis. In a few cases, investigating relatively non-complex inclusion populations has resulted in semi-quantitative presentation of the data. Also investigations have been conducted to distinguish between inclusion size classes.

The quantification possibilities are limited and restricted to certain inclusion populations and at least a general approach using the currently available international reference material has only been occasionally successful. A major portion of the International Reference materials have, due to their artificial composition, a relatively complex inclusion population.

Spark preference and plasma energy observations for detecting inclusions

The complexity of the inclusion population requires dedicated plasma energy and pulse form conditions to obtain optimum results. SPECTRO can advise you which parameters to use for your individual application. The figures below give an impression of the preference of the spark to attack the interface of sharp edged shaped inclusions before going to rounder shaped inclusions. Complex inclusions, like Al₂O₃ surrounded by MnS, require higher energies to break through the outer (MnS) shelf.

Spark preferences and energy needed to detect inclusions

Preference 1: Sharp edges
Preference 2: Rounder shapes

Al₂O₃ surrounded by MnS:
Higher energy needed to break through the outer MnS shelf.

Al₂O₃: Low energy can be applied
Simultaneous Display of “Normal Elemental Analysis” and “Single Spark Evaluation”:
### Guaranteed Precision and Detection Limits Fe-Base

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The data in this report was achieved under optimum operating conditions using the best suitable wavelengths and homogenous samples.
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* For further information regarding the determination of oxygen in low alloy steel please see application note Arc/Spark-22.
## Guaranteed Precision and Detection Limits Fe-Base

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The data in this report was achieved under optimum operating conditions using the best suitable wavelengths and homogenous samples.
Long Term Stability Data – One Week
(each point represents the average of 5 single measurements)

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Long Term Stability Data – One Month
(each point represents the average of 5 single measurements)

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The Analysis of Iron and Steel

SPECTRO ARC/SPARK Report


Summary

This report shows the capabilities of the new SPECTROLAB (M10) in terms of Limit of Detection, precision and stability for iron and steel applications. It also includes SPECTRO’s approach of using SSE technology related to correlation between elements, the identification of inclusions or segregation (qualitative) and the detection of soluble vs. insoluble portions of relevant elements.

The long term stability results shown are based on the average of five single measurements. During these measurements no recalibration or standardization was performed. Between the first section (one week) and the second section (one month) a limited number of measurements (1,000) were conducted.