LAB REPORT OES 29

Q8 MAGELLAN

Determination of Soluble and Insoluble Elements in Low Alloy Steels

The demand for cleaner steels is continuously increasing. Aluminum plays an important role for the steel cleanliness, and its detailed analysis can supply additional information about the steelmaking process and the steel purity. Aluminum and other elements like Calcium, Titanium, Boron can form non-metallic inclusions (insoluble oxides or nitrides) during the deoxidation step and in the secondary metallurgy. This is especially true for low alloy, Al-killed steels, where aluminum is used for deoxidation during the steelmaking, showing a high tendency for inclusion formation. As a result of the deoxidization, steel shows some insoluble aluminum content, manifesting in the form of aluminum oxide inclusions with high hardness. These inclusions lead to defects in the final steel product, compromising the steel quality and generating extra costs. Therefore, it is important to obtain not only the absolute content of elements like aluminum, calcium, boron and titanium in steel, but also whether they are distributed and alloyed homogeneously inside the metal (soluble) or in form of inclusions (insoluble).

The Q8 MAGELLAN, in combination with the new evaluation features in ELEMENTAL. SUITE software, allows not only for the analysis of the total content of these inclusion-forming elements, but also for the "separation" of this total amount into soluble and insoluble fractions.

The Q8 MAGELLAN provides fast analysis with no additional cost for your production, using standard sample preparation.

The Principle – Soluble & Insoluble Element Determination Single Spark Evaluation (SSE)

While analyzing the sample, the spark (one of thousands during the analysis) is attracted to the grain boundaries in the metal lattice. This is the preferred location of inclusion and since the cation concentration (Al, Ca, B, Ti) in these inclusions is much higher compared to the bulk metal, this leads to an increased signal. This is demonstrated in Figure 2 and 3 by comparing the distribution of single spark signals for aluminum during a full inclusion analysis/metal cleanliness inspection (MCI). Higher Al-signals in inclusions lead to an asymmetric single spark distribution (see Figure 3), where in contrast the distribution in a clean, inclusion-free steel is symmetrical (see Figure 2). During a full MCI, the single spark distributions are processed and statistically evaluated to derive a full set of information, including: element fractions, inclusion sizes and numbers, size distribution, and the stoichiometric composition of inclusions.

Figure 1
Steel quality control for every production step

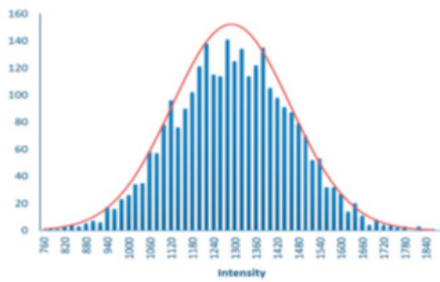


Figure 2
Symmetrical Al-intensity single spark distribution of clean, inclusion-free steel.

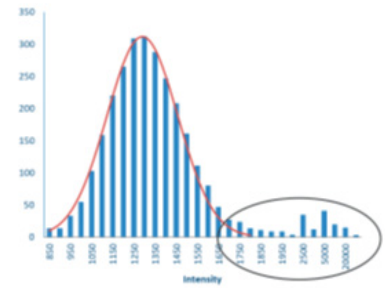


Figure 3
Asymmetric tail (at higher Al-intensities) on the single-spark distribution indicates inclusions that effect steel cleanliness.

The unique SSE capabilities of the Q8 MAGELLAN allow the derivation of the total and soluble fractions of inclusion forming elements utilizing a statistical filter in a less sophisticated and faster approach compared to a full inclusion analysis. As a result of this evaluation, the different fractions of total and soluble elements can be derived, based on the availability of standards with certified concentrations of total and soluble contents. These results enable the calculation of the insoluble fraction.

Sampling and Sample Preparation

The chemical composition of different stages of the steel refining process and casting is monitored by determining the composition of the steel samples taken from the liquid steel.

The surface of the sample is prepared by grinding or milling to have a flat and homogeneous surface. All samples used for the data collection were prepared following standard sample preparation procedure with grinding machine, using silicon-carbide paper, BS118 P60.

Certified Reference Material

Certified Reference Material (CRM) is reference material characterized by a metrologically valid procedure for one or more specified properties. It is accompanied by a certificate that provides the value of the specified property, its associated uncertainty, and a statement of metrological traceability.

Performance Evaluation

Measurements were made on low alloy steel CRMs with certified total and soluble element concentrations on a Q8 MAGELLAN. The certified element concentrations for the reference materials are compared with the average values of seven measurements in Table 1.

Table 1 Comparison of certified with measured values on CRM sets with certified total and soluble element contents.

CRM	Al_{total}		$Al_{soluble}$		B_{total}		$B_{soluble}$		Ti_{total}		$Ti_{soluble}$	
	certified	meas.	certified	meas.	certified	meas.	certified	meas.	certified	meas.	certified	meas.
11717a-1	0.0078	0.0077	0.0069	0.0068	0.0004	0.0004	0.0002	0.0002				
11717a-2	0.026	0.025	0.024	0.023	0.0012	0.0012	0.0011	0.0010				
11717a-3	0.298	0.296	0.295	0.293	0.0020	0.0021	0.0018	0.0018				
11717a-4	0.216	0.214	0.214	0.206	0.0096	0.0092	0.0085	0.0083				
11717a-5	0.104	0.102	0.101	0.100	0.0074	0.0073	0.0071	0.0067				
11717a-6	0.051	0.050	0.050	0.048	0.0049	0.0052	0.0047	0.0042				
11717a-7	0.024	0.022	0.022	0.021	0.0033	0.0036	0.0031	0.0029				
11719-1	0.019	0.020	0.017	0.018					0.016	0.016	0.015	0.014
11719-2	0.296	0.296	0.292	0.285					0.161	0.162	0.154	0.155
11719-3	0.019	0.019	0.016	0.016					0.024	0.024	0.023	0.022
11719-4	0.160	0.159	0.155	0.153					0.132	0.135	0.128	0.125
11719-5	0.036	0.037	0.034	0.035					0.029	0.030	0.028	0.025
11719-6	0.0021	0.0024	0.0016	0.0014					0.0010	0.0013	(0.0008)	0.0009
181A	0.016	0.016	0.014	0.0014								
183A	0.150	0.153	0.141	0.143								
184A	0.022	0.022	0.016	0.017								
185A	0.060	0.060	0.054	0.056								
186B	0.042	0.040	0.038	0.037								
187B	0.027	0.023	0.025	0.021								
188A	0.093	0.091	0.083	0.081								
189A	0.041	0.038	0.039	0.036								

Correlation with Certified Reference Material

Plots of the certified vs. measured values for aluminum, boron and titanium (see Figures 4, 5, and 6) show a perfect linear correlation (> 99%) with each measured value falling within the specified uncertainties of the CRM. The expanded combined uncertainty with a coverage factor of 2 on the certified values was used to generate the y-error bars.

Other inclusion-forming elements like calcium (Ca) can easily be integrated into the total vs. soluble concept when suitable CRMs or user provided samples are available, provided the required PMT detector is installed in the Q8 MAGELLAN.

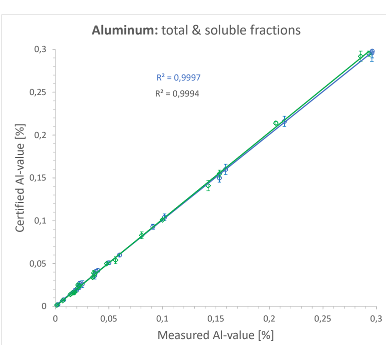


Figure 4

Linear fit between certified and measured values for Al-total (blue) and Al-soluble (green).

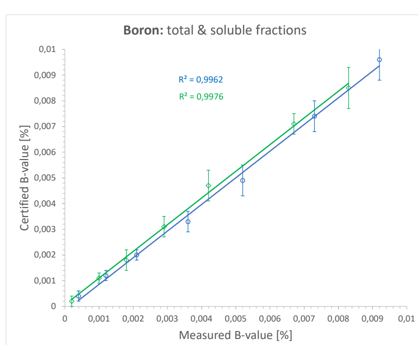


Figure 5

Linear fit between certified and measured values for B-total (blue) and B-soluble (green).

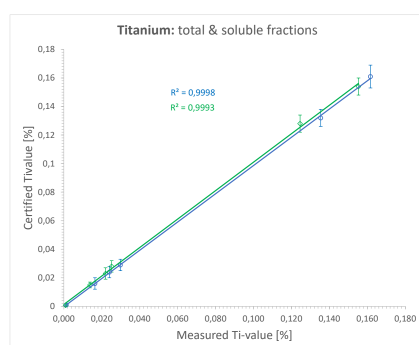
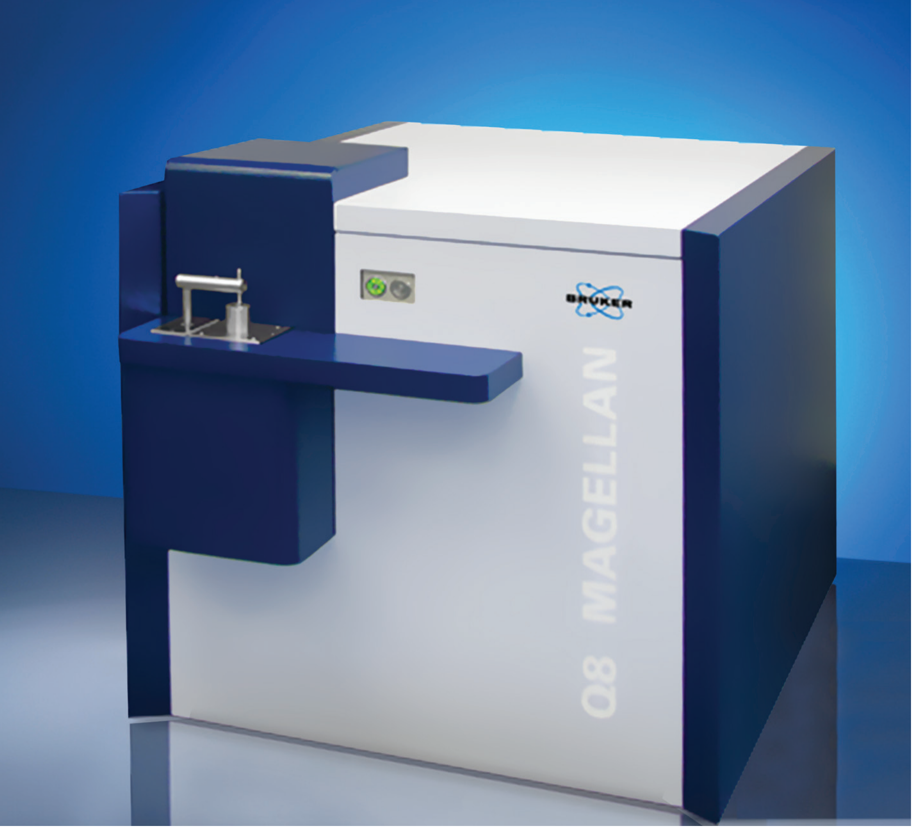


Figure 6

Linear fit between certified and measured values for Ti-total (blue) and Ti-soluble (green).



Conclusion

The Q8 MAGELLAN, in combination with the option to determine total and soluble fractions of an extended element set in ELEMENTAL.SUITE, provides the fast answers you need using the very latest, state-of-the-art technologies. Soluble vs. insoluble analysis becomes integrated into the normal analysis process with no extra time or special conditioning required.

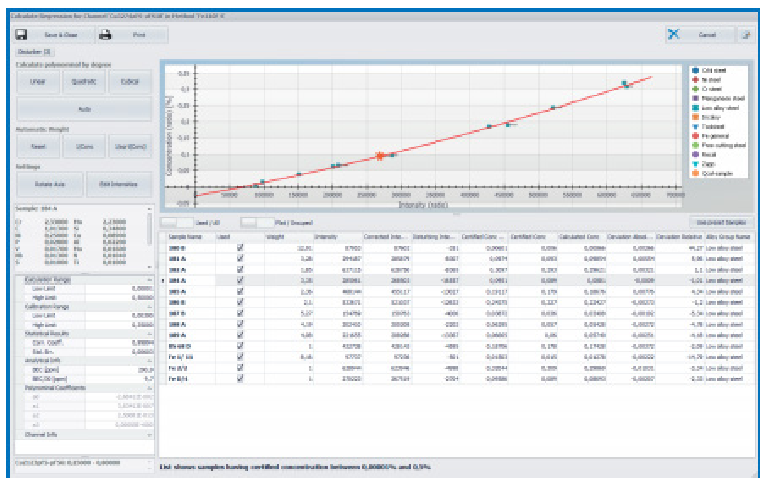
The analysis is fast, shows highly accurate, and well-suited for the daily routine analyses where steel cleanliness is being an important quality criterion.

ELEMENTAL.SUITE

ELEMENTAL.SUITE software assists you in your daily work. Automated average and limit checks ensure safe operation. Saving, printing, and reporting your analyses can be done with one click.

ELEMENTAL.SUITE also supports re-evaluation of raw data and has an integrated Result Viewer, which makes an additional LIMS system superfluous in most cases.

Designed for maximum usability and flexibility, **ELEMENTAL.SUITE** features a plug-in-based architecture to cover and adopt your analytical requirements - now and in the future.



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