WDS

Light element determination in steel using a parallel beam WD spectrometer

Application Note WDS #005

Introduction

The determination of the content of light elements and their spatial distribution in the material is of great interest to all those who manufacture and control steel products.

Light element determination is generally considered difficult for microanalysis techniques such as EDS and classical WDS. In contrast, the XSense is a parallel-beam WDS which is optimized for the low-energy region. With its grazing incidence optic and selected multilayer diffractors it has unrivalled high sensitivity for light elements.

This application note presents examples for the analysis of carbon, nitrogen, boron and oxygen in steel.

Light elements in steel

Stainless steel is an alloy of iron with various other elements such as chromium, nickel, and many other metals, usually in trace amounts. However, light elements are also important alloying constituents that influence the steel properties, intentionally or unintentionally. The contents of these elements must be kept within narrow limits in steel production, as they have a decisive effect on the formation of certain metallic and non-metallic phases and the general properties of the material. Microanalytical investigation of the content and distribution of light elements in steel is therefore of great importance for development and production monitoring as well as for failure analysis.

Challenges and solutions for light element analysis

The detection and quantification of light elements by X-ray microanalysis techniques is more challenging than for other elements. Light elements have a low fluorescence yield, and the relatively sparse low-energy photons are easily absorbed in the sample and detector window. Therefore, the count rates for light elements detected by EDS and classical WDS are markedly low, and the peak overlaps in EDS are significant.

The XSense spectrometer instead is highly sensitive to the low-energy X-rays and can achieve much lower detection limits for the light elements. This fact is due to the technical design, which uses a grazing incidence mirror optic to capture the X-rays very close to their source of origin, and because of the multilayer Bragg diffractors designed for highest performance in the low energy range.

Carbon

Carbon (C) is the most important and influential alloying element in steel. With increasing carbon content, the strength and hardenability of the steel increase, whereas its elongation, forgeability, weldability and machinability decrease [1].

Figure 1 shows the C distribution maps for two types of steel. AISI 440C is a carbon-rich steel with an average steel content of more than 1 wt.%. The map documents that carbon in this steel is concentrated in abundant chromium carbides with 5.7 wt.% C (green phase). The dual phase steel DP600 has relatively low absolute carbon concentrations.

Nevertheless, the two constituent phases martensite (green, 0.3 wt.% C) and ferrite (red, 0.01 wt.% C) could be well distinguished by WDS mapping based on their C content. Both maps were acquired with the BRML80 diffractor within only 5 minutes acquisition time to reduce the effects of carbon deposition.

Determination of carbon contents by microanalysis is difficult due to contamination issues: hydrocarbons, which are ubiquitous in the SEM chamber, are readily cracked in the electron beam, resulting in immediate deposition of C on the sample in the area of analysis.

Therefore, additional technical measures are required if the absolute C content is to be measured. In addition to plasma cleaning, it is advisable to use an air jet to blow the newly forming C away from the analysis spot and a cooling finger in the SEM chamber to trap hydrocarbons.

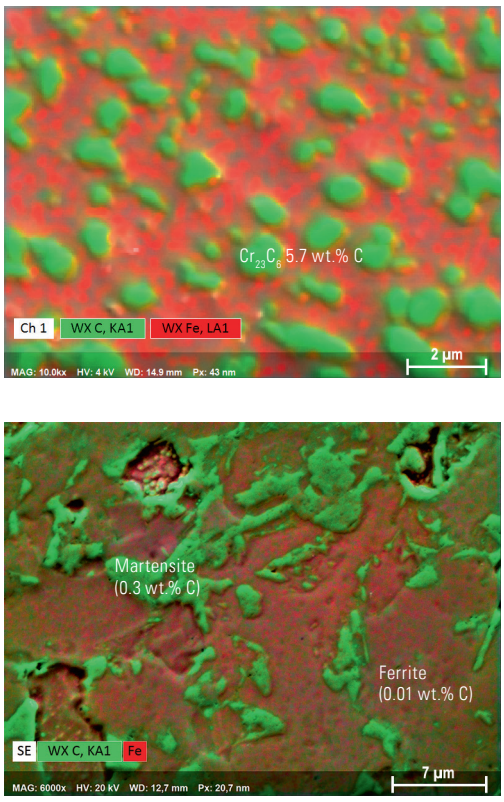


Figure 1
WDS element maps showing the carbon (C) distribution in two different steels. Top: steel with carbide inclusions, certified reference steel AISI 440C. Bottom: dual phase steel with (relatively C-rich) martensite and (C-poor) ferrite. DP600 sample courtesy of TU Eindhoven.

Nitrogen

The element nitrogen (N) can occur both as a steel pest and as an alloying constituent [1]. It is harmful because it reduces toughness through precipitation processes and leads to sensitivity to aging and brittleness. As an alloying element, nitrogen extends the gamma range and stabilizes the austenitic structure. In austenitic steels, it increases strength and, above all, yield strength and mechanical properties under heat. Nitriding can achieve a high surface quality through nitrite formation.

Figure 2 shows the determination of trace element contents of nitrogen in 15 certified steel samples (Acerinox) with WDS. The plot of measured vs. accepted values indicates that contents up to 100 ppm N can be well determined with WDS, whereas these low contents are all far below the detection limit for EDS. Quantitative analyses were performed at 5 kV, 10 nA, with BRML80 diffractor and 60 s dwell time on the peak.

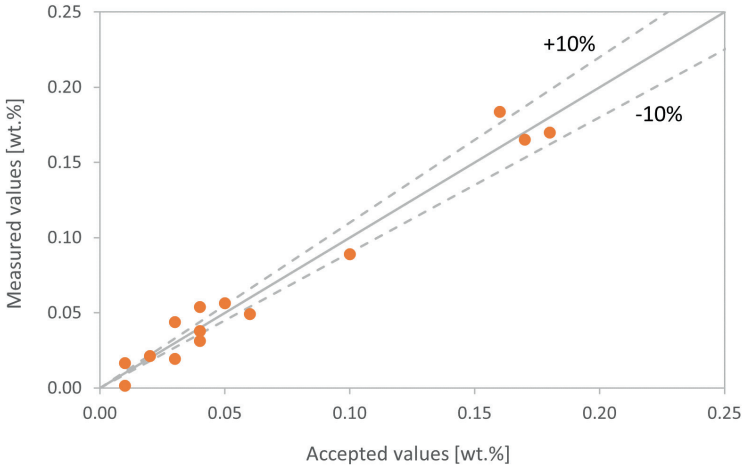


Figure 2
Nitrogen (N) contents determined in 14 certified steel samples (reference steels from Acerinox) are well in accordance with the accepted values and considerably below the EDS detection limit.

Boron

Boron-induced precipitation improves the strength properties of high-temperature steel grades in the elevated temperature range [1]. In structural steels, boron (B) improves through-hardening and thus causes an increase in core strength in case-hardening steels. Austenitic 18/8 CrNi steels can be brought to higher yield strengths and strengths by precipitation hardening with boron. Since boron has a high effective cross-section for neutron absorption, it is used to alloy steels for regulators and shielding of nuclear power plants.

The study of the cross-section of a Ni-P-B dispersion layer on steel substrate 100Cr6 represents a characteristic application for the light element B (Figure 3). The element distribution maps show the formation of different Fe and Ni phases in the dispersion layer and the contact zone. The measurement conditions for the 600 x 450 pixel (30 x 22 µm) map are 5 kV, 16 nA and 10 min acquisition time for WDS using the BRML200 diffractor.

The second example presents the combined WDS-EDS map of trace amounts of boron in a so-called boron-steel (Figure 4). The relative boron enrichments refer to submicron cementite phases containing 0.4 wt.% B, in contrast to the steel matrix containing 0.2 wt.% B. Map acquisition time was 120 min at a resolution of 300 x 225 pixels.

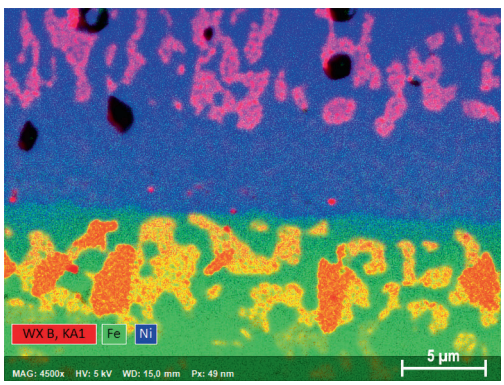


Figure 3
Combined WDS-EDS element distribution map of a Ni-P-B dispersion layer on a steel substrate. Sample courtesy of Ms. Nurul Amanina Binti Omar, HS Mittweida & TU Ilmenau.

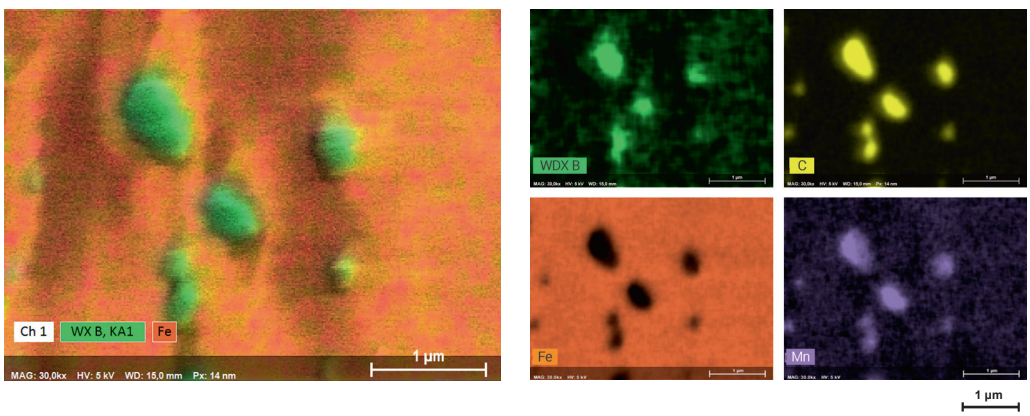
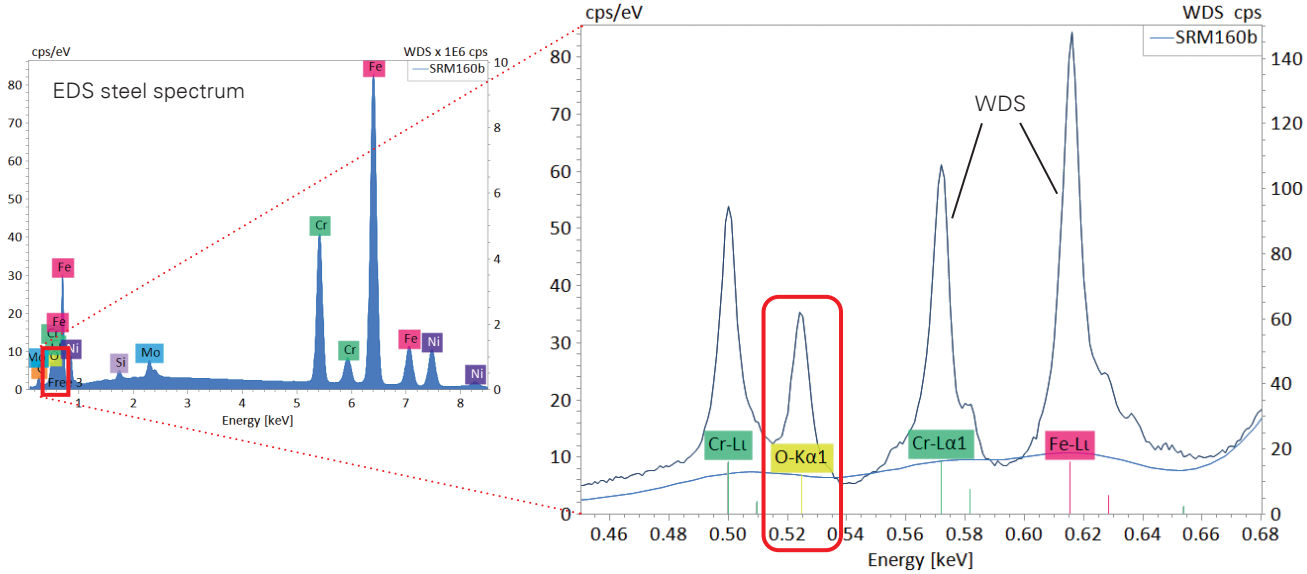


Figure 4
Element distribution maps for boron (B) as trace element in steel. The relative enrichment is bound to carbide phases in the submicron range. Sample courtesy of Dr. M. Amirthalingam, IIT Madras.



Oxygen

This element ages the steel and deteriorates, in particular, the notched impact strength [1]. In addition, oxygen produces so-called red fractures and favors the so-called wood fiber fractures.

For X-ray spectrometry, the presence of chromium makes it difficult to detect traces of oxygen in stainless steel. Due to the severe overlap between O-K and Cr-L peaks, low oxygen contents are not visible in EDS spectra. Only WDS can resolve the O-Kα peak and the Cr-L peaks in steel (Figure 5) and enable oxygen determination. Analytical conditions are 20 kV and 75 nA, using the BRML30 diffractor and 60 s dwell time for each step of the energy range scan.

Light elements are difficult to determine using classical X-ray-based microanalysis techniques, especially for EDS.

The XSense is a parallel-beam wavelength dispersive spectrometer with high-performance grazing incidence optics and dedicated multilayer diffractors optimized for the low X-ray energies.

This technical design results in the highest count rates for the low-energy photons, making the QUANTAX WDS system the most suitable microanalysis tool for the determination of light elements in steel, even in trace amounts and in situations where strong peak overlaps for EDS exist.

Figure 5
EDS and WDS spectra of a stainless steel (certified reference steel SRM160b) with focus on the low X-ray energies where Cr-, Fe- and O peaks overlap.

Conclusion

Knowing the contents and distribution of light elements in steel is very important, as this has a great influence on the properties of the material.

Reference

[1] Extracted and translated from „Wissenswertes über die Legierungs- und Begleitelemente im Stahl", Verlag Stahlschlüssel Wegst GmbH, http://www.stahlschluessel.de/de/info/allgemeines.aspx

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