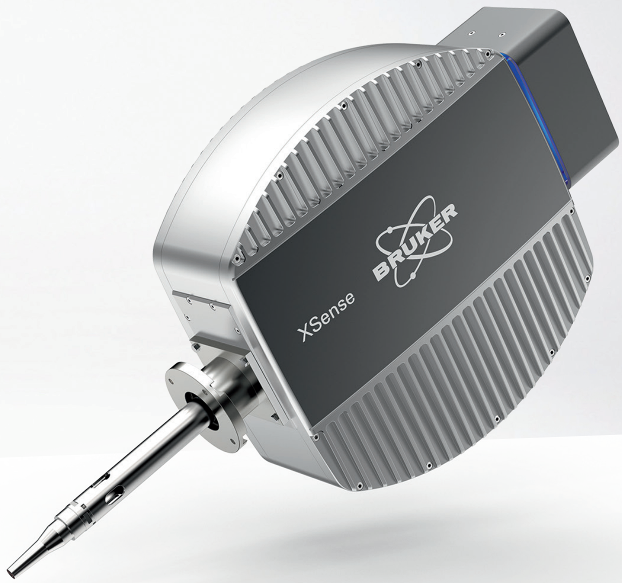

WDS

Determination of trace element concentrations in steel using QUANTAX WDS for SEM

Application Note # WDS-04

Introduction

Trace elements may have a critical influence on the properties, workability and durability of steel. Therefore, the determination of trace element concentrations in steel is of substantial relevance for quality assurance and inspection in the related industries and civil services.

EDS and WDS on SEM or EPMA are the analytical methods of choice when spatial resolution is of interest. Since WDS achieves a 10 times higher peak to background ratio for many X-ray lines when compared to EDS, trace element concentrations as low as 100 µg/g or below can be achieved.

Sample

There are innumerable steel types available on the market which vary in quality, purpose and composition. For this study various steels with certified compositions were investigated: SRM 160b, SRM 12h, SRM 13g, SRM 73c, SRM 101g, and SRM 663. Trace elements of interest are silicon and phosphorous which range from 0.18 wt.% – 1.08 wt.% (Si) and 0.006 – 0.020 wt.% (P), respectively, in the above mentioned standards.

Methods

The measurements were performed using a FE-SEM equipped with both, a Bruker XFlash® 6 I 10 ED spectrometer and a Bruker XSense WD spectrometer using a grazing incidence parallel beam optic. Qualitative trace element and major element determinations were done by WDS energy range scans at 20 kV and 60 nA applying the PET and TAP diffraction crystals. WDS-based Si and P quantification acquisitions were carried out at 20 kV, 180 nA beam current and 120 s peak time with metallic Si and InP used as standards. Major elements were quantified by standardless EDS, matrix corrections were made by φ(ρZ).

Results

WDS can clearly resolve the line series of Cr-L, Fe-L, Mn-L, and Ni-L whereas they strongly overlap in EDS (Figure 1a). Using QUANTAX WDS, the elements Fe, Cr, Ni and Mn can be quantified based on their L-line series X-rays. The ability for L-line series based quantification facilitates low-kV analyses when aiming for high spatial resolution and low depth penetration during the analysis.

WDS is perfectly suited for trace element determination with limits of detection which are an order of magnitude lower than those of EDS. This is due to significantly higher peak to background ratios (P/B) for WDS when compared to EDS. Energy range scans for silicon (Si) and phosphorus (P) in steel standard SRM160b clearly document the presence of these elements in the sample (Figure 1b). The quantitative results for Si and P in various steel standards are presented in Table 1.

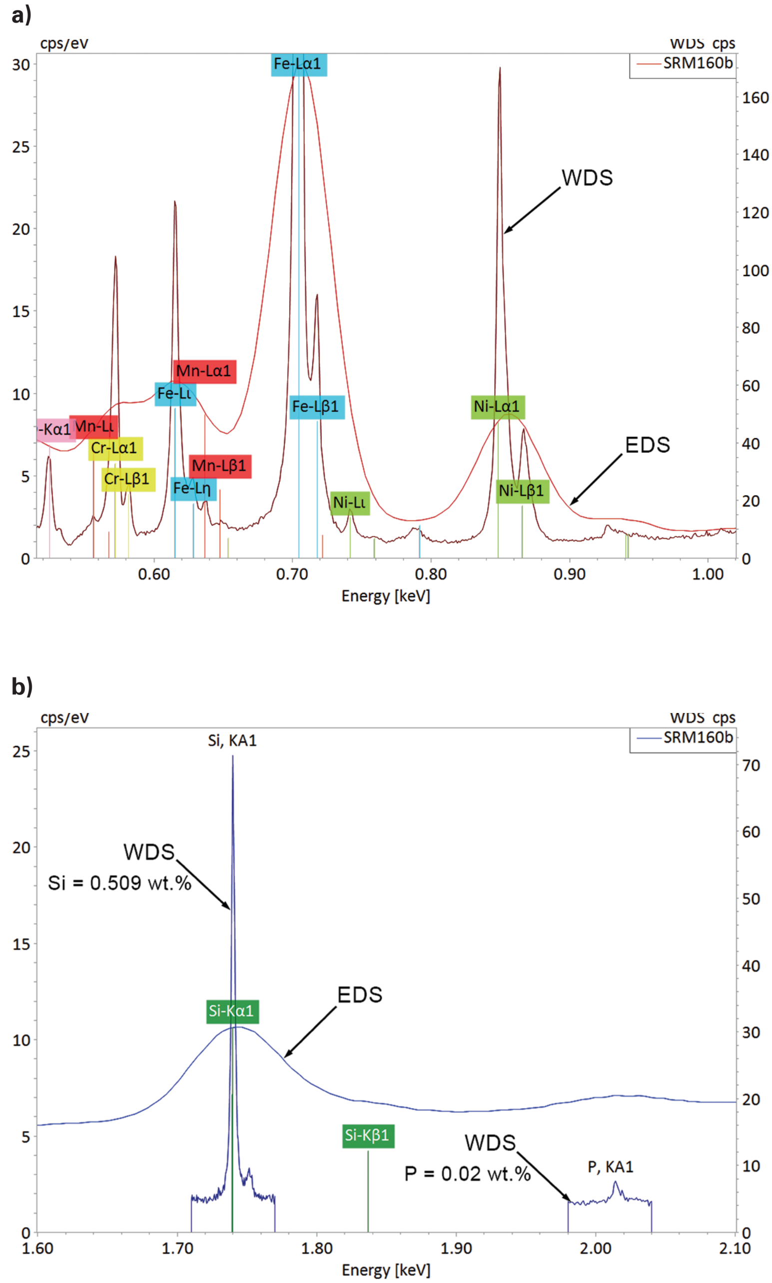

Figure 1
Partial X-ray spectrum of stainless steel SRM160b showing EDS and WDS results from a combined measurement:
a) major elements in the energy region 0.5 – 1.0 keV,
b) trace elements Si and P in the energy region 1.6 – 2.1 keV.

Sample	Si				P			
	Certified value [wt.%]	Result [wt.%]	1 Sigma [± wt.%]	Deviation [wt.%]	Certified value [wt.%]	Result [wt.%]	1 Sigma [± wt.%]	Deviation [wt.%]
SRM 13g	0.355	0.338	0.021	-0.017	0.006	0.004	0.002	-0.002
SRM 663	0.74 ± 0.01	0.757	0.022	0.017	0.029 ± 0.005	0.028	0.002	-0.0014
SRM 73c	0.181	0.177	0.006	-0.004	0.018	0.018	0.001	0.000
SRM 12h	0.235	0.241	0.009	0.006	0.018	0.018	0.002	0.000
SRM 101g	1.08 ± 0.01	1.003	0.035	-0.077	0.007 ± 0.001	0.004	0.003	-0.003

Table 1
Certified values and quantitative results for Si and P determined on standards with the XSense WD spectrometer.

Conclusion

- WDS shows significantly higher P/B ratios for the respective elemental X-ray lines compared to EDS.
- High P/B ratios yield in very low detection limits for WDS which can be 10x better than EDS.
- QUANTAX WDS is a powerful tool for the analysis of trace elements in steel.
- QUANTAX WDS and EDS are fully integrated in the ESPRIT software package.
- QUANTAX WDS and EDS can complement each other for the analysis of steel.

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