



Lab Report XRF 153

S6 JAGUAR

- Quality Control of Mild Steel

Introduction

Mild steel has low carbon concentrations and is therefore ductile. In addition it has also very low concentrations of other alloying elements, such as Cr, Ni, Mo and W. It is less resistant against corrosion, but due to its machinability, weldability and the low price it is often used in automotive and engineering for car parts, structural elements and fences. Trace elements such as Cu, Sn or Pb are critical because it will lead to early corrosion.

Sequential wavelength-dispersive X-ray fluorescence (WDXRF) spectrometry is today well

established for the analysis of metals in quality and process control. This application is challenging because alloying elements occur from the ppm range to high percentages and severe line overlaps will influence accurate trace determination. Especially mid size central laboratories are in need of an analyzer to cover a wide range of materials without compromise on data quality, but the small number of samples per day is not justifying a big WDXRF spectrometer. This report outlines the performance of the S6 JAGUAR for quality control of metals.

Instrument

The S6 JAGUAR WDXRF spectrometer is equipped with the HighSense goniometer, its closely coupled beam path and 400 W excitation power. It offers outstanding sensitivity for every single element in this class of instruments, which lead to high analytical precision. It covers the entire concentration range from the ppm up to 100% due to the HighSense detector, which is vital for elements such as Nickel in low and high alloy steels. For reducing line overlaps the S6 JAGUAR can be equipped with a fourth crystal LiF 220, which enhances the spectral resolution of traces, such as Cr, Mn, Fe, Co and Ni in mild steels. In case of smaller parts the S6 JAGUAR can be equipped with sample masks for smaller diameters than 34 mm.

Equipped with a 24 position EasyLoad sample magazine and TouchControl the S6 JAGUAR ensures optimal productivity. With its unique SampleCare technology and the added vacuum pump it offers lowest cost-of-operation and optimal instrument uptime. By adding TouchControl the S6 JAGUAR is easy to operate and ensures data integrity by its failsafe operation.

Preparation

Metal samples are cutted into shape. The surface is then finely ground; for better accuracy a polishing will lead to better data. This sample preparation is simple and quick with affordable instrumentation.

Measurement

Each element is analyzed with an optimal set of instrument parameters: Light elements are excited with low voltage at maximum power, while all heavy elements starting from Ca upwards are best excited with 50 kV. For the elements Al and Si the PET crystal is used and for the element range Ti to Pb the LiF200 is applied. To answer the demand for better trace analysis and better line separation an LiF220 can be used for Mn, Cr, Ni, Cu and Co. This can be vital if traces are becoming important. The typical measurement time was between 5 to 102 for the minor elements and up to 20 s for trace elements. The measurement parameters are shown in table 1.

Table 1: Set of measurement parameters of the S6 JAGUAR for Nickel laterite

Name	Voltage (kV)	mA	Filter	Crystal	Detector	Peak
Al KA1	30	13.3	No filter	PET	FlowCounter	144.811
Si KA1	30	13.3	No filter	PET	FlowCounter	109.064
P KA1	30	13.3	No filter	PET	FlowCounter	89.433
S KA1	50	8	No filter	PET	FlowCounter	113.035
Ti KA1	50	8	No filter	LiF (200)	FlowCounter	86.079
Cr KA1	50	8	No filter	LiF (220)	HighSenseXE	107.023
Fe KA1	50	8	No filter	LiF (200)	HighSenseXE	57.488
Mn KA1	50	8	No filter	LiF (220)	HighSenseXE	95.127
Ni KA1	50	8	No filter	LiF (200)	HighSenseXE	48.638
Ni KA1/Alt-220	50	8	No filter	LiF (220)	HighSenseXE	71.204
Cu KA1	50	8	No filter	LiF (200)	HighSenseXE	44.997
Ni KA1	50	8	No filter	LiF (200)	HighSenseXE	48.638
Ni KA1/Alt-220	50	8	No filter	LiF (220)	HighSenseXE	71.204
As KA1	50	8	No filter	LiF (220)	HighSenseXE	33.966
Mo KA1	50	8	No filter	LiF (220)	HighSenseXE	20.294
Sn KA1	50	8	No filter	LiF (220)	HighSenseXE	13.999
Sb KA1	50	8	No filter	LiF (220)	HighSenseXE	13.419
W LB 1	50	8	No filter	LiF (220)	HighSenseXE	37.133
Pb LB 1	50	8	No filter	LiF (220)	HighSenseXE	28.262

Calibration

A set of 8 international certified reference materials (CRMs) were used to prepare the calibration for the mild steels calibration. Two calibration curves, one for Cu and one for MnO, are shown in figure 1 and 2.

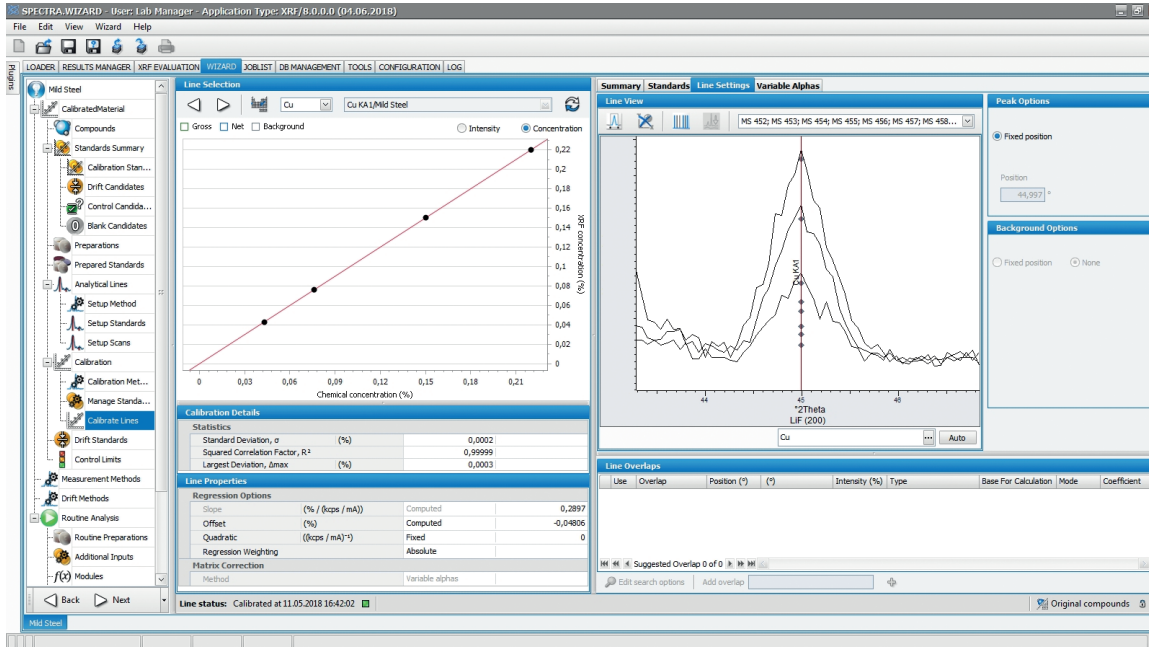


Figure 1: Calibration curve for Cu in mild steel, occurring in trace concentration level

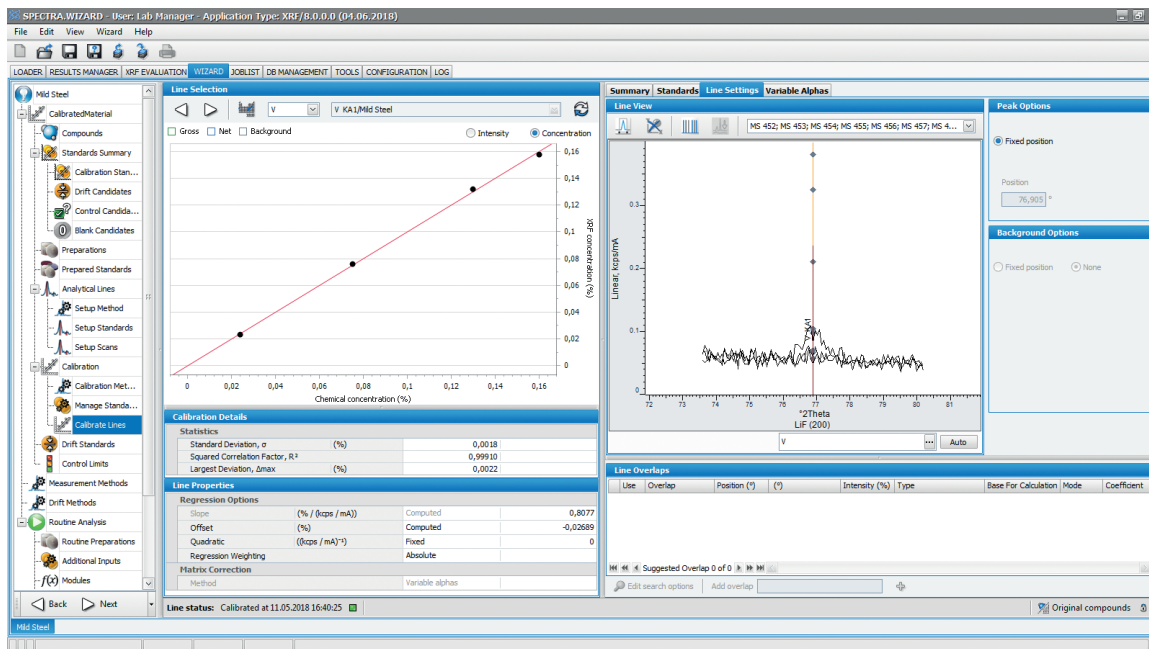


Figure 2: Calibration curve for V

Results

For successful quality control analytical precision is vital. The S6 JAGUAR shows its performance with a 17fold repetition test for one sample, shown in table 2. For the most important elements the relative standard deviation

is typically less than 1 %, traces shows higher variation, but at this low concentration levels R.S.D. of less than 5% are excellent

Table 3: Concentration ranges used for the nickel laterite calibration

	Al (%)	Si (%)	P (%)	S (%)	Ti (%)	V (%)	Cr (%)	Mn (%)	Fe (%)	Ni (%)	Cu (%)	As (%)	Mo (%)	Sn (%)	W (%)	Pb (%)
# 1	0.017	0.23	0.052	0.062	0.029	0.008	0.216	0.11	98.599	0.242	0.043	0.019	0.155	0.075	0.23	0.005
# 2	0.016	0.235	0.054	0.063	0.03	0.009	0.217	0.112	98.613	0.246	0.044	0.018	0.156	0.076	0.218	0.005
..
# 16	0.015	0.228	0.053	0.062	0.028	0.008	0.219	0.11	98.363	0.243	0.042	0.019	0.155	0.075	0.226	0.004
# 17	0.014	0.22	0.054	0.063	0.028	0.008	0.217	0.109	98.707	0.242	0.045	0.019	0.156	0.071	0.223	0.004
Mean value	0.015	0.229	0.054	0.062	0.028	0.009	0.218	0.110	98.614	0.242	0.044	0.019	0.155	0.075	0.224	0.004
Abs Std Dev.	0.001	0.004	0.001	0.001	0.001	0.000	0.001	0.001	0.100	0.002	0.001	0.001	0.001	0.002	0.003	0.001
Abs Std Dev.	7.0	1.8	1.6	1.0	3.3	4.9	0.5	1.0	0.1	0.8	3.0	4.0	0.6	2.2	1.2	22.4

Conclusion

The S6 JAGUAR shows high analytical precision when analyzing mild steel samples for material characterization and quality control. With its 400 W excitation power and the HighSense goniometer, the sensitivity for trace elements, such as Cu, Sn or Pb, is excellent. This enables to test material qualities and prevent the use of low quality mild steel with high Cu and Sn, which would tend to faster corrosion. The high analytical accuracy also enables the quick access of material grades and alloy types.

Bruker AXS is continually improving its products and reserves the right to change specifications without notice. Order No. DOC-L80-EXS153. © 2018 Bruker AXS.

Bruker AXS GmbH
info.baxs@bruker.com

Worldwide offices
bruker.com/baxs-offices

Online information
bruker.com/s6jaguar

www.bruker.com

