



Lab Report XRF 85

S2 RANGER

Quantitative Analysis of Al-Si Cast Alloys

This report details the performance and applicability of the S2 RANGER with XFlash® for the analysis of Al-Si cast alloys.

Introduction

Aluminum alloys containing silicon are very versatile materials with wide-ranging use from the automotive to the food industry. Control of the elemental composition is routinely performed during alloy melting, prior to casting and as a final product check to validate suitability for an application or customer requirements.

Testing is usually performed with either wavelength dispersive X-ray fluorescence (WDXRF) analysis or optical emission spectroscopy (OES). Thanks to advances made in the fourth generation of XFlash® silicon drift detectors (SDD), energy dispersive X-ray fluorescence (EDXRF) can now be used for this application. EDXRF lowers the cost per sample as well as the space and infrastructure requirements for

the instrument. Never before was it possible to use count rates in excess of 100,000 cps while retaining the resolution needed to discriminate between Al and Si $K\alpha$ emission lines and keeping the dead time of the detector below 15%.

Instrumentation

The S2 RANGER is an all-in-one benchtop EDXRF spectrometer with TouchControl™, an easy-to-use touch screen interface. This system requires only electrical power. Its unique XFlash® detector easily processes count rates of 100,000 cps while ensuring excellent resolution, low dead time and high signal stability. The compact design of the S2 RANGER includes a vacuum pump for excellent light element performance. It also includes an automatic x-y sample changer.

Table 1: Measurement conditions

Voltage [kV]	Beam Filter	Live Time [s]	Elements Analyzed
20	none	100	Al, Si
40	Al 500 μm	100	Cu, Mn, Cr, Ni, Zn, Ca, Ti, Pb
50	Cu 250 μm	100	Sn

Table 2: Calibrated concentration ranges

Element	Concentration Range [wt. %]
Al	Matrix
Si	9.60 – 15.30
Fe	0.20 – 1.09
Cu	0.026 – 0.72
Mn	0.02 – 0.90
Cr	0.01 – 0.17
Ni	0.02 – 0.66
Zn	0.02 – 0.67
Ca	0.000 – 0.007
Ti	0.01 – 0.36
Pb	0.00 – 0.12
Sn	0.01 – 0.19

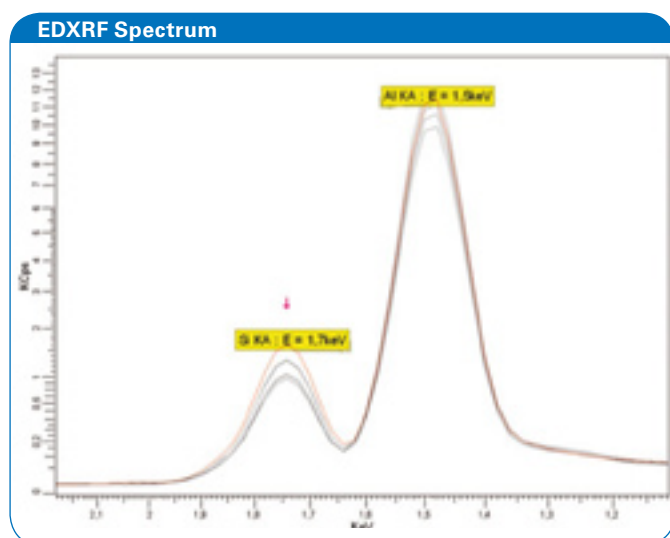


Figure 1: Spectral resolution of Al and Si K α

Sample Preparation

The Al-Si cast metal sample was obtained with a coquille, producing a cylindrical sample with a maximum diameter of 40 mm. The cylinder was cut into 40 mm diameter disks of up to 50 mm height. The analytical surface was flattened and smoothed using a rotating cutting mill.

Measurement Parameters

The following measurements were performed under vacuum. Using the predefined conditions of EQUA ALL, standardless quantitative analysis software included with every S2 RANGER with XFlash, allowed quick setup by a non-expert user. Only the live time was extended to achieve the required precision.

The current was automatically adjusted to a count rate of 100,000 cps to ensure optimum counting statistics. The detector's resolution was less than 129 eV at Mn K α 1 at 100,000 cps.

Calibration

A set of 6 internationally certified reference material (CRM) standards from Alusuisse and MBH was used for the calibration of Si, Fe, Cu, Mn, Cr, Ni, Zn, Ca, Ti, Pb, and Sn.

With aluminum being the major matrix element and silicon as the principal neighboring element, the peaks need to be well resolved in order to be determined with good accuracy and precision.

Prior applications of EDXRF employed empirical profile deconvolution or peak fitting techniques, both of which add uncertainty to the measured intensity. The S2 RANGER with XFlash does not rely on any such techniques since it can easily resolve the neighboring characteristic emission lines of Si K α and Al K α . It is especially noteworthy that the Al peak is fully separated from the Si peak while being a factor 5 higher in intensity.

The calibration was performed with SPECTRA^{EDX} software operated from a PC connected to the S2 RANGER. This integrated XRF spectroscopy package offers a wide range of matrix and interference corrections.

All CRM standards were used for the calibration. Sensitivities were calculated for each element after using appropriate matrix correction by least squares refinement of either absolute or relative deviation.

As an example, the analysis of one CRM, MBH Certified Standard G06H6G is shown. This CRM was not used for the calibration and therefore also not included in the training set.

The achievable accuracy is shown in table 3. Table 4 shows the precision of the measurement using the initially selected 100 s per condition with respect to the certified values for the CRM (Ca was not certified as was Al)¹

Repeatability was determined by measuring the same sample 10 times with the sample changer, removing and reloading the sample after each measurement.

Conclusion

The S2 RANGER with XFlash opens up a new performance class for EDXRF: Quantification of 12 elements in 5 minutes' counting time using 3 different excitation conditions and yielding repeatability for Si of better than 0.3% relative and 0.1% relative for Al. Compared to other methods the EDXRF spectrometer S2 RANGER is covering wide concentration ranges for all elements, achieving very good precision. Therefore the S2 RANGER is an attractive solution for the analysis of cast Aluminium products and it is in addition also suitable for the analysis of raw materials for production.

Table 3: Accuracy of the calibration

Sample	Si [wt. %]	Fe [wt. %]	Cu [wt. %]	Mn [wt. %]	Cr [wt. %]	Ni [wt. %]	Zn [wt. %]	Ca [wt. %]	Ti [wt. %]	Pb [wt. %]	Sn [wt. %]	Al [wt. %]
Certified Value	12.1	0.75	0.36	0.42	0.08	0.31	0.02	N.A.	0.14	0.09	0.09	Balance
Average n=10	12.2	0.734	0.351	0.44	0.081	0.307	0.018	0.018	0.142	0.09	0.09	86.0

Table 4: Measurement

Sample	Si [wt. %]	Fe [wt. %]	Cu [wt. %]	Mn [wt. %]	Cr [wt. %]	Ni [wt. %]	Zn [wt. %]	Ca [wt. %]	Ti [wt. %]	Pb [wt. %]	Sn [wt. %]	Al [wt. %]
G06H3G-1	12.2	0.733	0.351	0.443	0.083	0.308	0.0185	0.017	0.143	0.090	0.095	85.9
G06H3G-2	12.2	0.732	0.351	0.444	0.081	0.306	0.0178	0.019	0.143	0.090	0.094	85.8
G06H3G-3	12.2	0.734	0.350	0.444	0.081	0.305	0.0180	0.016	0.140	0.090	0.094	86.0
G06H3G-4	12.3	0.732	0.350	0.446	0.080	0.307	0.0183	0.018	0.142	0.088	0.097	86.1
G06H3G-5	12.2	0.731	0.350	0.444	0.081	0.308	0.0184	0.020	0.140	0.090	0.093	86.0
G06H3G-6	12.2	0.736	0.352	0.439	0.081	0.307	0.0182	0.018	0.141	0.089	0.093	86.0
G06H3G-7	12.3	0.736	0.352	0.448	0.081	0.309	0.0180	0.021	0.141	0.090	0.095	86.2
G06H3G-8	12.2	0.733	0.352	0.445	0.081	0.307	0.0180	0.020	0.142	0.089	0.095	86.0
G06H3G-9	12.3	0.736	0.352	0.443	0.081	0.306	0.0178	0.018	0.145	0.090	0.095	86.0
G06H3G-10	12.2	0.735	0.352	0.442	0.081	0.307	0.0183	0.016	0.140	0.091	0.094	86.0
Average	12.2	0.734	0.351	0.444	0.081	0.307	0.0181	0.018	0.142	0.090	0.095	86.0
Std.Dev.	0.0	0.002	0.001	0.002	0.001	0.001	0.0003	0.002	0.002	0.0008	0.001	0.1
Rel.Std.Dev.	0.26	0.27	0.28	0.54	0.77	0.35	0.70	9.77	1.19	0.92	1.25	0.11

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¹The given calibration data are typical values and depend strongly on the reference materials used, in the optimal sample surface preparation and on the measurement conditions.

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