

X-RAY FLUORESCENCE

Fast and reliable quality control of Ferrosilicon

Lab Report 171

Introduction

Ferrosilicon (FeSi) is an alloy containing two major elements, silicon (Si) and iron (Fe). In steel production and casting it can serve several purposes: deoxidizing, preventing loss of carbon (C), removing harmful impurities (e.g., S, N), and even modifying the solidification process. To achieve the desired effect, certain purity standards need to be met.

The purity or quality of Ferrosilicon is determined by the concentration level of contaminants, such as aluminum (Al), phosphorous (P), sulfur (S), calcium (Ca), titanium (Ti), zirconium (Zr), and lead (Pb).

Energy-dispersive X-ray fluorescence (EDXRF) analysis is an easy and quick technology to accurately monitor and verify the Ferrosilicon composition, helping to ensure cost-efficient steel production.

Instrumentation

The S2 PUMA Series 2 EDXRF spectrometer is an excellent solution for your Ferrosilicon analysis. The combination of a 50 Watt X-ray tube with closely-coupled beam optics and the new HighSense™ detector technology enables optimal sample excitation, resulting in outstanding analytical performance (Figure 1).

In contrast to other methods like AAS (Atomic Absorption Spectrometry) and ICP-OES (Inductively Coupled Plasma – Optical Emission Spectrometry), a simple one-time calibration makes the S2 PUMA ready for your application. This calibration is then checked daily by running a control sample. Once the system is calibrated, the TouchControl™ user interface makes it really easy to run routine samples after minimal introduction.

Sample Preparation

Ten grams of the calibration standards and the unknown samples were weighed out and pulverized using a tungsten-carbide (WC) ring and puck mill for 3 min. The samples were then pressed at 30 tons for 30 s using a 40 mm die and 1.0 g of wax binder. The result are robust and homogeneous pellets (Figure 2) that can be measured in vacuum mode for the best detection of lighter elements (Al and Si) combined with the lowest cost of operation (no Helium!). The preparation is simple and reproducible. It can be performed with any grinder and pellet press.

Figure 1

HighSense™ beam path of the S2 PUMA Series 2.

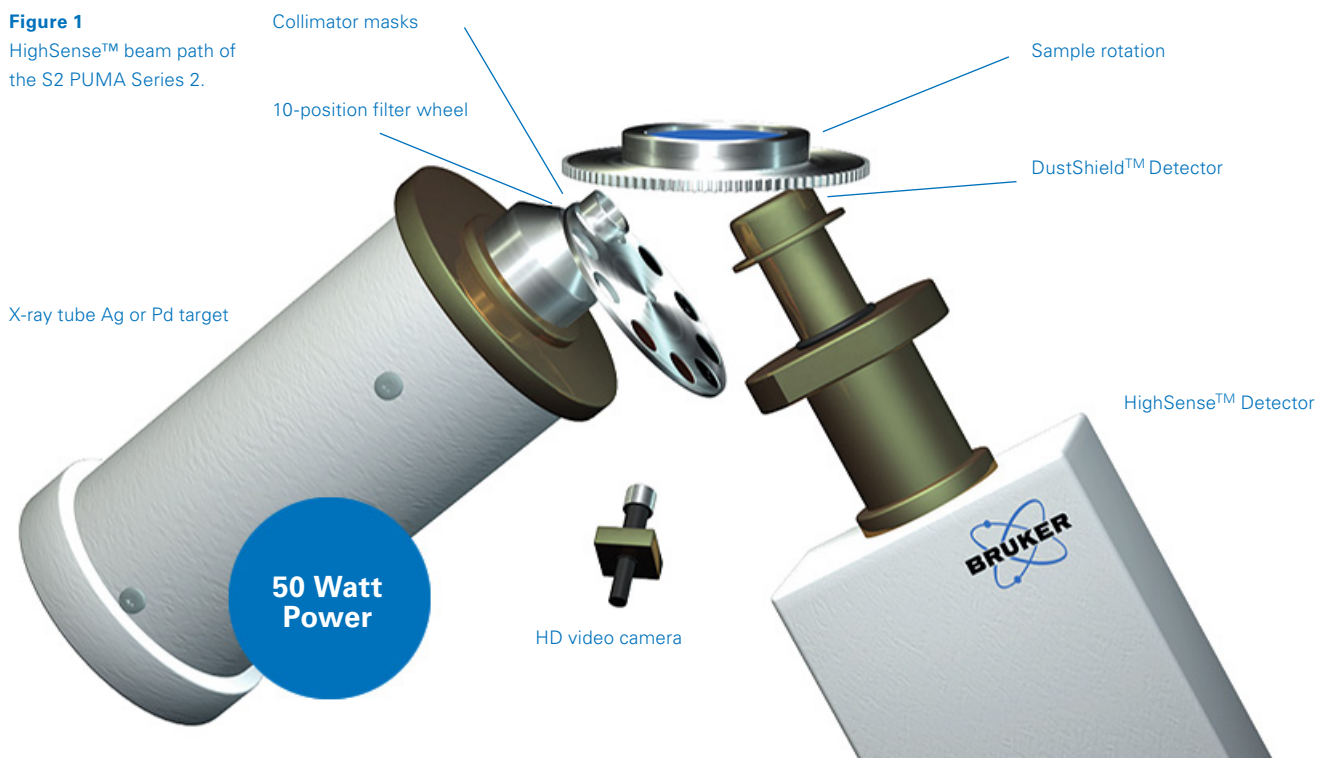


Figure 2

Example for finished pressed powder pellets.

Table 1

Measurement Parameters.

Elements	Atmosphere Mode	Voltage [kV]	Current [mA]	Filter	Rotation	Time [s]
Al, Si, P	Vacuum	20	Automatic	None	Yes	180
Ca, Ti, Fe, Zr, Pb	Vacuum	40	Automatic	500 µm Al	Yes	180

Calibration Details

A combination of certified reference materials (CRMs) standards and validated secondary standards (10 in total) were used for building a dedicated Ferrosilicon calibration for eight elements, covering wide concentration ranges (Table 2).

The calibration was performed with Bruker's intuitive spectrometer software, SPECTRA.ELEMENTS. This integrated XRF spectroscopy package guides the operator smoothly through the calibration process and uses smart matrix and interference correction models to achieve optimal performance.

Table 2

Calibration range of the FeSi standards.

Element	Concentration Range [wt.%]		
Si	48	to	79
Fe	12	to	51
Al	0.05	to	1.5
P	0.002	to	0.3
Ca	0.002	to	0.3
Ti	0.02	to	0.15
Zr	0.001	to	0.015
Pb	0.001	to	0.015

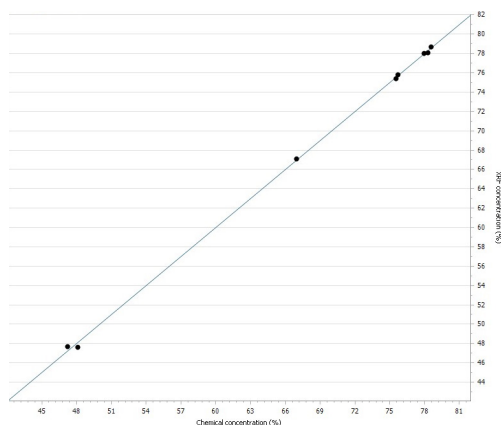


Figure 3

Example calibration curve: Si.

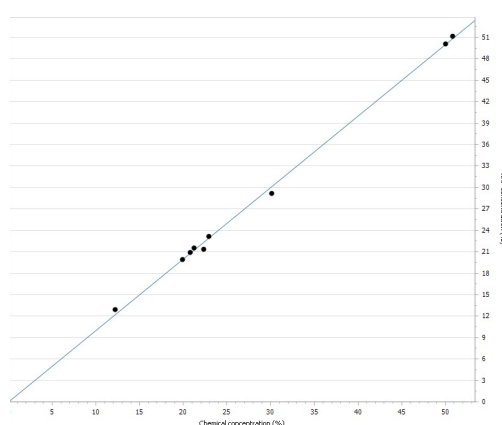


Figure 4

Example calibration curve: Fe.

Table 3

Precision test. (a) Certified value, (b) reference value, (c) informative values.

NIST SRM 58a	Al [%]	Si [%]	P [%]	Ca [%]	Ti [%]	Fe [%]	Zr [%]	Pb [%]
01	0.901	73.06	0.016	0.269	0.053	25.52	0.002	0.002
02	0.938	73.09	0.017	0.264	0.049	25.48	0.002	0.002
03	0.933	73.03	0.017	0.269	0.049	25.47	0.002	0.002
04	0.939	72.98	0.016	0.273	0.052	25.47	0.002	0.002
05	0.933	72.99	0.017	0.274	0.051	25.47	0.002	0.002
06	0.934	72.98	0.015	0.263	0.049	25.46	0.002	0.002
07	0.962	72.99	0.017	0.268	0.053	25.46	0.002	0.002
08	0.946	72.96	0.016	0.272	0.049	25.46	0.002	0.002
09	0.959	73.00	0.015	0.265	0.050	25.44	0.002	0.002
Certified	0.953 (b)	73.13 (a)	0.0105 (b)	0.271 (b)	0.051 (b)	25.24 (a)	< 0.005 (c)	--
Average	0.938	73.01	0.016	0.269	0.051	25.47	0.002	0.002
Rel. Std. Dev.	1.9 %	< 0.1 %	5.1 %	1.5 %	3.4 %	0.1 %	2.2 %	10.6 %

SampleCare™

Bruker's unique SampleCare technology (Figure 5) protects the vital system components in the event of sample breakage or liquid cup leakage. The sample detection system prevents the measurement of liquid or powder samples under vacuum. SampleCare is key for high system uptime and short & easy maintenance.

Conclusion

This lab report highlights the outstanding capabilities of the S2 PUMA Series 2 for quality control of Ferrosilicon and similar material. Equipped with modern software solutions and state-of-the-art hardware, the S2 PUMA is ideally configured to enable best-in-its-class analytical performance.

The S2 PUMA EDXRF spectrometer is ready for challenging industrial environments and applications with its TouchControl interface and its SampleCare technology. The 22-position XY Autochanger enables high sample throughput while keeping all flexibility. Mixed batches can be loaded (liquid, powder, solid, ...) and samples can be added at any time. You need to change priorities and quickly analyze a production sample? No problem with the S2 PUMA.

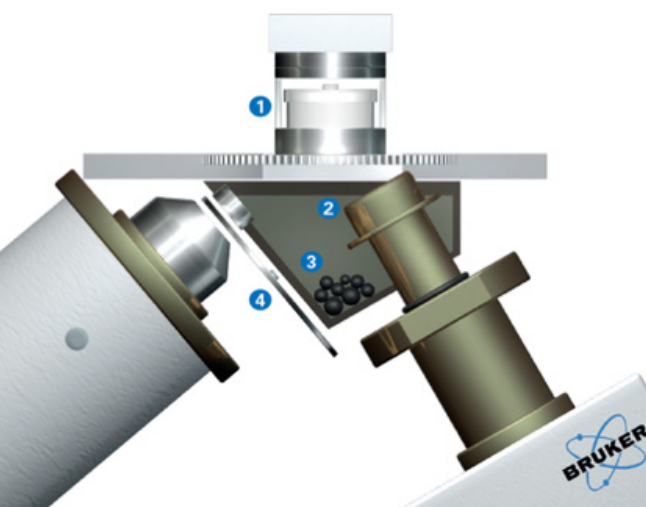


Figure 5 SampleCare™:

1. Grabber with automatic sample detection.
2. DustShield™ detector cap.
3. Dust reservoir.
4. Filter wheel.



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