Application Note WDXRF1111

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XRF1111 - FeO analysis in iron ore sinters using the XRD channel on multi-channel XRF spectrometer Simultix15

Introduction

The content of ferrous oxide (FeO) representing divalent iron (Fe^{2+}) in sintered iron ore must be controlled to improve the reducibility and control the temperature in blast furnaces in iron production processes.

Fe^{2+} in iron ore sinter is typically determined by the titrimetric method. Since samples must be dissolved with acids and the acidic solution of the samples is titrated with potassium dichromate solution, acidic and harmful waste liquid must be disposed of, which is costly and time-consuming.

There is a good correlation between the contents of FeO and Fe_3O_4 (magnetite: $Fe^{2+}Fe^{3+}{}_2O_4$) in sinters. FeO content can be determined by analyzing Fe_3O_4 by the X-ray diffraction method (XRD).

Rigaku Simultix 15, a multi-channel simultaneous wavelength-dispersive XRF (WDXRF) spectrometer, can be equipped with an XRD channel for FeO analysis measuring the diffraction peak of magnetite. This means it is not necessary to use a separate XRD system to analyze FeO.

For this analysis, sample preparation is very simple. Powder samples are pressed into rings or cups at high pressure (100 – 300 kN) to form pressed powder briquettes (disks). The interpersonal error is small and the total analysis time is short because of the simple sample preparation.

This application note demonstrates quantitative analysis of FeO content using the XRD channel, as well as XRF analysis results of sinters using the Simultix 15.

Instrument

The Simultix15 is a simultaneous WDXRF spectrometer with multiple-channels, which enables simultaneous measurement of all the elements in analysis samples. The instrument is designed for rapid analysis with high reliability and stability for routine analysis for production control.

The Simultix 15 is equipped with either a 4 kW or a 3 kW X-ray tube and fixed channels optimized for the elements to be measured. Configurations with up to 30 fixed channels (optionally, 40 channels) are available for simultaneous analysis.

The Simultix 15 configured for FeO analysis is equipped with a 4 kW X-ray tube, the XRD channel for FeO analysis and up to 24 fixed channels for XRF analysis.

An intelligent Automatic Sample Changer (ASC), capable of placing up to 48 samples, is optionally available for high-demand jobs.

The operation software has been improved to facilitate daily-use operation. The integrated “Flowbar” in quantitative analysis guides users through the calibration procedure.

Sample preparation and measurement

Each eight gram sample was pressed into a PVC ring (I.D. 31 mm) at 80 kN.

Measurements were performed in vacuum on the Simultix 15 configured for FeO analysis. When measurement with the FeO XRD channel is performed, an irradiation parallel slit is inserted between the X-ray tube and the sample. For measurements with XRF channels, the parallel slit is not used. This irradiation parallel slit is programmatically controlled. The excitation condition is 50 kV and 60 mA. The counting time is 40 seconds each for XRD and XRF measurements.

Calibration

House standards of sinters were used for calibration. The calibration curve of FeO using the XRD channel is shown in Figure 1. The calibration accuracy is 0.34 mass%.

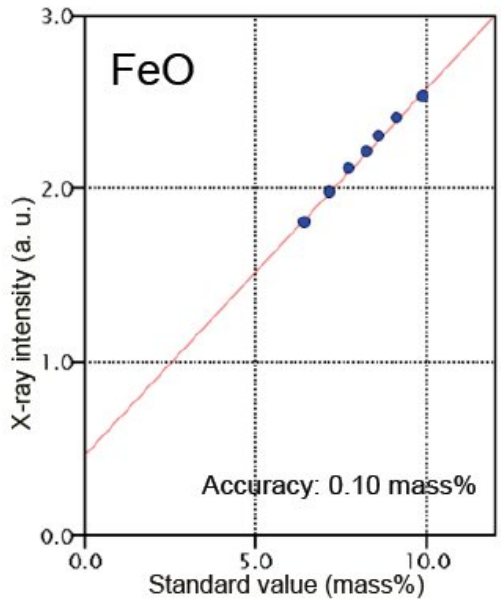

Figure 1: Calibration curve of FeO using FeO XRD channel.

The calibration curves of the other components using the fixed XRF channels are shown in Figure 2.

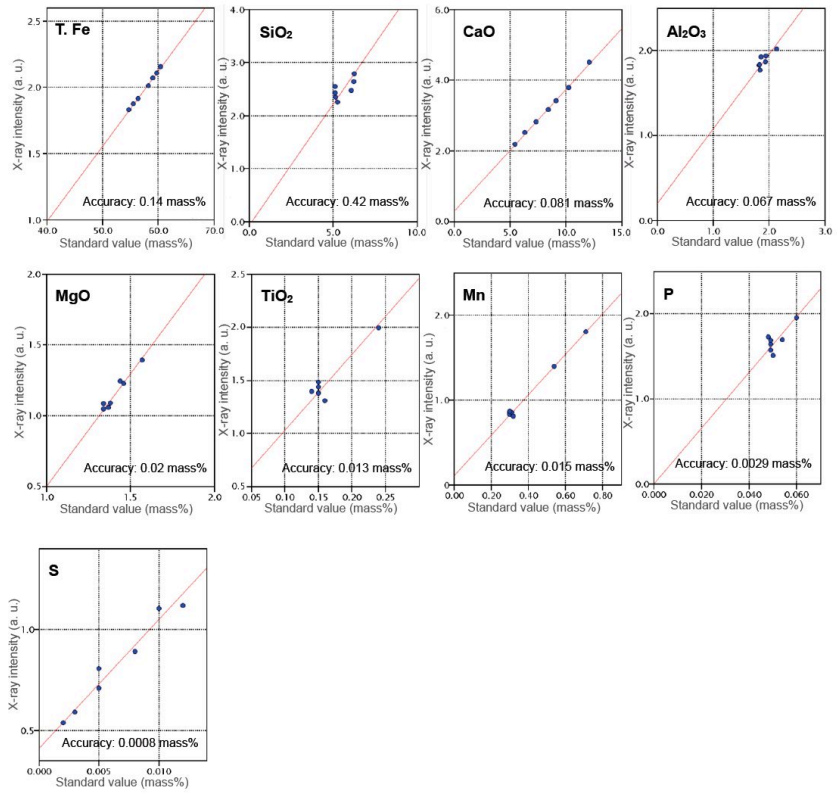

Figure 2: Calibration curves of sinter using fixed XRF channels.

The accuracy of calibration is calculated by the following formula:

$$Accuracy = \sqrt{\frac{\sum_i (C_i - \hat{C}_i)^2}{n-m}}$$

C_i: calculated value of standard sample
$\hat{C}_i$: reference value of standard sample
n : number of standard samples.
m: degree of freedom (linear 2, quad. 3)

Repeatability test

A repeatability test was performed with 10 measurements. The results of the 10 repeat measurements are recorded in Table 1.

Table 1: Repeatability test results

	FeO (XRD)	T.Fe	SiO_2	CaO	Al_2O_3	MgO	TiO_2	Mn	P	S
1	6.52	55.23	5.25	6.25	1.968	1.40	0.144	0.497	0.0598	0.0101
2	6.50	55.19	5.26	6.24	1.971	1.40	0.144	0.496	0.0607	0.0098
3	6.51	55.20	5.25	6.25	1.967	1.40	0.144	0.496	0.0593	0.0100

4	6.53	55.19	5.25	6.25	1.968	1.39	0.144	0.496	0.0598	0.0102
5	6.50	55.19	5.26	6.24	1.964	1.40	0.144	0.496	0.0599	0.0096
6	6.48	55.21	5.25	6.25	1.965	1.40	0.144	0.496	0.0600	0.0102
7	6.49	55.18	5.24	6.25	1.970	1.40	0.143	0.496	0.0600	0.0098
8	6.51	55.20	5.25	6.25	1.971	1.40	0.144	0.496	0.0599	0.0104
9	6.45	55.18	5.25	6.25	1.971	1.40	0.144	0.496	0.0602	0.0100
10	6.48	55.22	5.26	6.25	1.963	1.40	0.145	0.496	0.0605	0.0095
Average	6.50	55.20	5.25	6.25	1.968	1.40	0.144	0.496	0.0600	0.0100
Std. dev.	0.023	0.016	0.005	0.003	0.0028	0.003	0.0005	0.0005	0.0004	0.0003
R.S.D.%	0.36	0.03	0.09	0.05	0.14	0.22	0.33	0.10	0.67	2.82

Conclusion

The Simultix15 equipped with an XRD channel for FeO analysis gives excellent analysis results for FeO in sinters, as well as for the other components analyzed by the fixed XRF channels.

X-ray fluorescence spectrometry is a rapid, precise and accurate analysis method that meets the requirements of the steel industry. It can also minimize cost and time in iron ore and sinter analysis. A multi-channel system is best suited for plant laboratories, where a high volume of sample analysis is required for quality and process control.

The combination of XRF and XRD analysis channels in the Simultix15 is the best solution for production and quality control of agglomeration process for the ingredients of iron making.

Related products

Simultix15

High-throughput tube-above multi-channel simultaneous W DXRF spectrometer analyzes Be through U.