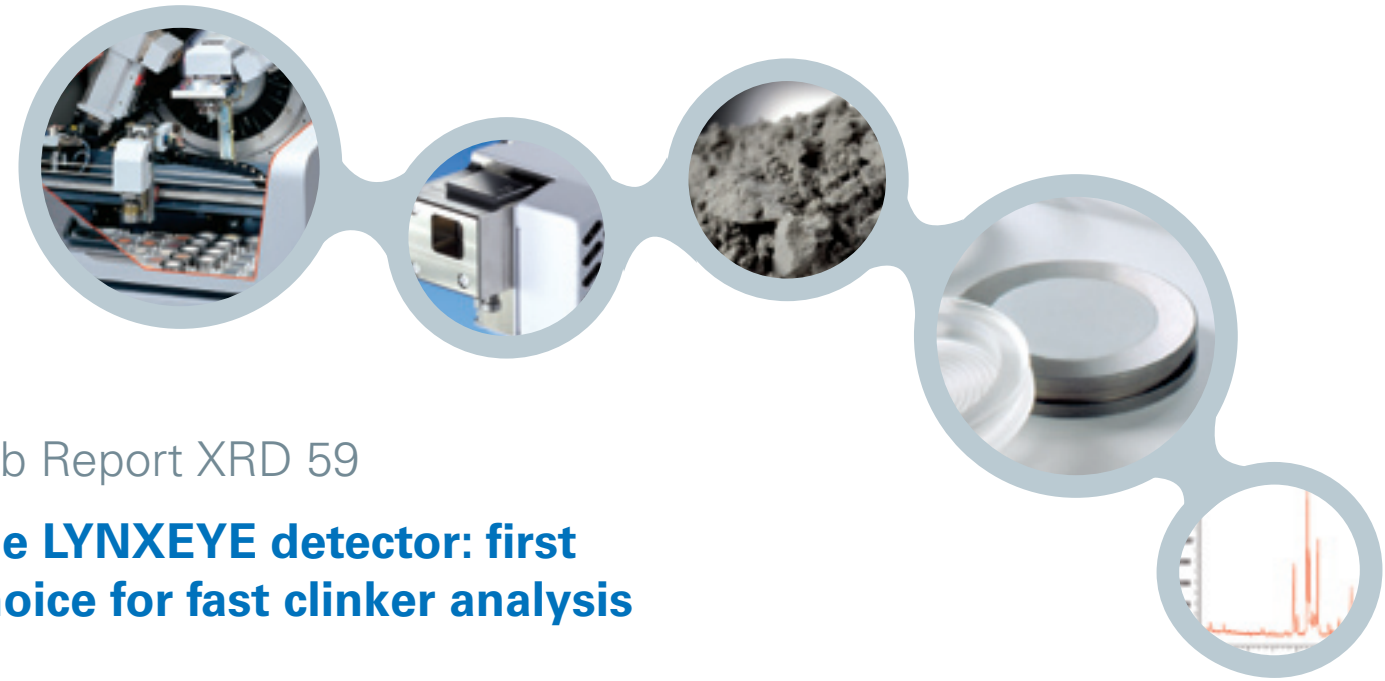
Bruker AXS



Lab Report XRD 59

The LYNXEYE detector: first choice for fast clinker analysis

Introduction

The combination of XRD measurements with Rietveld analysis currently provides the most powerful method available for automated quantitiative phase analysis in the cement industry. For clinker analysis, Bruker AXS offers market-leading software (see grey text box below) and hardware solutions. In addition, to meet the increasing needs of higher sample throughput in industrial applications, a fast and affordable Super Speed detector, the LYNXEYE™ (Fig. 1) is available.

This Labreport deals with the accuracy and precision which is achievable by combining TOPAS Rietveld analysis and fast data acquisition with the D4 ENDEAVOR (Fig. 2) diffractometer equipped with the unique LYNXEYE detector.

The LYNXEYE is a 1-dimensional detector, based on Bruker AXS' compound silicon strip technology. Compared to a simple point detector, the LYNXEYE dramatically increases measured intensities without sacrificing resolution and peak shape. A Diffraction Solution equipped with a LYNXEYE records a typical powder pattern in approximately 1/200th of the time required using a point detector.

For this study, a cement clinker was re-evaluated that was used for a Rietveld Round Robin organized by the Verein Deutscher Zementwerke e.V. (German Cement Works Association) in 2005. Sample preparation was done by grinding and pressing the sample in a standard steel ring using the POLAB®APM unit from Polysius. This sample was measured ten times (instrumental setup in Tab. 1) to determine the reproducibility of the analysis. The measurement time was less than 5 minutes. After each measurement the sample was removed from the measurement position and reloaded. The calculation time with TOPAS (Version 4.2) was less than 5 seconds using a Pentium 2.8 GHz Processor.

Figure 1: The LYNXEYE detector

TOPAS' speed of calculation and the stability of this new generation of refinement software is the decisive step to overcome the limitations of traditional Rietveld programs. The seamless integrated Fundamental Parameters Approach (FPA) allows a physically correct description of the measurement data, which finally ensures getting accurate analytical results. There is no need for a reference measure. It is not only the most successful Rietveld software; it is meanwhile the scientific and industrial standard in the world of cement.

Configuration of the Diffractometer

Instrument	D4 ENDEAVOR
Measurement circle	401 mm
Tube	2.2 kW Cu long fine focus
Tube Power	40 kV / 45 mA
Primary optics	Divergence slit fixed to 0.5° 4° Soller slit Air-scatter-screen
Rotation sample stage	30 rpm
Secondary optics	4° Soller slit Ni Cu - Kß filter
Detector	LYNXEYE Detector opening 4°
Step size	0.02
Time per step	0.1 sec
Measurement range	10° to 65° (2Theta)
Total measuring time	4 min 37 sec

Table 1: D4 ENDEAVOR Set up with LynxEye detector

Figure 2: The D4 ENDEAVOR diffractometer

Measurement Data

Fig. 3 shows a typical XRD scan of the VDZ Clinker sample. Note the overall intensity and the background at low angles. The entire scan time was 4 minutes 37 seconds.

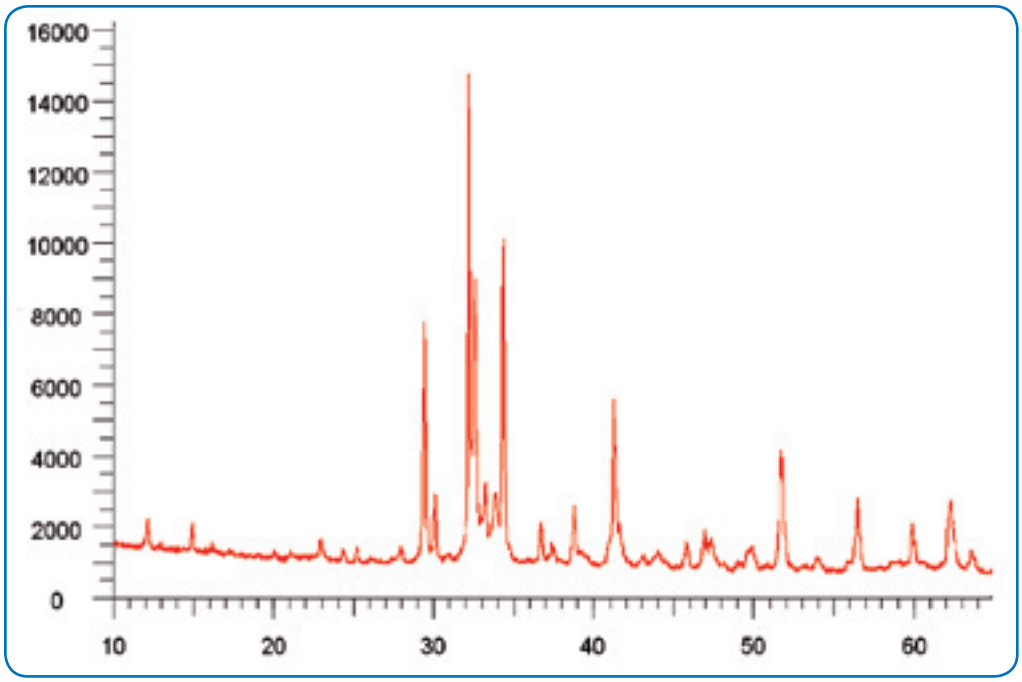


Figure 3: LynxEye measurement data of the VDZ Clinker sample (counts versus °2Θ)

	Alite	Belite	C_3A ortho	C_3A cubic	C_3A sum	Ferrite	Periclase	Freelime	Arcanite	Quartz
Measurement 1	70.7	8.1	4.8	3.0	7.8	10.7	0.5	1.5	0.7	0.0
Measurement 2	70.2	8.5	4.6	3.1	7.7	10.8	0.5	1.5	0.7	0.0
Measurement 3	70.0	8.4	4.7	3.1	7.9	10.8	0.6	1.5	0.9	0.0
Measurement 4	70.4	8.1	4.9	2.9	7.8	10.8	0.7	1.5	0.7	0.0
Measurement 5	70.1	8.5	4.9	2.9	7.7	10.8	0.6	1.5	0.7	0.0
Measurement 6	70.1	8.5	5.0	3.1	8.0	10.5	0.6	1.5	0.7	0.0
Measurement 7	70.1	8.5	4.8	2.9	7.7	10.8	0.6	1.6	0.8	0.0
Measurement 8	70.0	8.6	4.6	3.1	7.8	10.7	0.7	1.5	0.8	0.0
Measurement 9	70.2	8.4	5.0	2.9	7.9	10.8	0.6	1.5	0.6	0.0
Measurement 10	70.2	8.6	4.9	3.1	8.0	10.6	0.6	1.5	0.7	0.0
Mean	70.2	8.4	4.8	3.0	7.8	10.7	0.6	1.5	0.7	0.0
Std. Dev. (1 σ)	0.20	0.18	0.12	0.10	0.11	0.11	0.06	0.02	0.07	0.00

Table 2: Reproducibility test TOPAS analysis of the VDZ Clinker sample (values in weight %)

Results

Table 2 gives the TOPAS Rietveld results for all measurements as well as the mean value and the standard deviation given in one sigma. The reproducibility for the Calciumslicates Alite (C_3S) and Belite (C_2S) is about 0.2 wt. %. For the interstitial phases, the Aluminate (C_3A) and the Ferrite (C_4AF) phase it is about 0.1 wt. % and for the minor phases even well below 0.1 wt. %. No other existing method for clinker phase quantification allows this level of analytical performance. Changes and trends in the running process can be clearly recorded without ambiguity.

These reproducibility tests are part of the Bruker AXS standard acceptance tests to guarantee results of the highest quality. The unique 1-dimensional LYNXEYE detector offers the fastest measurements, together with accurate and precise phase quantification. TOPAS Rietveld analysis is meanwhile not only a valuable tool for clinker quality and process control, it also has shown its value in other parts of the whole production process. Examples include hotmeal and bypass control, or the determination of the sulphate phases and the limestone filler in the finalized cement (CEM I).

Author

Rainer Schmidt
Bruker AXS, Karlsruhe, Germany

All configurations and specifications are subject to change without notice. Order No. DOC-L88-EXS059.
© 2009 Bruker AXS. Printed in Germany.

Bruker AXS GmbH
Karlsruhe, Germany
Phone +49 (721) 595-2888
Fax +49 (721) 595-4587
info@bruker-axs.de
www.bruker-axs.de

Bruker AXS Inc.
Madison, WI, USA
Phone +1 (800) 234-XRAY
Phone +1 (608) 276-3000
Fax +1 (608) 276-3006
info@bruker-axs.com
www.bruker-axs.com