Application Note B-XRD1140

View on rigaku.com

B-XRD1140 - Accurate and highly precise quantitative analysis of cement samples using Rietveld refinement

Introduction

Properties of cement, such as curing time and strength, depend on the clinker components. Optical microscopy and X-ray diffraction analysis are used as quantitative analysis methods of clinker components (crystalline phases). In particular, quantitative analysis using X-ray diffraction combined with Rietveld refinement is widely used in cement research and quality control as a quick and easy method. Precise and accurate analyses are required to evaluate correct cement characterization. Also, high-speed measurements are required since free lime and other components may change depending on the environment. We evaluated the quantification accuracy and precision of Rietveld refinement by performing tenfold iterative measurements with the MiniFlex benchtop X-ray diffractometer equipped with the D/teX Ultra2 high-speed 1D X-ray detector. One measurement took approximately five minutes. The NIST clinker standard reference material (NIST2688) was used for the measurement.

Measurements and results

Rietveld refinement was performed using a template function from the SmartLab Studio II integrated X-ray analysis software. The template function allows the analysis of multiple data sets under the same conditions by saving information about crystal phases, the initial values of refinement parameters, and the refinement procedure in advance. Figure 1 shows the quantitative results and the precision of ten measurements, in addition to the certified NIST2688 values. Crosses correspond to 10 quantitative values and black bars denote the certified value and its uncertainty for each crystalline phase. Table 1 shows the average of the quantitative values for each component (Standard deviation: 1σ (n = 10)) and the certified value. Figure 1 and Table 1 show that the quantitative values for all crystalline phases are at and around the certified value, indicating that the obtained quantification values are highly accurate. In addition, the standard deviation in the quantitative values are extremely small, implying that the quantitative analysis was very precise. These results prove that the high-speed 1D X-ray detector provides high-intensity and high-quality measurement data in a short period of time, and the Rietveld refinement with the template function enables accurate and highly precise quantitative analysis of cement samples.

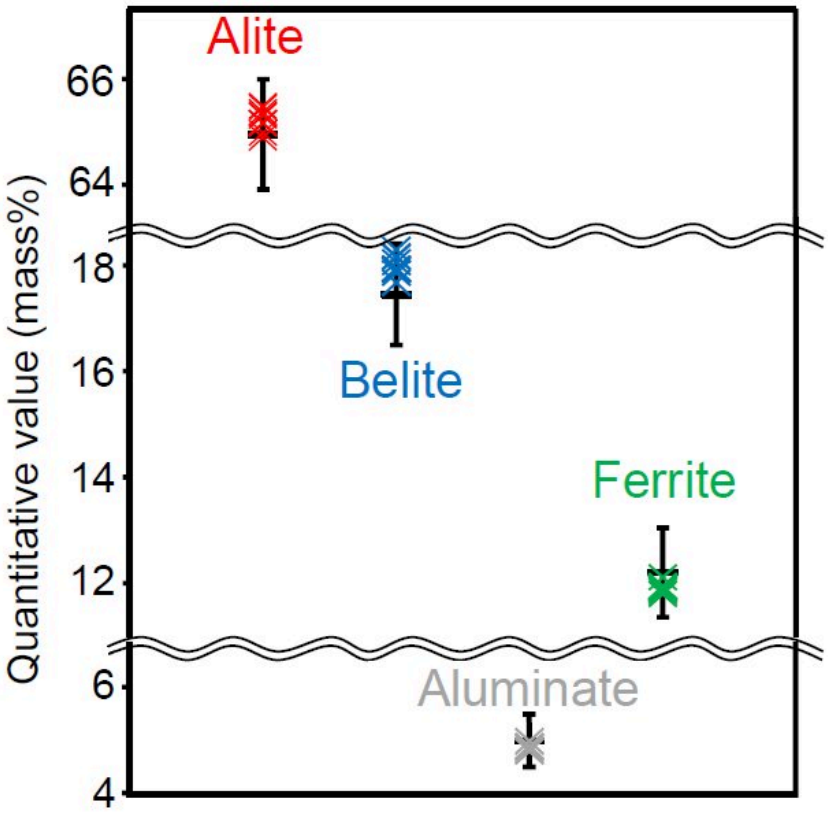

Figure 1: Quantitative results and precision of each crystalline phase in NIST2688 by Rietveld refinement

Table 1: The quantitative values and certified values of NIST2688 (Units: mass%, Standard deviation: 1σ (n = 10))

Crystalline phase	Quantitative value	Certified value
Alite	65.23 ± 0.20	64.95 ± 1.04
Belite	17.99 ± 0.18	17.45 ± 0.96
Aluminate	4.87 ± 0.07	4.99 ± 0.50
Ferrite	11.9 1± 0.08	12.20 ± 0.84

Related products

MiniFlex

New sixth-generation general purpose benchtop XRD system for phase i.d and phase quantification

SmartLab

Advanced state-of-the-art high-resolution XRD system powered by Guidance expert system software

SmartLab SE

Highly versatile multipurpose X-ray diffractometer with built-in intelligent guidance

SmartLab Studio II

Windows-based software suite for Rigaku's X-ray diffractometers