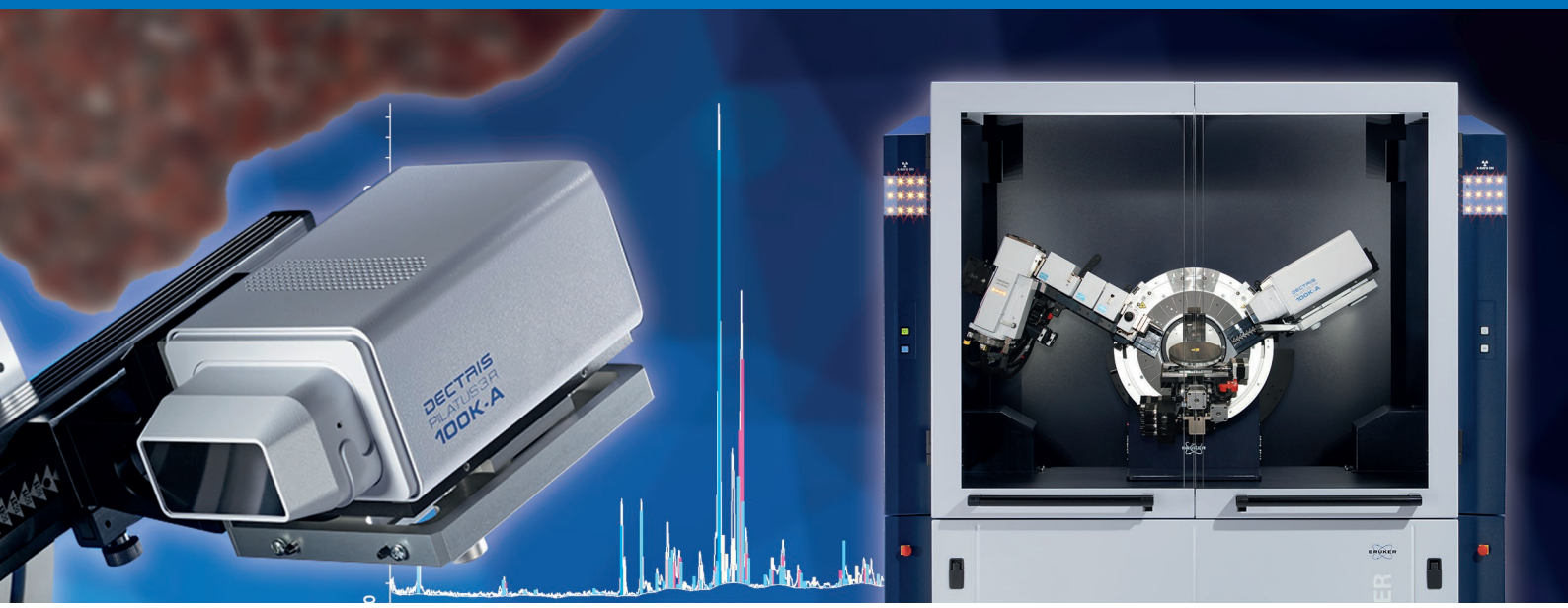

Application Report XRD 21

D8 DISCOVER with PILATUS3 R 100K-A micro XRD² of Quartz Monzonite

The D8 family of diffraction solutions combined with the PILATUS3 R 100K-A hybrid photon counting (HPC) pixel detector is an innovative x-ray diffraction (XRD²) solution that is uniquely suited for multipurpose modern materials research characterization. In this report, we present the capabilities of this system in a two dimensional diffraction configuration for the analysis of coarse grained geological samples for applications such as phase identification (Phase ID) and quantitative phase analysis.

Introduction

X-ray diffraction is unique in its ability to identify and quantify the structural phases present in a sample; this is in contrast to the elemental constituents identified with many other techniques. When analyzing geological samples, sensitivity to the structure of various phases is exceptionally important as many phases have the same elemental building blocks, differing only in the way that those blocks are arranged. Frequently, it is advantageous to leave the sample in an as-collected state, rather than reducing it to a powder in a ball mill, to allow further petrographic analysis. Two dimensional detectors are uniquely suited for this analysis as the large diffraction space coverage can capture reflections from crystallographic phases with large grain sizes and preferred orientations that would otherwise be missed.

Measurement

A sample of quartz monzonite, a coarse grained igneous rock (Figure 1), was collected and mounted such that a flat section of the sample was aligned to the center of the instrument. Measurements were performed in reflection geometry with a D8 DISCOVER equipped with a spot-beam source and PILATUS3 detector. Two coupled scans from 10˚ to 70˚ in 2θ with an increment of 5˚ and 2 minutes per step were performed. The XRD² data was collected with a sample to detector distance of 33 cm. A 300 µm primary beam collimator was used to define the incident beam diameter. The scan shown in Figure 2a was collected with the sample statically positioned at the X in Figure 1, while the scan shown in Figure 2b was collected with X and Y oscillation of +/-2 mm and Phi rotation of 3 rpm, equivalent to the circle in Figure 1.

Results

The two dimensional scattering pattern was visualized and integrated to a one dimensional diffractogram with DIFFRAC.EVA. Phase ID and a quantitative Rietveld analysis was performed on this 1D pattern.

Strong single crystal reflections are observed in Figure 2a. Motion of the sample successfully removes the coarse grain effects common to plutonic rocks, resulting in Debye rings typical for powdered samples (Figure 2b). The integrated diffractogram of this pattern is shown in Figure 3. The identification of the crystalline phases present in the sample can easily be performed, and the peak positions and the relative intensities match the data base entries. In addition to the relative intensity data present in the one dimensional diffractogram generated from the collected XRD² data, the static two dimensional image can be used to supplement the phase identification process. By identifying diffraction rings with similar uniformity, subsets of the reflections can be identified and searched against.

The one dimensional diffractogram was further analyzed for phase quantification in DIFFRAC.TOPAS. Effects such as preferred orientation and axial divergence are minimized or eliminated due to the large gamma coverage of a two dimensional detector. The result of this analysis is shown in Figure 4, positively identifying the rock as quartz monzonite.

Author

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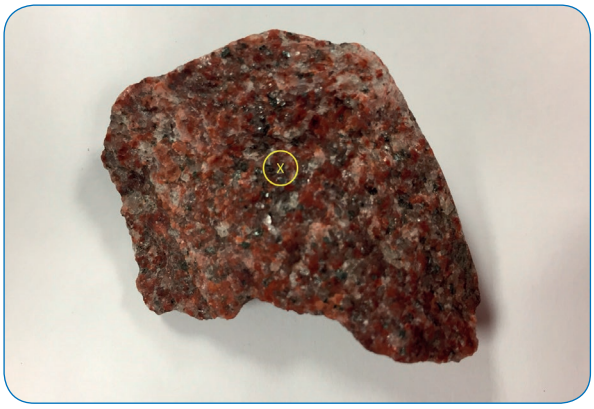

Figure 1. Quartz monzonite rock sample. The yellow X indicates the position where Figure 2a was collected, while the circle indicates the region rastered over resulting in image 2b.

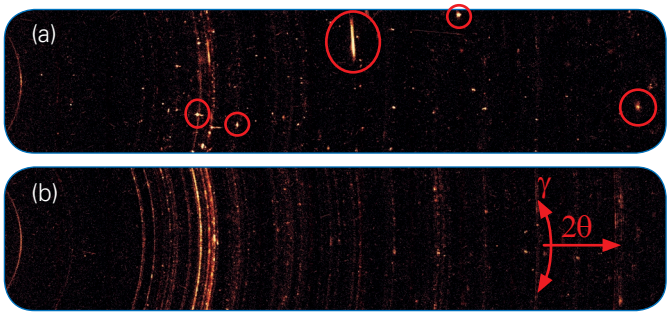

Figure 2. Resulting images from a coupled scan without (a) and with (b) X, Y and Phi motion. Red circles indicate some single crystal reflections.

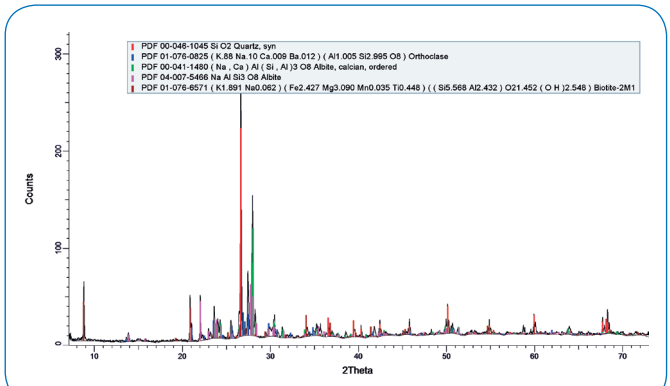

Figure 3. Phase Identification in DIFFRAC.EVA of an integration of the image collected in Figure 2b.

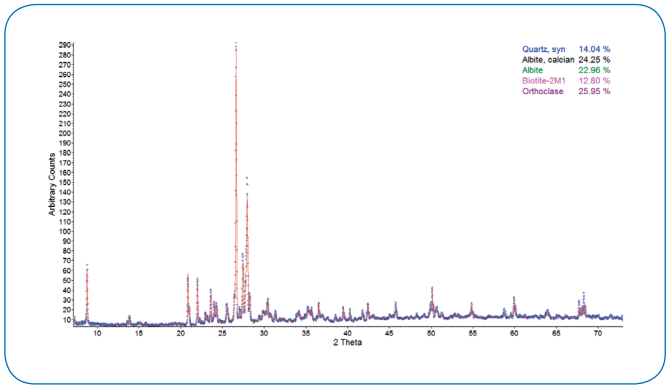

Figure 4. Quantitative Rietveld Refinement in DIFFRAC.TOPAS of an integration of the image collected in Figure 2b.

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