Application Note XRD 616

µXRD in Geology with EIGER2 R 500K

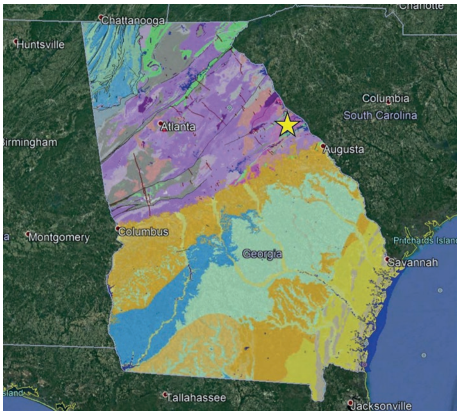
In-Situ Study of Lazulite from Graves Mountain, Georgia, USA

X-ray diffraction is a technique commonly used by geologists for qualitative and quantitative mineralogy. In this report we investigate an in-situ lazulite crystal along with the surrounding matrix using a combination of Bragg-Brentano and microdiffraction. By using microdiffraction in combination with rotation of the sample in a pseudo-Gandolfi mode (phi rotation and psi oscillation) it is possible to reveal trace mineral phases that cannot be identified using traditional powder methods alone.

Report Summary

- The sample was characterized with large area Bragg-Brentano with 1D detection and small area Microdiffraction with 2D detection
- Microdiffraction revealed differences in composition and morphology of the matrix and inclusion phases
- Rotation in phi and psi allows access to additional reflections for large crystallite in-situ specimens

The traditional bulk mineralogy approach is to reduce the sample to a homogenous powder and analyze with a divergent beam (Bragg-Brentano) geometry. Grinding is not always a practical option, due to rarity of a given specimen or the desire to characterize the spatial relationship of multiple phases within an assemblage. Microanalysis techniques offer the ability to analyze the sample in its native state, easing analysis of the individual constituents.



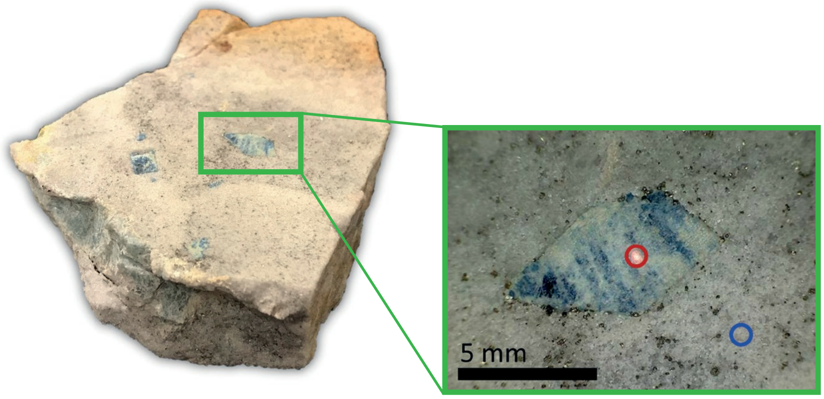


Figure 1. Geological map of Georgia, USA[1]. Origin of lazulite sample indicated by small yellow star.

Figure 2. Photographs of lazulite specimen. Bragg-Brentano data was collected from the region indicated by the green square. Microdiffraction data were collected from the two regions indicated by red and blue spots.

Introduction

The lazulite sample used in this report was obtained from Graves Mountain, a NE/SW striking monadnock located in eastern Georgia, USA (Figure 1). Part of the Carolina Slate Belt, the low- to medium-grade metavolcanics of Graves Mountain were mined for kyanite to be used in high-alumina refractory products. In addition to kyanite, the locale is known to mineral collectors for its world-class twinned rutile crystals, iridescent hematite coatings on quartz, lazulite, pyrite, and pyrophyllite.

Experimental

Diffraction data were collected using a D8 ADVANCE diffractometer equipped with copper (Cu) radiation, TRIO multipath optic, Compact CradlePlus, and EIGER2 R 500K detector.

Bragg-Brentano

Traditionally, mineralogical specimens are measured using a Bragg-Brentano geometry. A wide area scan of the rock slab (Figure 2, green box) was collected using a Bragg-Brentano geometry with a line beam and a scanning 1D detector mode (Figure 3). The specimen was rotated in phi during the measurement. Quartz from the matrix dominates the diffraction pattern and trace amounts of variscite and pyrite can also be identified - but the phases associated with lazulite are absent despite visual confirmation.

This approach works well for powder specimens, but fine detail of in-situ specimens can be lost in noise or, in this case, missed completely as would be the case with the lazulite crystal in this example.

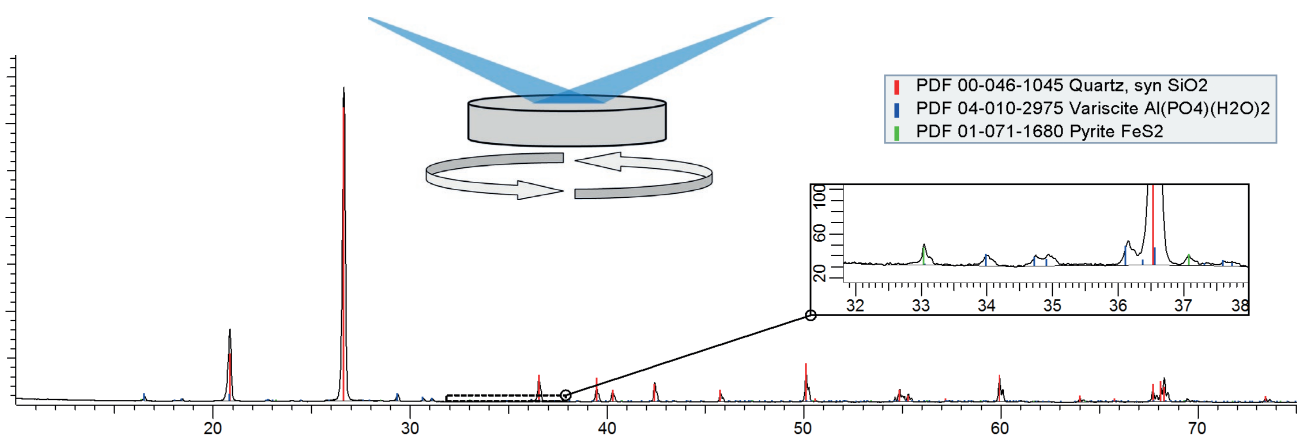


Figure 3. Wide area scan in Bragg Brentano geometry (inset schematic). Because the illuminated area is so large, the diffraction signal is dominated by the matrix, primarily quartz. Trace amounts of variscite and pyrite are also identified.

Microdiffraction

A 2D microdiffraction experiment was used to better understand the crystalline nature of the lazulite. The lazulite crystal and quartzite matrix (Figure 2, red and blue circles) were measured in-situ using a 0.5 mm spot beam and the EIGER2 in scanning 2D mode (Figure 4). During the measurement the specimen was rotated in phi at 3 rpm and oscillated in psi from 0˚ to 35˚ (pseudo-Gandolfi) to access more reflections. Microdiffraction confirms the matrix is primarily fine-grained quartz, while the large lazulite crystal contains intergrowths of polycrystalline variscite, metavariscite, and gypsum (Figure 5).

Conclusions

The blue lazulite crystals of Graves Mountain Georgia contain small polycrystalline mineral inclusions of variscite, metavariscite, quartz, and gypsum.

Microdiffraction can be used to investigate a smaller region of interest of in-situ rock slabs and thin sections. By utilizing rotation and tilt of the sample, the sample can be maintained in a pristine state while collecting high quality powder diffraction data from a small area. The EIGER2 detector is capable of seamless transitions from 1D to 2D geometries, making it easy to collect both traditional powder scans as well as microdiffraction data.

[1] USGS Georgia Geologic Map Data, https://mrdata.usgs.gov/geology/state/state.php?state=GA

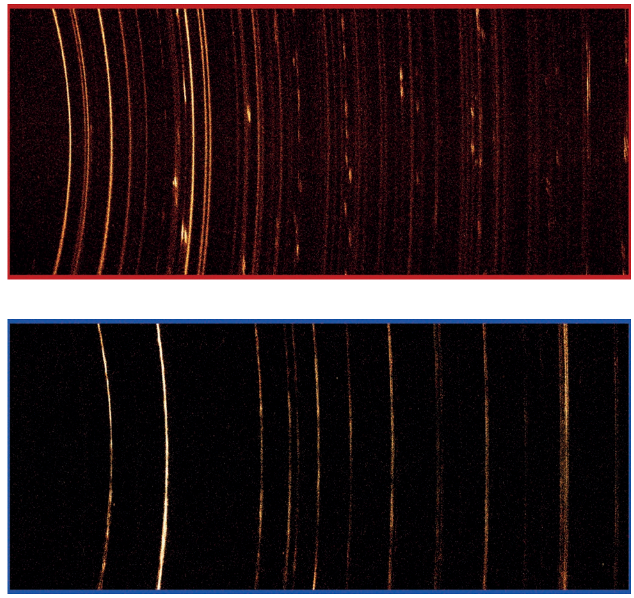


Figure 4. Scanning 2D frames for microdiffraction of the lazulite porphyroblast (top, red frame) and quartzite matrix (bottom, blue frame). The spottiness of the lazulite phase is indicative of a large single-crystal with smooth diffraction rings indicating fine-grained intergrowths.

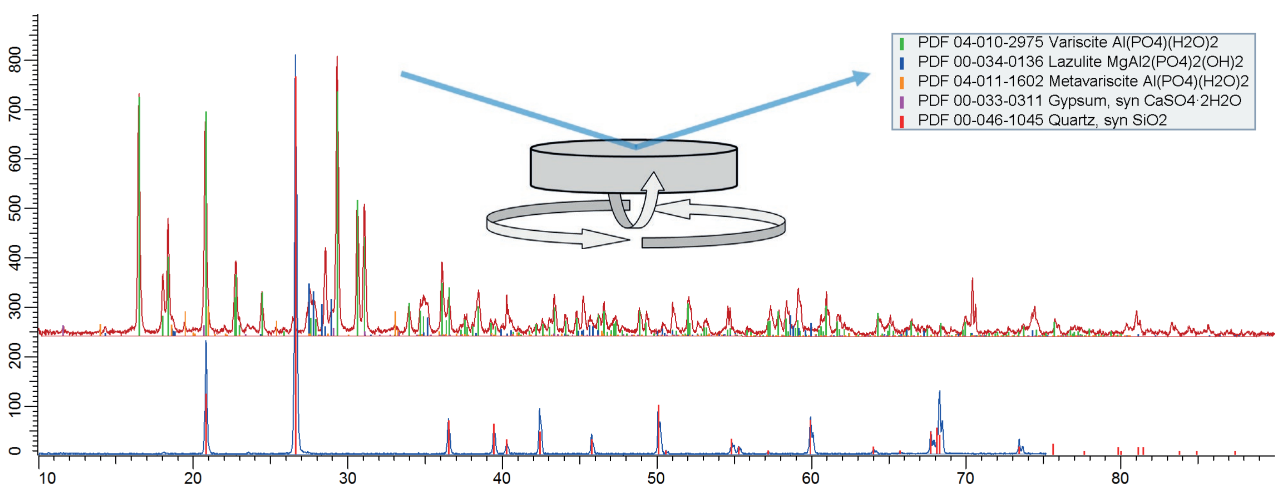
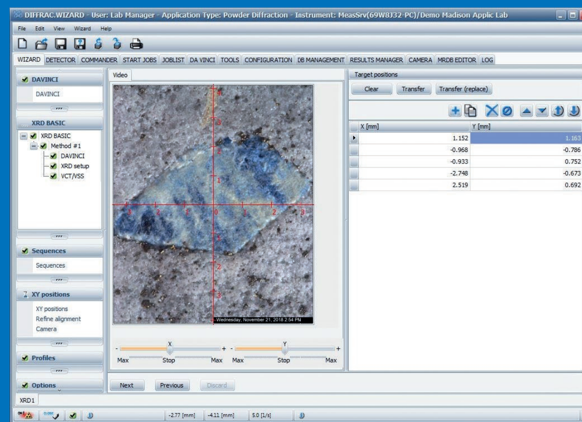


Figure 5. Integrated data from microdiffraction geometry (inset schematic). The matrix data (blue) is predominantly quartz and the data from the lazulite inclusion (red) shows multiple mineral phases.

DIFFRAC.SUITE Workflow for Microdiffraction

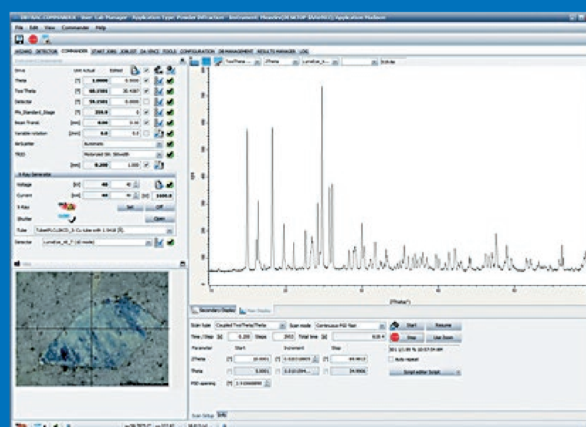
PLAN in DIFFRAC.WIZARD

- Set up multiple measurements with individual X, Y, and Z coordinates
- Large area mapping with rastering and sequencing
- Configure random motions in phi and/or psi to improve sampling statistics



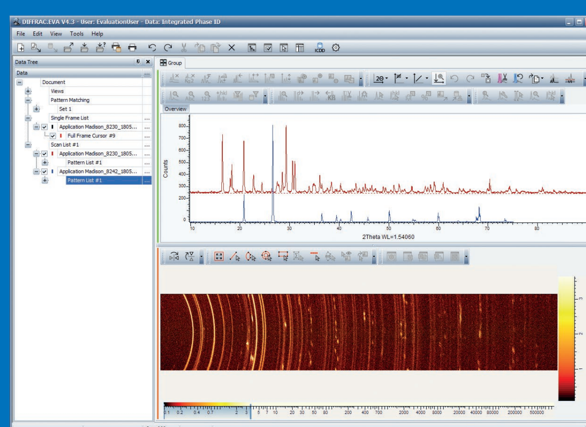
MEASURE in DIFFRAC.MEASUREMENT

- Direct measurement control or launch pre-defined experiment methods
- Real-time data monitoring in both 1D and 2D detector modes
- Integrated camera plugin for verifying sample positioning



ANALYZE in DIFFRAC.EVA

- Supports 0D, 1D, and 2D data
- Create customized report layouts and templates
- Phase Identification with a variety of commercial and user-defined databases



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