Application Note M0-001

Microdiffraction – µXRD Analysis of Geode Interior

Microdiffraction is x-ray diffraction analysis on a small amount of sample or a small area of a larger sample. Microdiffraction requires special XRD instrumentation, including a point focus source, special sample holders for large or irregularly shaped parts, and ideally a two dimensional area detector to observe a large section of the Debye ring (diffraction pattern). The 2D detector is essential in collecting data on trace phases, weak signal, and/or large individual grains which have a high degree of orientation.

Figure 1 Quartz geode with black crystalline material in center.

In this particular example, a quartz geode was cracked open exposing a thin black film. This presented a perfect application for µXRD since it was desirable to keep the sample intact rather than to attempt to scrap out the black deposit. Figure 1 shows one half of the broken quartz geode with the black deposit. Figure 2 shows the sample mounted on a Bruker D8 Discover system.

Phase identification with search/match was performed on the resulting XRD pattern, and the results were quantified using RIR (Reference Intensity Ratio) values and Rietveld Refinement.

The table below shows the domination of the quartz in the calculation of Wt%, and it is most likely that the quartz signal is from the white colored foundation itself. The Wt% is also normalized for the absence of quartz, and the results shown in the last column of the table show that the deposit is primarily composed of iron oxide hydroxide in the orthorhombic form of Goethite, and Aluminum hydroxide, in the monoclinic form of Gibbsite.

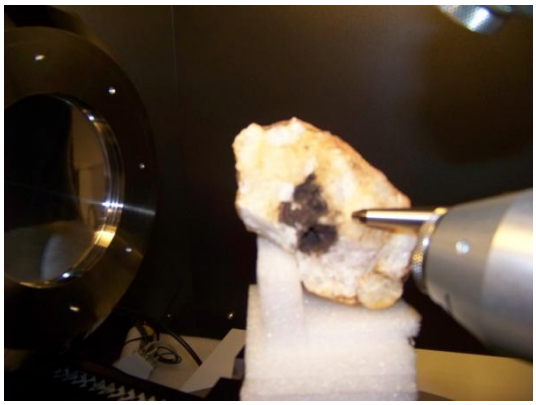
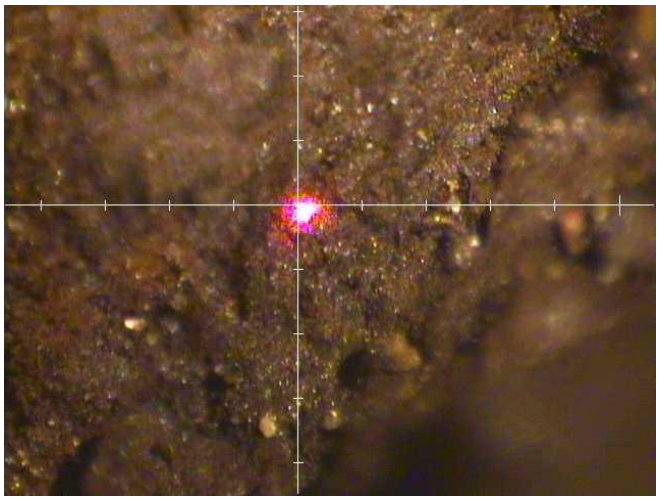


Figure 2 Geode mounted on D8 system

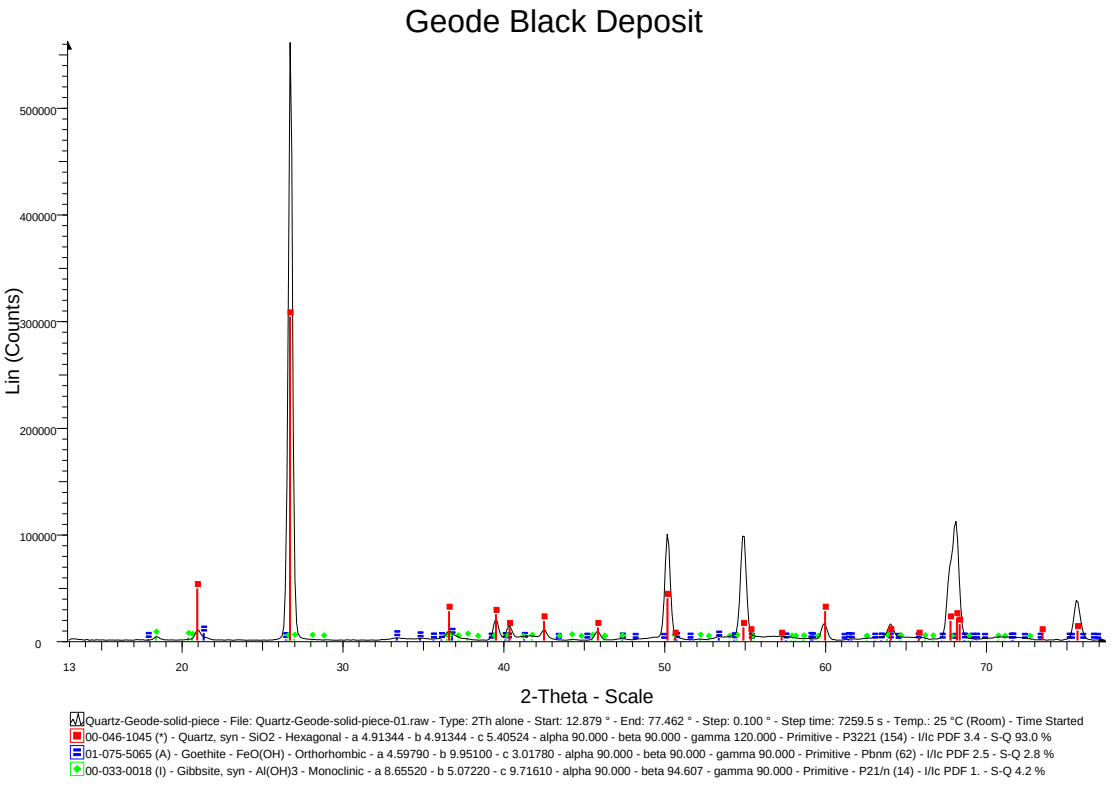
Phase (compound)	Structure	Quantitative Wt %	Normalized Wt%
Quartz – SiO_2	Hexagonal	89.6	N/A
Goethite – FeO(OH)	Orthorhombic	4.1	39.4
Gibbsite – $Al(OH)_3$	Monoclinic	6.0	57.7
Iron Titanium Oxide- Fe_2Ti_4O	Cubic	0.3	2.8



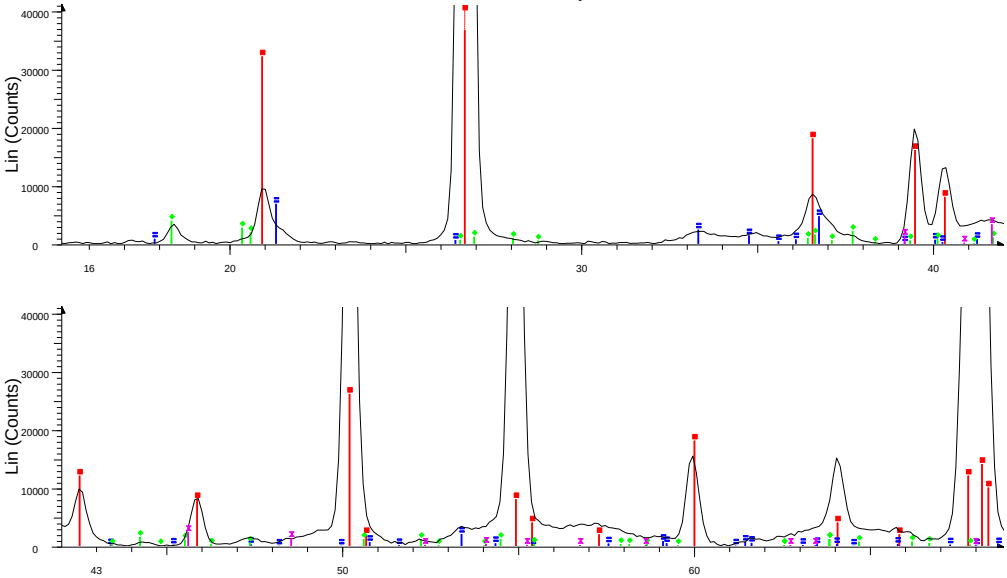
Note that one of the primary crystallographic phases is Iron Oxide Hydroxide (Goethite), which is dark brown or black in color. The small trace amount of cubic Iron Titanium Oxide is less certain and less significant.

Note that figure 3 shows the zoomed in position of the point source (approximately round) x-ray beam positioned on the deposit. Beam sizes ranging from 50 micros to 500 microns in diameter are typically used, and depend upon the size of the area under investigation. X-ray Diffraction Patterns follow.

Figure 3 Image of area collected inside the mounted geode



Geode Black Deposit



Quartz-Geode-solid-piece - File: Quartz-Geode-solid-piece-01.raw - Type: 2Th alone - Start: 12.879 ° - End: 77.462 ° - Step: 0.100 ° - Step time: 7259.5 s - Temp.: 25 °C (Room) - Time Started

00-046-1045 (*) - Quartz, syn - SiO2 - Hexagonal - a 4.91344 - b 4.91344 - c 5.40524 - alpha 90.000 - beta 90.000 - gamma 120.000 - Primitive - P3221 (154) - I/Ic PDF 3.4 -

01-075-5065 (A) - Goethite - FeO(OH) - Orthorhombic - a 4.59790 - b 9.95100 - c 3.01780 - alpha 90.000 - beta 90.000 - gamma 90.000 - Primitive - Pbnm (62) - I/Ic PDF 2.5 -

00-033-0018 (I) - Gibbsite, syn - Al(OH)3 - Monoclinic - a 8.65520 - b 5.07220 - c 9.71610 - alpha 90.000 - beta 94.607 - gamma 90.000 - Primitive - P21/n (14) - I/Ic PDF 1. -

00-005-0696 (N) - Iron Titanium Oxide - Fe2Ti4O - Cubic - a 11.29700 - b 11.29700 - c 11.29700 - alpha 90.000 - beta 90.000 - gamma 90.000 -