Application Note: 51560

Use of the DXR Raman Microscope to Generate a Micron-Level Map of an Amethyst Sample

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Key Words

- Dispersive Raman Spectroscopy
- Hyperspectral Mapping
- Library Searching
- Multivariate Curve Resolution (MCR)

Introduction

Dispersive Raman spectroscopy is a powerful analytical tool that is capable of identifying a broad range of compounds and is used in a number of industries. The technique requires minimal sample preparation while providing easy access to the far IR, making it a method of choice for inorganic analysis. Additionally, with a 1-µm excitation laser spot size, dispersive Raman microscopy is ideal when exceptionally good spatial resolution is required.

The DXR Raman microscope is a full-featured instrument configured with high-quality optics, a high-precision motorized stage and the OMNIC™ software suite, which includes the powerful Atlµs™ mapping and analysis tools. With its emphasis on the underlying technology that supports point-and-shoot capabilities, the DXR Raman microscope brings the full power of Raman microscopy into the hands of the non-expert user, without sacrificing performance.

Raman mapping can be used to produce detailed information on chemical distributions in a wide range sample formats. In the pharmaceutical industry, for example, tablet mapping yields valuable information about component distribution and particle size. Raman microscopy is also widely used for the detection and analysis of micron-sized defects and impurities in polymers, papers, glasses and other materials.

This application note demonstrates the power of this technology in the analysis of an interesting geological specimen. The same basic principles of analysis are readily applicable to many other samples.

Analysis

The sample is an amethyst from the Amethyst Mine Panorama of Thunder Bay, Ontario, Canada. Visual inspection (Figure 1) revealed a brown material on the non-fractured surface and purple coloration typical of amethyst on freshly-fractured surfaces. The sample was mounted on a microscope slide for analysis. Analysis of multiple samples using the DXR Raman microscope can be automated using multi-well plates and OMNIC Array Automation software.

Figure 1: Amethyst sample

Visual Microscopy

Brightfield illumination of the sample at 50X magnification (Figure 2) shows a variegated surface. Switching to the darkfield illumination option on the DXR Raman microscope reveals the surface structure in the sample (Figure 3). A mosaic of darkfield images (Figure 4) shows the distribution of the surface features. Some of these may have come from weathering, oxidation or deposition while underground, and may account for the brown surface coloration of the mineral.

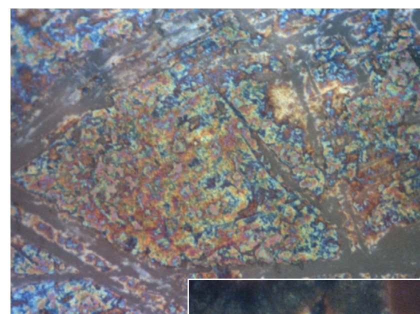

Figure 2: Brightfield image of amethyst sample at 50X magnification

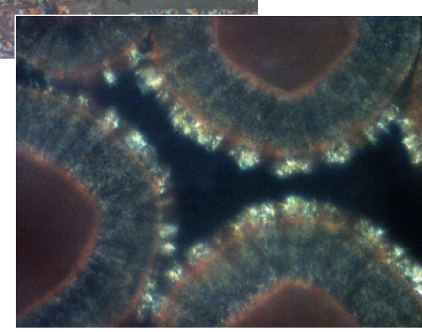

Figure 3: Amethyst sample under darkfield-illumination at 50X magnification

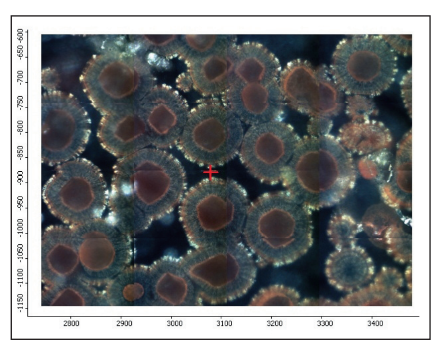

Figure 4: Image mosaic of the amethyst sample. Darkfield illumination, 100X magnification, Atlµs software

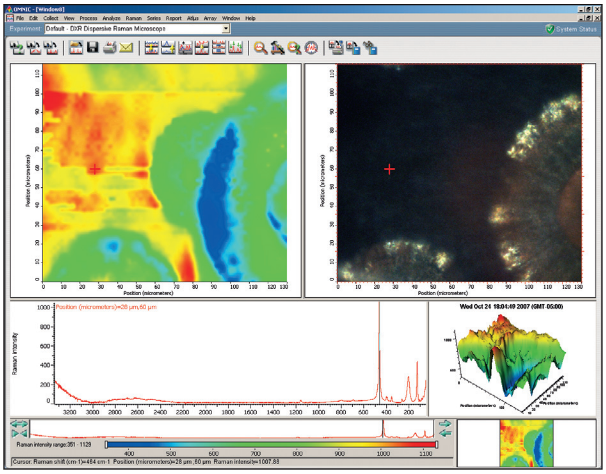

Figure 5: Result from mapping a section of the surface of the amethyst sample. Top right: visual image of mapped area; top left: chemical map based on the intensity of the spectral band at 466 cm^{-1}. (DXR Raman microscope, 780 nm excitation laser, full-range grating, 2-µm steps)

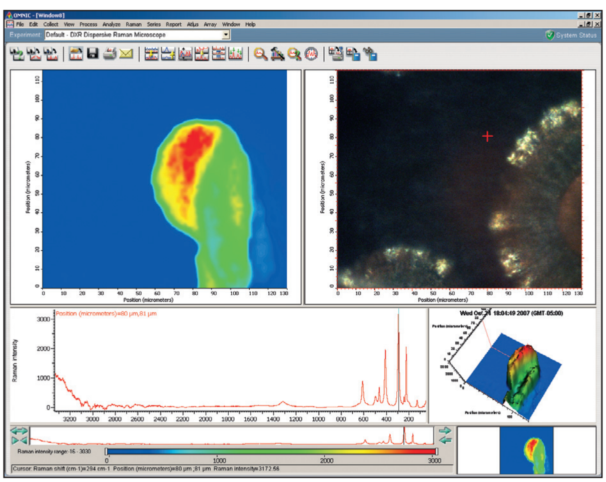

Figure 6: A second chemigram generated from the same Raman map as in Figure 5 showing distribution of a component with a major Raman band at 294 cm^{-1}

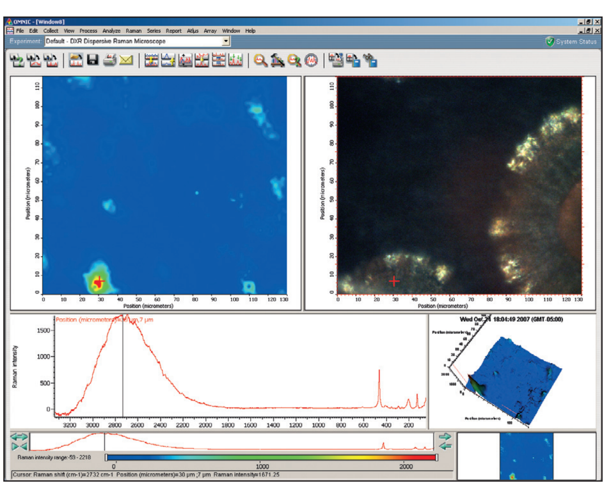

Figure 7: A third chemigram generated from the same Raman map as in Figure 5, showing distribution of a third, minor component characterized by a broad Raman feature at 2750 cm^{-1}

Raman Chemical Mapping

Raman hyperspectral mapping complements the visual image by providing information about the chemical composition (Figure 5). OMNIC Atlμs mapping software provides multiple tools for analyzing the data.

The Raman spectrum at the bottom of the screen reflects the composition of the material at the position of the cross-hairs in the image. If a peak is selected in that spectrum, the chemical map, or chemigram, at the top left of the screen shows the relative intensity of that peak across the sample. Moving the slider across the spectrum and moving the cross-hairs across the image, makes it easy to survey the composition of the mapped sample. The example in Figure 5 shows the chemigram generated by selecting a major peak at 466 cm^{-1}. Figure 6 shows the results of selecting a different region in the image. The Raman spectrum of this material is quite different from that in Figure 5. The chemigram generated from the spectral peak at 294 cm^{-1} is also different. Moreover, there is a distinct region in the chemigram in Figure 6 that is not obvious in the visual image. Distribution of a third chemical component is shown in Figure 7.

Component Identification – Library Searches

The chemical components in a map can be identified by comparing their spectra with spectra from known compounds. Spectra from Figures 5, 6 and 7 are searched against spectra in a library of geological samples. Figure 5 gave a 98.7% match with amethyst, while Figure 6 was a good match for hematite. The spectrum in Figure 7 matches quartz, although the broad feature at 2750 cm^{-1} is not fully explained.

A chemical map generated by calculating the degree of correlation with a library spectrum for amethyst is shown in Figure 8a. This confirms the presence of amethyst in the sample and also shows the presence of significant features that are not amethyst. A correlation map against a spectrum of hematite is shown in Figure 8b, confirming that this is also a major component of the sample.

A third component is revealed by generating a correlation map (Figure 8c) against the spectrum of the feature identified in Figure 7.

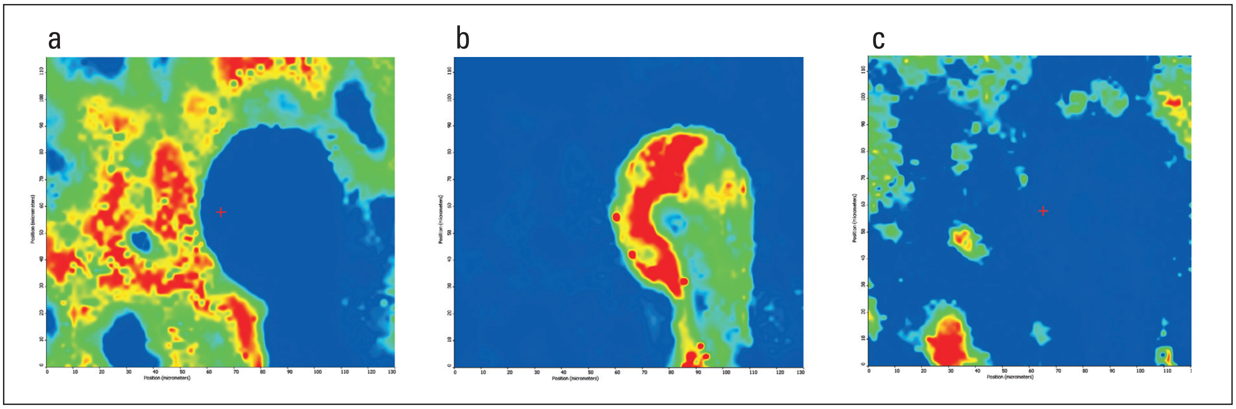

Figure 8: Maps generated by the calculated degree of correlation with library spectra of (a) amethyst; (b) hematite. The map in (c) was generated by calculating the correlation with the spectrum of the feature mapped in Figure 7.

Component Identification – Multivariate Curve Resolution

Atlμs also provides powerful functions such as multivariate curve resolution (MCR) that can extract components from chemical maps and facilitate component identification, even in cases where there is no prior knowledge about the chemical components in the sample. MCR analysis of the amethyst sample identified three components (Figure 9). Correlation maps (Figure 10) against the three components closely resemble the maps in Figure 8. A library search confirms a close match between components 1 and 2 with amethyst and hematite, respectively. A correlation map using component 3 is similar to that generated by the quartz-like feature in Figure 8c. A library search using component 3 resulted in an 88.9% match with quartz, however the quartz spectrum does not include the broad feature centered around 2750 cm^{-1}. Analysis of that region using OMNIC InterpretIR+™ suggests the presence of another silicate, or amorphous silicate.

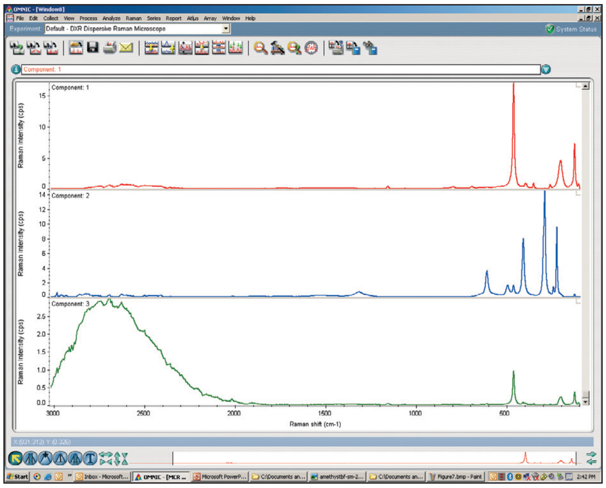

Figure 9: The three components of the amethyst sample generated using multivariate curve resolution of the hyperspectral mapping data collected using OMNIC Atlμs mapping software.

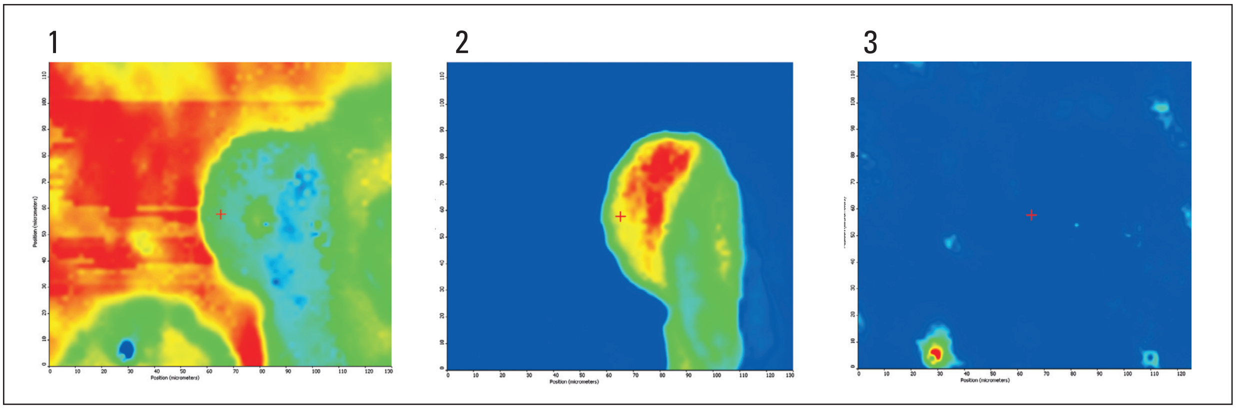

Figure 10: Correlation maps using the three components in Figure 9 generated using MCR analysis; component 1; component 2; component 3

Conclusions

The DXR Raman microscope, combined with OMNIC Atlμs software and library searching capabilities, takes full advantage of the power of Raman microscopy in this analysis of an interesting geological sample. High-quality optics provide an arresting visual image, which is powerfully complemented by the detailed chemical image supplied by Raman analysis. Library searching, correlation mapping and MCR analysis combine to identify and map the components in this sample.

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