Using the Correlate™ module to study a mineralogical section with Raman and SEM

Introduction

You can now correlate the highly specific chemical information shown in Raman images with complementary results from other imaging techniques, using Renishaw's Correlate module. The Correlate module enables you to analyse the same sample positions in multiple microscopy systems and overlay the resulting images.

The Correlate module, available in Renishaw's WiRE™ Raman spectroscopy software, includes the following integrated tools:

- Coordinate Manager to import and transform coordinates between different systems (e.g. a scanning electron microscope (SEM) and an inVia™ confocal Raman microscope)
- Batch Measurements to automate Raman data collection at multiple positions
- Image Alignment Tool to align and overlay images from multiple

Here, correlated SEM and Raman analyses were performed on a mineralogical section. This section contained a dissolution layer in a mineralised limestone. This layer was analysed to provide information on residual mineral species, to help understand ore nucleation processes.

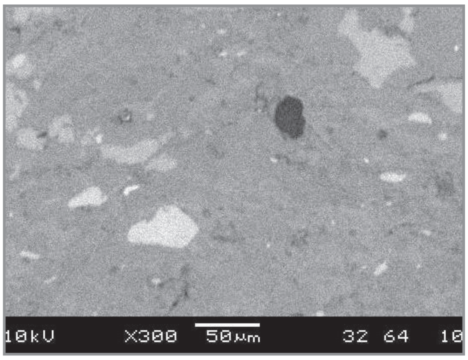

Figure 1. SEM image of geological sample

SEM images

The backscattered electron (BSE) SEM images show information on the atomic number of the elements in the sample; where the image is brighter, the element is heavier.

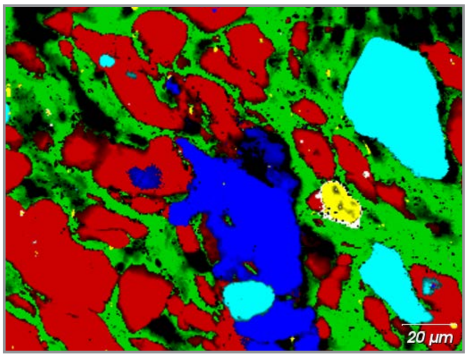

Figure 2. Raman image of geological sample

Raman images

Raman images show chemical and structural information on each mineral; where the image is coloured, a specific component or compound is present. The minerals identified in the Raman images were rutile, anatase, brookite, apatite, amorphous carbon, quartz and calcite. The Raman images clearly show the additional chemical specificity (e.g. titanium dioxide polymorphs), with many minerals not visible in the SEM image clearly identified in the Raman image.

Acquiring the SEM and Raman data

SEM images of four positions on the mineralogical sample were initially acquired using a BSE (JEOL JSM 6060 LV), revealing differences in elemental atomic numbers composition over the analysed areas. The sample was then moved to an inVia Qontor confocal Raman microscope for Raman analysis. We used Coordinate Manager to transform coordinates and to enable analysis at the same four positions. The Batch Measurement capability in the WiRE software was used to run the same measurement centred at each position, with LiveTrack™ maintaining focus throughout this process.

Raman images of the identified minerals were generated and overlaid with SEM images using the Image Alignment Tool. The rotation and scale of the images were pre-adjusted from the transformed coordinates to accurately overlay the Raman images on the SEM images. This experimental workflow is summarised below.

Analyse samples in SEM
- Acquire images at position of interest.
- Record central coordinates of images and reference markers.

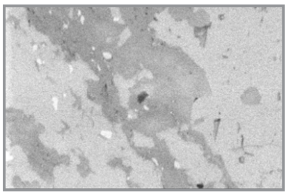

Figure 3a. SEM-BSE image of geological sample

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Move sample to inVia Raman microscope

Figure 3b. The inVia Qontor confocal Raman microscope

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Calibrate coordinates using WiRE's Coordinate Manager
- Find reference markers and record stage positions.
- Calibrate inVia coordinates of positions of interest.

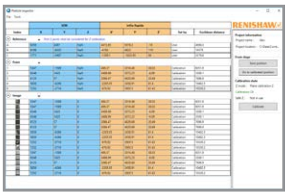

Figure 3c. A screencapture from the WiRE software

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Map multiple locations
- Maintain focus with LiveTrack.
- Use Batch Measurements to repeat the same measurement at each position.

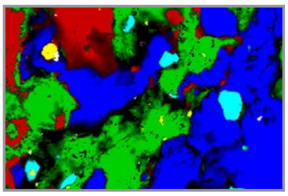

Figure 3d. A Raman image of the geological sample

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Overlay results using Image Alignment Tool

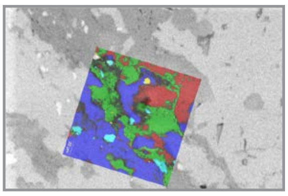

Figure 3e. A Raman image overlaid upon the SEM-BSE image of the same geological sample

Overlaying SEM and Raman images

Four positions were analysed using the SEM and inVia. Individual and overlaid SEM and Raman images are shown in Figure 4. Context of these images relative to the larger scale structure of the sample are shown in Figure 5.

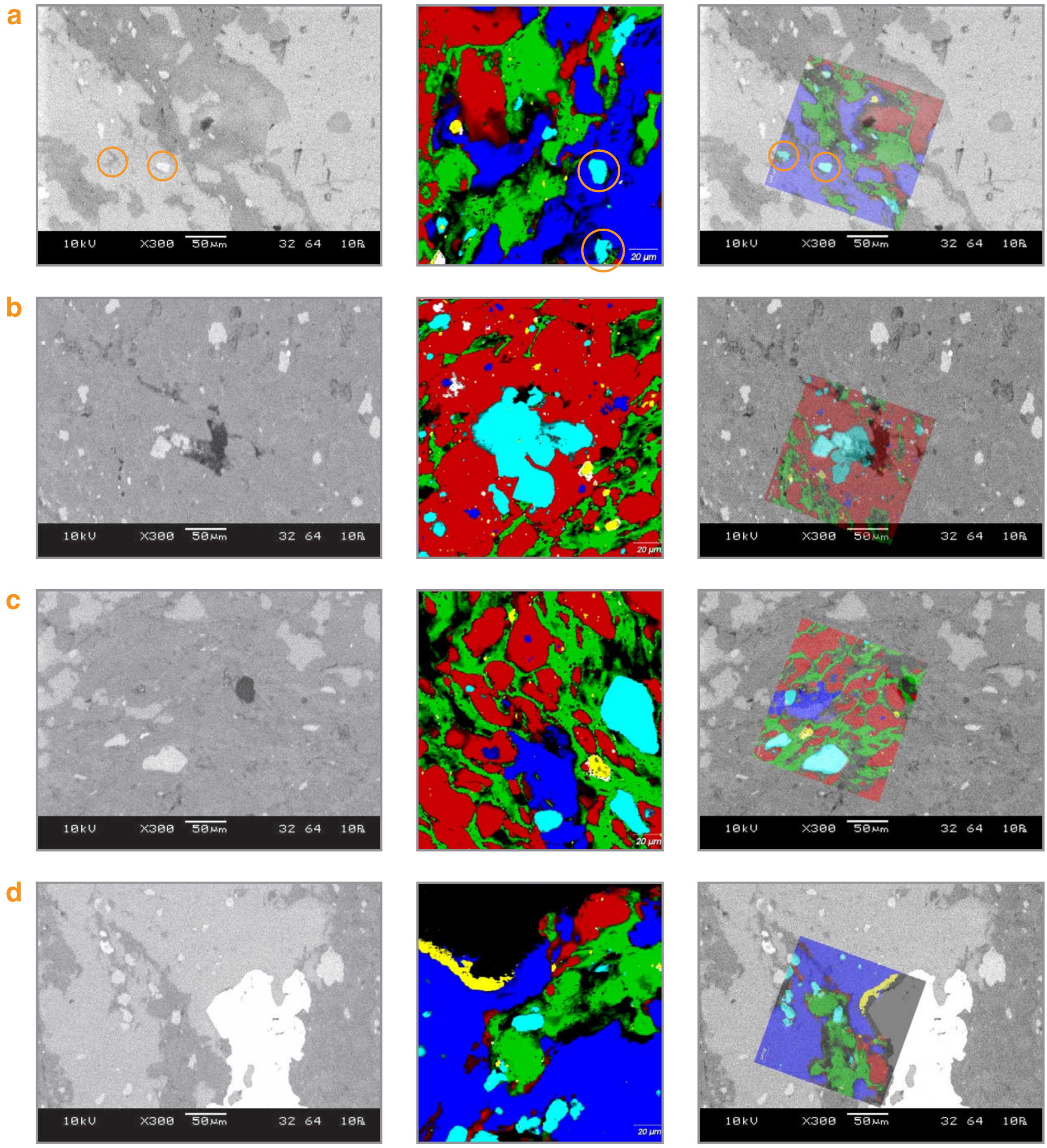

Figure 4. (L to R) SEM-BSE, Raman, and overlaid SEM and Raman images acquired at four positions (a to d) on the geological sample. The colours in the Raman images correspond to the identified components: rutile (yellow), anatase (white), apatite (light blue), carbon (green), quartz (red) and calcite (dark blue)

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Correlating images

The correlated information from the SEM, Raman and white light images reveals:

- Bright areas in the SEM image generally correlate with rutile, anatase and apatite, which contain heavier elements than the other identified minerals. The darker areas in the SEM images correlate with quartz and carbon, which are composed of lighter elements.
- The Raman image in Figure 4(a) shows clear domains of apatite, a calcium phosphate mineral. Subtle changes in the Raman spectra from these two domains are observed, in addition to the brightness change seen within the SEM image. Correlating the two techniques together has therefore revealed apatites of a different elemental composition; where the apatite appears darker in the SEM image, it may be composed of heavier elements (different end-member ratios).
- The SEM image in Figure 4(c) shows a significant area of featureless, grey material. In the Raman image, however, a detailed distribution of quartz and carbon is revealed.
- In many areas of the SEM image no contrast is seen. When viewing the overlaid Raman image, it is clear that many areas contain a wide mineral assemblage.

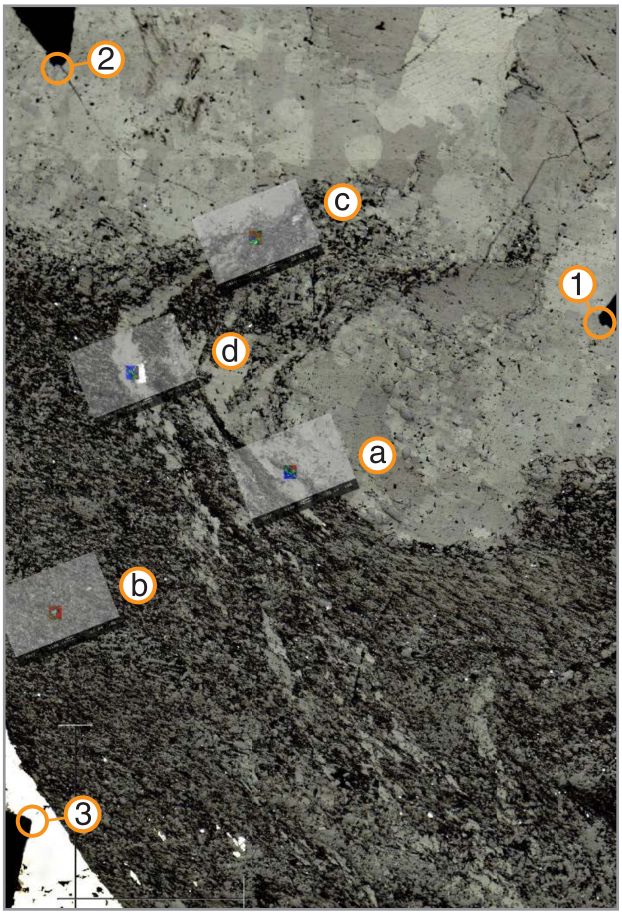

Figure 5. While optical microscope images are not required to use the features of the Correlate module, this inVia white light montage of the dissolution layer in the mineral section was generated and, by matching the microscope's coordinate systems with the Correlate module, overlaid with SEM and Raman images (seen in Figure 4) of each analysed position (a – d). Specific points on the reference makers (1 – 3) were used to transform coordinates between the SEM and the inVia system at a high precision

Conclusion

By correlating Raman, SEM and white light images, Renishaw's Correlate module has provided a detailed insight into the composition and structure of a dissolution layer in mineralised limestone. This can help to understand ore nucleation processes in mineral deposits where dissolution is present.

Acknowledgements

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Renishaw. The Raman innovators

Renishaw manufactures a wide range of high performance optical spectroscopy products, including confocal Raman microscopes with high speed chemical imaging technology, dedicated Raman analysers, interfaces for scanning electron and atomic force microscopes, solid state lasers for spectroscopy and state-of-the-art cooled CCD detectors.

Offering the highest levels of performance, sensitivity and reliability across a diverse range of fields and applications, the instruments are designed to meet your needs, so you can tackle even the most challenging analytical problems with confidence.

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