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FIB-SEM Instrument with Integrated Raman Spectroscopy for Correlative Microscopy

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Confocal Raman Microscope (CRM) is integrated with Scanning Electron Microscope (SEM) and its standard analyzers such as Energy Dispersive X-ray spectroscope (EDX), that can be further equipped with Focused Ion Beam (FIB). This yields valuable chemical information about molecular composition and chemical bonds in the sample on top of the high resolution SEM image, elemental composition map by EDX, nano-prototyping capability by FIB, etc.

In presented system, confocal setup of the Raman Microscope provides lateral resolution of 360 nm (with the 532 nm excitation laser). This is a high standard in the world of light microscopy, however, electron microscopy offers resolution more than 2 orders of magnitude better. Combining the CRM spectral image and high resolution SEM image acquired in-situ is therefore of great benefit. State-of-the-art Raman analyzers inside SEM use parabolic mirror for focusing the primary laser beam on the sample and collecting the Raman-scattered light. The lateral resolution of these systems typically does not exceed 2-5 μm. We achieve the resolution comparable with stand-alone instruments by integrating a full confocal light microscope with SEM.

The integrated system is capable of Raman imaging which is an important property. When just a single spectrum is acquired, one can never be sure, whether the position calibration is off. Besides lateral scanning, vertical movement is also supported, which allows non-destructive 3D tomography of laser transparent samples. Combination of CRM chemical analysis and SEM high resolution imaging makes this tool ideal for use in chemistry, medicine, biology, geology, forensic science and many other fields.

The integration is feasible with two types of electron columns: conventional (LYRA) and immersion (GAIA). The immersion column [1] is recommended for non-conductive or fragile samples, because it offers better resolution at low acceleration voltages (1 nm at 15 kV and 1.4 nm at 1 kV). Its three-lens design is equipped with a Schottky field-emission gun and it offers multiple display modes (for ultra-high resolution, large field of view or increased depth of focus) as well as a field-free mode for investigating magnetic samples.

Modification capability is ensured by FIB with three different columns to choose from. FIB also enables 3D tomography techniques by sequential FIB slicing followed by subsequent analysis to create 3D objects with analytical information such as material contrast, elemental composition, crystallographic information, etc. This is complementary to the above mentioned 3D non-destructive CRM tomography.

Raman – SEM integration is an addition to the previously reported FIB-SEM integration with Scanning Probe Microscope (SPM) and Time-Of-Flight Secondary Ion Mass Spectrometer (TOF-SIMS) [2]. Wide variety of techniques integrated in a single tool is advantageous especially for correlating multiple measurements and analysis of the same sample area in-situ. This meets the ever-growing needs of

today's nanotechnology for nano-scale analysis, imaging and modification in a single universal instrument [3].

References:

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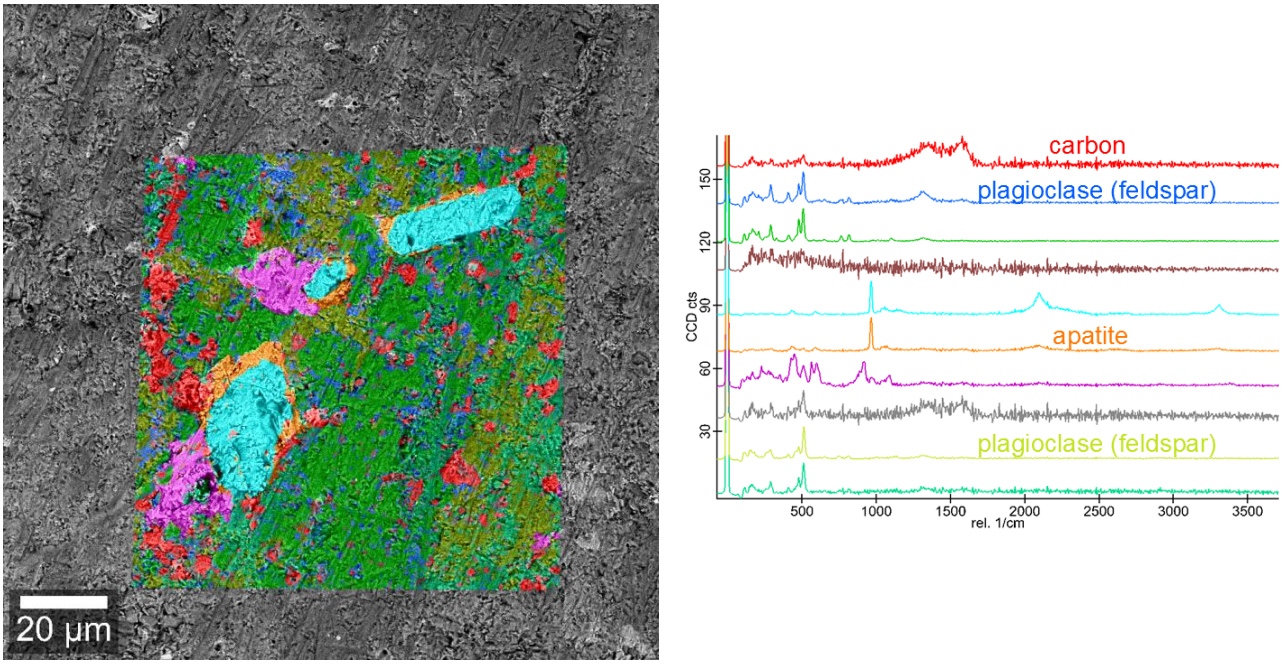

Figure 1.: Left: Overlayed SEM (in grey) and Raman (in color) micrographs of diorite geological sample. Right: Raman spectra corresponding to the various phases on the left picture including carbon, plagioclase (feldspar) and apatite.

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