European Mineralogical Conference
Vol. 1, EMC2012-599-5, 2012
European Mineralogical Conference 2012

Synergism of coupling μ-XRD^2 with μ-Raman: Next generation structural characterization

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The use of X-ray optics, microfocus X-ray sources, and large 2-dimensional detectors (XRD^2) has significant improved spatial resolution, measurement times, and information density of today's laboratory X-ray diffractometers. Measurement times of a few seconds and spot sizes below 100 μm are readily becoming considered *state-of-the-art.*$^{(1)}$ However, as conventional bulk X-ray analysis becomes highly spatially and temporal resolved, a paradigm change in the way we utilize the method and the way we interpret data is necessary.

Unlike bulk X-ray powder diffraction, we cannot always assume that in μ-XRD samples a large number of randomly oriented crystals exists – with the grain size in the micrometer range and the sampling volume in the tens of micrometer we are faced with semi-single-crystal diffraction for many samples. Consequently, quantitative and textural analysis of the XRD pattern becomes very difficult and the measured and calculated *(hkl)*-dependent reflections may differ greatly. Even sample rotation only partially mitigates this issue and, at the same time, sacrifices spatial resolution significantly. On the other hand, we still are limited by Bragg lattice reflections and albeit the great many advantages of μ-XRD we have to remember that amorphous or liquid phases remain very little suited for this method.

Considering the challenging task to characterize heterogeneous materials, such as complex mineral assemblies in geoscience or advanced nanocomposites in materials science, we see not only a need to utilize μ-XRD the best possible way, but to synergistically add an additional characterization tool to overcome fundamental limitations to X-ray diffraction. Given the small size of a fiber optical head and the non-destructive nature, we chose μ-Raman spectroscopy for complementary μ-XRD/μ-Raman studies and integrated the Raman system in a commercial laboratory microdiffractometer. The Raman system now enables, for the same sample at the same time and at the same location as the μ-XRD measurement, a spatial resolution down to the μm-range and enables microscopic investigation of the sample using exchangeable microscopic optics. Also, from the other point of view, adding μ-XRD to Raman has several distinct advantages: it expands the range of materials that can be studied beyond only Raman active substances (*e.g.*, including now also metals), it adds additional accuracy and information density since Raman modes may depends strongly on sample orientation and polarization effect, and the extensive PDF databases overcome the very limited number of entries in current Raman databases. As also the sampling volume of μ-XRD and μ-Raman differ in size and shape, we will discuss additionally the problem of the different excitation volume of both methods and discuss this aspect in the light of heterogeneous samples.

We will demonstrate high resolution phase mappings of heterogeneous material systems as performed in a setup which integrates the fiber-optical measurement head of a inVia Raman microscope from RENISHAW in a BRUKER D8-Microdiffractometer. Further advantages like the identification of organic substances, amorphous phases, structurally bound water, and large single crystals are also shown, representing a significant step forward in the analyses of complex samples.

Literature

(1) C. Berthold, A. Bjeoumikhov, L. Brügemann: Fast XRD^2 microdiffraction with focusing X-ray microlenses, Part. Part. Syst. Charact. 26 (3): 107-111 (2009)