Simultaneous μRaman spectroscopy and X-ray μDiffraction at ID13

Richard J. Davies, Manfred Burghammer, Christian Riekel.

European Synchrotron Radiation Facility, Grenoble, France.

Raman spectroscopy and X-ray scattering are complementary techniques which greatly benefit from combinatorial use. Whilst one provides information relating to molecular bond energies and orientations, the other provides information on crystallographic structure and morphology. Thus, not only do they provide differing information, but this relates to quite different length scales within a materials' hierarchical structure.

Although many applications benefit from combining X-ray and Raman techniques, the majority do not require their in situ combination. Most materials appear homogeneous when studied using macroscopic beams, eliminating the need for accurate sample placement. For dynamic studies, duplicating experimental conditions during sequential experiments is usually feasible when the results are inherently averaged. By contrast, the use of microfocus techniques necessitates an in-situ approach to data collection. Over micron length scales most materials appear highly heterogeneous which makes precise sample alignment critical. Meanwhile, under dynamic experimental conditions, microprobes can be highly sensitive to local variations. In such cases, only an in situ data collection strategy can ensure meaningful results. Not only does this need to provide the capability for simultaneous measurement, but it also requires a common sampling position.

The ESRF-ID13 beamline has developed a globally unique setup of combined μRaman/μDiffraction including μSAXS. Its in situ μRaman setup offers on-axis laser beam delivery with X-ray and laser beams sharing a common focal position on the sample and similar spot sizes. The system has been recently upgraded to allow routine offline use with the addition of a stand-alone microscope. This provides the added possibility of characterizing samples either before or after X-ray experiments in order to locate regions of interest or monitor radiation damage.