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Redox and speciation mapping of rock thin sections using high spatial resolution full-field imaging technique

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Because of their complex genesis, natural rocks are the most often heterogeneous systems, with various scale-level heterogeneities for both chemistry and structure. In the last decade, the dramatic improvements of hyperspectral imaging techniques provided new tools for accurate material characterisation. Most of these micro- and nano- analytical techniques rely on scanning instruments, which offer high spatial resolution but suffer from long acquisition times imposing practical limits on the field of view. Conversely, full-field imaging techniques rely on a fast parallel acquisition but have limited resolution.

Although soft X-ray full-field microscopes based on Fresnel zone plates are commonly used for high resolution imaging, its combination with spectroscopy is challenging and 2D chemical mapping still difficult. For harder X-rays, lensless X-ray microscope based on simple propagation geometry is easier and can be readily used for 2D spectro-microscopy.

A full-field experimental setup was optimized at the ESRF-ID21 beamline to image iron redox and speciation distributions in rocks thin sections. The setup comprises a Si111 or Si220 (ΔE = 0.4 eV) monochromator, a special sample stage and a sensitive camera associated with a brand new GGG:Eu light conversion scintillator and high magnification visible light optics. The pixel size ranges from 1.6 to 0.16 μm according to the optic used.

This instrument was used to analyse phyllosilicates and oxides of metamorphic sediments coming from the Aspromonte nappes-pile in Calabria. Iron chemical state distributions were derived - from images of 1000 $\times$ 2000 $\times$ 30 μm^3 rock thin sections - by subtraction of absorption images above and below the Fe K-edge. Using an automatic stitching reconstruction, a wide field image (4$\times$3 mm^2 with a 1 μm^2 resolution for a total of about 12 millions pixels) of Fe_{total} elemental distribution was produced. Moreover, μ-XANES analyses (more than 1 million individual μ-XANES spectra) were performed from 7100 to 7280 eV with an energy resolution of 0.3 eV. This spectral resolution allows fine pre-edge features to be clearly observed. A redox mapping was then derived from these full-field μ-XANES acquisitions. It highlights different mineral generations that crystallized at different redox conditions. Redox mapping has also been coupled with electron μ-probe analyses in order to calculate accurately the P-T path of the studied rock.