A novel three-dimensional elemental imaging method based on full-field X-ray fluorescence detection technology

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A novel three-dimensional (3D) elemental micro-imaging method was tested at beamline P06 of PETRA III, based on full-field micro-XRF detection technology. As opposed to using a single-element energy-dispersive detector and microbeam excitation in a confocal arrangement [1,2], for these 3D-XRF experiments a unique energy-dispersive CCD-detector (SLCam [3]) was installed and characterized at P06 in combination with excitation by a linearly focused X-ray beam. During the 3D micro-XRF experiments the Kirkpatrick-Baez (KB) X-ray mirror system was used with the vertically focusing mirror moved out of the beam path, allowing the generation of a linearly focused beam, oriented in the vertical plane. The horizontal beam size was determined by a knife-edge scan using a 50 µm gold wire while the vertical beam size was determined using a PCO 4000 high resolution CCD camera which was positioned along the primary beam, downstream of the sample position. The obtained sheet-beam measured 5 µm horizontally by 1.37 mm vertically (FWHM) , which was used to illuminate and excite a vertical cross-section of the sample volume of interest at an energy of 18.2 keV.

The technique was tested on diamonds containing inclusion clouds and larger (few 100 µm) inclusions. By scanning through the diamond in the z-direction (towards the detector) (see Fig.1) and recording 2D images at each position, a 3D micro-XRF data-set of the entire volume of interest of the diamond is obtained, including the diamond inclusion(s). Diamond P11 (Machado River, Brazil). was analyzed using 41 vertical slices with 5µm steps and counting time of 30 min per slice, leading to a full 3D elemental imaging measurement time of 21h per scanned volume. The relatively long counting time per slice was necessary in order to achieve adequate elemental XRF intensities. The installation of the new multilayer monochromator at P06 will reduce the acquisition time for a full 3D data-set by a factor of ~50, making the collection of 3D elemental distributions within 0.5 - 1h possible for this type of geological samples.

One representative 2D full-field micro-XRF slice is shown in Figure 2A for the Fe-Kα distribution. The intense Fe hotspots are located in the surrounding of a large inclusion with sizes of a few microns. Figure 2B shows the reconstructed 3D image obtained from the single 2D XRF slices of the largest inclusion of diamond P11, while Fig.2B illustrates the measured XRF sum-spectra corresponding to various Fe/Ni/Cr rich clusters within the 3D data-set.

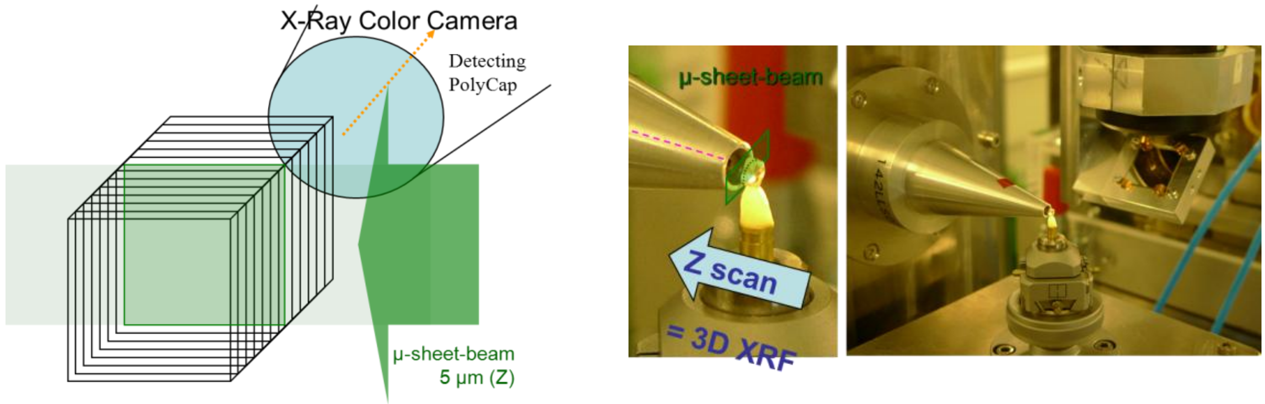

Figure 1: Schematic overview of the working principle of 3D-XRF using a sheet µ-beam (left). Full-field XRF detector (SLCam [1]) polycapillary optics, oriented towards the excited vertical cross-section within the sample. A sequence of 2D XRF images are collected by a single linear scan of the sample, allowing the reconstruction of the full 3D elemental distributions (right).

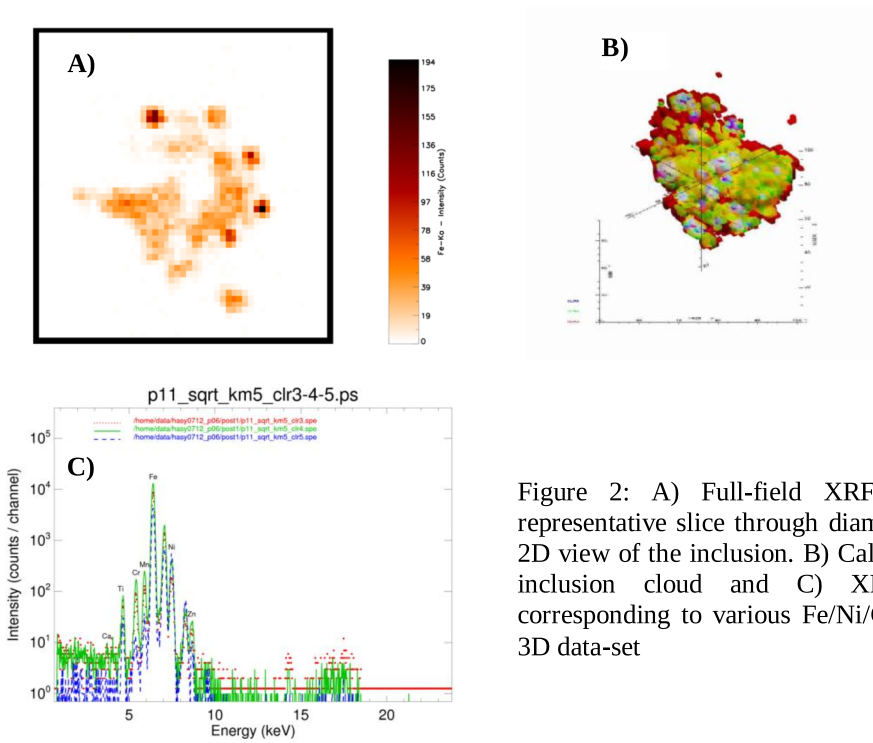

Figure 2: A) Full-field XRF image (Fe-Kα) of one representative slice through diamond P11, showing detailed 2D view of the inclusion. B) Calculated 3D view of the P11 inclusion cloud and C) XRF spectral distributions corresponding to various Fe/Ni/Cr rich clusters within the 3D data-set

References

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