Station B16 at the Diamond Light Source is a versatile test facility for trying out new experiments with synchrotron X rays of energies in the range 4 to 20 keV: either as white beam or monochromatic, and either unfocussed or focussed. We have used collimators, of sub-micrometre cross-section, for white radiation [1]; and, more recently, monochromatic radiation (11.6 keV) together with a linear series of 14 compound refractive lenses, giving an elliptical focal spot size 6.0 μm by 7.2 μm, to image inclusions in natural and synthetic diamonds. Specimens were scanned on an X-Y stage and images were obtained both in transmission and by X-ray fluorescence energies characteristic of chosen metallic elements. The X-ray fluorescence spectra at each pixel were also recorded.
The diamonds were selected for the variety of their inclusions to test the imaging capability of scanning X-ray microscopy: four diamonds from the Finsch Mine (South Africa), one from Udachnaya (Siberia) and one from Orapa (Botswana), as well as some synthetic diamond grit particles. The Finsch diamonds were polished plates, 2–3 mm in thickness and 5 mm in diameter. One was a 'coated' stone with a clear core and a cloudy overgrowth containing numerous small black sulphide or graphite inclusions; one had a cloudy cube-shaped core; one was a twin (a 'macle') of peridotitic paragenesis containing purple chrome-rich garnet inclusions; and another was of eclogitic paragenesis which contained garnet inclusions as well as a pale olive-green cloudy region of tiny unidentified inclusions. We found that the cuboid growth sectors of this eclogitic diamond contained nickel.
The two other diamonds, a rounded dodecahedron of about 2 mm in diameter from the Udachnaya mine containing an inclusion of chromite, and an octahedron of about 2 mm in size from the Orapa diamond mine, containing a sulphide inclusion, were also selected for imaging experiments on the inclusions. Many inclusions were indeed imaged and the X-ray fluorescence spectra were recorded. These experiments demonstrated the power of scanning X-ray microscopy to identify non-destructively the various chemical elements contained in inclusions lying well within thick diamond specimens.
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Hard X-ray diffraction scanning tomography with sub-micrometer spatial resolution.

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To perform 3D crystallographic phases imaging inside polycrystalline single phase but also multi phases samples, a new method combining X-ray powder diffraction with scanning tomography has recently been proposed [1-3]. One major advantage of this technique (written here Xray Diffraction-Computed Tomography (XRD-CT)) is that contrary to others methods no *a priori* knowledge on the phases present in the sample (crystallographic structure ...) is required. The spatial resolution of this technique is directly linked to the beam size incoming on the sample and micron-scale resolution has already been demonstrated [1]. The capability of the method to reach higher spatial resolution and therefore to access nanomaterials characterization is linked to the focusing capabilities of the beamline. Since the XRD-CT technique is based on powder diffraction methods, Rietveld refinement can be partly included in the data processing just like for 2D-XRD. A previous study based on a quite similar approach for XRD-CT data treatment has been recently proposed [2]. We report in this paper the first attempt to perform XRD-CT measurements on the ID22NI hard X-ray nanoprobe of the European Synchrotron Radiation Facility (ESRF) with a beam size of 150×220 nm [4]. The studied sample is a small spherical (about 50 μm in diameter) annealed UMo particle and a multiphases interface buried about 5 μm under the surface has been especially characterized. Moreover the interest of Rietveld method for analyzing the XRD-CT data will be evaluated and the possibility to derive from such an approach the weight fraction of the various phases inside each voxel will be discussed. Finally, the advances provided by this experiment on our understanding of the UMo/Al metallurgy will be presented.

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