

# Three-dimensional confocal $\mu$ -XANES on mineral inclusions in natural diamonds

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## Introduction

The majority of natural diamonds are formed in the lithospheric upper mantle (< 200km depth). During the growth of the diamond, fluids, mineral and rock fragments can be trapped in the diamond host. These inclusions are shielded from the environment during the transport of the diamond towards the Earth surface, preserving their original composition as captured. Inclusions in natural diamonds can therefore provide direct information on the physical and chemical conditions in the deep Earth down at least to the upper part of the lower mantle [1-2]. Only a few sources, e.g. Juina (Brazil) and Kankan (Guinea) provide diamonds from superdeep sub-lithospheric mantle regions of the deep Earth which originate at depths of several hundreds of km. Determination of the trace-level rare-earth elements composition of the mineral phases in the diamonds can allow the determination of the inclusions' origin (sedimentary, hydrothermal, metasomatic or carbonatitic). Performing micro-XANES of the lower atomic number minor/major elements of the inclusions can provide us identification of the main mineral phases present.

Due to the high scattering power of the diamond host, fluorescence XANES in a confocal detection scheme is required in order to improve signal-to-scatter background ratios. Moreover, the confocal detection mode allows performing a truly 3D spatially resolved XANES analysis. In the example below, this is illustrated on a Juina (Brazil) diamond (RS69) containing a cloud of microscopic (1-10  $\mu\text{m}$ ), possibly liquid inclusions. Within this diamond, Fe-K confocal micro-XANES spectra were recorded on 18 different inclusions. The confocal micro-XANES spectra show a depth dependence in the composition of the Fe mineral phase in the diamond.

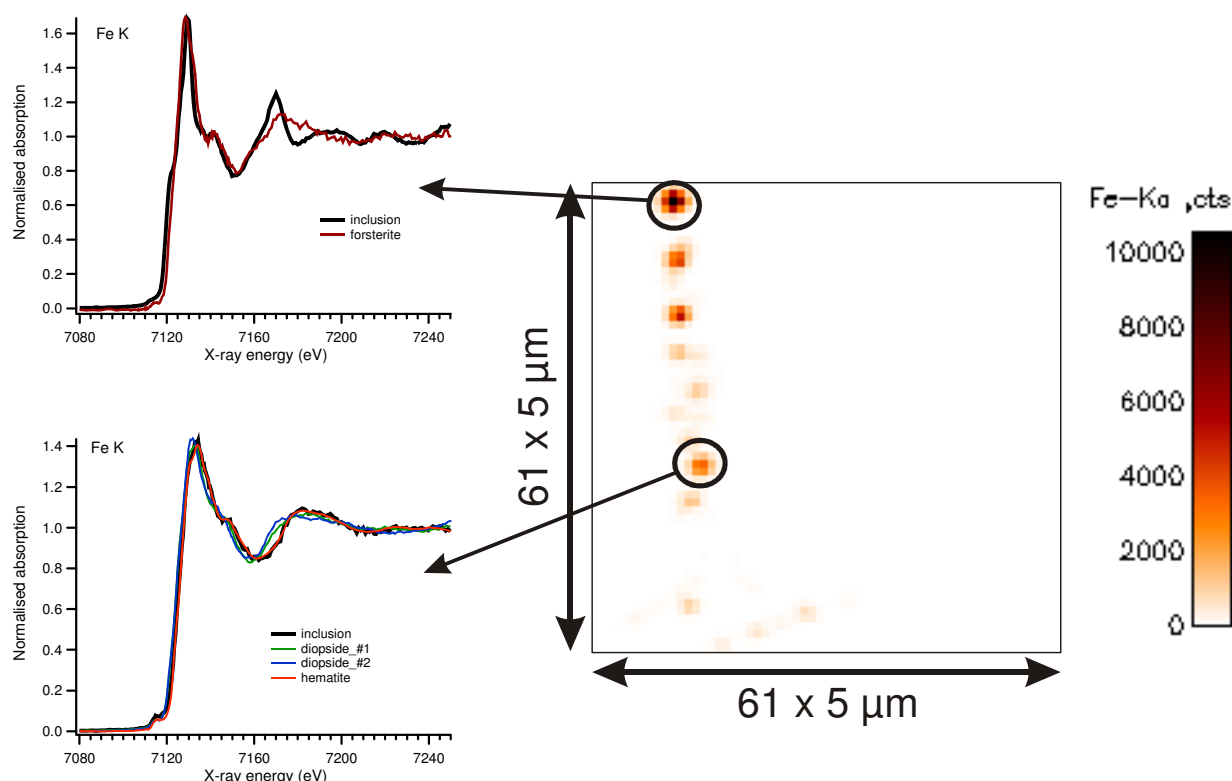
## Experimental

The Fe-K confocal  $\mu$ -XANES spectra were recorded at beamline L of the DORIS III storage ring in fluorescence mode using a Si(111) monochromator. The fluorescence radiation was recorded using a vortex-EX SDD detector (SII, 50 mm<sup>2</sup>, 350  $\mu\text{m}$  crystal thickness, measured energy resolution 183 eV at Fe-K $\alpha$ ). The monochromatic beam was focused by a polycapillary lens (XOS inc., USA) resulting in a beam size of about 21  $\mu\text{m}$  (FWHM) at the Fe-K edge. The confocal detection was achieved by positioning a second polycapillary half-lens (XOS inc.) in front of the energy dispersive detector. The measured confocal depth acceptance was about 26  $\mu\text{m}$  (FWHM) for Fe K $\alpha$  radiation. The vertical FWHM of the confocal ellipsoidal detection volume was 17  $\mu\text{m}$ .

## Results

The diamond surface was polished close to the internal inclusion region. A vertical confocal plane was recorded perpendicular to the polished surface through the cloud of inclusions. The resulting Fe-K $\alpha$  intensity image is given in Figure 1, showing a line of separated inclusions at a depth of about 50  $\mu\text{m}$  underneath the polished surface on the right. Confocal Fe-K micro-XANES spectra were recorded on a selection of these inclusions: representative spectra for the upper part and the lower part of the map are given in Figure 1, left. A compositional change along the inclusion depth can be observed. The spectrum for the lower part is identified as originating mainly from hematite

(Fe<sub>2</sub>O<sub>3</sub>). The spectrum for the upper part is not yet unambiguously identified, but from comparison with Fe XANES reference spectra given by Wilke et al. [3], the measured spectrum on the one hand closely resembles to that of a periclase mineral (the cubic form of MgO). On the other hand, from the set of Fe bearing minerals (excluding periclase) which was measured as references, the best agreement was found for forsterite (Mg<sub>2</sub>SiO<sub>4</sub>), again a Mg-O compound.



**Figure 1:** Fe-K confocal  $\mu$ -XANES spectra for the two inclusions identified on the confocal  $\mu$ -XRF map shown on the right (excitation energy 7200 eV, 5 s LT, 61 (5  $\mu$ m)  $\times$  61 (5  $\mu$ m)).

## Conclusions

The confocal  $\mu$ -XANES detection mode allows a truly 3D spatially resolved analysis of mineral inclusions within natural diamonds. As an example, Fe-K confocal  $\mu$ -XANES spectra were recorded from a diamond containing a cloud of mineral inclusions. A compositional change of the inclusions stream within the diamond from a magnesium oxide (or magnesium silicate) to iron oxide was observed.

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