EUROPEAN SYNCHROTRON RADIATION FACILITY
INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

Experiment Report Form

	Experiment title: XAS investigation of rare, Fe-rich Deep Earth diamond inclusions	**Experiment number**: 26-01-1140
Beamline: BM26A	**Date of experiment**: from: 18/04/2018 to: 22/04/2018	**Date of report**: 21/06/2018
Shifts: 12	**Local contact(s)**: Dipanjan Banerjee	*Received at ESRF:*

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Report:

High pressure minerals trapped in super deep (sub-lithospheric) natural diamonds are unique messengers of deep Earth. Synchrotron radiation based confocal micro-XAS and micro-XRF enable to obtain quantitative insitu information on elemental composition, chemical and structural state within the still sealed microscopic inclusions [e.g. 1-3]. The elemental and associated chemical state patterns for selected elements (e.g. Fe) are crucial indicators to decipher the origin and processes associated with the formation of these exciting deep Earth samples. Furthermore it will help to reconstruct the redox-conditions of the deep Earth mantle including the important mantle transition zone, which may act as a barrier or even sink of diverse lithologies [2].
These investigations represent the extension of our earlier elemental and chemical state imaging studies on Fe bearing inclusions using 3D confocal micro-XAS/XRF which contributed to the discovery of a hydrous ringwoodite inclusion in a Juina diamond: a mineral never before found on Earth which may point to a vast reservoir of water in the mantle [1,4]. The proposed project will focus on a unique suite of (Fe,Mg)-Oxide inclusions.

The new scanning micro-XAS setup developed in the framework of the DUBBLE LTP in collaboration with Dr. W. Bras and UGent XMI will be used to perform the confocal 3D micro-XANES experiments. During

these experiments a polycapillary optics (XOS Inc., Albany, USA) based confocal setup will be applied, as was tested and optimized in a series of pilot experiments in collaboration with DUBBLE and Ghent University Xray Microspectroscopy and Imaging (XMI) group to extend the bulk X-ray Absorption Spectroscopy (XAS) capabilities of the DUBBLE BM26A beamline towards chemically selective X-ray micro-imaging methods by 3D micro-XAS/XRF [5]. The implemented microfocus setup allows for the selective data acquisition from a 10×10×12 μm³ volume in a surrounding sample matrix to obtain 3D resolved elemental and chemical speciation information, without the need for intensive post-processing data recombination algorithms as is customary in conventional tomography.

During the experiment, several Fe-rich diamond inclusions were measured. Inclusions of interest were located by cross-sectional confocal XRF slicing methods, after which XANES profiles were measured on points of interest (Figure1).

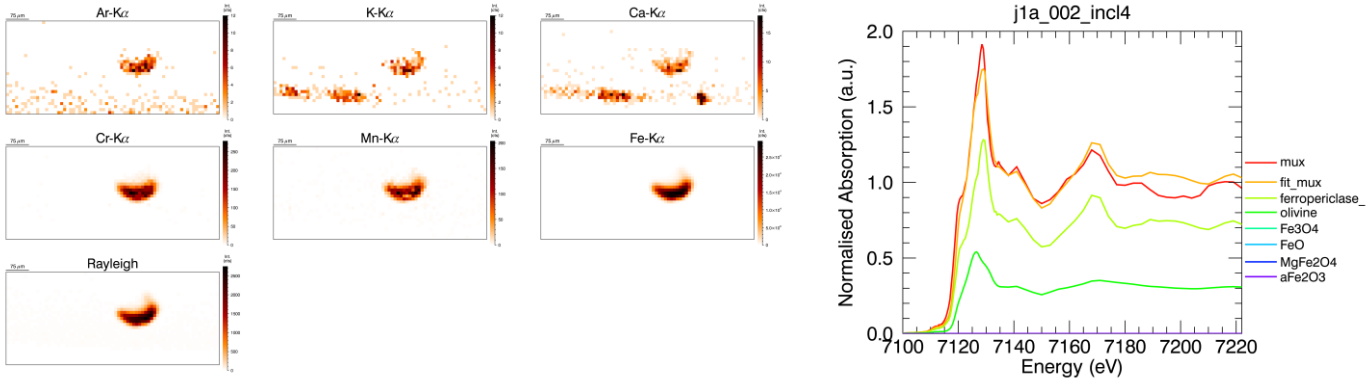

Figure 1: Confocal μXRF cross-sectional slice of a diamond inclusion, close to the diamond surface (left) along with a XANES profile denoting ferropericlase composition measured in the Fe rich region of the inclusion (right)

Additionally, 3D confocal XRF images were measured for a select few inclusions in an attempt to identify mixed phase inclusions (Figure 2). Due to the time required for such 3D scans and the limited available beamtime, this approach could not be followed for each inclusion.

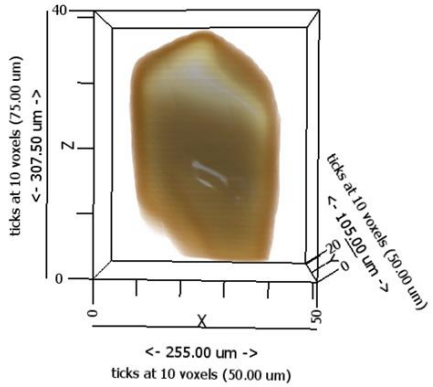

Figure 2: Confocal μXRF 3D Fe-K_a image of a diamond inclusion (j1a_002)

The data acquired during this beamtime will be cross-referenced with earlier obtained data and will provide important information on the content of these diamond inclusions.

[1] Pearson, D.G., et al. NATURE, 507, 221–224 (2014).
[2] Brenker FE, et al., EARTH AND PLANETARY SCIENCE LETTERS 236 (3-4): 579-587 (2005).
[3] Brenker, F.E., et al., SCIENCE LETTERS, Volume: 260, Issue: 1-2, Pages: 1-9 (2007).
[4] Silversmit G; et al., ANALYTICAL CHEMISTRY, Vol. 83, Issue: 16, 6294-6299 (2011).
[5] Silversmit G, et al., PHYSICAL CHEMISTRY CHEMICAL PHYSICS Volume: 12, issue 21, 5653-5659 (2010).