First in-situ micro X-ray fluorescence measurements at the new Extreme Conditions Beamline P02.2 at PETRA III

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In the last decade, X-ray micro-fluorescence analysis (XRF) has become a versatile tool in high pressure research as it allows studying trace element contents of coexisting phases in diamond anvil cells (DAC). Recently, combined micro-XRF/XRD analysis at extreme conditions has been shown to be a very powerful extension to micro-XRF as elemental reorganisation can then be directly linked to structural changes. A significant technical improvement of in-situ XRF was achieved by the use of collimating capillaries with an adequate focal distance for DACs [1]. By these optics, the solid angle of fluorescence detection is restricted to values between 150 and 300 μm FWHM and thus reduces the scatter signal from the diamond anvils in the XRF spectrum (Fig 1). This enables optimum XRF signal detection even in thick diamonds without recess geometry and extends the pressure range of micro-XRF in DACs to up to 135 GPa [2].

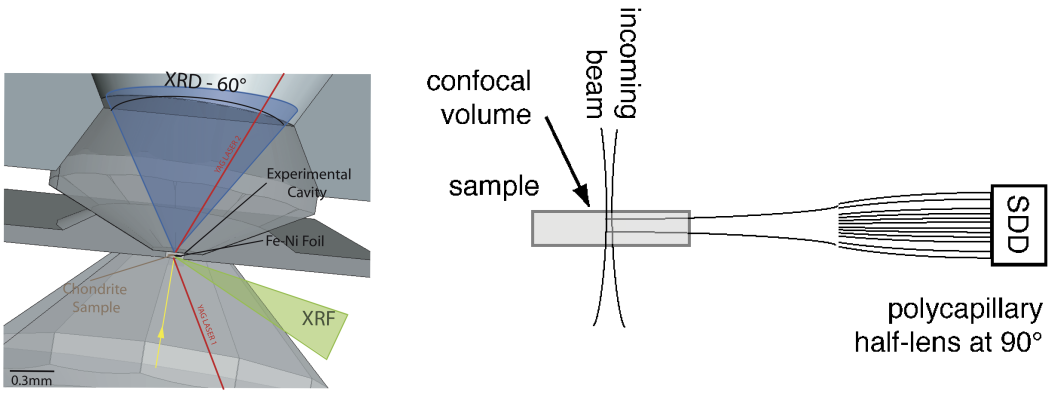

Figure 1: Schematic view of a diamond anvil cell with the X-ray beam paths (left) and of the confocal XRF set-up (right; not to scale).

In this study, we realised a fluorescence set-up at the new Extreme Conditions Beamline P02.2 at the storage ring PETRA III in parallel to the existing X-ray diffraction (XRD) set-up and used it for first experiments. For the measurements, the excitation energy was set to 42 keV which allows to detect elements with Z up to 58 (Ce) via K-shell fluorescence. The beam was focused to a spot size smaller 2 μm. XRF detection was achieved using a collimating polycapillary half-lens mounted on a Vortex silicon drift detector. The capillary was characterised at beamline L at DORIS III (Fig. 2). At both beamlines, the fluorescence signal was recorded at 90° to the incoming beam using a Vortex SDD detector. At P02.2, the fluorescence spectra were processed with a Struck SIS3302 ADC. This set-up was first tested for a reference sample and then applied for two different ”types” of DAC: one externally heated hydrothermal DAC with recess geometry (in-house design, see also [3]) and a laser-heated DAC with diamonds with different culet sizes [2]. Reference samples showed that parallel detection of elements with Z between 26 and 58 is feasible (Fig. 3a). However, the lower cut-off energy of the spectrum had to be set as high as 6 keV to optimise for the fluorescence detection at high energies. This shows that a Vortex SDD - despite the fact that the Si chip is too thin - is not well-suited for fluorescence detection of high energy photons. To optimise the fluorescence detection, detectors with thicker and higher Z element crystals will be tested for the

application. However, XRF from a fluid loaded into a hydrothermal DAC, showed that in-situ trace element detection of Ba and La is already possible with the chosen set-up (Fig. 3b).

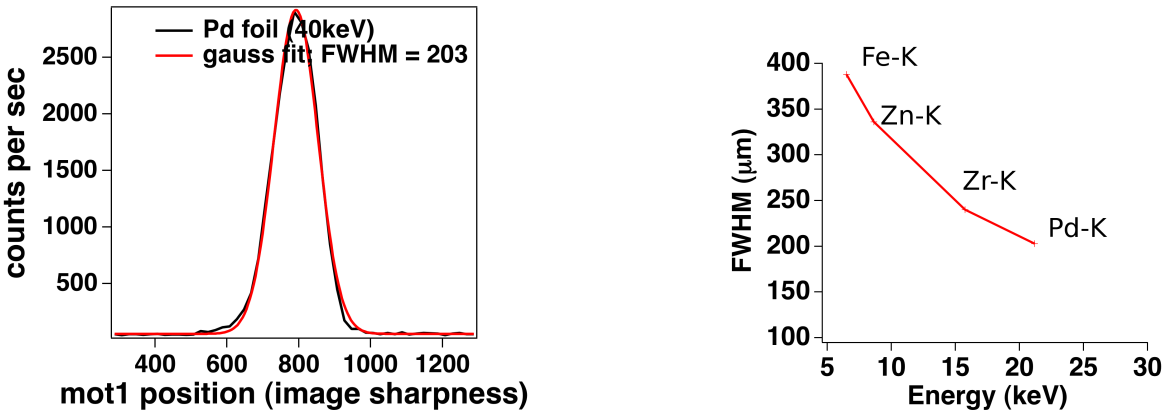

Figure 2: Characterisation of the confocal set-up with the detector capillary at beamline L: the results are based on measurements of foils at excitation energies of 20 keV or 40 keV. The incoming beam was focused on the sample with a single bounce capillary, producing an incident X-ray beam size of 10 μm. The fluorescence radiation of the foils was detected with a SDD. (a) The confocal volume was measured by depth scans of different foils (Fe, Zn, Zr and Pd) and was determined by (b) Gauss fits of the profiles.

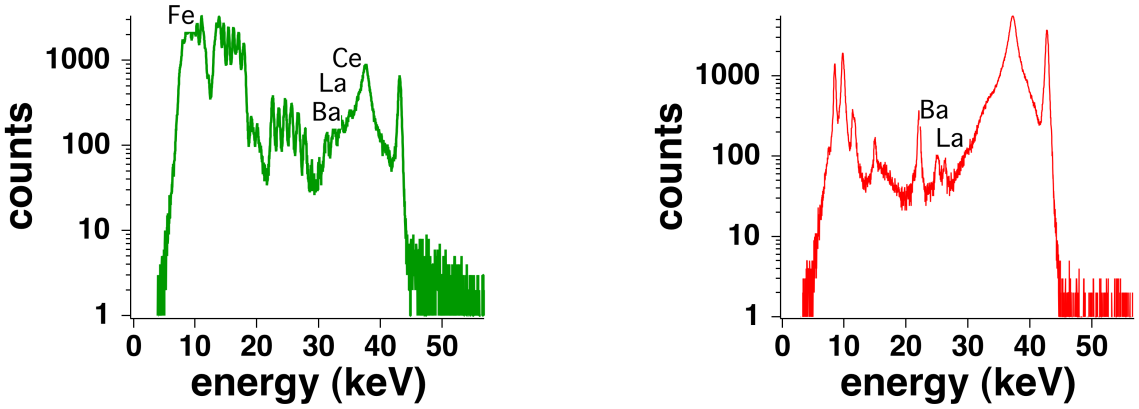

Figure 3: Fluorescence spectrum of 100 μm thick standard reference material NIST610 (a) with confocal capillary with 100 sec exposure time. The sample is a silicate glass doped with elements with Z = 26 - 72 at concentration levels of 500 ppm. Fluorescence spectrum of a standard reference solution with Ba and La (b) loaded into a hydrothermal DAC. Concentration level of Ba and La around 1000 ppm, sample time 300 sec.

References

[1] M. Wilke, K. Appel, L. Vincze, C. Schmidt, M. Borchert, S. Pascarelli (2010) Journal of Synchrotron Radiation 17, 669.

[2] S. Petitgirard, M. Borchert, D. Andrault, K. Appel, M. Mezouar, H.-P. Liermann, (2012) Review of Scientific Instruments 83, 013904.

[3] M. Borchert, A. Ehnes, I. Schwark, A. Watenphul, K. Appel (2011) HASYLAB annual report, this volume.