A con-focal setup for micro-XRF experiments using diamond anvil cells

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In this contribution we introduce an experimental setup to perform con-focal micro X-ray fluorescence measurements in situ in samples at high temperatures and pressures in diamond anvil cells (DAC) (e.g. Schmidt et al. 2007). The con-focal arrangement is used to suppress the background in X-ray fluorescence (XRF) spectra that stems from elastic and inelastic scattering of the diamond anvils. The setup is based on a focusing optic in the incident beam that reaches a spot of 5-10 \( \mu \)m and a focusing poly-capillary in front of an energy-dispersive solid-state detector. The detector poly-capillary is designed to work at a very long working distance of 50 mm in order to collect the radiation from the center of the DAC at 90° to the incident beam. The probing volume is defined by the two foci and has a size of ca. 300 \( \mu \)m at 8 keV and 150 \( \mu \)m at 19 keV as measured by scans through thin metal foils. Comparison of XRF spectra acquired with a usual detector collimator and spectra recorded with the detector capillary shows a strong suppression of XRF signal generated outside the probed volume, i.e. XRF from the gasket material and signal from elastic and Compton scattering by the diamond anvils. The ratio of the Zr K-alpha fluorescence peak to the peak of the Compton scattering changes from 0.5 (collimator) to 1.26 (detector capillary) for a ca. 1000 ppm Zr standard solution and an incident beam energy of 20 keV. For a standard solution containing ca. 1000 ppm Hf, the ratio of the L-alpha to the Compton signal increases to 6 using the detector capillary and an incident beam energy of 9.7 keV. Thus, the con-focal setup substantially improves the fluorescence to background ratio. This will result in higher sensitivities for dilute elements in the sample chamber of the DAC. Furthermore, the possibilities of interference of the sample's signal with signal from the sample environment are greatly reduced. In a broader sense, the setup can also be applied to other confined samples that require long working distances.

Schmidt et al. (2007) Lithos 95, 87-102