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Design of an anaerobic sample chamber for fluorescence measurements compatible with the Lytle detector

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A sample chamber has been developed that is compatible with the commercially available Lytle ion chamber with soller slits. The key features are (i) the sample position can be shifted vertically without changing the geometry with respect to the soller slits and ion chamber, (ii) the gas-tight structure makes it possible for experiments to work with samples that require anaerobic conditions.

Keywords: XAFS; instrumentation.

1. Introduction

Fluorescence-mode X-ray absorption spectroscopy has been frequently measured using a combination of soller slits and an ion chamber in Stern–Heald geometry (Stern & Heald, 1979). At the 10ID beamline at the Advanced Photon Source (http://mrcat.iit.edu/) many users perform quick-XAFS scans, with each measurement taking 2–5 min. Since quick-XAFS scans are possible with high-intensity X-ray beams, we need to be able to: (i) script the sequence for automatic XAFS measurements for several samples loaded on a sample holder, (ii) change the sample position for each measurement in case of damage owing to high-intensity X-rays, (iii) change the $Z-1$ filters from outside of the sample chamber, and (iv) run the sample under different types of controlled atmosphere. In order to meet all these conditions we have designed a sample chamber compatible with the existing sollar slits and ion chambers.

2. The sample chamber

Fig. 1 shows a schematic of the sample chamber. An outer box is made of aluminium plates screwed together. Incoming and outgoing X-ray beams pass through a NW25 nipple flange, that enable it to be connected to flight paths with a vacuum flange with X-ray windows. For simplicity, most of the seals used ultra-torr seal or epoxy resin, though welding the chamber or machining from a single piece might be required for some applications under special conditions. The fluorescence window can be easily replaced depending on the energy of the X-rays or the experimental conditions. The slot for the $Z-1$ filters is accessible from outside the chamber by sliding from the side or from the top, so that optimization is possible without opening the sample chamber. The sample is mounted on a sample holder, attached with a rod extending from a linear slide system shown in the figure. The sample holder is connected to the motorized shaft with a vertical motion without in-plane rotation of the sample holder. We use Haydon linear actuators, which do not require shaft couplers (http://www.haydonkerk.com/). The motors can easily be driven with a standard Steppak SPD6B bipolar driver at 0.5 A 4 V DC with excellent reproducibility. The linear slides are attached to the lid, which can be sealed with a rubber gasket on the grooved flange. In the design, maximum care has been taken with the relative geometry

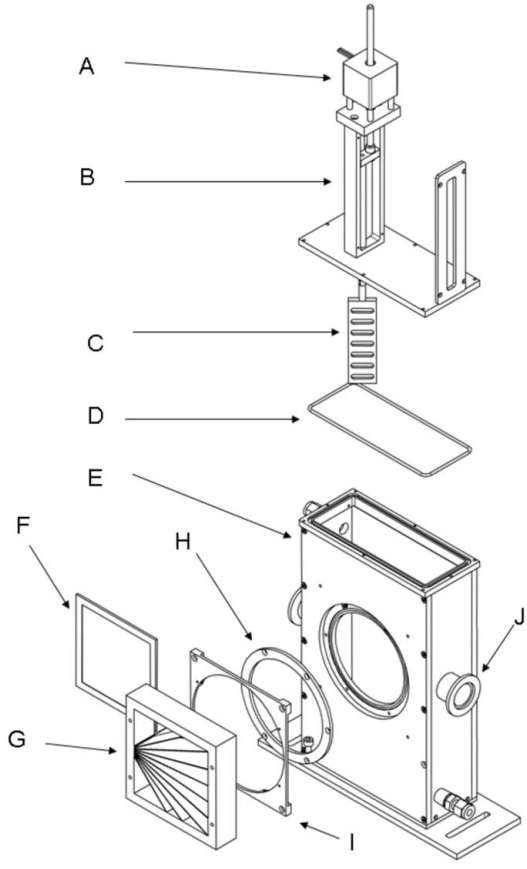

Figure 1
Design of the sample chamber for the Lytle detector. A, Haydon motor; B, linear slide; C, sample holder; D, rubber gasket; E, sample chamber; F, $Z-1$ filter; G, soller slits; H, filter guide; I, soller slits; J, NW25 nipples.

of the sample position and soller slits such that fluorescence photons can be selected with good efficiency.

This was used successfully to measure Zn and Se *K*-edge XAFS of ZnSe semiconductor thin films possessing a thickness gradient deposited on Si (100) wafer and quartz glass. It was also used for measuring the Ru *K*-edge of a solution with a density gradient along its depth.

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References

Stern, E. A. & Heald, S. M. (1979). *Rev. Sci. Instrum.* **50**, 1579–1583.