Multi-channel collimator, a tool for high-pressure angular-dispersive XRD experiments

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Introduction

The investigation of matter under extreme conditions is one of the main issues addressed at synchrotron radiation sources. Among the various experiments focused on the investigation of the properties of matter at non-ambient conditions (e.g. high pressure / high temperature) studies of highly disordered materials (as liquids and amorphous) turn out to be one of the most challenging. The reason is lack of information obtained from the sample attributed mostly to the following experimental difficulties:

1. Liquids and amorphous materials exhibit diffuse, low intense x-ray diffraction signals sometimes several orders of magnitude lower compared to crystalline counterparts.

2. The investigated sample volume is usually very small, depending on the applied pressure and the type and construction of the cell.

3. The weak diffraction signal is furthermore contaminated by the inevitable presence of a sample container like a gasket staying in the path of the direct beam.

One can therefore understand that the analysis of such low intense and contaminated diffraction signals becomes very frustrating and in many cases even impossible. In our previous work [1] we demonstrated the functionality of the new Paris-Edinburgh press allowing in-situ high pressure/temperature studies, adapted recently to the PETRA experimental beamlines. In the same article we expressed the need for a spatial collimator capable to filter out unwanted scattering from the diffracted signal.

The schematic drawing in Fig. 1 shows how spatial collimator filters the diffracted signal. The direct beam transmitted through the gasket assembly (the sample is inserted in the middle) is sequentially diffracted from each individual part. The total angular dispersive diffraction signal is at the end a sum of the contributions from each layer. In general the separation of the individual terms is complicated by the geometry changing with pressure which renders absorption corrections very difficult. The collimator inserted between sample and detector acts as a spatial filter transmitting only the signal from a geometrically restricted volume (where the sample is located) while the rest is being absorbed. This is equivalent to the crossed beam technique used in energy-dispersive X-ray experiments.

1 in case of large volume cells like Paris-Edinburgh one needs to take into account additional heating elements, sample protective layers, and thermocouple.
Design of multichannel collimator and supporting system

The collimator in general consists of two concentric sets of slits sitting on a base plate, shown in Fig. 2a. The bulk shape of collimator was made from a single block of stainless steel by machining, while the slits were cut using wire electroerosion. The inner (width 100 µm) and outer (300 µm) slits are located 5 and 20 cm from the sample, respectively. Following reference [2] the angular separation between the slits was set to be 0.81°. As follows from the collimation principle (see sketch on Fig.1) the high accuracy specified for the collimator shape coupled to precise alignment is crucial in order to eliminate parasitic scattering. The precise alignment can be done by four independent translation motors connected to the collimator see Fig. 2b. The overview photograph taken at the PETRA1 beamline (Figure 2c) shows the whole assembly (collimator and P-E press) prepared for in-situ measurements.

First test results

In order to examine the functionality and efficiency for background suppression, an X-ray powder diffraction measurement was performed on a LaB$_6$ [3] sample. The measurements were carried out at HASYLAB on the experimental station PETRA1. The sample encapsulated in an amorphous boron gasket surrounded in addition by a graphite cell and BN protective layers was illuminated by a well collimated 1x1mm X-ray beam of 21keV photon energy. The diffracted signal was filtered by well aligned multi-channel collimator focused to the sample volume, operates in a sweeping mode (forth and back rotation 10x) within an angular range 1.62° and rate 0.03 °/s) during the data acquisition time. The corresponding XRD patterns recorded by a 2D detector

\[ \text{alignment in respect to sample and direct beam} \]
\[ \text{see Figure 1c in [1] for typical sample assembly for HP measurement} \]
(mar345 Image plate) were in the next step integrated into 2Theta space by using Fit2D [4]. Figure 3a shows the sample’s 2D XRD pattern recorded for 3 minutes without the collimator. On the picture the most intense Debye-Scherrer rings belong to LaB$_6$ and BN but in addition many intermediate rings fill the space in between. These rings originate from the front and back side of the gasket and graphite cell. Furthermore, the pattern shows bright areas “the ears” diverging from the centre of the beamstop. This is fluorescence radiation from the sample. The shape is determined by the presence of the upper and lower anvils. The XRD pattern from the same sample using the multi-channel collimator is shown in Figure 3b. Compared to the above the pattern is significantly cleaner from intermediates as well as from the ears. After radial integration (Figure 3c) the differences become even more pronounced. The pattern without collimator shows very complicated and intense background composed of many Bragg peaks. The background furthermore shows a diffuse liquid-like pattern with oscillations over the whole 2Theta range. Such background oscillations would make a structure analysis of amorphous materials practically impossible. The filtered XRD pattern measured in presence of the collimator shows much better peak/background ratio, the background itself looks far more simple with only a few low intensity peaks. We hope in future measurements we will be able to suppress the background even more by reducing the beam size, improving the alignment procedure and by using a convenient beam stop just behind the fist collimator row. The last remark concerns the total sample measurement time. In the presented experiment the total time to get the filtered XRD pattern extends over the measurement without collimator by factor of 10.

Figure 3: Raw data without collimator (a) and with collimator (b). The XRD patterns of a LaB$_6$ sample encapsulated in the gasket, measured without and with collimator (a).

References