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# Collimation of x-ray diffraction under high pressure at Beijing Synchrotron Radiation Facility

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**Abstract**
A collimation system was designed to reduce the scattering background in the energy-dispersive x-ray diffraction (EDXD) experiments at the high-pressure station of Beijing Synchrotron Radiation Facility. The results showed that the collimation system could improve the signal-to-noise ratio substantially. Some better data were obtained which were very difficult to access before in EDXD experiments. The system provides a useful technique for experimentation at pressure higher than 100 GPa.

## 1. Introduction

X-ray diffraction experimentation is a very important technique in research on the equations of state and phase transitions under high pressure. At the beginning of the 1990s, use of the energy-dispersive x-ray diffraction (EDXD) technique was achieved at Beijing Synchrotron Radiation Facility (BSRF). However, for nanocrystalline or low-$z$ material, the diffraction signal is usually weaker than the noise signal that comes from the Compton scattering and other scattering background in the hutch. In such experiments, there is a strong noise background with some low and blurry peaks associated with the sample in the diffraction pattern. Another source of noise is the scattering of x-rays by the gasket. The situation becomes worse in the case of ultrahigh pressure ($>$100 GPa), since the peaks from the sample become weaker and broader.

A collimation system was designed to solve these problems. The collimation of the x-ray diffraction beam is an important step in the EDXD experiment. It is essential for the location of the rotation centre, precision of angles and the resolution of spectra. In the past, there has only been a slit before the window of the detector, which can only limit the angle of the diffracted beam. The location of the rotation centre and diffraction beam cannot be achieved using solely this method, so a system for collimation of the diffraction x-ray beam was designed and installed to solve these difficulties. With this collimation system, the scattering backgrounds were reduced efficiently and the signal-to-noise ratios of the data for dilute samples were improved in high-pressure EDXD experiments.

  

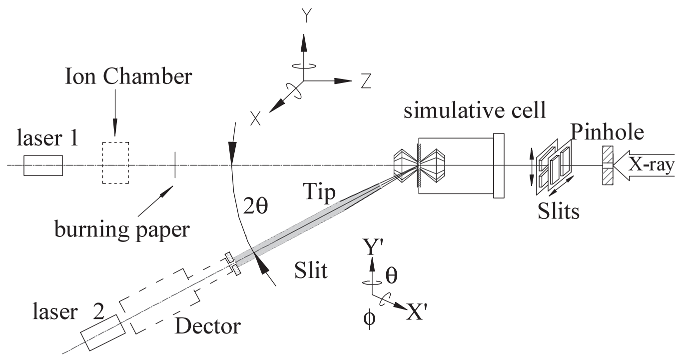


**Figure 1.** A sketch of the collimation system.

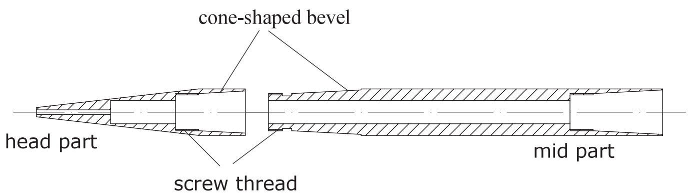


**Figure 2.** Connection of the head part and mid-part of the tip.

## 2. Structure of the collimation system

A sketch of the system is shown in figure 1. The system for collimation of the diffraction beam is a cuprous pipe (tip) with a five-dimension ($X$, $Y$, $Z$, $\theta$, $\phi$) movement. The five-dimension control device, which can be controlled by the PC, consists of three stepper motorized stages to remotely control the translation of the tip in the $X$-, $Y$- and $Z$-directions, and a motorized pint-sized mount with 0.5 $\mu$rad resolution that controls the yaw and pitch.

The tip consists of three parts: the head part, mid-part and cover part. In the head part, there is a hole of 0.5 mm diameter, which is sealed by 2–3 mm thick lead, and a hole with diameter of 130–300 $\mu$m is drilled in the lead. The head part of the tip can be changed easily according to the requirements of the experiment. A screw thread and a cone-shaped bevel were designed and connected for collimation at the coupling part between the head part and mid-part, as shown in figure 2.

At BSRF, two lasers were used for establishing the datum line of the experimental beam. Laser 1 is located on the $Z$-axis to present the incident beam datum line; laser 2 is positioned on the rotation arm of the detector to simulate the diffraction beam datum line.

The steps for setting up the datum lines are:

(1) The incident x-ray beam is visualized by using burning paper, a simulative cell (there is a hole at the centre of its top; the laser and x-ray beam can pass through the hole), slits and a pinhole. Then laser 1 is adjusted to pass through all the elements, and then it presents the incident beam.

(2) Laser 2 on the rotation arm of the detector is adjusted to present the simulative incident beam when the rotation arm of the detector is turned to zero degrees ($2\theta = 0$; this degree is only mechanical zero, not the real zero degrees) and then clear the angle indicator. The diffraction datum line and incident datum line are on the same line at this time.

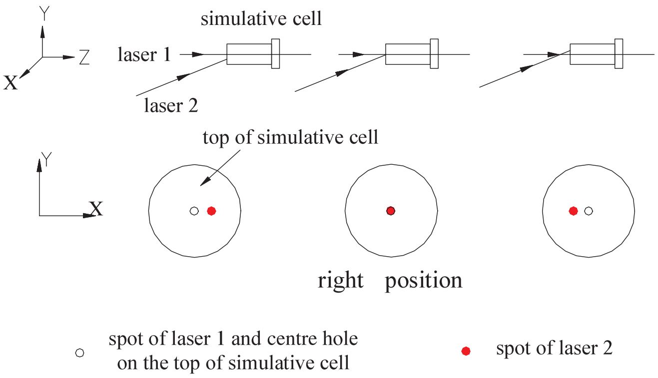


**Figure 3.** Adjusting the position of the simulative cell.
(This figure is in colour only in the electronic version)

(3) The five-dimension control device is used to locate the tip, let the beam of laser 2 pass the tip and then the simulative cell and slit.
(4) The precision of the collimation by laser is not sufficient. The position of the tip will be checked using the ion chamber, which is set before laser 1 after step 2. The PC controls the position of the tip by adjusting the stepper motorized stages and the motorized pint-sized mount. The ion chamber monitors the intensity of the x-ray passing through. The position of tip where the ion chamber gets the maximum signal is the best position.

The steps of setting up the rotation centre are:

(1) Turn the rotation arm to $2\theta$ degrees, and set the position of the simulative cell (during experiments, the gasket in the DAC is in the same plane as the simulative cell's top, which is set up here) on the $Z$-axis. After passing the tip, the spot of laser 2 always transits the small hole at the centre of the simulative cell's top, where the spot of laser 1 transits also. The different positions of the spot of laser 2 and the simulative cell are compared in figure 3.
(2) Turn to a different value of $2\theta$ and correct the position of the simulative cell continually; then the simulative cell is positioned at the rotation centre.
(3) The precision of the location of the rotation centre by the laser is not sufficient. The position of the rotation centre will be checked using the detector monitoring the diffraction signal.

During the experiments, the head part of the tip should be close to the cell; the window of the detector should also be close to the tail of the tip.

## 3. Result

The scattering background arises from the Compton scattering and other scattering sources in the hutch as described previously. Before adding the collimation system, the signal received by the detector is limited only by a slit before the window of the detector, so there is a big acceptance angle for the noise. When using the collimation system, the scattering background is reduced efficiently and the signal-to-noise ratio is clearly improved.

This collimation system was adjusted in February 2001. In May 2001, it was applied in most experiments, and some better data were collected. Diffraction patterns without and with the tip are shown in figures 4 and 5, respectively. This shows that the collimation system can

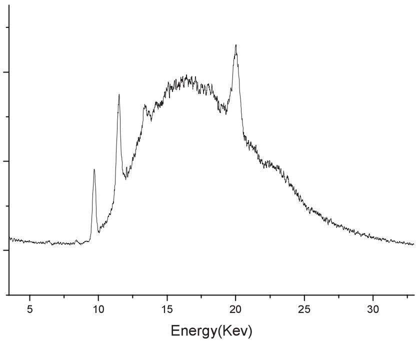


**Figure 4.** The nano-wire material without a tip under $P = 80$ GPa.

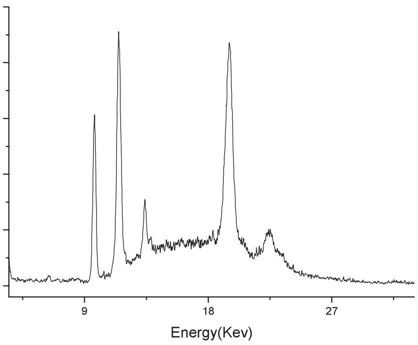


**Figure 5.** The nano-wire material with a tip under $P = 80$ GPa.

reduce the ambient background effectively and improve the signal-to-noise ratio of the data remarkably. The peaks of samples can be distinguished clearly.

## Acknowledgment

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