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A high pressure XRD setup at ADXRD beamline (BL-12) on Indus-2

Tapas Ganguli, A.K.Sinha[1], Chandrabhas Narayana*, Anuj Upadhyay, M.N.Singh, P.Saxena, V.K.Dubey, I.J.Singh, Sendhil Raja, H.S.Vora, S.K.Deb
Raja Ramanna Centre for Advanced Technology, Indore 452013, INDIA
* Jawaharlal Nehru Center for Advanced Scientific Research, Bangalore, INDIA

E-mail: anil@rrcat.gov.in

Abstract. A high pressure XRD measurement setup in the angle dispersive geometry has been setup in the Angle Dispersive X-ray diffraction beamline (BL-12) in Indus-2 synchrotron facility. The X-Ray beam is collimated inside the diamond anvil cell (DAC) using a pair of cross-slit collimators and 100 micron orifice in a 400 micron thick Ta sheet. With the use of an adaptive optics to ensure a converging beam at the sample position, the need of an x-ray beam collimator is eliminated making the alignment of the DAC quite easy. The alignment of the DAC with respect to the incident x-ray beam is made by placing it on a computer controlled sample mounting and alignment stage developed specifically for this setup. Interactive software has been developed to make the alignment of the X-ray through the DAC very easy and accurate. NIST standard LaB6 powder was used for test runs, and a few fine pieces of gold served as pressure calibrator. The data was recorded on a MAR345 Image plate detector.

1. Introduction

High pressure XRD measurements are extremely important to determine the structural properties of materials under extreme condition and provide information about the bulk modulus, stable phases etc. Recently, we have set up a high pressure diffraction measurement setup at the Angle dispersive X-Ray diffraction (ADXRD) beamline (BL-12) of the Indus-2 synchrotron. The setup has been used with a Mao-Bell type Diamond Anvil cell (DAC) for the first set of demonstration experiments. Some user measurements at high pressures have also been carried out in the recent past. In this paper the design of the high pressure measurements has been explained.

2. Experimental setup

The beamline, installed on a bending magnet, is designed to operate in the photon energy range of 5-25 keV, with an adaptive optics. The pre-mirror focuses the x-rays in the vertical plane and the second crystal of the double crystal monochromator focuses in the horizontal plane. The focal spot of the beam is about 500 microns x 500 microns. The length of the beamline is approximately 33m, with a defocusing of 1:3. In the high pressure measurements, the X-Ray beam is focussed at a point slightly behind the sample position. At a distance of about 195 mm in front of the focus point, a pair of cross-slits placed 80 mm apart with an opening of 300 microns is used to collimate and define the beam. Between the cross-slits and the focus point, a Ta orifice is placed to define the size and the position of the beam. Figure 1 shows the schematic of the beamline and the collimating scheme followed in this work**.**

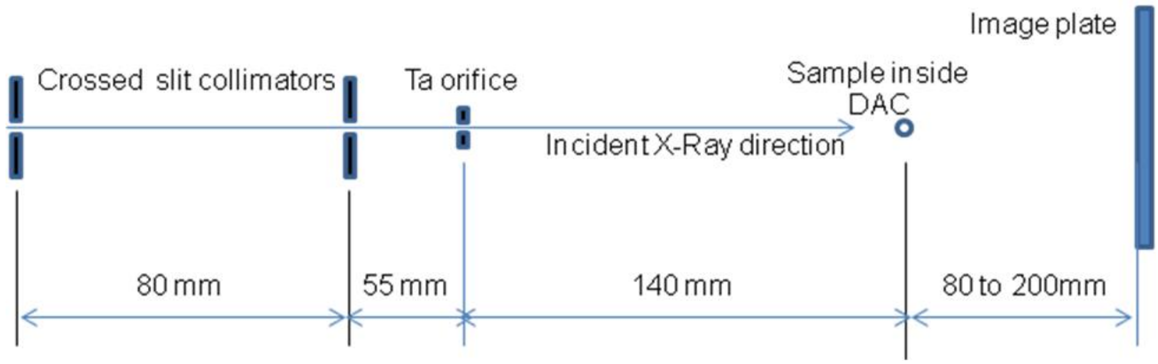

Figure 1: The schematic with the distances between the collimating components and the DAC used in the high pressure setup.

This orifice is made by laser drilling a 100 micron hole in a 400 micron thick Ta sheet. An Yb-Silica fibre laser with a pulse energy of 0.2 mJ and pulse width of 20 nanosecond operating at a rep rate of 20 kHz, was used to ablate the hole in the Ta sheet. The Ta sheet was mounted in a fixture to hold it flat at the focus of the scan lens and the holes trepanned by moving the focussed laser spot (20 micron dia) in a circular fashion using a XY galvo-scanner under computer control. The thickness of the Ta sheet ensures that all the X-Ray, even for the highest photon energy in the beamline (25 keV) get completely absorbed inside the sheet. The position of the orifice is observed through a camera and fine adjustments in its position are made manually to maximize the intensity through the orifice.
A very crucial aspect of the setup, is the proper alignment of the DAC with the X-Ray beam. The typical cross-sectional area of the sample volume inside the DAC is about 150 microns in diameter. This is required to be aligned with approximately 100 micron diameter of the incident X-Ray beam. To achieve this, a computer controlled sample mounting stage has been developed specifically for this work. In this system, the DAC is placed on a computer controlled x-y motion stage. The DAC is then scanned perpendicular to the X-ray beam (x-y plane) initially in a 10mm x 10mm area at 100 micron step, and finally in 2.5 micron step. The area of the scan is selectable through an user friendly software. Behind this stage, a Si photodiode (AXUV-100) is placed to monitor the X-Ray intensity through the DAC. The X-ray intensity, as read from the Si photodiode, is plotted as a function of the x-y position of the DAC using the same software (a contour plot). Figure 2 shows a screen shot of the X-Ray beam locator software that plots the X-ray intensity as a function of the DAC position. The user can then move the x-y stage carrying the DAC to the desired location on the click of a mouse button.
After the alignment of the DAC the diffraction measurements are taken on an Image plate, measurements are taken on a MAR345 Image plate detector. Figure 3 is a picture of the complete setup during the experiments. However, this DAC mounting and alignment stage can be easily modified for other designs and sizes of DACs.

3. Results
Initial measurements to test the suitability of this system have been carried out on this setup using the NIST standard LaB_6 powder. Figure 4 shows the data as a function of pressure. Au powder was used for the calibration of the pressure inside the cell. The data was taken on the image plate,

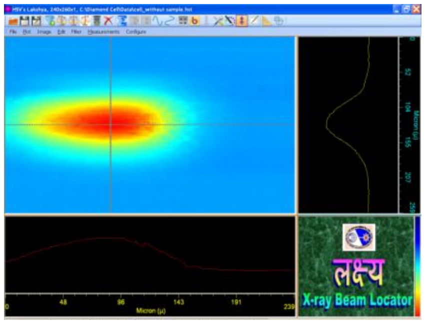

Figure 2: A view of the X-Ray beam locator software

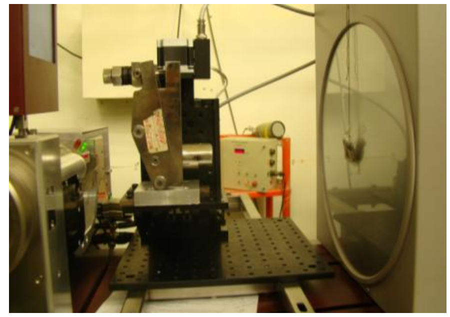

Figure 3: Picture of the DAC on an automated x-y scanner

with an acquisition time of about 20 mins. The samples were placed inside a Mao Bell type DAC with a gasket diameter of about 150 microns. No significant gasket peaks are observed in the data. This is due to the following two points that have been taken care of in this work: 1) the X-Ray beam was used in the collimating geometry so that after passing through the Ta orifice the final beam at the sample is smaller than the orifice diameter, and 2) the precision x-y scanner stage ensures that the X-Ray beam passes exactly through the centre of the DAC. Figure 4 shows the diffraction patterns of LaB_6 as a function of pressure. The absence of the gasket peaks and the shift of the Au peaks with pressure are clearly seen. The cubic phase (atm. pressure) peaks of the LaB_6 are highlighted in the data.

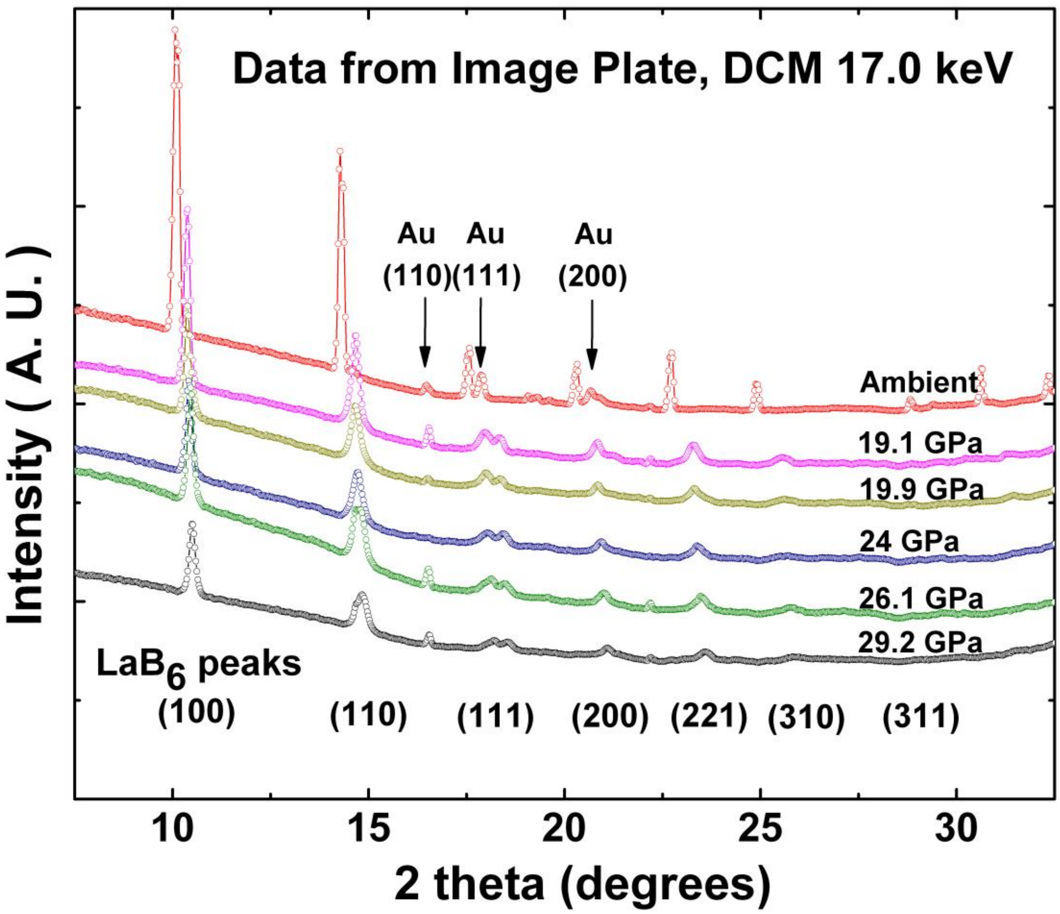

Figure 4: The XRD pattern of LaB_6 as a function of pressure.

4 Conclusions

We have installed and tested a high pressure XRD arrangement at the ADXRD beamline at Indus-2. This setup is available for high pressure measurements for users.

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