A.2: Development of a High pressure XRD setup at the ADXRD beamline on Indus-2

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.

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.

This orifice is made by laser drilling a 100 micron hole in a 400 micron thick Ta sheet. An Yb-Silica fibre laser (pulse energy: 0.2 mJ, pulse width: 20 nanosecs, rep rate: 20 kHz) was used to ablate the hole in the Ta sheet. The Ta sheet was mounted 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 upto 25 keV (max. in the beamline) 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. Figure A.2.1 shows the schematic of the setup.

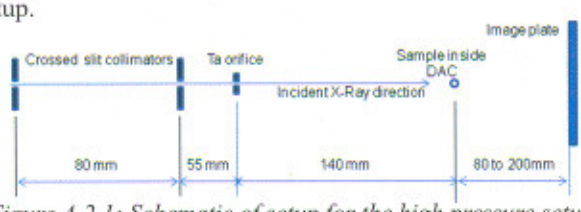

Figure A.2.1: Schematic of setup for the high pressure setup.

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 10 mm x 10 mm 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). The user can then move the x-y stage carrying the DAC to the desired location on the click of a mouse button.

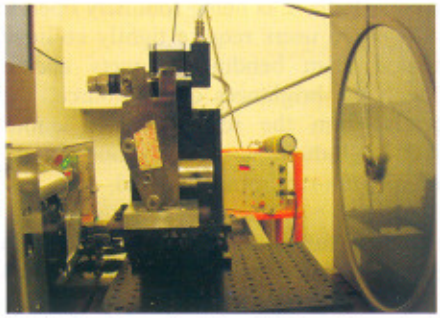

Fig. A.2.2: Photograph of the DAC on the automated x-y scanner

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 A.2.2 shows 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.

Initial measurements to test the suitability of this system have been carried out on this setup using the NIST standard LaB_6 powder placed inside a Mao Bell type DAC with a gasket of 150 μm. Figure A.2.3 shows the data as a function of pressure. Au powder was used for the calibration of the pressure inside the cell. 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. 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

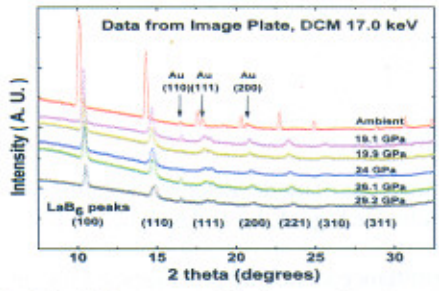

Fig. A.2.3: The XRD pattern of LaB_6 as a function of pressure

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