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An energy dispersive X-ray diffraction station at a VEPP-4 synchrotron beam line for structural studies at high pressure

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Abstract

The energy dispersive X-ray diffraction (EDXRD) station at a VEPP-4 synchrotron beam line is presented. The first results obtained with a high pressure chamber at the VEPP-3 synchrotron beam line are shown.

Two intrinsic advantages of energy dispersive X-ray diffraction (EDXRD) of polycrystalline samples are fixed scattering angle geometry and fast data collection because of the use of the whole energy spectrum of the incident primary beam. This also makes it an ideal tool for the investigation of materials at non-ambient conditions. Synchrotron radiation with a high intensity, low divergence beam and a smoothly varying energy spectrum further enhances the above features, especially at high pressures. That is why EDXRD stations for structural studies under pressure are becoming popular at various synchrotron radiation centers in the world.

The EDXRD station will be installed on channel number 7 of synchrotron radiation of the storage ring VEPP-4 (Fig. 1). Channel number 7 takes radiation from magnet IV (field 9.4560 kG). The critical energy and wavelength are equal 22.6 keV and 0.55 Å, respectively. The distance between the SR source and the first collimator is equal to 36.85 m, and the distance between the SR source and the sample is equal to 42.45 m.

This report gives preliminary results for an EDXRD system that was installed on the VEPP-3 synchrotron under the Indo-Russian Long Term Programme of cooperation in science and technology (ILTP). The VEPP-3 and VEPP-4 storage rings are located at Novosibirsk Russia and the nominal operating energies are 2 GeV and 7 GeV respectively. Fig. 2 gives its spectral photon flux.

The EDXRD set-up consists of the following subsystems:

1) Adjustable aperture primary beam collimator.
2) A Mao–Bell type diamond anvil cell (DAC) mounted on x–z–θ–ϕ stages (Fig. 3).
3) A conical slit between the DAC and the solid state detector to select a suitable scattering angle.
4) A data acquisition, control and processing set-up consisting of a solid state detector, an amplifier, an ADC, and the following NIM standard interface modules: multichannel analyzer (MCA), CAMAC modules for the control of the stepping motors, and a personal computer (PC AT/386, 25 MHz).
5) The necessary software for data acquisition, control and analysis.

The primary beam collimator was made up of two orthogonal rectangular slits of adjustable widths; as a whole it could be positioned normal to and centered on the beam by appropriate translation, tilt and rotation stages. The variable aperture helped in alignment of the spectrometer and in eliminating extraneous peaks from the gasket and DAC parts in the collected spectra.

The used diamond anvil cell is a modification of the standard Mao–Bell design [1]. Here one of the diamond anvil backing plates is half spherical and the other is cylindrical. The ~ 0.3 carat diamond anvils fixed on them can be tilted and translated to make them parallel to each other and to center the anvil faces on the piston and cylinder axis on which the backing plates are fixed. In the present cell the backing plates are made of hardened steel. The x–z–θ–ϕ stages served to locate the cell in the center of the beam to within ~ 5 m. Due to space limitations in the VEPP-3 experimental hall and the limited photon flux above 30 keV, a conical slit which collects a larger portion of a Bragg cone than a point slit was employed to gather the intensity data. It defined a scattering angle of $\theta \sim 6°$ and was directly attached to the DAC. This conical slit

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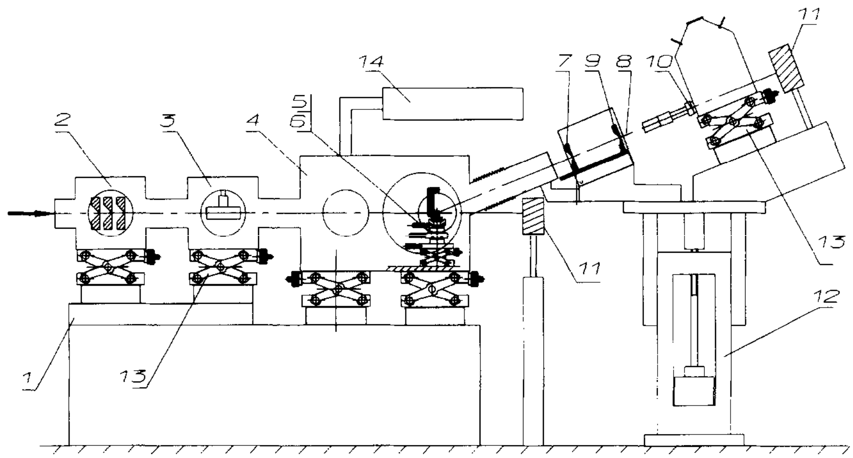

Fig. 1. The schematic diagram of the energy dispersive X-ray diffraction station on the beam line of VEPP-4: 1 – platform; 2 – input collimation system; 3 – ionization chamber; 4 – experimental box; 5,6 – diamond anvil cell mounted on x–z–θ–ϕ stages: 7,8 – slits; 9 – output collimation system; 10 – solid state (Ge) detector; 11 – beam stopper; 12 – mechanism for vertical movement of detector; 13 – mechanism of vertical movement; 14 – laser system for measurement pressure.

slide-fits into the DAC and its position can be adjusted so that the cone apex coincides with the sample position (the diamond face).

The subsystems (1), the CAMAC interface modules and the crate together with the software for the control of the stepping motors fabricated by INP were integrated in systems (2), (4) and (5), designed, fabricated and tested at BARC, and were installed at beam line 5B of VEPP-3. The conical slit was designed at BARC but fabricated at INP.

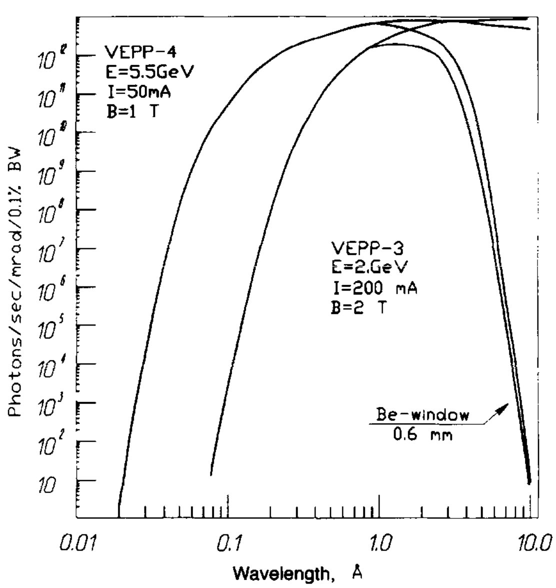

Fig. 2. The spectral photon flux from storage rings VEPP-3 and VEPP-4.

The hardware of the data acquisition, control and analysis system is configured to take advantage of the high counting rate available with synchrotron radiation and to make the PC available for processing of the accumulated spectra data, while the data acquisition is on in a standalone mode. The heart of the system is a multichannel analyzer (MCA) module for data collection, an on-line display of the spectra and data reduction and transfer to the PC for further analysis. The MCA is a microcomputer (8085) controlled with 4 K channel memory. The fast data accumulation is implemented using direct memory access (DMA). A Si(Li) detector with a measured resolution of

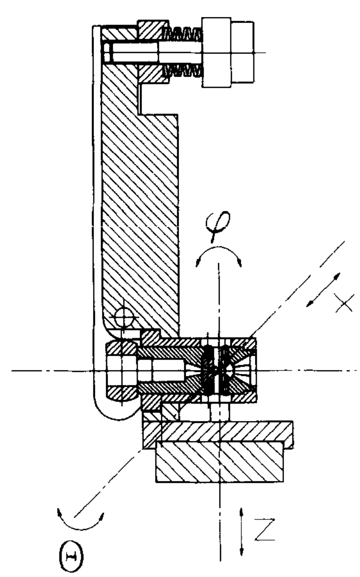

Fig. 3. Diamond anvil cell.

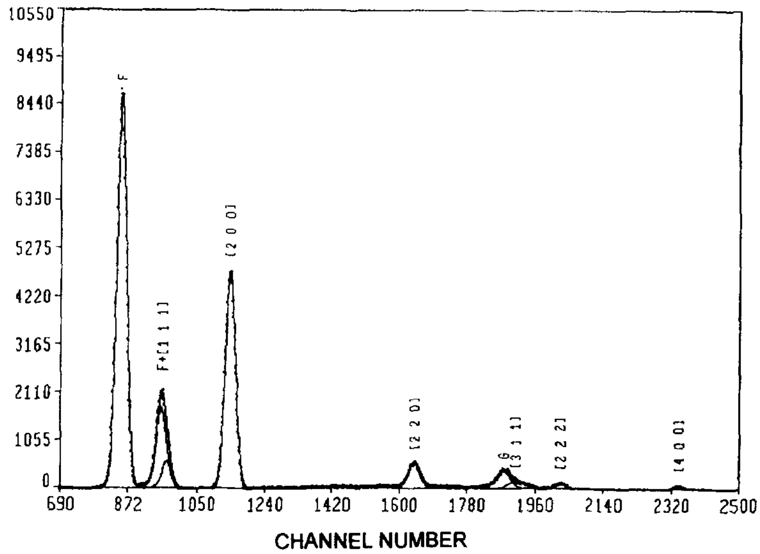

Fig. 4. RbCl in the NaCl phase, $\theta = 5.314$, energy = 0.8787 + CN × 0.01466 (CN-channel number). VEPP-3 operating at 2 GeV.

168 eV at 13.9 keV was used for the energy analysis of the diffracted radiation. The output, after appropriate amplification and pulse shaping, is fed to an ADC which is interfaced to the MCA. The module can be a software set (from the PC key board) to collect one 4 K spectrum or sequentially collect four 1 k spectra (for kinetic studies) at present. The software controlling the module is versatile with menu driven options for live display of the spectra on the PC terminal and data handling commands like scaling, smoothing, peak search and data transfer to the PC for further fit peak analysis. Another interface card has been plugged into the PC which controls all stepping motors used for adjustments of the input collimator and the DAC. It uses CAMAC modules with software menu driven options.

The following software developed at Trombay was used for processing of the collected spectra. These programs were available on the PC used for the data collection:

a) ECALC – for energy calibration of the ADC from a standard source (say ^{241}Am) spectrum.

b) EBRAGG – for finding the scattering angle by least squares refinement of the recorded Bragg peak positions for a sample of known lattice parameters.

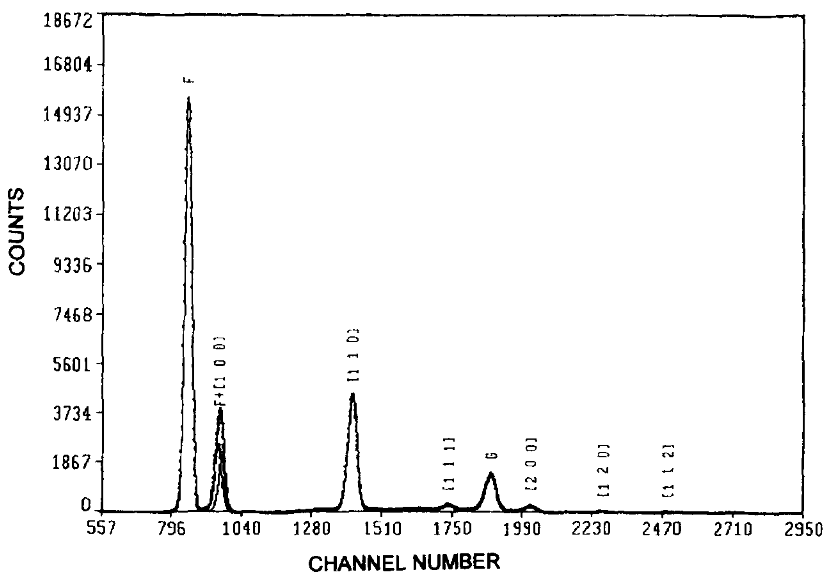

Fig. 5. RbCl in CsCl phase (above 2.7 GPa).

c) FIT – for determining the peak positions, half widths and integrated intensities by non-linear least square fitting of the observed data. Gaussian peak shapes are used to fit the individual peaks. It can resolve overlapping peaks and constrain parameters of some peaks (a very useful feature in separating fluorescence peaks from Bragg peaks).

d) DSPACE – for calculating the space group allowed d-values, Bragg energies and corresponding channel numbers from lattice parameters of a sample.

e) CREF – for least squares refinement of the cell parameters from an indexed powder pattern.

A Reitveld least squares program for analysis of Bragg intensities was also available, but it was not used in the present experiments.

In a typical experimental run, initially the primary beam collimator was positioned in the beam with a ~ 500 μm aperture, and the transmitted beam intensity was maximized by monitoring it by an ionization chamber. Then the DAC was positioned in the beam and the above process was repeated by adjusting the DAC only with the help of x–z–θ–ϕ remote controlled stages. The spectra were then collected, the aperture of the beam was progressively reduced to 75 μm and the DAC was appropriately adjusted to reduce the extraneous peaks due to the gasket.

The data collected from RbCl in the spring steel gasket demonstrating the NaCl to CsCl structural transition under pressure in it is shown in Figs. 4 and 5 respectively. The pressure was determined from its known equation of state [2]. It is obvious from these figures, that the data collected at VEPP-3 is of superior quality with good statistics, and better resolution up to a higher range of d-values. The data collected at higher energies will be further improved when we use an intrinsic Ge detector.

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