High Pressure II
Microbeam Techniques

MS18.02.01 THEORY AND PROBLEMS OF MICROFOCUSING: D. Labergerie, European Synchrotron Radiation Facility, 38043 Grenoble, France.

Because of the unusual index of refraction ($n < 1$) of X-Rays in matter it is impossible to use classical refractive optics such as lenses to focus such beams. Nevertheless, some optics working either by reflection (mirror) or diffraction (bent crystal, multilayers, Bragg Fresnel lenses) can be successfully used to focus X-ray beam to a few microns.

This presentation will deal with focusing mirror and the optimization of different parameters such as: substrates (energy related), mirror shape (spherical, or aspherical), optical quality (roughness and slope errors), and also selected geometries (Kirkpatrick-Baez, Wolter types) to maximize the flux at the sample.

Using geometrical considerations the focal spot size can be calculated as a function of all these parameters, and this at any plane along the optical path. The comparison between the analytical approach and ray tracing simulations: the program Shadow will be discussed.

As an example results obtained with a novel device developed at the ESRF: bimorph mirror will be presented.Two such mirrors will be soon installed on the High Pressure Beamline in a Kirkpatrick-Baez configuration. This new active mirror made of piezo-electric ceramic: have several advantages over conventional bent mirrors.

MS18.02.02 MICROFOCUSING USING K-B OPTICS FOR GEOCARS-APS: FIRST RESULTS Peter J. Eng, Mark Rivers, B.X. Yang*, Tom Duffy CARS, University of Chicago, Chicago, IL 60637, *Present address: Argonne National Laboratory, Argonne IL 60439

We present the test results of micro-focusing a continuous spectrum (4KeV - 65KeV) of x-rays using two, 100 mm long, actively bent mirrors in a Kirkpatrick-Baez geometry, as well as design results for much longer mirrors (~1m) optimized for producing small beams at the Advanced Photon Source. The mirrors are figured by applying in situ two different moments on the ends resulting in a surface figure that approximates an ellipse. We have demonstrated the ability to doubly focus NSLS bending magnet x-rays from 4 KeV to 13 KeV to a spot size less than 4 microns in diameter with a net gain of 2000 over a similar size beam produced with slits. In the bending magnet test the beam was focused in the vertical direction with a high quality rhodium coated Si mirror with a RMS surface roughness and slope error less than 2 and 2rad, respectively. The horizontal mirror consisted of uncoated float glass with significantly greater roughness and slope error. This combination of mirrors worked extremely well, pointing the direction for inexpensive micro-focusing optics. Additional high energy focusing tests were carried out at the NSLS superconducting wiggler beamline X17. In this case a continuous x-ray spectrum was both vertically and horizontally focused using the Si, and W coated float glass, mirror receptively. In this double focusing tests a cutoff energy of 60KeV (at an incident angle of 1mrad) was achieved with a FWHM focal spot size of 10microns horizontal by 3.0microns vertical and a net gain of 150. The system is now routinely used for energy disperseive high pressure diamond anvil cell diffraction measurements at X17, with a substantial improvement in signal to noise compared to beams produced by slits.

MS18.02.03 MICROFOCUSING EXPERIMENTS ON A 2ND GENERATION SOURCE. T. Kikegawa and O. Shimomura Photon Factory, National Laboratory for High Energy Physics, 1-1 Oho Tsukuba, Ibaraki 305, Japan

At the Photon Factory new experimental stations, BL-18C and BL-13B2, have been constructed for high pressure studies using diamond-anvil cells (DAC). Two different focusing optics are exclusively designed for an angle dispersive X-ray powder diffraction experiment with an Imaging Plate as a two-dimensional detector. BL-18C, a bending-magnet beamline, is equipped with a computer-controlled fixedexit double crystal monochromator and a pair of cylindrical mirrors. The monochromator consists of a pair of Si(111) flat crystals with (+n,-n) configuration. The Pt-coated fused quartz mirrors can focus an X-ray beam to about 0.08 mm x 0.03 mm in the Kirkpatric-Baez optics with a cutoff energy of about 25 keV.

A simple focusing optics is adopted for BL-13B2, the end station of the multipole wiggler beamline. The design is strongly restricted by the space problem; i.e. no extra space for further optical components on the existing beamline. Thus inside the experimental hutch only a curved crystal monochromator of the Johann geometry (Matsushita & Hashizume, 1983; Takeshita, 1995) is installed at 33m from the source point. An asymmetically cut Si(220) curved crystal monochromator is commissioned to obtain demagnified images of the syncrotron radiation source. The focus size is approximately 0.10mm in horizontal direction at the focus point 1.5m from the monochromator with the energy of 30keV.

The high pressure studies with DAC are carried out in both experimental stations. However, experiments at simultaneous high pressures and high temperatures are performed using a carbon dioxide laser heating device in BL-13B2.

References: Matsushita, T. and Hashizume, H. Handbook on Synchrotron Radiation, edited. by E.E.Koch (North-Holland, 1983), 1, 261 Takeshita, K (1995) Rev.Sci. Instrum. 66(2), 2234-2240.

MS18.02.04 MICROCRYSTAL DIFFRACTION, DIFFICULT BUT POSSIBLE. Larry W. Finger, Geophysical Laboratory, 5251 Broad Branch Road, N.W., Washington DC 20015-1305, USA

Since the earliest availability of radiation from synchrotron sources, researchers have been attempting to use the high brightness of such facilities to study the crystal structure of materials that do not form large crystals. Because of the correlation between X-ray scattering and atomic number, the dimensionless scattering power, $S = (F_{000}/V_U)^2 V_C \lambda^3$, is a better measure of the strength of the scattered intensity than the volume alone. In the formula, F_{000} is the number of electrons per unit cell, V_U is the volume of the unit cell, V_C is the volume of the crystal, and λ is the wavelength. For SiO_2 (quartz) with a crystal size of 50 μm, and wavelength of 1 Å, $S = 8 \times 10^{16}$. This value is near the minimum for laboratory instruments. If the crystal size is reduced to 1 μm, then $S = 8 \times 10^{12}$, which is near the lower limit for diffraction from a light-element crystal at present synchrotron sources. Advances in design of insertion devices and focusing optics will reduce the lower limit for the scattering power.

The major difficulties with microcrystal diffraction are those of manipulating and mounting micron-sized crystals, ensuring that backgrounds are minimized, and providing an incident beam that is homogeneous on the scale of the "sphere of confusion" of the diffractometer. Despite these severe problems, recent developments in area detectors, and in beamline optics for insertion device lines have made it possible to obtain reliable intensities from very small crystals.