14-Diffraction Physics and Optics

14.01 – X-ray Optics for Synchrotron Radiation

MS-14.01.01 X-RAY OPTICS FOR THIRD GENERATION SYNCHROTRON SOURCES: NEW DEVELOPMENTS AND FIRST EXPERIENCE AT THE ESRF. Andreas K. Freund, European Synchrotron Radiation Facility Grenoble , France

The low emittance and high brilliance of third generation synchrotron sources require a substantial research and development effort in the domain of x-ray optics in order to be capable of preserving the beam quality from the source along the beamline until the sample under study.
At the ESRF we have developed new techniques for cooling, focusing, polarization, etc. During the past seven months the performances of several optical elements under full load have been studied on four different beamlines fed by undulators and high powder wigglers.
Results of these test experiments will be presented for various devices and techniques such as microfocusing by Bragg-Fresnel optics and by glass capillaries, focusing by bent crystals, progress in adaptive mirror optics, cryogenic cooling, diamond coated optics, thin crystal monochromators.
This presentation will be followed by an outlook on ongoing projects and on the most likely performances of future devices.

MS-14.01.02 HIGH HEAT LOAD OPTICS AT THE ADVANCED PHOTON SOURCE. By W. K. Lee* and D. M. Mills, Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60439, USA.

At 100 ma ring current, the 2.5 m insertion devices at the Advanced Photon Source (APS) are capable of producing up to 5 kW total power and up to 150 W/mm^2 peak power density. Optical elements placed in such a beam will suffer considerable thermal distortions unless they are properly cooled. These distortions will dilute the brilliance of the synchrotron radiation. The APS has embarked on an aggressive program designed to develop solutions for this problem. The techniques being developed include liquid metal cooling, novel crystal geometries, cryogenic cooling and novel materials. One of the more promising approaches to this problem is the inclined crystal, which has been pioneered and developed by the APS staff. Recent experimental and computer modeling results of the inclined crystal performance indicate that this may provide an acceptable solution to the high heat load problem for the APS. Details of this will be presented along with a brief overview of the APS high heat load program.

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MS-14.01.03 PRODUCTION OF 0.1 TO 5 MICRON DIAMETER X-RAY BEAMS AND THEIR APPLICATION TO X-RAY CRYSTALLOGRAPHY. By D. H. Bilderback*, S. A. Hoffman, & D. J. Thiel, CHESS and Applied Physics, Cornell University, USA.

We have produced x-ray beams of 0.1 to 5 micron dimension by passing larger diameter x-ray beams through a hollow tapered glass tube. The x-rays are squeezed to a smaller cross section as they undergo successive total reflections from the smooth inner wall of this 'x-ray funnel'. The capillaries we have pulled are made from leaded glass (density=5.2 g/cm^3) tubing with an inside bore of either 20 or 40 microns. Typical lengths for these tapered needles are 10 to 20 cm. Samples are placed within a few microns of the tip to minimize the effect of a slightly diverging x-ray beam. There are many uses for these miniature hard x-ray beams including diffraction, imaging, spectroscopy, etc., from a small part of a sample.

We are just beginning to investigate the x-ray crystallography impact of this technology on the study of tiny crystals. The kind of information that can be obtained from crystallography includes not only the orientation, 3-d structure, mosaicity, but also information on the local stress of a crystallite in a larger polycrystalline matrix of material.

One of the first questions to answer is how small of a sample can be studied? This is a question of the combined strength of the x-ray source, the gain of the x-ray optics, the scattering power of the sample, and the efficiency of the detector. Since the observed diffracted intensities are generally proportional to the volume of sample illuminated, the smallest samples will always demand a very powerful x-ray source and efficient optics. To date with our optics, we have observed intensity gains of up to 1000 and have made beam sizes down to 0.1 microns.

We have recently made Laue diffraction images with a 5 micron diameter beam, utilizing white bending magnet radiation from CHESS on a biological single crystal, lysozyme. Well-exposed and spatially-resolved Laue patterns were obtained on film with a 120 second exposure time, but currently the main problem is radiation damage. The sample had to be translated every 25 seconds during the exposure period in order to keep exposing fresh volumes to a damaging x-ray beam. With cryogenic cooling added, we hope that stationary exposures from even much smaller diameter beams will be possible. If so this method may lead to the study of the 3-d structure of medically important crystals that refuse to grow to a size big enough to handle by more standard methods.

With materials science type of samples (silicon and gold single crystals), we have been able to obtain Laue patterns in 300 seconds of exposure for beam sizes 0.3 micron diameter or smaller. Well shaped round Laue spots from a (100) silicon wafer showed the divergence of our small beam to be less than 8 mr. We observed Laue streaks from a somewhat stressed or slightly disordered 500 Angstrom thick gold crystal foil. The volume illuminated was 0.3 micron diameter by 0.05 microns, or $3.5 \times 10^{-3}\ \mu^3$. The Laue spots were radially streaked, consistent with a mosaic spread of several degrees.

These and other experiments that are underway with this microbeam technology will be discussed.

MS-14.01.04 PERFECT-CRYSTAL X-RAY-OPTICS FOR PRODUCING AND CHARACTERIZING CIRCULAR POLARIZATION WITH SYNCHROTRON RADIATION. By T. Ishikawa*, K. Hirano and S.Kikuta, Department of Applied Physics, University of Tokyo, Japan.

Dynamic x-ray diffraction leads to different dispersion relations between σ and π states which is well known as *diffractive birefringence*. Perfect crystal x-ray phase retarders based on this effect are, in principle, attainable for all conceivable diffraction geometries, i.e. reflection and transmission in both Bragg and Laue geometries. Properties of the phase retarders in each geometry are discussed.

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Transmission phase retarders of Bragg and non-absorptive Laue geometries have a remarkable characteristics that (i) phase retardation has opposite sign on the opposite tails of the Bragg peak, so that we can easily switch the photon helicity by rotation around a single axis, (ii) phase retardation depends on the deviation from the exact Bragg condition, which is slowly varying on the far tails of the Bragg peak. These directly lead to realization of energy-tunable production of circularly polarized x-rays and fast switching of photon helicities. Both have been shown experimentally by using Bragg-transmission phase-retarder. In addition to the production of circularly polarized x-rays, phase retarders can be used for the complete determination of the unknown polarization state. By this, polarization state including naturally polarized component were determined for x-rays from elliptical multipole wiggler installed on NE1 beamline of the Accumulation Ring of TRISTAN. Transmission phase retarders using Borrmann effect was also experimentally characterized. Helicity switching capability utilizing crystal symmetry was demonstrated. Several beamline optics for production of circularly polarized x-rays from linearly polarized synchrotron radiation are discussed. Double-crystal monochromator with inclined scattering plane followed by a phase retarder, which make (+,-,+) parallel setting as a whole, is one of the practical solution for the design of the beamline for high flux as well as well-defined circular polarization. Another practical solution is to use transmission phase retarders with inclined scattering plane in a conventional monochromatic beamline equipped with a usual vertically dispersed double-crystal monochromator under certain conditions.

MS-14.01.05 CHARACTERIZATION OF YB_{66} CRYSTALS FOR USE AS A NEW SOFT X-RAY MONOCHROMATOR WITH SYNCHROTRON RADIATION. By Z.U. Rek(1)*, M. Rowen(1), Joe Wong(2), T. Tanaka(3), 1) Stanford Synchrotron Radiation Laboratory, SLAC, Stanford, CA 94309, USA, 2) Lawrence Livermore National Laboratory, Livermore, CA 94551, 3) National Institute for Inorganic Materials, Tsukuba, Ibaraki, 305 Japan.

YB_{66}, a complex binary semiconducting compound with a cubic crystal structure and lattice parameter of 23.44Å , was selected for monochromatization of soft X-rays in the 1-2 KeV energy range (Wong, Shimkaveg, Eckart, Tanaka, Rek,Tompkins, Nucl. Instr. and Meth.,1990, A241,243). The crystals were grown by an indirect heating floating zone method (Tanaka, Otani, Ishizawa, J. Crystal Growth, 1985, 73,31). Characterization of crystal grain structure and growth-induced defects as a function of crystal growth parameters was performed with white beam X-ray topography and rocking curve measurements. By numerous modifications of the growth parameters, crystals perfect enough were grown to be used as a double crystal monochromator to measure high resolution XAFS spectra of Mg, Al and Si in a number of compounds and minerals. The measurements were performed on the JUMBO beam line at SSRL. The results demonstrate the excellent performance of YB_{66} crystals as a new type of crystal monochromator for this energy range. The effects of crystal heating by synchrotron radiation were observed and studied. Recent experiments in crystal growth of YB_{66}, results of crystal characterization and performance of this material as an actual monochromator will be discussed.

PS-14.01.06 GIAR-FILMS FOR NUCLEAR RESONANT FILTERING OF SYNCHROTRON RADIATION. By K. Kaneko, Y.Kashiwase*, I.Nishida, M.Kogiso, M.Mori, M.Minoura, X.W.Zhang+ and T.Kado++, Department of Physics, College of General Education, Nagoya Univ. Chikusa, Nagoya 464-01, +Nat. Lab. for High Energy Physics (KEK), Oho, Tsukuba,Ibaraki 305,++Gov. Indus. Res. Inst., Chugoku, Kure, Hiroshima 737-01, Japan.

For the purpose of producing filters for monochromatization of synchrotron radiation to band width 10^{-7}-10^{-8}eV, GIAR (Grazing Incidence Antireflection)-films are designed to suppress enormous background scattering by electrons in the angular region where nuclear resonant reflection amplitude is large near the critical angle of the total reflection. The purpose of this study is to prepare and develop GIAR-films by investigation of the structure as well as X-ray interference between reflections from the films. An about 250-Å-thick Fe film coated with about 70-Å-thick Al film was deposited on a flat surface of a quartz glass plate by evaporation. The reflectivity curve of the specimen was measured by means of the optical system installed on the BL-14B of the Photon Factory (KEK). The radiation of 0.86025 Å was obtained using finally the 10 6 4 double reflections from a channel-cut Si crystal. It is made clear by the experiment that a GIAR-film with low reflectivity minima about $4x10^{-4}$ around glancing angle 5 mrad can be produced. Good simulation of the curve fitted to an observed interference pattern is obtained by assuming a three-layer model with boundary roughnesses 10-20 Å between layers.

PS-14.01.07 COHERENT INTERACTION OF MULTIPLE-BEAM DIFFRACTION IN A CRYSTAL WITH CIRCULARLY POLARIZED X-RAYS. By Qun Shen* and K.D. Finkelstein, CHESS, Cornell University, Ithaca, New York, 14853, U.S.A.

A novel x-ray diffraction technique has been developed over the past few years here at CHESS. The idea is to combine circularly polarized x-rays with coherent multiple-beam diffraction (the Renninger effect) in a crystal. There are two main effects that can occur in a multi-beam diffraction process. One is the phase sensitive interference among the multiple Bragg excitations and the other is polarization state mixing which results from the double Thomson scattering mechanism. The combination of these effects can produce an interference intensity that involves both the phases of the structure factors and the phase difference between the **s** and **p** components of the incident beam polarization.
This research has resulted in two main areas of applications. The first is to determine noncentrosymmetric phases or to detect noncentrosymmetry in a crystal using elliptically or circularly polarized x-rays [Shen & Finkelstein, Phys. Rev. Lett. 65, 3337 (1990); Shen, SPIE Proceedings 1550, 27 (1991)]. An experiment on a GaAs crystal has demonstrated that determination of the polarity or the chirality of a noncentrosymmetric crystal is possible by using both right- and left-handed elliptically polarized synchrotron radiation. The second application is to use the multi-beam diffraction from a crystal of known structure to measure the degree of circular polarization of an x-ray beam [Shen & Finkelstein, Phys. Rev. B45, 5057 (1992)]. This unique way of characterizing circular polarization at low-to-medium x-ray energies has created a great deal of interest in the x-ray and synchrotron communities. Because the multi-beam interference intensity depends on all three Stokes-Poincare parameters, a complete characterization of the incident beam polarization is possible. The relatively high throughput of such analyzer may allow us to perform polarization-in/polarization-out measurements in magnetic and/or resonant scattering experiments to obtain a complete analysis of the scattering cross-section.

PS-14.01.08 DELTA-CRYSTAL - A PROGRESS IN X-RAY OPTICS AND A CHALLENGE FOR CRYSTAL GROWERS. By H. Bradaczek, G. Hildebrandt and W. Uebach, Institut für Kristallographie, Freie Universität Berlin, Germany.