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PARABOLIC REFRACTIVE X-RAY LENSES FOR MICROSCOPY AND MICROANALYSIS

M. Kuhlmann [1] C. G. Schroer[1] B. Benner[1] T. F. Guenzler[1] J. Meyer[1] B. Lengeler[1] C. Rau[2] T. Weitkamp[2] A. S. Simionovici[2] A. Snigrev[2] I. Snigreva[2]
[1]II. Physikalisches Institut, Aachen University of Technolgy (RWTH), D-52056 Aachen, Germany [2]ESRF, B. P. 220, F-38043 Grenoble, Cedex, France

The development of parabolic refractive x-ray lenses has opened new possibilities for hard x-ray microscopy and microanalysis at synchrotron radiation sources. Using aluminium, we have fabricated parabolic refractive x-ray lenses of high quality that are used for distortion free, magnifying imaging with sub-micrometer resolution. Combined with tomographic techniques, this allows one to reconstruct the three-dimensional inner structure of a sample at sub-micrometer resolution with minimal sample preparation. The lenses can be used to image the synchrotron radiation source onto a sample in a reducing geometry, generating an intensive hard x-ray microbeam at the sample position. The microbeam allows one to perform hard x-ray analytical techniques with a spatial resolution in the micrometer and sub-micrometer range, such as fluorescence and absorption spectroscopy, diffraction, or small angle scattering. Recently, we have succeeded to make parabolic refractive x-ray lenses of beryllium. These lenses are more than one order of magnitude more transparent for hard x-rays than aluminium lenses. In addition, they have a larger aperture that can lead to a higher resolution (down to 50nm) and a larger field of view (about 1mm) in imaging experiments. The beryllium lenses have been characterized in first experiments. Their optical performance is compared to that of the aluminium lenses.

Keywords: X-RAY LENSES X-RAY MICROSCOPY X-RAY MICROANALYSIS

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QUANTITATIVE X-RAY PROJECTION ULTRAMICROSCOPY USING A SCANNING ELECTRON MICROSCOPE

S.W Wilkins[1] S.C Mayo[1] P.R. Miller[1] T.J. Davis[1] T.E. T.E. Gureyev[1] D. Paganin[1,2] A. Pogany[1] A.W. Stevenson[1] D. Gao[1] D.J. Parry[1]
[1]CSIRO, Manufacturing Science & Technology, PB33 Clayton Sth MDC, Vic 3169, Australia [2]School of Physics & Materials Engineering, Monash University, Clayton, Vic 3169, Australia

We outline a new approach to X-ray projection microscopy using a scanning electron microscope (SEM) as host that exploits phase contrast to boost the quality and information content of images. These developments have been made possible by the combination of a high-brightness field-emission-gun (FEG) based SEM, direct detection CCD technology and new phase retrieval algorithms. Using this approach we have been able to obtain spatial resolution of better than 0.2 mm and have demonstrated novel features such as: i) phase-contrast enhanced visibility of significant image features (e.g. edges and boundaries), ii) energy-resolved imaging to simultaneously produce multiple quasi-monochromatic images using broad-band polychromatic illumination, iii) implementation of micro-tomography, iv) rapid and robust phase/amplitude-retrieval algorithms to enable new quantitative modes of microscopic imaging. Widespread applications are envisaged to fields such as materials science, biological and biomedical research and microelectronics device inspection. Some illustrative examples will be presented. The quantitative methods described here are also very relevant to projection microscopy using other sources of radiation such as visible light and electrons.
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Keywords: X-RAY MICROSCOPY, PHASE CONTRAST, MICROTOMOGRAPHY

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SOFTWARE FOR NEUTRON SINGLE CRYSTAL DIFFRACTOMETER TriCS

O. Zaharko M. Koennecke J. Schefer
Laboratory for Neutron Scattering ETH Zurich and Paul Scherrer Institutre VILLIGEN PSI 5232 SWITZERLAND

The thermal neutron single crystal 4-circle diffractometer TriCS at SINQ is designed for investigations of chemical, commensurate and incommensurate magnetic structures as well as phase transitions driven by temperature, magnetic field or pressure. The instrument is equipped with three He^3-filled area detectors and an Eulerian cradle. It can operate in the equatorial and inclined geometries similar to D19 at ILL [1]. Software for TriCS includes new data collection and data analysis routines. The algorithm for the data collection aims to record simultaneously a maximal number of reflections in each omega scan at preset χ and φ angles. The calculation of the reflection positions is based on the matrix approach [2] extended to the inclined geometry. Data analysis is performed in two steps. Firstly a local maximum search is performed through a measured data set. Suitable strong and not overlapped reflections are analysed without any assumption about the crystal lattice with the dynamic mask procedure [3] and stored as templates in a library for later reference. The position of these reflections can be used for indexing or UB matrix refinement. Then the integration of intensity at positions read from a list is performed by matching a template constructed from nearby strong library reflections. The routine also supports non-integer indices.
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NEUTRON DIFFRACTION STUDY OF THE CRYSTALLINE-STATE PHOTORACEMIZATION OF A BULKY ALKYL GROUP IN A COBALOXIME COMPLEX

T. Ohhara[1] S. Ikeda[1] I. Tanaka[2] N. Niimura[2] Y. Ohashi[3]
High Energy Accelerator Research Organization (KEK) Institute of Materials Structure Science Oho 1-1 TSUKUBA IBARAKI 305-1018 JAPAN
[1]Institute of Materials Structure Science, KEK [2]Advanced Science Research Center, JAERI [3]Dept. of Chemistry and Materials Science, Tokyo Institute of Technology

The chiral bulky alkyl group in some bis(ethoxycarbonyl)ethyl cobaloxime complexes are known to be racemized by exposure to visible light with retention of single crystal form. The mechanism of this reaction has been interested because the structural change of such a bulky alkyl group in crystal lattice is thought to be a model of enzymatic or catalytic reactions which also proceed in restrained environments. In this work, we prepared a single crystal of [(R)-1,2-bis(ethoxycarbonyl)ethyl-d1(α)]-(pyridine-d5)cobaloxime-d14 complex and carried out a single crystal neutron diffraction measurement after 3 days exposure to red light in order to elucidate the mechanism of the photoracemization. The single crystal neutron diffraction measurement was carried out with BIX-3 diffractometer equipped with a neutron imaging plate. The crystal size was 2.0x1.5x0.7mm and 2700 unique reflections were collected by 6 days measurement. The neutron diffraction data have been analyzed up to 0.86 Å resolution. The final R factor was 12.73%. The result showed that the 30% of the chiral bulky alkyl group was inverted to the opposite configuration and the deuterium atom bonded to the chiral carbon atom of the alkyl group was also bonded to the chiral carbon after inversion. This result indicated that the crystalline-state photoracemization of the bulky alkyl group proceeds with Hula-twist motion of the alkyl radical produced by exposure to visible light.

Keywords: SINGLE CRYSTAL NEUTRON DIFFRACTION CRYSTALLINE STATE REACTION COBALOXIME