PS15.01.08 EXPERIMENTAL WAYS OF STUDY OF ATOMIC MOTION IN OXIDES OF TRANSITION METALS. D. A. Kovtun*, A. P. Kovtun**, V. P. Krasnolutskiy*, M. F. Kupriyanov**, A. P. Naumov*, *Institute of Physics, Rostov State University, 194 Stachki St., 344090, Rostov-on-Don, Russia, **Department of Physics, Rostov State University, 5 Zorge St., 344104, Rostov-on-Don, Russia

The influence of different atomic space distribution on X-ray diffraction pattern is shown. According to Guinier's concept [1] and some arguments briefly given in the work it is assumed that chemical state in oxide of transition metals is structurally and energetically degenerate and can be presented as a quantum superposition of a set of mutually orthogonal elementary "dipole" states localized inside first coordinate sphere of metal atom. In each such state the metal atom is shifted with respect to the center of octahedron (approx. 0.05-0.15 Å). Due to small sizes of the so called "white" spot the radiation sources must emit monochromatic X-rays with the short wavelengths ($E_{ph} > 100$ keV).

We have analyzed two suitable radiation sources:

- the radiation of isotopes
- synchrotron radiation

The problems and ways of elastic photon registration are also discussed taking into consideration secondary radiation.

1. Comes R., Lambert M., Guinier A., Commun. Solid St. Phys., Vol. 6, p. 715 (1968)

PS15.01.09 STUDIES OF PROCESSES OF DIFFRACTION OF HIGH-MONOCHROMATIC GAMMA-RADIATION IN REAL SINGLE CRYSTALS. Alexander Kurbakov, Alexei Sokolov, Department of Condensed Matter, Petersburg Nuclear Physics Institute, 188350, Gatchina, Russia

Diffraction of high-monochromatic short-wavelength (0.003nm) gamma- radiation was used to experimental study of diffraction process in real single crystals.

To check-up the modern theoretical elaborations in the field of creation of the general theory of diffraction , in additional to recent gamma-ray and X- ray experimental tests of Kato's statistical dynamical theory by measuring the period of the intensity beats and values of integral intensities as a function of the sample thickness or wavelength that can be applied only to highly perfect crystals, we carried out experiments on both quite perfect crystals and crystals with relatively high distortions of the crystal lattice formed during crystal growth and/or different technological operations. It was used dislocationless Si and Ge single crystals, Si undergone special purposeful influences to their crystal structure and Ge after plastic deformation. Also we investigated single crystals of quartz, which as Si has rather high degree of structure perfection, but principally different, from point of influence to diffraction process, low-dislocation defect structure. Mainly it was used the possibility of measuring the absolute values of integral reflecting power in the condition of a high angular resolution for several orders of reflection (hkl).

So we had possibilities to realise the conditions for changing the values of static Debye-Waller factor E in all range from 1 to 0 and measuring the dependencies of E upon reflection vector for various types of lattice defects.

A detailed discussion of the applicability of Kato's statistical dynamical theory (with account of last theoretical elaborations) and other modern theories to shortwavelength gamma-ray diffraction experimental data are given.

PS15.01.10 STUDY OF LOSS IN ABSORPTION OF X-RAYS IN THIN NATURAL DIAMOND CRYSTALS AROUND LAUE DIFFRACTION MAXIMA. Krishan Lal, S. Niranjana, N. Goswami, A. R. Verma, National Physical Laboratory, New Delhi-110012, India

Significant loss in absorption of X-rays in thin diamond crystals,with $\mu t << 1$, and having varying degrees of crystalline perfection has been observed at and near Laue diffraction maxima by using a high resolution X-ray diffraction technique. The specimen crystals were {111} platelets. A Five Crystal X-ray Diffractometer was used in three crystal configuration with Mo $K\alpha_1$ exploring X-ray beam. The specimens were oriented in Laue geometry for diffraction from ($\bar{2}20$), ($\bar{4}40$), ($\bar{1}11$), ($\bar{1}\bar{1}3$) and ($\bar{2}\bar{2}4$). Three beams are observed when the crystal is oriented for diffraction [e.g. Lal et al, Solid State Commun. **96** 33 (1995)]. The X-ray intensity measurements were made by using a scintillation counter coupled to a timer counter. It was mounted on a track and positioned to alternatively receive forward diffracted beam, plus the residual direct beam and the diffracted beam. Total transmitted intensity was obtained by adding intensities of all the three beams. Plots of total transmitted intensity as a function of glancing angle showed peaks at the diffraction peak positions. This enhancement in the transmitted intensity is due to a loss in the absorption of the exploring beam when diffraction takes place. This is identical to the Borrmann Effect. However, in the present case the crystals were thin with $\mu t \sim 0.3$ only. Also, their degree of perfection could be rather low. In a typical crystal, with $\mu t = 0.29$, the value of μ dropped from the usual 0.219 mm^{-1}, to 0.075 mm^{-1} [($\bar{1}11$)]; 0.136 mm^{-1} [($\bar{2}20$)]; 0.117 mm^{-1}[($\bar{1}\bar{1}3$)]; 0.16 mm^{-1} [($\bar{2}\bar{2}4$)] and 0.198 mm^{-1} [($\bar{4}40$)]. The half width of its diffraction curve being 100 arc sec[for ($\bar{2}20$)], which is 87 times the theoretical half width for a perfect diamond crystal. Nearly perfect diamond crystals give a half width of ≤ 10 arc sec. So far as known to us this is the first time that such a loss in absorption at diffraction peaks has been reported for such thin and not so perfect crystals. These results indicate a coupling between the diffracted and the forward diffracted beams.

PS15.01.11 LABORATORY-BASED DIFFRACTION SIGNAL ENHANCEMENT BY THE USE OF POLYCAPILLARY X-RAY OPTICS. C. A. MacDonald, W. M. Gibson, Center for X-Ray Optics, University at Albany, Albany, NY 12222

Polycapillary x-ray optics are shaped arrays consisting of hundreds of thousands of hollow glass capillary tubes. X rays are conducted along the tubes by total external reflection at glancing incidence. Gently curved arrays can be used to redirect, collimate or focus the x-ray beams. The primary benefit provided by the use of polycapillary optics with conventional laboratory-based diffraction sources is the ability to transform x rays emitted over a large angular range into a beam with a much smaller angular divergence. The output angular divergence may be to some extent controlled by the specific system design, but is near the critical angle for total external reflection, about 4 mrad at 8 keV.

The optics can also provide significant benefit apart from collimation, by reducing background, suppressing high energy photons, and providing more convenient alignment geometries. The high energy photon suppression results from the inverse dependence of the critical angle on photon energy. Significant suppression of the $K\beta$ peak relative to the $K\alpha$ peak has been demonstrated for Cu radiation.

Insertion of capillary optics into existing diffraction systems does not provide maximum benefit compared to re-engineered system. However, preliminary measurements performed in these conditions show significant signal gains. Large area thin film diffraction signals were shown to increase by a factor of 3 to 8 when a collimating optic with a linear capture angle of 0.15 rad, 12.5μm channel size, and output area of 3.1 cm was placed into a standard Bragg-Bretano diffractometer. Capillary optics provide even larger signal gains for very small samples. More than an order of magnitude signal increase was achieved for a 0.3 mm Lysosyme crystal by employing an optic with a 0.1 rad capture angle and 5 mm output diameter. This paper will present a review of the broad range of applications of capillary optics to diffraction systems.

PS15.01.12 ON THE X-RAY DIFFRACTION BY PERFECT ABSORBING CRYSTALS.

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Previous measurements of the (111) intensities diffracted by a perfect Ge crystal in the Bragg case, at frequencies of the incident radiation close to the K absorption edge, were considerably higher than those calculated by the dynamical theory of X-ray diffraction[1]. The theory was modified so that the Kramers-Kronig dispersion relations be satisfied for each value of the glancing angle of the incident beam. In this way the photoelectric absorption contribution f'_{dyn} to the real part of the form factor depends on the glancing angle as the imaginary part does. f'_{dyn} is equal to the product of the intensity of the internal wavefield at the absorbing K-electrons by the contribution f'_{at} predicted by the anomalous dispersion theory of the individual atom (for simplicity the effects of the crystal field on the matrix elements of the absorption transition are neglected). The corresponding Darwin-Prins curves are higher than those foreseen by the present form of the dynamical theory of absorbing crystals and the integrated intensities are 20-30% greater. For example the relative calculated integrated intensities of the (111) Bragg reflection by a thick Ge crystal for a frequency of the incident beam 7.64 eV higher than the frequency of the absorption edge are about 1.58, 1.44 and 1.12 by taking the absorption contributions to the real part of the form factor equal to 0, f'_{dyn} and f'_{at} respectively. The dynamical theory in its present form is a good approximation if the absorption contribution to the real part of the form factor is much smaller than its basic part. It is proposed that this theory be modified to take into proper account the dispersion relations. An important conclusion is that $f'_{dyn} \approx 0$ (the effect of the anomalous dispersion is wiped out) in that part of the interference region where the absorption is small. Since the internal wavefield depends on the absorption contribution to the real part of the form factor, a consistent value of this contribution can be obtained either by a numerical solution of the equation of f'_{dyn} or by an iteration procedure(applicable for incident frequencies a few eV away from the absorption edge) of the same equation. A comparison with the above mentioned experimental results is satisfactory. The proposed modified theory can be readily extended to the Laue case, to different absorption phenomena and to the diffraction of other types of radiation by perfect crystals.

1) A. E. Merlini, Il Nuovo Cimento **15 D** (1993) 169.

PS15.01.13 OBSERVATION OF PHASE CHANGE OF X-RAY POLARIZABILITY BY THE ROCKING CURVES IN THE BRAGG CASE.

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Rocking curves of the transmitted beam for GaAs 600 diffraction in the symmetric Bragg case were measured just below the K-absorption edge of Ga by using X-rays from synchrotron radiation. When X-ray energy is 9eV below Ga K-absorption edge, the rocking curve shows the asymmetry: the intensity in the lower angle region of the exact Bragg angle is larger than that in the higher angle region. The reversed asymmetry is observed in the rocking curve at 3eV below Ga K-edge, i.e. intensity in the higher angle region is larger than that in the lower angle region. The critical energy of this reversal is about 6eV below Ga K-edge.

Based on a dynamical theory of X-ray diffraction with absorption effect taken into account[1)], the diffracted and the transmitted intensities were calculated. By comparing the measured rocking curves with the calculated ones, it is elucidated that the asymmetry of the transmitted rocking curves described above depends on the value of the phase difference δ, which is the difference of phase factors of the Fourier transforms of the real and the imaginary components of the X-ray polarizability. The phase difference δ is a function of crystal structure as well as anomalous scattering factor. Similar asymmetric rocking curves in the Laue case have been reported by Fukamachi et al.[2)]. It is pointed out that this change of the asymmetry is potentially useful in the phase determination of the structure factors.

1) T. Fukamachi & T. Kawamura, Acta Cryst.(1993), A49,384.
2) T. Fukamachi, R. Negishi, S. M. Zhou, M. Yoshizawa, T. Sakamaki, T.Kawamura, T. Nakajima, Acta Cryst.(1996), in press.

PS15.01.14 POLYCAPILLARY X-RAY OPTICS FOR MACROMOLECULAR CRYSTALLOGRAPHY.

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Polycapillary x-ray optics have shown great potential in macromolecular x-ray crystallography. Incorporation of polycapillary x-ray optics into existing x-ray sources yields significant increases in beam intensity compared to simple collimation. The optic used in this work collects x-rays from the source over a large solid angle (6° capture angle) and redirects them into a quasi-parallel beam (<0.2° divergence) of 5 mm diameter. Using this optic coupled to a modified RU200 rotating anode source, we recently produced a gain in flux through a 0.3 mm collimator of more than an order of magnitude over a comparable graphite monochromated source. Using large, microgravity grown lysozyme crystals as a "standard", we collected full data sets with and without Ni filtering of the primary beam (the high energy rejection of the optics allows measurements to be done without further filtering). Without filtering, a complete data set, to a minimum of 1.54 Å resolution, was obtained within 13.4 hours with a final Rmerg of 7.29 % (based on all observations). The data were collected with a Siemens Multiwire detector, read by FRAMBO and analyzed using SAINT. We have made similar measurements with a prototype optic which produces a weakly convergent beam (< 1° convergence), and further increases the flux gain to more than 100. Full data sets using this optic have also been collected. Details of the data reduction and analyses, as well as applications to structure determinations, will be presented.

PS15.01.15 X-RAY HOLOGRAPHY WITH ATOMIC RESOLUTION

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One of the basic problems in crystallography is that in the conventional diffraction experiments only the intensity of the scattered radiation is recorded, its phase is lost. In holography [1], the scattered radiation is mixed with a reference wave and the resulting interference pattern is recorded. The hologram contains