

PS-13.02.08 DETERMINATION OF THE VOLUME RATIO OF DOMAINS NEAR THE STRUCTURAL PHASE-TRANSITION TEMPERATURE.

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After passing through a cubic-to-tetragonal phase transition, a crystal generally consists of three kind of domains which are specified by the direction of the *c* axis. A single reciprocal-lattice point in the cubic phase splits into a set of points in the tetragonal phase with domains; the domain volume ratio can be determined from the relative intensities of the split points. Near the transition of second order, the splitting is so small that we need a high-resolution diffraction technique. To the high-angle double-crystal X-ray diffractometry (HADOX) of a 2 θ -resolved version, a four-circle goniometer has been introduced in order to align the specimen crystal in any orientation; by this, three-dimensional intensity distribution in the reciprocal space can be measured with high resolution. In the previous HADOX experiments of the original version, anomalous temperature dependence was observed near the 105 K transition in SrTiO₃; the 004 peak of the tetragonal phase gradually disappeared in a temperature range of 10 K below the transition. For this phenomenon, there were two possible explanations: a change in domain orientation and two-phase coexistence. In the present experiment, the 400, 040 and 004 positions in the cubic phase were examined; the intensity distribution was measured as a function of temperature. It is found that the total intensity at the three positions is constant for both 400 and 004 peaks, although the intensity of individual 400 and 004 peaks varies. This means that the previous observation was due to a change in domain orientation.

PS-13.02.09 IMAGING-PLATE X-RAY TOPOGRAPHY ANALYSIS SYSTEM FOR CHARACTERIZING GROWTH STRIATIONS IN SILICON CRYSTALS.

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Using synchrotron plane wave x-ray topography, local lattice distortion due to growth striations in Si crystals was quantitatively determined at a sensitivity of 10⁻⁸ (Kawado, Kojima, Maekawa & Ishikawa, 1991). Since x-ray topographs were taken on photographic plates, with local variations in the diffracted x-ray intensity being indirectly obtained by microphotometer measurements of variations in the plate's optical density, x-ray exposure and development processing had to be carefully controlled to obtain linearity between the optical density and sample rotation angle. To overcome this disadvantage, an imaging-plate (IP) x-ray topography analysis system has been developed and applied to a quantitative analysis of local lattice distortion due to oxygen striations in magnetic Czochralski (MCZ) Si crystals.

The experimental arrangement for plane wave x-ray topography was set up on beam-line 15C at the Photon Factory. Using the double-crystal monochromator and the asymmetric-reflection collimator, a large-size (50 × 50 mm) incident x-ray beam ($\lambda = 0.1126$ nm) was realized with an angular divergence of ~0.1 arcsec and a wavelength spread of ~3 × 10⁻³. The sample crystal, which was a 10 mm-thick undoped MCZ Si plate with [100] surface orientation perpendicular to the growth axis [001], was aligned to give a symmetric 800 reflection in the Bragg geometry; thereby constructing a non-dispersive (+n, -n) setting between the collimator and the sample crystal.

A series of x-ray topographs were taken at 0.05-arcsec-intervals at the slope of the rocking curve's low-angle side in two sample configurations where the sample crystal was rotated 180° around the surface normal. High-resolution type IP's (Fuji DL UR-III) were used to record the x-ray topographs. A PIXsysTEM (Oikawa, Mori, Takano & Ohnishi, 1990) was used in its high-resolution mode as an IP reader, with signals being logarithmically amplified, digitized into

12-bit data, and then transferred to a data processing system that was constructed on a 32-bit UNIX engineering work station (Sony NEWS). Calculations for lattice distortion measurements were performed using the method proposed by Kikuta, Kohra & Sugita (1966) to separate the lattice spacing variation $\Delta d/d$ from the orientation variation $\Delta\alpha$. The resultant one-dimensional profiles and two-dimensional images of $\Delta d/d$ and $\Delta\alpha$ variations were printed out by a laser beam printer and with a grey scale by a full-color printer, respectively. Examples of two-dimensional images are shown in Fig. 1, where enhanced contrast gives a increase in variation. This newly developed analysis system, having excellent features such as high speed data process, easy operation and availability of large amount of data, has offered more quantitatively reliable characterization of growth striations in as-grown Si Crystals.

References

- Kawado, S., Kojima, S., Maekawa, I & Ishikawa, T. (1991). Defects in Silicon II, Edited by Bullis, W.M., Gösele, U. & Shimura, F., p. 65. Pennington: Electrochemical Society.
Kikuta, S., Kohra, K. & Sugita, Y. (1966). Jpn. J. Appl. Phys. 5, 1047.
Oikawa, T., Mori, N., Takano, N. & Ohnishi, M. (1990). J. Electron Microsc. 39, 437.

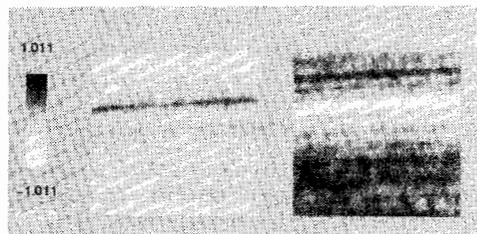


Fig. 1. Two-dimensional images showing $\Delta d/d$ and $\Delta\alpha$ variations in an MCZ Si crystal.

PS-13.02.10 THE STUDY OF DIFFRACTION CHANGES OF KTP UNDER A DC FIELD WITH SYNCHROTRON RADIATION WHITE BEAM TOPOGRAPHY.

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Single crystal KTiOPO₄ (KTP) is an important nonlinear optical material (F. C. Zumstay, J. D. Bierlein, T. E. Gier, J. Appl. Phys., 1976, 47, 4980). It exhibits a quasi one dimensional Potassium ion conductivity along its crystallographic *c*-axis (J. D. Bierlein, C. B. Arweiler, Appl. Phys. Lett., 1986, 49, 917). In recent years, Yang et al. (H. G. Yang et al., Phys. Rev. B, 1988, 37, 1161) and Yang (Yang Zhen, Chinese Phys. Lett., 1987, 4, 533) found that when DC voltage is applied along the *c*-axis of KTP crystals, it produces the same phenomena as these in α -LiIO₃ single crystals. The last investigation of these phenomena shows that these can be explained by a grating theory (Y. Y. Li, Solid Stat. Ionics, 1988, 31, 99) and classical extinction theory (Q. Li, Y. Zhen, Z. Krist., 1988, 183, 265) respectively, but there are still some controversy especially about the strong anisotropy of the neutron diffraction intensity enhancement effect; On the other hand, the mechanism of the neutron diffraction in crystals is quite similar to that of X-rays. Thus, a Synchrotron Radiation (SR) White Beam Topography investigation of the KTP crystal becomes necessary to see if the diffraction of X-rays is also changed.

The specimen, a good KTP single crystal grown from flux melt, was cut into a rectangular slice with one pair of surfaces parallel to the crystallographic plane *a*, and the other two pairs of surfaces parallel to the planes *b* and *c*, respectively. After grinding and chemical polishing, the specimen linear dimensions are 0.5 × 15.0 × 3.7 mm³ along the *a*, *b*

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and c axes. The SR White Beam Topography experiment was carried out on 4W1A Beamline of National Synchrotron Radiation Laboratory of BEPC. The direction of incident X-ray was perpendicular to the slice surface and parallel to the a-axis. In order to obtain a dynamic information, a transmitted White Beam Topography was taken every 30 second. A $V_+ = 70$ Volts was applied to the C surfaces which were covered with silver paste for good electrical contact, this voltage corresponds to an average field strength $E_+ = 190$ V/cm.

From these topographs, one can see that :

1. Many dark straight lines parallel to the c-axis was found, these lines randomly distributed over all slice surface.
2. The longer the time of application of DC field, the darker and wider the lines.
3. When DC field was removed, these dark lines became light and narrow gradually until they disappeared.

From all the above results, it follows that under the application of a DC field, the crystal lattices of KTP was distorted because of the space charge accumulation in KTP.

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PS-13.02.11 CZOCHRALSKI GROWTH AND X-RAY TOPOGRAPHIC INVESTIGATION OF SALOL SINGLE CRYSTALS. By H. Klapper and G. Neuroth, Mineralogisches Institut, Universität Bonn, W-5300 Bonn, Fed. Rep. Germany.

Salol, $C_{13}H_{10}O_3$, crystallizes in the orthorhombic space group $Pcab$ with lattice parameters $a = 11.258$ Å, $b = 23.402$ Å, $c = 7.961$ Å. The melting temperature is 42°C . Until now large single crystals have been grown from solutions and from supercooled melts. These crystals exhibit a high growth anisotropy: They develop a plate-like habit with dominating $\{010\}$ pinacoid.

In the present study crystals were grown by the Czochralski method by pulling along various crystallographic and non-crystallographic directions. The aim was to study the growth behaviour and the typical arrangements of grown-in dislocations for different growth directions. The salol melt was held at about 0.5°C above the crystallization temperature (at about 42.5°C). Due to the low thermal conductivity of salol and low heat radiation, an additional cooling of the growing crystal by the air circulating in the growth chamber was necessary. By decreasing the temperature of the cooling air from about 40 to about 35°C , the diameter of the crystal could be increased from 5 mm (seed crystal) to about 30 mm. Typical pulling and rotating rates were 0.6 mm/h and 8 rev./min. Crystals of up to 100 mm length and 30 mm diameter and of excellent optical perfection were obtained.

The crystals were cut into slices (thickness ca. 1 mm) parallel to the pulling direction. Thus the slices contain a part of the seed crystal, the region of first growth on the seed, the crystal cone and the grown crystal until the end of growth. This allows to follow - within one specimen - the development of grown-in defects from the start to the end of the growth.

The growth defects were imaged by using the X-ray topographic technique of A.R. LANG (CuK α radiation). They are mainly grown-in dislocations which originate from inclusions (gas bubbles) formed in the zone of first growth, in particular at the edges of the seed crystal. Other sources of dislocations are steps in the shoulder (cone) of the crystal boule. Such steps frequently appear when the diameter increase is too fast. The grown-in dislocation lines take a course roughly normal to the (local) growth front. Since this interface is (due to the air cooling mentioned above) concave against the melt, the dislocation lines do not grow out through the side faces, but are "focussed" towards the axis of the crystal boule. This leads to an increase of the dislocation density along the axis of the boule. Nearly dislocation-free crystals can be obtained by careful control of the seeding-in procedure and the diameter increase.

In a few cases reactions of crossing dislocation lines have been observed. Dislocation lines with opposite Burgers vectors annihilate in the crossing region. Dislocations with different Burgers vectors b (e.g. $b = [100]$ and $[101]$) form two nodes connected by a new dislocation line segment ($b = [00\bar{1}]$) of lower energy per unit length. The sum of Burgers vectors of the dislocations entering the node is zero (theorem of F. C. Frank, Bristol).

PS-13.02.12 X-RAY STRUCTURE DIAGNOSIS OF SEMICONDUCTOR MQW AND SUPERLATTICES. BY Z.H.Mai*, C.F. Cui, J.H.Li and J.T.Ouyang, Institute of Physics, Chinese Academy of Sciences, Beijing 100080, China.

Semiconductor multi-quantum well (MQW) and superlattice (SL) systems are important materials for novel device applications. Recent studies have shown that the structural parameters and the perfection of the material systems are the key factors to improve the physical properties of devices. $Al_xGa_{1-x}As/GaAs$ MQW, Ge_xSi_{1-x}/Si and $In_xGa_{1-x}As/GaAs$ superlattices grown by MBE method were systematically investigated by x-ray double-crystal diffraction, x-ray grazing incidence diffraction and x-ray topographic methods.

Both coherent and incoherent interfaces between the two components of the Ge_xSi_{1-x}/Si superlattices were observed. The experimental rocking curves of one sample having 15 periods shows that in addition to the substrate peak there is a family of periodic SL reflections due to the presence of a periodic strain in the epitaxial structure. Moreover, each satellite was accompanied by a set of interference fringes (Fig.1). By fitting computer-simulated double-crystal x-ray diffraction rocking curves to the experimental data, it is determined that there exist twice abrupt variations in both the component thicknesses ratio t_1/t_2 (t_1 and t_2 are the thickness of the Ge_xSi_{1-x} and the Si layers, respectively) and the fraction x , being analogous to ABA structure (Table 1).

The structural parameters of 15 periods $In_{0.18}Ga_{0.82}As/GaAs$ strained layer superlattice were also determined by x-ray double-

Table1. Simulated parameters

	N	x	$t_1(\text{Å})$	$t_2(\text{Å})$
A	1-3	0.294	51	64
B	4-11	0.234	47	69
A	12-15	0.294	51	64