PS-13.02.08 Determination of the Volume Ratio of Domains Near the Structural Phase-Transition Temperature. By T. Koga, 2. Lu, Y. Suehiro* and A. Okazaki, Department of Physics, Kyushu University, Fukuoka 812, Japan.

After passing through a cubic-to-tetragonal phase transition, a crystal generally consists of three kinds of domains which are specified by the direction of the c axis. A simple 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 (HADDOX) of a 20-improved 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 HADDOX experiments of the original version, anomalous temperature dependence was observed in the 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 004 and 004 peaks varies. This means that the previous observation was due to a change in domain orientation.


Using synchrotron plane wave x-ray topography, local lattice distortion due to growth striations in Si crystals was quantitatively determined by analysis of X-rays transmitted through the 105 kV transmission electron microscope (Siemens, 1986) with the 400 line of Si[112] orientation. The experimental arrangement for plane wave x-ray topography was set up on beam-line 1SC at the Photon Factory. Using the double-crystal monochromator and the asymmetric-reflection collimator, a large-size (50 × 20 mm) incident x-ray beam (λ = 0.126 nm) was realized with an angular divergence of ±0.1 mrad and a wavelength spread of ±3 × 10⁻⁶. The sample crystal, which is a 10 mm-thick undoped MCZ (Mitsubishi Chemical, 1986) with a surface orientation perpendicular to the growth axis [001], was aligned to give a symmetric reflection in the Bragg geometry, thereby constructing a non-diversive (+n, -m) setting between the collimator and the sample crystal.

A series of x-ray topographs were taken at 0.05 arcsec-intervals on 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 SXIPs (Fujifilm, 1989) were used to record the x-ray topographs. A PIIXsYTEM software (Okawka, Mori, Takano & Ohnishi, 1990) was used in its high-resolution mode as an IT 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, Kojima, and Sugita (1996) to separate the lattice stretching variation 4α/α from the orientation variation 4α. The obtained one-dimensional profiles and two-dimensional images of 4α/α and 4α variations were printed out by a laser beam printer and with a gray scale by a full-color printer, respectively. Examples of two-dimensional images are shown in Fig. 1, where enhanced contrast gives a direct increase in variation.

This newly developed analysis system, having excellent features such as high speed data processing, easy operation, and availability of large amount of data, has offered more quantitatively reliable characterization of growth striations in as-grown Si crystals.

Fig. 1. Two-dimensional images showing 4α/α and 4α variations in an MCZ Si crystal.


Single crystal KTP (KTP) is an important nonlinear optical material (P. C. Zorynta, J. H. Burstein, T. E. Gier, J. Appl. Phys., 1976, 47, 4087). It exhibits a quadratic one-dimensional polarization dependence along its crystallographic c-axis (J. D. Burstein, C. H. Azeichner, Appl. Phys. Lett., 1966, 15, 341). In recent years, Yang et al. (H. G. Yang et al., J. Appl. Phys., 1988, 27, 1161) and Yang, Yang, and Zhen (Chinese Phys. Lett., 1987, 4, 535) found that when DC voltage is applied along the c-axis of KTP crystals, it produces the same phenomena as those in LiNbO3 single crystals. The laser investigation of these phenomena shows that these can be explained by a grating theory (Y. Y. Li, Solid State Ionics, 1998, 100-102) and classical extinction theory (Y. L. Li, Y. Zhen, Z. Kristallogr., 1991, 185, 255), respectively, but there is still some controversy especially about the strong anisotropy of the neutron diffraction intensity enhancement effects. 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) Beam Beam Topography investigation of the KTP crystal becomes necessary to use the diffraction of X-rays as a tool.

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 2.5 × 15.9 × 3.7 mm along the a, b, and c directions.
13-Defects, Microstructures and Textures

The growth defects were imaged by using the X-ray topographic technique of A.R. LANG (CuKα 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 feeding-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]) formed two nodes connected by a new dislocation line segment (b = [001]) 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).


Salol, C₇H₆O₃, 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. 0.1 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.

PS-13.02.12 X-RAY STRUCTURE DIAGNOSIS OF SEMICONDUCTOR MQW AND SUPERLATTICES. By ZH-Mai*, JF. Cui, JH. Li and Z. Tsung. Institute of Physics, Chinese Academy of Sciences, Beijing 100080, China.

Semiconductor multi-quantum well(MQW) and superlattices(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. Al₈₀Ga₂₀As/GaAs MQW, Ga₃₅Se₇₅/Si and In₀₅Ga₄₅As/GaAs superlattices grown by MBE method were systematically investigated by x-ray double-crystal diffraction, x-ray grating incidence diffraction and x-ray topographic methods.

Both coherent and incoherent interfaces between the two components of the Ga₃₅Se₇₅/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-calculated 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 t₁/t₂ (t₁ and t₂ are the thickness of the Ga₃₅Se₇₅ and the Si layers, respectively) and the fraction x, being analogous to ABA structure (Table 1).

The structural parameters of 15 periods In₀₅Ga₄₅As/GaAs strained layer superlattices were also determined by x-ray double-

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