

## 13-Defects, Microstructures and Textures

361

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.

We are grateful to Prof. Liu Yaogang for growing KTP crystals and to the National Laboratory of Synchrotron Radiation of BEPC for experimental support.

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  $Pc_{2h}$  with lattice parameters  $a = 11.258 \text{ \AA}$ ,  $b = 23.402 \text{ \AA}$ ,  $c = 7.961 \text{ \AA}$ . The melting temperature is  $42^\circ\text{C}$ . 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  $0.5^\circ\text{C}$  above the crystallization temperature (at about  $42.5^\circ\text{C}$ ). 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  $35^\circ\text{C}$ , 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 $\alpha$  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	$t_1(\text{\AA})$	$t_2(\text{\AA})$
A	1-3	0.294	51	64
B	4-11	0.234	47	69
A	12-15	0.294	51	64

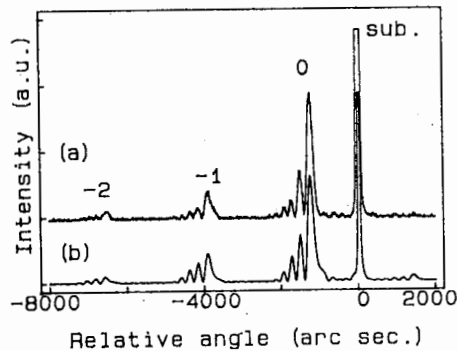


Fig.1 X-ray diffraction rocking curves  $Si_{1-x}Ge_x/Si$  superlattice: (a) experimental; (b) simulated.

crystal diffraction rocking curves, combined with computer simulation. Its perfection was characterized by x-ray plane wave topographic technique using both synchrotron radiation and laboratory x-ray sources. X-ray topographs were taken at different positions of 224 asymmetric reflection rocking curve. The experimental results show that there exist a kind of novel regular dislocation network on the interface between the substrate and the epilayer. They were determined to be pure edge dislocations with the Burger's vectors  $\frac{a}{2}[110]$  and  $\frac{a}{2}[\bar{1}10]$ , respectively. By analysing the x-ray diffraction rocking curves, the following perfection information of our sample was obtained: the mean strain relaxation  $R = 31.7\%$ ; the mean mismatch  $f = 0.55\%$ ; the dislocation density  $\rho \sim 2.71 \times 10^7 \text{ cm}^{-2}$ .

PS-13.02.13 CHARACTERIZATION OF InAs AND InGaAs ON (110) GaAs BY X-RAY DIFFRACTION AND TOPOGRAPHY. By L. Hart\*, P. F. Fewster<sup>1</sup>, X. Zhang and D. W. Pashley<sup>2</sup>, Interdisciplinary Research Centre for Semiconductor Materials, Imperial College, London, UK, <sup>1</sup>Philips Research Laboratories, Redhill, UK, <sup>2</sup>Department of Materials, Imperial College, London, UK.

There has recently been interest in semiconductor growth on (110) orientated surfaces. Strain relief in heteroepitaxial layers grown on (110) substrates is affected by the surface geometry since, in the [001] direction the {111} slip planes are inclined to the surface, while in the [1-10] direction, the {111} planes are perpendicular to the surface. Transmission electron microscopy has shown that strain relief of InAs on (110) GaAs is asymmetric, with 60° misfit dislocations in the [1-10] direction, giving rise to large tilts, and Lomer type dislocations in the [001] direction.

X-ray diffraction and topography have been used to determine relaxation and tilt in a range of samples of varying thicknesses of InAs and InGaAs, grown by molecular beam epitaxy on (110) GaAs. Reciprocal space mapping around the 220, 620 and 331 reflections enabled separation of components of strain and tilt in the [110], [1-10] and [001] directions respectively. From the reciprocal space maps, it was possible to determine both the macroscopic, "average" tilt and the spread of microscopic, "local" tilts. The relaxation and tilt in the [001] direction were found to increase with sample thickness: a 400Å InAs layer was almost fully relaxed and the average tilt was 1.0°, with a spread of 1.8°. In the [1-10] direction, however, relaxation was high even for the thinnest samples studied (30Å) but there was negligible tilting. Topographs taken from the same areas studied by diffractometry gave additional information on the size of the tilted regions.

PS-13.02.14 MINUTE STRAIN FIELDS IN AN AS-GROWN FZ Si CRYSTAL CONTAINING D-DEFECTS. By S. Kimura\*, T. Ishikawa and J. Matsui, Microelectronics Research Laboratories, NEC Corporation, Japan.

The nature of D-defects in FZ Si crystals is still unclear since non-destructive characterization techniques have not been effective in observing D-defects in "as-grown" crystals, due to their small size and minute strain. In a previous work (Kimura, Ishikawa, Mizuki & Matsui, *J. Cryst. Growth*, 1992, 116, 22-26), we had shown that minute tensile strain fields around A-defects in an as-grown FZ Si crystal can be quantitatively detected using plane-wave X-ray topography with extremely collimated X-rays, combined with the oscillatory profile of the diffraction curves in the Laue geometry. In the present work, therefore, this technique was applied to an as-grown FZ Si crystal containing D-defects. We examined a (111) wafer prepared from an undoped FZ Si crystal containing D-defects. The X-ray topographic measurements were performed at BL-15C of the Photon Factory at the National Laboratory for High Energy Physics. In this experimental arrangement, successive asymmetric 220 and  $\bar{2}\bar{2}0$  diffractions from the first and second collimator crystals produced incident X-rays with an angular divergence of about 0.01 arc sec for  $\lambda = 0.735 \text{ \AA}$  (Ishikawa, *Acta Crystallogr.*, 1988, A44, 496-499). Contrast analysis of the topographs gives following results: The D-defects region in an as-grown FZ Si wafer was thereby non-destructively imaged for the first time; D-defects had only been observed previously using X-ray topography following copper decoration or a preferential etching technique using Secco's etchant (Yamagishi, Fusegawa, Fujimaki & Katayama, *Semicond. Sci. Technol.*, 1992, 7, A135-A140). Furthermore, in the D-defect region, tensile strain with  $\Delta d/d$  less than  $1.5 \times 10^{-5}$  exists. Finally, the spatial distribution of this strain is not uniform, while copper decorated D-defects show a uniform distribution.

PS-13.02.15 CHARACTERIZATION OF THE CELLULAR GROWTH STRUCTURE OF GaAs - 0.2 at % In BY X-RAY TOPOGRAPHY. By F. Minari\* and B. Billia, Laboratoire Matériaux Organisation et Propriétés, ass. CNRS, Univ. Aix-Marseille 3, France.

In order to obtain large GaAs single crystals by the Czochralski technique, Indium is usually added into the melt to reduce the density of grown-in dislocations. Under optimal growing conditions, the moving liquid-solid interface remains plane, but due to morphological instabilities it can turn to a cellular structure. The case of GaAs/In is particularly interesting because the cells are partially faceted, i.e. they are made of portions of {111} planes connected by rough regions, in opposition with entirely faceted or entirely rough cells occurring in other materials. Moreover, in Czochralski growth, the well-known phenomenon of striation takes place due to periodic fluctuations of In content at the interface. These striations, which are revealed as sharp fringes by X-Ray Topography on a plane section of the ingot, act as a natural marker of the interface (plane as well as faceted) at constant intervals of time. Using the X-Ray images of these striations, we studied the birth, the evolution and the geometrical characteristics of the cellular structure on sections parallel and perpendicular to the [001] growing axis. Our observations show that:

- a unique local perturbation near the center of the initially flat interface initiated the whole cellular structure,
- no individual dislocation is visible within the cells, but such defects may be present at the junctions between cells,
- the fourfold symmetry of the cells expected on (001) sections is strongly altered by convection in the melt,
- dynamical roughening is evidenced at the edges of a cell undergoing overgrowth by neighbouring cells, which increase in size,
- whatever the extension of the facets (i.e. the cell-size) the width of the rough grooves between cells is remarkably constant while solidification proceeds,