



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. Fewster¹, X. Zhang and D. W. Pashley², Interdisciplinary Research Centre for Semiconductor Materials, Imperial College, London, UK, ¹Philips Research Laboratories, Redhill, UK, ²Department 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 1.5×10^{-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,

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- minute misorientations are present between the different parts of a given cell,
- the contrasts of the regions connecting the facets at the tip of the cells and in the grooves between them are different. This phenomenon is not related to the sign of a possible local curvature of the net planes in these regions. Up to now it is not elucidated and electron microscopic observations are needed which could give further experimental information about the general question of the transition between plane and rough interface at the facet edge.

PS-13.02.16 X-RAY SECTION TOPOGRAPHY STUDY OF BRAZIL TWIN BOUNDARIES IN NATURAL AMETHYST. By Z. Baran^{*}, Instituto de Fisica, Universidade Federal da Bahia, Salvador, Ba., Brazil and B. Capelle, Laboratoire de Minéralogie-Cristallographie, Université P. et M. Curie, 75252 Paris, France.

A natural amethyst crystal twinned according to the Brazil twin law was investigated by means of X-ray section topography in order to explain the unexpected contrast between alternating dextrogyre and levogyre lamellae formed at the natural rhomboedral face (0111) of this Brazilian amethyst crystal. It was also studied by transmission projection topography, recorded with Mo K α radiation and $\mu t=0,3$ (Z. Baran *et al.*, 1990, *Acta Cryst.*, A46, p. C-430). Under such a condition of relatively low absorption, the contribution of anomalous dispersion to the image contrast should be rather small. It was found, however, that the Brazil twinning, which can also be considered as an inversion twinning, can be observed by means of X-ray topography.

The Brazil twin consists of dextrogyre and levogyre lamellae in parallel orientation with (1120) or {1011} as composition plane. This is really a parallel growth (Lu Taijing and I. Sunagawa, 1990, *J. Cryst. Growth*, 99, 1232). In a previous paper (Z. Baran *et al.*, 1987, *Phys. Stat. Sol. (a)*, 101, 9), a difference in the lattice spacing in the direction perpendicular to the rhomboedral growth face was observed by X-ray double-crystal topography between the twin lamellae and the twin boundary layer, and shown to be equal to $\Delta d/d = -2.10^{-5}$. This fact agrees well with a probable model of accommodation layer which is formed between the adjacent twin lamellae. As a consequence, lattice deformations can be also expected along the twin boundaries. To investigate the nature of contrast images in our projection topographs, a series of section topographs was taken at different positions. These section topographs show that the interface or the boundary is detectable by X-ray topography. The observed very complicated contrast image is not similar to that occurring in the Brazil twin boundary in natural quartz, as it was observed by Yang *et al.*, (1986, *Phys. Stat. Sol. (a)* 97, 411), which is characterized by an hour-glass-shaped system of fringes. Thus, it is presumed, the source of the image contrast of the twin boundary is to be associated with strain. This corresponds to the last item of the classification of the surface defects separating two perfect or nearly perfect regions of the crystal (A. Authier, 1977, in *Crystal Growth and Materials*, Chap. II.3, ed. by E. Kaldis and H.J. Scheel, North Holland Publ., p.530). The enhanced black-and-white contrast observed on the X-rays projection photographs along the Brazil boundaries shows that a strong strain field exists within the twin

lamellae, reflecting the same structure factor modulus (i.e. no domain contrast). As indicated by our section topographs, the observed contrasts do not present a dynamical but rather a kinematical character due to strain concentrations at the boundary. An attempt is made to explain the nature of those boundaries by means of a detailed discussion of the recorded topographs.

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PS-13.02.17 VIBRATION MODES STUDIES USING SYNCHROTRON X-RAY TOPOGRAPHY. B. Capelle, J. Détaint, J. Schwartzel, A. Zarka^{*}, Y. Zheng. Laboratoire de Minéralogie-Cristallographie, Universités P. & M. Curie (Paris VI) et Paris VII, CNRS 09,4 place Jussieu, 75252 Paris Cedex 05, France. Centre National d'Etudes des Télécommunications, PAB/BAG/MCT, 92220 Bagneux, France.

X-ray topography has been extensively used to assess the quality of crystals, in particular for quartz, and it is now a usual technique to characterize vibration modes. More recently, the availability of synchrotron radiation has brought new possibilities to this technique: the high flux of X-ray radiation permits to observe simultaneously several diffraction patterns with different diffraction vectors g and to analyse the spatial structure of vibration modes; the synchrotron pulsed X-ray beam can be used to excite X-ray synchronized vibrations in resonators and it allows to reveal the time structure of vibration modes. Synchrotron X-ray topography appears thus to be a well adapted method of analysing vibration mode shapes. Synchrotron X-ray topography permits also to verify experimentally theoretical models and to get informations not yet considered theoretically. For particular cases, complex boundary conditions and coupling piezoelectric constants may lead new components in the displacement fields. Experiments at the LURE-DCI storage ring (France) have been carried out to characterize mode shapes in quartz resonators, but also in berlinite and lithium tantalate, and in connection with the presence of different crystal defects. Interesting cases showing stationary trapped and coupled components, were evidenced by classical X-ray topographs in quartz AT plano-convex resonators. These stroboscopic topographs provide interesting experimental data for the investigation of non-linear effects in quartz. In a similar way, quartz of other cuts (BT and SC), berlinite and lithium tantalate have been investigated and instructive informations were obtained concerning mode lateral anisotropy, coupled components *etc.* Other important cases studied by synchrotron X-ray topography concern crystal defects (such as dislocations and growth bands) which affect mode shapes in a most complicated way, and external conditions such as stress and temperature. Through these studies, it can be seen that synchrotron X-ray topography provides useful experimental data concerning diverse aspects of mode shapes in resonators. Different factors affecting mode shapes such as coupling mechanisms, crystal defects and non-linear effects can be finely analysed by this technique.

PS-13.02.18 IMAGE TREATMENT OF SYNCHROTRON TOPOGRAPHS. By Y. Epelboin^{*}, M. Pilard, A. Soyer, Lab. Minéralogie-Cristallographie, Universités P.M. Curie et Paris VII, U.A. 09 CNRS, 75252 Paris Cedex 05, France, e-mail: epelboin@lmcp.jussieu.fr

The development of synchrotron facilities means that the processing and analysis of images recorded either on films or by means of TV cameras must be enhanced to extract the maximum number of features from a single image. The experiments are too costly and the time of experience too short to lose part of a possible information. Different mathematical treatments may be used for the same image if one wants to study different features.

In preliminary experiments for the ESRF, conducted at LURE/DCI, we have recorded digitized images directly from a X-Ray camera or later from films using an ordinary video camera. We have studied the influence of the digitization process on the quality of the images and we have investigated various techniques of image treatment such as Fourier filtering or maximum entropy. Wavelets are under investigation.