

## 13-Defects, Microstructures and Textures

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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<sup>\*</sup>, 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 $\alpha$  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<sup>\*</sup>, 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<sup>\*</sup>, 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.

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We will discuss these techniques, showing their possibilities and limitations. For instance we will show how it is possible to correct a varying background which often exists in the topographs, due to extended strains, and which severely prevents from observing small defects in the black areas of the image or enhance the visibility of an anisotropic feature. It is also possible to study an anisotropic texture in the images. Fourier and entropy techniques are complementary. We will compare their results and try to build a strategy for image analysis.

Two kinds of treatments may be considered. On-line analysis for standard features such as background correction, off-line treatment to extract special features. The scientist, having the knowledge of the contents of a topograph, is the only person able to choose between the various means of analysis. Thus a full investigation of a given topograph is a long process which must be applied to selected images only after a first rapid analysis.

## PS-13.02.19 BRAGG-CASE IMAGES OF STACKING FAULTS.

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Bragg-case synchrotron double-crystal images of stacking faults have been studied in a synthetic diamond. The topographs taken on the tails of the rocking curve showed well pronounced interference fringes arising from the stacking faults: the first such observation in Bragg diffraction geometry. The fringes were strongly dependent upon the angular setting, being invisible at the rocking curve maximum but gaining in contrast and becoming more closely spaced further from the maximum. These experimental images were compared with predictions of plane-wave dynamical theory and a reasonably good correspondence was obtained when the finite beam divergence was taken into account. It was found that the theoretical fringe sequences depended upon the type of stacking fault, and confirmed that the stacking faults observed were of intrinsic type.

## PS-13.02.20 DOMAIN WALLS IN FERROELECTRICS. By M. Takagi\* and S. Suzuki. 1-10-6, Tsurumaki, Setagaya, Tokyo. Sanyo Tsukuba RC, Tsukuba, Japan.

Images of imperfections which arise from the dynamical diffraction effect are observed on X-ray topographs of perfect or nearly perfect crystals. If a mosaic spread of a specimen crystal is a few minute of arc (which is ten times as large as that of perfect crystals), intensity in a Lang section topograph is uniform and the intensity is proportional to the integrated intensity of the Bragg reflection. Images of antiparallel domain walls and of the regions in the intermediate states of polarization reversal have been observed on the section topographs of ferroelectric  $\text{NaNO}_2$  and thiourea crystals with such a mosaic spread. Structure of domain walls and of the intermediate state regions have been determined from dependence of the contrast of the images on the indices of Bragg reflection  $hkl$ . Studies of domain walls for  $\text{NaNO}_2$  and thiourea, and of the intermediate state for  $\text{NaNO}_2$  have already been reported and additional studies have been made on the polarization reversal of thiourea and on change in images of domain walls of  $\text{NaNO}_2$  near the Curie temperature  $T_c$ . Results of these series of studies have made clear the structures of  $180^\circ$  domain walls and the polarization reversal procedure.

Atomic positions in positive domain and those in negative domain shift relatively. For a perovskite-type crystal such as  $\text{BaTiO}_3$ , the relative shifts are as small as the amplitudes of thermal vibration, so that the two opposite domains are expected to be connected smoothly at an antiparallel domain boundary without any special layer. For some ferroelectrics such as  $\text{NaNO}_2$  and thiourea the amount of the relative shift is as large as  $0.5 \sim 1 \text{ \AA}$ . For such ferroelectric crystals, thick  $180^\circ$  domain walls which connect the two opposite domains without lattice strain are expected. The structures of domain walls determined are as have been expected. Fig.1(a) is the c-projection of the unit

cell of  $\text{NaNO}_2$  and (b) is that of domain walls where  $\text{NO}_2$  radicals are rotated by about  $50^\circ$  or  $130^\circ$  around the c-axis from the original orientation. Fig.2 is the projection of the unit cell of ferroelectric thiourea, where full circles and the dotted circles represent the atomic positions in the positive and the negative domains respectively. The domain wall structure of thiourea determined is a modulated superstructure with long periods along the c-axis. In the structure, direction of thiourea molecules gradually varies along the c-axis between the two directions shown in Fig.2.  $180^\circ$  domain walls of  $\text{NaNO}_2$  and thiourea are about  $1 \mu\text{m}$  thick and in the domain wall structure atoms and molecules are at the halfway positions between the two opposite domains. On application of d. c. electric fields to  $\text{NaNO}_2$  and thiourea, polarization reversal takes place via intermediate states. Structure of the intermediate state resembles the structure of domain wall.

The structure of domain walls of thiourea resembles that of the higher temperature phase above  $T_c$ , and topographic observation on  $\text{NaNO}_2$  at  $T_c$  suggests the same correlation for  $\text{NaNO}_2$ . These structural correlations would be applied for any ferroelectric for which the atomic shift between the two opposite domains is far larger than the amplitudes of thermal vibration.

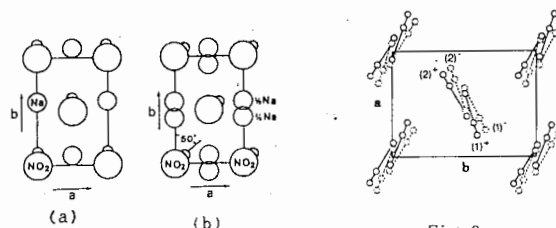


Fig.1

Fig.2

## PS-13.02.21 STRUCTURE FACTORS OF LAYER SYSTEMS - EXAMPLE: BRAGG REFLECTORS (GaAs/AlAs)/GaAs.

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Shifts of the atomic positions inside a layers system have a strong influence on the diffracted intensities even for high order satellites and, hence, have to be described correctly. At these high orders the Fourier components of the term  $\exp(-ihu)$  ( $h$ : diffraction vector;  $u$ : shift in the atomic positions as compared to a reference lattice) might be negligible but neighboring high Fourier orders of the electric susceptibility  $\chi_h(r)$  are combined with low orders of the exponential. For that reason the concentration profile inside a layer stack of a quasi binary compound cannot be calculated straightforward from the diffraction curve.

On the other hand due to intermixing of Fourier orders the structure factors also depend on the phases of the Fourier components. If the satellite reflections are well separated a simulation procedure based on the integrated intensities of the satellites and on the Fourier transform of the mo-