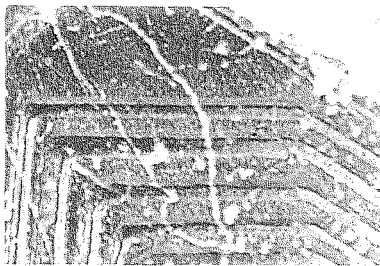


**07.1-07** STRUCTURAL STUDIES OF THE GROWTH STRIPES ON NATURAL FACES OF AMETHYST CRYSTAL. By Z. Baran (b), K. Godwod (a), T. Waminski (a): (a) Institute of Physics, Polish Academy of Sciences, Warsaw, Poland; (b) Universidade Federal Da Bahia, Instituto De Fisica, Salvador, Bahia, Brasil.

The growth-dependent structure of amethyst crystals has been investigated by Schlössin and Lang. They focused their interest on samples cut off from the interior of crystals with imperfect lamellae coinciding with laminated Brazilian twinning (H.H. Schlössin & A.R. Lang, *Phil. Mag.* (1965) **12**, 283). Our studies deal with oriented {1011} growth faces of an amethyst taken from the mine in Bahia, Brasil. The aim was to determine the uniformity of the interplanar spacings and the distribution of impurities on the natural faces, i.e. whether they were undisturbed by mechanical or chemical treatment. The task was accomplished using four techniques: 1) divergent beam X-ray topography (the oscillating slit method) (J. Auleytner, *Acta Phys. Polon.* (1971) **A39**, 379); 2) parallel beam X-ray topography in the double-crystal spectrometer; 3) electron probe microanalysis (EPMA); 4) polarizing microscopy. The following results were obtained. First, imaging of the sample of the crystal bulk reveals the laminated structure due to Brasil twinning. The twinned crystals show on the faces, appearing as growth stripes. Secondly, divergent beam X-ray topogrammes showed an increase of the total X-ray reflection power at the stripe boundaries and thus a significant disturbance of those regions. Thirdly, a relative change of the lattice constant,  $\Delta d/d$ , at the stripe boundaries was determined by the Yoshimura method (J. Yoshimura et al., *J. Cryst. Growth* (1979) **46**, 691). It was found from parallel beam X-ray topogrammes taken in the double-crystal spectrometer ( $\text{CuK}\alpha_1$  ( $\text{Si}_{533}\text{-SiO}_2$   $40\text{Å}$ )). The calculated half-width of the reflection curve for this arrangement, having a remarkable dispersion, should be  $14''$ , and it was  $\sim 22''$  when measured for the amethyst; the difference indicated the presence of the internal strain macrofield. Two series of topogrammes were recorded for azimuths  $0^\circ$  and  $180^\circ$ , and for different angular positions of the amethyst which covered the rocking curve (Ilford G5 films and exactly the same exposure times were used). Three different types of twin boundaries were distinguished: 1) with the same lattice constant as the undisturbed crystal; and 2, 3) with  $\Delta d/d$  equal to  $-1.8 \times 10^{-5}$  and  $-1.3 \times 10^{-5}$ , respectively. The EPMA was used to recognize the concentration profiles of four elements: Na, Al, Mn and Fe in the direction perpendicular to the stripes; the average atomic concentrations were of 120, 750, 550, and 310 ppm, respectively. Within the accuracy of 30 ppm there was no increase of Fe content at the stripe boundaries.



Parallel beam X-ray topogramme of the amethyst sample.

**07.1-08** MORPHOLOGY AND STRUCTURE OF SYNTHETIC MICROCRYSTALS OF MAGHEMITE ( $\gamma\text{-Fe}_2\text{O}_3$ ). By H. Batis\*, Ch. Leclercq\*\*, M. Boudeulle\*\*\* and P. Vergnon\*, \* Laboratoire associé au C.N.R.S. n° 231, \*\*\* Equipe de Recherche associée au C.N.R.S. n° 600, Université Claude Bernard, Lyon I, 69622 Villeurbanne Cédex, and \*\* Institut de Recherche sur la Catalyse (C.N.R.S.), 69626 Villeurbanne Cédex (FRANCE).

Small grains of ferric oxide are prepared from vapour phase reaction by hydrolysing iron trichloride in a hydrogen-oxygen flame. The experimental reactor allows to control the reaction conditions: the size of the particles may be varied from 15 to 80 nm. Either hematite ( $\alpha\text{-Fe}_2\text{O}_3$ ) or maghemite ( $\gamma\text{-Fe}_2\text{O}_3$ ) may be obtained: maghemite results from the rapid cooling of liquid droplets formed in the flame, whereas hematite is obtained by direct condensation from the vapour phase to solid state.

The structure and the morphology of the small particles have been studied by X-ray diffraction and electron diffraction and microscopy. The lattice plane imaging coupled with microdiffraction has pointed out that maghemite grains are perfect single crystals which present an association of the {111}, {110} and {100} forms. For the largest grains ( $> 70$  nm), the {110} faces are often stepped by {111} planes. Twin crystals (Spinel law) with polysynthetic twinning are frequently observed. The crystal structure is cubic, with a P lattice mode: the distribution of the  $\text{Fe}^{3+}$  ions and the vacancies into the octahedral sites foreshadows the hematite structure.

These grains have been tested as catalysts in the partial oxidation of propene: the activity in acrolein formation seems to be related to the presence of the faceting observed on the large crystals.

**07.1-09** GRAIN SIZE, IMPURITY DISTRIBUTION AND INTERNAL STRAIN IN POLYCRYSTALLINE GaP AND InP INGOTS GROWN BY THE SSD METHOD. By F. Moravec and J. Novotný, Institute of Radio Engineering and Electronics, Czechoslovak Academy of Sciences, Prague, Czechoslovakia.

Bulk gallium phosphide and indium phosphide crystals were grown by the synthesis, solute diffusion (SSD) method (F. Moravec, J. Novotny, *J. Crystal Growth* (1976) **33**, 90 and F. Moravec et al., *Kristall und Technik* (1980) **15**, 1105) including the SSD method improved by the application of a thermal screen for temperature gradient modification (F. Moravec, J. Novotny, *J. Crystal Growth* (1980) **52**, in press). The crystals obtained were examined with respect to grain size, impurity distribution and internal strain.

The average grain size was evaluated in dependence on impurity contents, ingot diameter, temperature gradient and the shape of bottom part of the crucible. The distribution of impurities in differently oriented crystal grains was studied by means of X-ray microprobe analysis and measurements of free electron concentrations and electrical conductivities. The differences in electron concentrations measured in various grains were found to be as high as one order of magnitude while the average concentration level was about  $10^{17}\text{cm}^{-3}$ . The internal strain of crystals was evaluated by means of ultrasonic velocity measurements. Acoustic emission caused by the temperature change of crystals was also studied. There is a marked difference in acoustic emission of polycrystals in comparison with single crystals.