

of relatively large blocks in a form of oriented "columns" grown on the seed crystal, which were strongly twisted around the 210 growth axis. The crystal grown in Space consisted of blocks, too, but the "columns" have smaller diameters and were much less twisted. Natural crystal walls, parallel to the 111 plane, had been found in some parts of the crystal surface. The density of dislocations was similar in all crystal blocks ( $10^7 \text{ cm}^{-2}$ ). A thin layer of vapour-grown crystal covered partially the side walls of the seed crystal. A constant ratio of the characteristic line intensities for the three elements along the crystal has been found. Analyses for the cross-section parallel to the growth axis have shown no significant differences in composition homogeneity between the crystal grown in Space and that on the Earth. The same refers to the cross-section in the plane perpendicular to crystal longitudinal axis, except for Pb and Se where the distribution of those elements showed somewhat smaller fluctuation. Some areas containing PbSe precipitates (density ca  $1 \times 10^3 \text{ cm}^{-2}$ ) in the seed crystal and also partially in the Earth-grown one were observed. The crystal grown in Space exhibited PbSe precipitates (density of  $1.5 \times 10^4 \text{ cm}^{-2}$ ) in the bulk, and inclusions of  $\text{Pb}_{0.5}\text{Se}_{0.17}\text{Te}_{0.24}$  situated on the plane parallel to the boundary between the grown crystal and the seed part.

**07.1-02** TWINNING OF EPITAXIAL DIAMOND FILMS GROWN FROM GASEOUS PHASE. By M.O. Kliya, A.E. Alexenko, B.V. Spitsin, Institute of Crystallography, Institute of Physical Chemistry of the USSR Academy of Sciences, Moscow, USSR.

Epitaxial diamond films were grown from the gaseous phase of carbon containing compounds at temperatures of about  $1000^\circ\text{C}$  and pressures less than 1 atm. The films were grown on the natural faces (111) of diamond as well as on (111), (110) and (100) surfaces of polished diamond substrates. Film morphology was studied at room temperature. Samples were taken out of the chamber after each stage of growing, which made it possible to observe the surfaces of the samples at different thicknesses. These repeated interruptions could lead to surface changes due both to cooling and to additional defects arising from the interruptions. Growth layers and specific square figures of growth were always seen on (100) substrates. Tangential growth of the layers and the formation of new ones can be seen from stage to stage. The generation of new layers always occurred at the same points. When the film is thinner than  $1-2 \mu\text{m}$ , the top of each square pyramid has a point which generates new layers. These defects grow with film thickness. When they reach some microns it becomes possible to conclude that each defect is a protrusion over the surface of a square pyramid. The protrusion is formed by triangular facets which form asymmetric pyramids turned towards one of the four [110] edges. All four orientations of the protrusion are equally probable. This fact allows one to suggest that the visible defects are diamond twins. The distribution of goniometer signals from the grown films corresponds to the locations of diamond facet projections corresponding to the four possible twin orientations according to the spinel law. The causes of this twinning under the spinel law are unknown. Extreme changes in

the conditions before and after interruption of the growth are of great importance. Some particles can be deposited on the as-grown film surface and can stimulate the generation of the twins. The number of twins increases with the number of the cycles of growth and with film thickness. Thus, a single-crystal film  $70 \mu\text{m}$  thick turns into polycrystalline. The loss of thermostability is clearly displayed at the locations of the twin clusters; they become dark brown or black after annealing. Film sections with isolated twins do not undergo any noticeable changes during two-hour annealing at a temperature of  $1400^\circ\text{C}$ . The structural changes which lead to the loss of transparency of the film begin in the sections with high twin density in the film, or at the film-substrate boundary, but not on the surface. The density of dark spots and their areas increase with the duration of the annealing. Different intensity of darkness can be seen in almost every dark spot, its bounds being clear. It may be concluded that the film structure changes layer by layer. It can be supposed that the process begins in the twin boundary and then spreads along the surface and into new layers.

**07.1-03** ADVANCES IN THE APPLICATIONS OF THE HOLOGRAPHIC INTERFEROMETRY TO THE STUDY OF CRYSTAL GROWTH FROM SOLUTION. By F. Bedarida and L. Zefiro (Istituto di Mineralogia), P. Ottonello and C. Pontiggia (Istituto di Fisica), Università di Genova, Italy.

Changes of concentration near a crystal growing from a supersaturated solution have been investigated in the past by classic optical interferometry. Holographic optical interferometry gives the possibility of working in larger volumes (some tens of  $\text{cm}^3$ ), where the convective movements affecting the growth process may be easily checked. In all these techniques, only a mean refractive index of the solution may be measured via the interference fringes produced by the overall optical path-length variations. A real three dimensional map of the refractive index variations is obtained from multi-directional holographic interferometry. Since the refractive index is generally a function not only of concentration but of temperature, too, the temperature variations near the interface of a growing crystal have been tested by thermocouple probes; values of the order of  $10^{-2}^\circ\text{C}$  have been measured during the growth of a  $\text{NaClO}_3$  crystal from a 1% supersaturated solution. Therefore, the interference fringes obtained experimentally may be related directly to the distribution of concentration near the growing crystal.