

07.X-04 METALLIC-, IONIC- AND INERT GAS MICROCLUSTERS
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Recently, the main problems underlying the experimental study of homonuclear particles with sizes between single atoms and the solid state have been solved: the formation of microclusters, their individual detection and the separation of beams with uniform cluster size. For the first time, agglomerates containing between two and several thousand atoms or molecules have been produced. The measured mass distribution spectra yield information about growth mechanisms, crystallography, magic numbers of the clusters and in the case of double ionisation the critical sizes for Coulomb-explosion.

07.X-05 CRYSTAL STRUCTURE OF THIN SOLID FILMS. By L.I.Man and S.A.Semiletov, Institute of Crystallography of the USSR Academy of Sciences, Moscow, USSR.

Thin solid films of various materials are widely used in modern science and technology. HEED is the main technique for investigation into their structure. There will be given a brief review of the results of such investigations with the emphasis at the correspondence between the structures of thin films and bulk specimens. In the majority of cases thin films and bulk specimens have the same structures. The absence of such a correspondence is due to impurities, inclusions and point defects (vacancies, interstitial atoms) whose concentrations exceed greatly those of equilibrium.

The results will be presented of the investigation into the character of the scattering of high energy electrons in polycrystalline thin solid films of various materials which have been carried out at the electron diffraction laboratory of the Institute of Crystallography of the USSR Academy of Sciences. It will be shown that kinematical theory with due regard for dynamical effects (e.g. after Blackman) can well serve the purposes of electron diffraction structure analysis. The effect of dynamical scattering on the accuracy of structure determination (potential peak heights and atomic coordinates) will be discussed as well as the possible refinement of thin film chemical composition from the potential peak heights in Fourier syntheses.

07.X-06 MAGNETRON SPUTTERING OF COMPOUND FILMS By W.D. Westwood and S. Maniv, Bell-Northern Research, Box 3511, Station C, Ottawa, Ontario, Canada, K1Y 4H7. Magnetron sputtering for the deposition of thin metal or alloy films has become established in a number of manufacturing processes because it provides high deposition rates at relatively low substrate temperatures. In the electronics, optical and energy industries, there is a need for an economic process for deposition of compounds. For example, Al_2O_3 is used as a dielectric and as an optical waveguide, ZnO is utilized in surface acoustic wave devices, Indium Tin Oxide is employed as a transparent conductor in liquid crystal display panels and as a heat mirror, and CdS, CdSe and amorphous hydrogenated silicon are candidates for solar cell fabrication. Reactive sputtering in magnetron systems has been investigated as a method for high rate deposition of these compounds. Magnetron sputtering offers some distinct advantages but careful control of the parameters is necessary to obtain the required film properties. H_2 can be used as a reactive gas without affecting the sputtering rate provided the ionization in the discharge is sufficiently high. When O_2 is the reactive gas, two stable modes of deposition are normal. In the first, oxygen is used simply as a dopant and does not affect the deposition rate; this has been used to modify the resistivity of CdSe films. In the second mode, an oxide layer forms on the target surface, causing a large decrease in deposition rate but providing fully reacted films; high resistivity ZnO films for SAW devices have been prepared. We have also developed a third mode in which the deposition rate is slightly reduced and the film properties are controlled. This technique has been used to prepare transparent conducting films of defect semiconductors, such as indium tin oxide. These different modes of magnetron sputtering offer new and interesting possibilities for the deposition of compounds.

07.1-01 REAL STRUCTURE CHARACTERIZATION OF PbSeTe CRYSTALS GROWN UNDER MICROGRAVITY CONDITIONS AND ON THE EARTH. By J.Auleytner, J. Bał, Z. Furmanik, K. Godwod, A. Jędrzejczak, E. Mizera, A. Szczerbakow and T. Warmiński, Institute of Physics, Polish Academy of Sciences, Warsaw, Poland.

The $\text{PbSe}_{1-x}\text{Te}_x$ crystals were prepared in Space and on the Earth, for comparison, by sublimation of a $\text{PbSe}_{0.5}\text{Te}_{0.5}$ charge crystal onto a single crystal seed having the same composition. Both seed and charge crystals for the two experiments had been prepared on the Earth from a crystal grown using the vapour phase transport technique. The growth technique (Markov and Davydov, *Nieorg. Mat.* (1975) 11, 1755) was adapted in the multipurpose apparatus "Crystal" to perform experiments in Space (Malinin and al., *Proc. 3rd European Symp.*, Grenoble, France, (1979) p.5 ESA Sp-142) and on the Earth. The crystal seed, 8 mm in diameter and 2 mm thick with 210 growth direction, was thermally adhered to the base of a cylindrical (with the same diameter) BaF_2 substrate crystal. The seed was fixed in a sealed quartz ampoule, 9 mm in inner diameter and filled with a noble gas, at a distance of 2 cm from the charge. The ampoule walls were not in contact with the crystal. The characterization of the real structure of PbSeTe crystals has been performed using the following X-ray and metallographic techniques: 1) Laue back-reflection method, 2) oscillating film method for the estimation of mosaic structure and dislocation density, 3) oscillating slit method (Auleytner, *Acta Phys. Polon.* (1971) A39, 379) for topographic imaging of block structure geometry, 4) EDAX and EPMA for a quantitative estimation of chemical composition, 5) metallographic observations. A difference has been found in the block structures of the crystals grown in Space and on the Earth. The latter consisted

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 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°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°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 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}°C have been measured during the growth of a 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.