11.1–27 INVESTIGATION OF X-RAY POLARIZATION EFFECTS IN DOUBLE-CRYSTAL TOPOGRAPHY.

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Double-crystal topography in the Bragg case was performed using synchrotron radiation and the new double-axis X-ray diffractometer (Nucl. Instr. and Meth.,190,593(1981)) installed at DORIS II/HASYLAB. With this instrument the reflecting plane of the double-crystal set C1 (reference crystal)-C2 (sample crystal) can be rotated continously with respect to the reflecting plane of the monochromator (two independent perfect Si crystals). Rotation axis is the beam incident on C1. It is thus possible to align the whole topographic set-up (crystals C1, C2, two monitor detectors and the film cassette) in the Bragg geometry for a given Θ_B^S angle on the inner bench of the diffractometer and to change the inclination angle α of the reflecting plane C1-C2 with respect to the polarization plane of the primary beam by rotating the bench about the monochromatic beam direction. For such a variation of α changes of the rocking curves widths (Γ_{RC} when rocking C1 and Γ_S when rocking C2) and changes of the penetration depth t_e of the beam during the reflection process have been predicted (Nucl. Instr.andMeth.,208,(1983)) when rocking of the strong α-dependence of relative σ and π contributions to the total reflecting power of the crystals. The effects should be the more pronounced the higher is the degree of polarization of the incident monochromatic beam and the closer to 45° is the sample Bragg angle Θ_B^S. The continous spectrum of the synchrotron radiation, its high initial degree of polarization (about 90%) and different combinations of reflections at the monochromator, C1 and C2 offered unique opportunities to study the problem at a variety of conditions nearly fulfilling both requirements.
Trial experiments for some almost perfect Si crystals with symmetric and/or asymmetric cuts, different reflections like (440),(551),(331), (422), some Θ_B^S in the range 39° to 51° (and λ in the range 0.63 Å to 1.92 Å respectively) and different α angles were carried out. Utilising reflections (400) or (800) and monochromator Bragg angles Θ_B^M very close to 45° in order to produce the monochromatic beam, its degree of polarization could be improved above the one given by the source itself. At such improved conditions and α angles close to 0° the measured rocking curves displayed a complex character (σ and π contributions superposed), the widths Γ_S (about one to two order of magnitude smaller than Γ_{RC}) decreased significantly for α approaching 0° and changes of topographic patterns were noticed. The patterns revealed the existence of some minute lattice strains connected with the crystal growth. The results seem to show that in a thin layer near the surface the stresses caused during the growth process are allowed to relax into minute strains which then are imaged on topographs.

Thus by taking topographs with different penetration depths t_e a variation of lattice deformations below a crystal surface can be explored.

11.2–1 DETERMINATION OF STRAIN DISTRIBUTIONS AND FAILURE PREDICTIONS BY NOVEL X-RAY METHODS.

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A number of X-ray methods were developed which make it possible to determine the distribution of elastic and plastic strains emanating from stress raisers. The determination of elastic strains is made by measurements of reflected intensities and rests on the theory relating the integrated reflectivity to lattice curvature. The theory covers the entire range from zero to infinity and takes into account, anomalous transmission and crystal anisotropy (Z. H. Kalman & S. Weissmann, J. Appl. Cryst. (1983) 16, 295). Using silicon as model material, strain gradients and strain distributions were measured from bent crystals containing stress raisers and the results were compared to calculations based on continuum mechanics. Good agreement was obtained between experiment and theory. The distribution of plastic strains is determined by double crystal diffractometry using a computer-aided rocking curve analyzer (CARCA). The plastic zones at the notch tips were mapped in terms of contourlines of excess dislocation densities and it was shown that in tensile-deformed, double-notched silicon the mapping of microplasticity by CARCA leads to the early disclosure of the future fracture path. Aided by pendellösung topography it was shown that strain hardened microplastic zones constrain residual, elastic strains and that the degree of strain hardening governs the magnitude of residual strains. Extending the results of single crystal studies and CARCA method (Yazici, Mayo, Takemoto & Weissmann, J. Appl. Cryst. (1983) 16, 89), to commercial aluminum alloys, cycled in air, and in corrosive environment, the accrued damage is determined and failure predictions are made nondestructively.

11.2–2 THE EFFECT OF REAL STRUCTURE OF GARNET ON ITS OPTICAL AND ELECTRICAL PROPERTIES.

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Electrical conductivity relation to temperature does not always follow the law of $\sigma = \sum_{i=1}^{i=n} \sigma_i e^{-E_i/kT}$ in real crystals where $E_i < E_{i+1} < \ldots < E_n$, though it is stated valid for ideal non-metal crystals. The cause of this is seen in processes of recapture of charge carriers by some defect trap levels as well as in chemical and physical transformations and in effects of concentration and charge gradients.

Studies have been made on $Y_3Al_5O_{12}$ and on $(TR_x, Y_{1-x})_3Al_5O_{12}$ obtained by horizontally directed growth technique (TR – Dy^{3+}, Lu^{3+}, Eu^{2+}).

Optical absorption spectra of these crystals in the range of 400–50000 cm^{-1} may be understood in known scientific graphology. The fine structure of Dy^{3+} spectrum (local symmetry D_2) is thought to be conditioned by forbidden transitions in the 4f–shell. The broad absorption bands observed at heterovalent substitution $Y^{3+} \rightarrow Eu^{2+}$ are in agree-

ment with interconfigurational transitions 4f–5d permitted by Laport's rule.

Study of electrical conductivity in the range of 300–1000 K has shown the dependence of $\sigma(T)$ in the process of the first heating to differ from the monoexponential one, as it has been stated above: $TK\sigma$ decreases in the range of 710–830 K and goes further to zero or reverses its sign. It should be stressed that $\sigma(T)$ topography do not depend on type or concentration of activator ion. All of the studied crystals have been found to possess similar c h a r a c t e r i s t i c parameters of the real charge transfer in $(Y, TR)_3Al_5O_{12}$ (starting temperature for the fast growth of σ, temperature range of changes $TK\sigma$, values of activation energy of conduction).

It wholly eliminates the idea of determining influence of type and valence of the activator ion on charge transfer processes, so the results obtained should be interpreted as follows. The dependence $\sigma(T)$ of YAG crystals and of solid solutions based on these garnets is thought to be determined by structural defects common for the garnet lattice. This approach makes one consider rational the observed differences in σ values and in sign and values of $TK\sigma$ in "anomalous" region as well as the absence of dependencies between $TK\sigma$ and ion-activator concentrations.

We consider the deviation of $\sigma(T)$ dependency from the exponential law to be caused by a process of recombination of charge carriers with oxigen vacancies.

11.2–3 X-RAY CHARACTERIZATION OF STACKING FAULTS IN CUBIC ZnS CRYSTALS GROWN FROM THE VAPOUR. By M.T. Sebastian and P. Krishna, Physics Department, Banaras Hindu University, Varanasi-221005, India.

An X-ray diffraction study is made to determine the nature of stacking faults present in vapour grown cubic ZnS crystals as well as cubic crystals obtained by solid state transformation from the 2H phase by thermal annealing. For this the point intensity distribution along the 10.L reciprocal lattice row of both kinds of disordered cubic (3C) crystals was recorded on a single crystal diffractometer. The observed intensity profiles are found to be asymmetrically broadened and do not show peak shifts indicating that stacking faults present in both as-grown and annealed crystals are predominantly twin faults distributed randomly. The experimentally obtained intensity profiles agree with those calculated theoretically for a random distribution of twin faults.

This experimental result is in accordance with the following model of the 2H-3C transformation. The transformation commences with the random nucleation of deformation faults in the 2H structure creating a large number of 3C nuclei within the 2H phase. These nuclei then grow further by non-random insertion of deformation faults occurring preferentially at two-layer separations. Since the cubic nuclei can have two twin orientations, the end product of the transformation is a heavily twinned cubic structure.

11.2–4 THE CRYSTAL STRUCTURE OF VANADIUM DITELLURIDE, $V_{1+x}Te_2$. By K.D. Bronsema, G.W. Bus and G.A. Wiegers, Laboratory of Inorganic Chemistry, Materials Science Centre of the University, Nijenborgh 16, 9747 AG Groningen, The Netherlands.

Vanadium ditelluride, $V_{1+x}Te_2$ ($0.04<x<0.11$) has a $Cd(OH)_2$ type structure with unit cell dimensions $a_h = 3.638$ Å and $c_h = 6.582$ Å above the transition temperature T_t of 482 K (for $x \approx 0$). Below T_t the structure is monoclinic, spacegroup C2/m, with cell constants $a_m = 18.984$ Å ($\approx 3a_h\sqrt{3}$), $b_m = 3.5947$ Å ($\approx a_h$), $c_m = 9.069$ Å ($\approx \sqrt{(3a_h^2 + c_h^2)}$), $\beta = 134.62°$. In this low-temperature form the vanadium atoms form double zig-zag chains with V-V distances of 3.316 Å, causing the Te lattice to be distorted. Due to the simultaneous occurrence of the distortion of the $Cd(OH)_2$ type structure of $V_{1+x}Te_2$ in three equivalent directions, complex diffraction patterns are observed. Similar patterns were found for the Nb and Ta ditellurides (Van Landuyt et al., phys. stat. sol., 41, 271 (1970)).

11.2–5 THE DEFECT STRUCTURE OF DOPED PbTe. By M. Schenk, Department of Crystallography, Section of Physics, Humboldt University of Berlin, Invalidenstr. 43, DDR-1040 Berlin, German Democratic Republic.

In PbTe, a narrow-gap semiconductor, doping seems to cause a complex defect structure, at least in some cases. Thereby the electrical activity of the dopants is influenced. In and Bi act as donors, Tl as an acceptor, while Ag should act as an acceptor (Aleksandrov et al., Kratkie soobščeniya po fizike No.7 (1981), 35–41). Point defects due to non-stoichiometry - e.g. lead vacancies in the case of p-type material, and tellurium vacancies and/or lead interstitials at the metal-rich side of the stability region, respectively - also cause an increase of the carrier concentration.
The results of precision lattice parameter, carrier concentration, and dopant concentration measurements show decreasing lattice constants due to increasing dopant concentrations of Bi, In, and Tl. That may be comprehensible for Bi^{3+} and In^{3+}, respectively, because of their small cation radius (compared with that of Pb^{2+}), and according to their lattice relaxation, but it is not intelligible in the case of the big Tl^+ ion, if there is not considered the formation of an association of Tl with a vacancy.
Ag increases the lattice parameter, which can be understood by a model of a complex formation in the following way: $Ag_{Pb}^{+} + Ag_i^{-} \longrightarrow [Ag_{Pb}Ag_i]^{o}$.
The electrical activity of such associates, considering point defects, are discussed.