13-Defects, Microstructures and Textures

PS-13.01.08 FROM SHEAR LAG TO ATOMIC FIT. By P.M. Bronsveld*and J.Th.M. De Hosson, Department of Applied Physics, University of Groningen, Nijenborgh 4, 9747 AG Groningen, The Netherlands.

Already for some time now people are trying to improve aluminum by adding a ceramic reinforcement as SiC or Al_2O_3 to make so-called DRA or discontinuously reinforced aluminum [J. of Metals, January 1993]. The idea then is to make use of the strength properties of the ceramic particle while maintaining the ductility of the base metal. Crucial is the transfer of the load from the base metal to the reinforcement. A whole series of macroscopic models have been applied, e.g. the shear lag model in which the load transfer takes place via a kind of press-fit between both phases. Residual stress measurements on the Al/SiC system using neutron diffraction [T. Lorentzen, J., Neutron Res. 1(1993)13] clearly show an initial strain in the aluminum base, subsequently taken over by the strain in the ceramic particles. In order to understand what happens on the atomic scale, a study has been undertaken applying high resolution electron microscopy. In this analysis one observes the atomic fit between the metal and ceramic structures and tries to interpret it starting from the distinctive space groups, unit cells, lattice parameters and orientation relationships. Subsequently one tries to measure the displacement between both phases and searches for misfit dislocations. It may happen that one particular type of atom diffuses towards the interface, e.g. Mg, being a surfactant [Delannay et al. J. , Mat. Sci. 22(1987)1], positions itself preferably at the interface and via the formation of MgO may give rise to a reaction layer causing a lower fracture toughness. We have studied the Al/SiC and Al/Al_2O_3 systems and found that when the production method is one in which fine powder is cold-pressed and subsequently hot-extruded, one finds indeed an interface being a more or less abrupt transition from metal to ceramic. The so-called liquid route of manufacturing gives a reaction layer which for the Al/Al_2O_3 system turns out to be the spinel $MgAl_2O_4$ as was confirmed by comparison of the experimental results with the EMS and MacTempas HREM simulation programs.

PS-13.01.09 TEXTURED THIN FILM OF C_{60} GROWN BY VACUUM DEPOSITION. By W.L. Zhou*, W. Zhao, Y.Q. Zhou, K.K. Fung and L.Q. Chen, Institute of Physics and Beijing Laboratory of Electron Microscopy, Chinese Academy of Sciences, P.O.Box 603, Beijing 100080, People's Republic of China

The reports of straightforward procedure for synthesizing macroscopic quantities of C_{60} and its superconductivity in alkali-doped solids have triggered an intense concentration of physicists, chemists, etc. As the thin films of C_{60} prepared by vacuum deposition are free from solvents and also have a potential application toward new electronic devices, it is very important to know the crystallization and structure of the thin film. Different thicknesses of C_{60} thin films on NaCl (001) substrates have been produced by us. The thin films were grown by sublimating the pure C_{60} powder to NaCl (001) cleavage surface with pretreatment of eliminating residual solvent at 10^{-5} torr. Transmission electron microscopy study showed that at the initial stage the C_{60} thin film tended to crystallize randomly with few hexagonal phases and as it grew thicker, it formed [110] texture thin film with multiply twin particles and fewer hexagonal phases. Twins, microtwins, stacking faults and five fold twin particles were studied by high resolution electron microscopy (HREM). As the C_{60} is bounded by van der Waals force, the lattice bending structure and disorder cores due to the strain releasing in five fold twin particles were found by HREM. The large cages of C_{60} experienced to the strong electron beam were also observed by HREM.

References
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PS-13.01.10 REASONABLY GOOD DISLOCATION IMAGES IN LACBED PATTERNS AND EFFECTS OF ITS STRAIN FIIELD ON THE REFLECTION CONTOURS. By Y. Xin* and X.F. Duan Beijing Laboratory of electron microscopy, Chinese Academy of Sciences, P.O. Box 2724, Beijing, 100080, China

Large-angle convergent beam electron diffraction (LACBED) comprises real and reciprocal space information and therefore is useful to the study of defects in crystals. Dislocations parallel to the reflection rocking curve contours have been studied by an improved LACBED technique. Shadow images of dislocations of reasonably good spatial resolution superimposed on LACBED patterns have been obtained at the diffraction plane. It has been found that only when the shadow image of the dislocation is parallel and close to a low-order reflection contour with $\mathbf{g}\cdot\mathbf{b}\neq 0$ can it be formed with reasonably good contrast in the LACBED patterns. Both the image of the dislocation and the changes of the reflection contours clear in one LACBED pattern make it convenient to study the strain field of a dislocation. The strain fields of dislocations in Si and GaAs crystals have been investigated. Different effects are observed.

PS-13.01.11 SUPERLATTICE VARIANTS IN $Sr_2CuO_2(CO_3)$ - AN ELECTRON MICROSCOPY STUDY
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The compound $Sr_2CuO_2(CO_3)$ has a layered structure based on that of perovskite lattice (a_p= 3.9 A); it consists of lamellae with the intra-lamellar stacking sequence: ... - (CuO_2-Sr-CO_3-Sr) - (CuO_2- ... . The lamella thickness is $c_p = 2a_p$; the basic lattice is double perovskite, primitive tetragonal (a_p, a_p, c_p).
Electron microscopy and diffraction investigations revealed three intensity levels of the diffraction spots (associated with three sublattices in reciprocal space); they can be related to the levels in the crystal structure: the basic structure, the modulated -Sr-CuO_2-Sr- block-layer structure, and the superstructure due to the CO_3 planar arrangement, (O. Milat, G. Van Tendeloo, S. Amelinckx, T.G.N. Babu, C. Greaves, submitted in J. Solid State. Chem.). The unit cell of the modulated block-layer structure is body-centred, while the superlattice cell of the CO_3 planar arrangement is found to be either body-centred or primitive, with the same cell parameters: $2a_p$, $2a_p$, $2c_p$.
The appearance of two types of superlattice cells can be accounted for by a model of the layered arrangement of CO_3-triangles between two enclosing block-layers (fig.1). A sense can be attributed to the arrangement of the CO_3-triangles and two opposite senses may coexist in the successive CO_3-layers within block-layers of the same underlying structure. The Sr-displacement modulation of the block layer structure is a consequence of the mutual interaction between the basic perovskite, and CO_3 planar substructures. Of the different superlattice variants, two special cases were frequently observed: the body-centred superlattice variant, where the CO_3-arrangements in all layers are of the same sense and the primitive superlattice variant, where the CO_3-arrangements of opposite senses alternate in successive layers. More complicated regular stackings (with a long period superlattice: $c_S = nc_p$) were also observed, and result in closely spaced satellites around the primitive superlattice spot positions (fig.2). An irregular stacking of the CO_3-layers induces streaking of the corresponding superlattice spots in the diffraction patterns.

All superlattice variants of the CO_3-arrangements are observed to be very susceptible to the disordering due to electron irradiation damage; first the sense of the CO_3-arrangement is lost and the superlattice variants disappear, the structure retains the symmetry of the body-centred modulated block-layer. Finally, since the displacement modulation in a block-layer is coupled to the degree of order in the CO_3 layers, it will also tend to disappear and eventually the higher symmetry of the basic perovskite structure will be achieved.

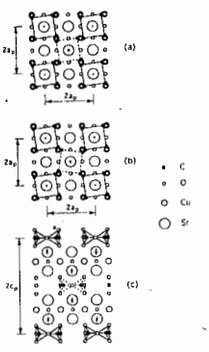

Fig.1.

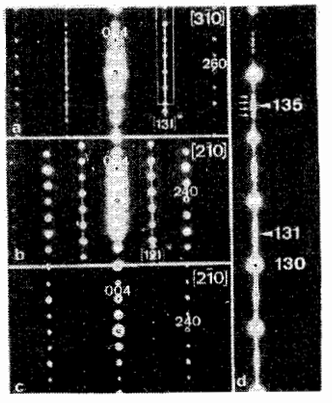

Fig.2.

Fig.1. Schematic of the two substructures in the $Sr_2CuO_2(CO_3)$ unit cells: (a) and (b) projections along [001] for the body-centred and primitive superlattice, respectively; (c) projection along [100] - the same for both cells. CO_3-layers are emphasized by C-C and C-O bonds.
Fig.2. Electron diffraction patterns of $Sr_2CuO_2(CO_3)$ along: (a) [310] zone; (b) and (c) [210] zone before and after irradiation, respectively; (d) enlargement of $[13l]^*$ row in (a).

13.02 – Characterization of Materials by Topography and High Resolution Diffractometry

MS-13.02.01 RECENT DEVELOPMENTS IN PLANE WAVE X-RAY TOPOGRAPHY By T. Ishikawa*, Department of Applied Physics, University of Tokyo, Japan.

Extremely high brilliance as well as sharp collimation in vertical direction of synchrotron radiation give a sufficient intensity of incoming beam of high resolution x-ray topography even after conditioned by perfect crystal x-ray optics for high collimation and high monochromatization. Plane wave x-ray topography, which originated in laboratory source as a version of double-crystal topography with asymmetric collimator, becomes much more easily accessible by using synchrotron radiation, because there are no vibration and heat source near the diffractometer, in addition to the high intensity of the incoming beam. At the Photon Factory, a multi-axis diffractometer for precision topography and high-resolution scattering was developed. Large area of the sample crystals, more than 50×50 mm^2, can be simultaneously exposed by expanding the narrow synchrotron beam vertically by using a single asymmetric collimator. Plane wave topography using high order reflections makes it possible to observe various inhomogeneities in state-of-art silicon crystals in as grown state. The white spectrum of synchrotron radiation makes the commercially available crystal plates such as (001) or (111) oriented as good asymmetric collimators for various reflections by a suitable selection of the wavelength. Successive use of asymmetric collimator can make the angular collimation of incident beam less than 1/200 of the intrinsic diffraction width. With this incident beam, oscillatory profiles in Laue case rocking curve were clearly observed. The small angular period of oscillation gives corresponding contrasts to minute strain fields in highly perfect silicon, which are known as equal-inclination fringes. Wavelength tunability of synchrotron radiation is useful not only for obtaining appropriate asymmetric collimator, but for realizing extremely asymmetric reflection at sample crystal. This enables us to make selective observation of crystal quality in the surface vicinity, as well as the localized strain field near the hetero-interfaces and thin epitaxial layers. Plane wave topography was expanded by adding an analyzer crystal after the sample crystal. By this, spatial mapping of dilation and inclination of lattice planes became possible.

MS-13.02.02 TOPOGRAPHY OF THE DIFFUSE SCATTERING CLOSE TO BRAGG PEAKS. By Paul F. Fewster, Philips Research Laboratories, Cross Oak Lane, Redhill, Surrey, U.K.

Diffuse scattering close to the Bragg condition arises from crystal imperfections. To interpret this scattering many theoretical models have been proposed to extract information on microdefects. With diffractometry alone there are uncertainties in the origin of this diffuse scattering due to instrumental artifacts and other defects of lesser interest. This paper discusses the importance of the instrumental probe and how a "δ-function type" probe (Fewster, J. Appl. Cryst. 1991, 24, 178-183) for topography gives an unanbiguous interpretation of the diffuse scattering. The scattered intensity distribution has been mapped to very high resolution in the vicinity of the Bragg peaks of good quality GaAs, device quality Si and nominally "dislocation-free" Ge substrate crystals. Topography has been used to show that the majority of the scattering emanates from surface damage or dislocations and not point defects or thermal diffuse scattering (TDS). These latter two contributors give rise to a very weak background intensity in the most highly perfect crystals.

A nominally "dislocation-free" Ge crystal was shown to have a very weak background intensity, 10^{-4} of 333 Bragg peak at 15"arc away. With this high resolution probe the relative strain and tilt associated with each image is obtained and the swamping influence of the Bragg scattering is minimised. This method has enabled the characterisation of the diffuse scattering and provided a rapid analysis of surface damage.

MS-13.02.03 IN SITU SYNCHROTRON TOPOGRAPHY STUDIES OF FERROELASTIC DOMAIN STRUCTURE AND PHASE TRANSITION IN LANTHANUM PENTAPHOSPHATE. By Z.W.Hu*, X.R. Huang, S.S. Jiang, and D. Feng, National Laboratory of Solid State Microstructures, Nanjing University, P.R. China

The ferroelastic domain structure and phase transition in Lanthanum Pentaphosphate(LaP_5O_{14}) have been studied. A set of normal ferroelastic domains, zigzag domains, growth bands, and growth sector boundaries are observed by white-beam synchrotron radiation X-ray topography(WBSRXT) and the domain walls are found to vanish and reappear with characteristics of a classical second-order phase transition on real time synchrotron topographs on cycling the sample between room temperature and above the transition temperature(T_c). However, a fluctuation of the number of domains or domain walls with temperature approaching T_c, is revealed and shown to be reversible by real time WBSRXT. An interpretation of this interesting phenomenon and theoretical analysis of the domain structure are presented, respectively, in terms of the classical Landau theory and group theory.