13-Defects, Microstructures andTextures

13.01 - Electron Microscopy of Defects, Microstructures and Textures

MS-13.01.02 Structural Study of Carbon Nanotubes
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This talk covers an introduction of carbon nanotubes which we discovered (Iijima, Nature, 354, 56(1991)), and some latest developments in nanotube studies. The materials draw much attention among many solid state scientists in physics, chemistry and materials science in various aspects because of their unusual size dependent properties. The tubules, studied by HRTEM and electron microdiffraction, are only a less than a few nanometers in diameter, and each tube consists of a few nesting cylinders of graphic carbon sheets. On each graphic tubule the carbon atoms hexagons form in a helical fashion about the needle axis. Electron diffraction patterns taken from individual nano-scale tubules are a main technique available for the structural studies. The technique was extended to observe single cylinder tubes with a diameter of around one nanometer. It emphasizes that we deal with electron scattering from a single atomic sheet with a nano-scale size.

Tubule morphologies were affected by occurrence of pentagons and heptagons in the hexagon sheet tubes (Iijima et al., Nature, 365, 776(1992)). The latter introduces negative gaussian curvature into a hexagon sheet network and causes various graphic structures (Mackay & Tormena, Nature, 352, 762(1991)). Non-hexagon rings play an important role in tubule growth and lead us to propose an open-end growth mechanism (Iijima et al., Phys. Rev. Lett., 69, 7100(1992)).

Other subjects include oxidation and capacitivity of the nanotubes (Ajayan & Iijima, Nature, 361, 333(1993)) which provide interesting crystallographic problems occurring specifically in a nanospace.

MS-13.01.03 INVESTIGATION OF APFEROIC SOLIDS RELATED TO THE ARCHIMEDEAN SPIRAL BY I.A. Bursill, Telur, J. Mod. Phys., 45, 219-226, 1960. These include clino-scheelite, pellogite (a clay) and cyllindrite is sulphide.

Further examples of synthetic materials related to normal crystals via conformal transformations, are found in sulphide catalyst powders as well as some derivatives of graphitized carbon, including nanotube variants.

Some recent results of high resolution electron microscopic studies of this family of aperiodic solids are presented.

MS-13.01.04 STRUCTURAL ANALYSIS BY DIFFUSE SCATTERING. By Yimeiu Zhu.
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YBa2Cu3O9 superconductors undergo a structural transition (from twin to tweed) when oxygen levels are sufficient depleted or a small fraction of the Cu atoms is replaced by certain tri-
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valent cations. The three-dimensional weak modulation, with components along the <110> and [001] directions, has been systematically studied by transmission electron microscopy in YBa2Cu3O7-x (M=Fe, Co or Al; x ≈ 0.025). We observed the streaking and splitting of both fundamental and satellite diffraction spots in selected-area diffraction mode and nano-diffraction mode, respectively. Careful analysis of the diffuse scattering patterns suggests that the <110> modulation can be attributed to pseudo-periodic domains with <110> twinning displacement, while the [001] modulation reflects the stacking of such domains on top each other along the c-axis. The size of the domain (or the period of the modulation) decreases with increasing dopant concentration. Intensity calculations of the twin images and diffuse scattering using a displacement wave model and Monte Carlo simulation show good agreement with the observations. The presence of off-stoichiometric oxygen, or variation in local orthorhombicity, was found to be responsible for such structural modulation.

Discussions and collaborations with Z.X. Cai, J. Tafso, M. Suenaga, and J.M. Cowley are gratefully acknowledged. This research was supported by U.S. Department of Energy under contract DE-AC02-76H000106.

MS-13.01.05 COHERENT CONVERGENT-BEAM ELECTRON DIFFRACTION

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Convergent-Beam Electron Diffraction (CBED) is a powerful technique to identify the symmetries of crystals and the characters of lattice defects. The explanations of the experimental results in these cases are based on the assumption that incident electrons falling on a specimen are incoherent. When a high coherence electron source (i.e. Field-Emission Gun; FEG) is used, interference fringes produced in the overlapping regions of CBED disks help to observe the interference fringes using a STEM microscope, although the fringes were distorted by the aberration of the probe forming lens. By using an Ultra-tomography microscope, 19(1988)31 observed the interference fringes in SiC as well as the overlapings of CBED disks. This group observed the fringe splitting of a lattice spacing 15 Å of 6H-SiC without the distortion using a new TEM microscope. The interference fringes showed a shift of a half period due to a glide plane. The resolution of the fringes 15 Å is rather large for applying this technique to other materials. We have obtained similar interference fringes of smaller lattice spacings of PbS, Sr3NiO4, Sr5IrO6, Lu7In5O12, Mo2O3 and TiO2(Brookite) using electron microscopes JEM2010F and JEM100CX-FEG equipped with a FEG. 

The minimum lattice spacing, from which we could obtain the interference fringes (right figure), is 2.7 Å or the lattice spacing of the 200 reflection of FeS2. The vertical fringes A corresponding to the spacing 3.4 Å are produced by the interference between the 020 and the 020 reflections. The horizontal fringes B corresponding to the spacing 2.7 Å are due to the interference between the 002 and the 020 reflections. 

MS-3.01.06 HIGH SPATIAL RESOLUTION OF DIFFRACTION INFORMATION IN LARGE-ANGLE CONVERGENT-BEAM ELECTRON DIFFRACTION PATTERNS FROM CROSS-SECTONAL SPECIMENS OF Ge,Si/ /Si STRAINED-LAYER SUPERLATTICES

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Fine diffraction lines in large-angle convergent-beam electron diffraction (LACBED) patterns were used to investigate the strain in a lattice misfit system because they are sensitive to small changes of the spacing between lattice planes. Since the probe is slightly defocused, equivalent to imaging the specimen with a convergent spherical wave, a low magnification X-ray diffraction plane of the microscope is superimposed on a high spatial resolution shadow-image of the specimen. In the case of cross-sectional specimens of Ge,Si/ /Si strained-layer superlattices (SLS) LACBED can give much information on local strain and misfit stress relaxation. It is important that the probe size, which defines the spatial resolution of the shadow-image, should be as small as possible. The elastic relaxation of strain in the very thin samples used for cross-sectional transmission electron microscopy (XTEM) must be taken into account. The diffraction lines in the GeSi layers are shifted from their position in the Si layers by amount determined by the misfit strain and the subsequent relaxation. We shall demonstrate that the line shifts in XTEM specimen caused by the effects of misfit strain and stress relaxation can be separated. This technique can therefore be used to profile strain and misfit stress relaxation in SLS.

PS-3.01.07 THEORY OF SPIRAL CRYSTALLOGRAPHY

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Spiral structures exist widely in nature: in both biological and inorganic materials. One of the most common examples occurs in molluscs species where the structure exhibits alternating visoral spirals known as parastichies. The latter always have numbers given by a Fibonacci series. Apart from their present day genetic origin, these types of spiral lattices were also obtained by close packing of pyramidal discs, just as the hexagonal pattern can be obtained by close packing of equal size discs and T. Pusil and T. Yama. Lettis. B2. 1265-1252(1998).

In this paper a generalized abstract model is developed, based on the mechanism of the close-packing model, to enable the critical role played by the initial conditions and the growth law to be analyzed. This model describes the growing disc by a one-dimensional single peak centro-symmetrical function f(θ). The spiral lattice aggregates are thus generalized as a multi-peak function P(i) obtained by adding f(θ) at the global minimum of the existing aggregate P(i)(θ). Both functions f(θ) and P(i)(θ) have the same periodicity, reflecting the angular divergence parameter of the spiral lattice. The periodicity may be limited, leading to the divergence interval, defined as the distance between successive global minima, may be chosen to be equal to the golden mean. This result agrees of course with our earlier graphical computer simulations.

A kinematic approach is attempted and its corresponding configuration of the aggregate is expressed as a function of the initial conditions in a general way. To this stage, the initial conditions, required for convergence, have been found within certain limits.

Such results are essential for the study of spiral growth in general; a way at a time. At this stage, the initial conditions, required for convergence, have been found within certain limits.