13-Defects, Microstructures and Textures

13.01 - Electron Microscopy of Defects, Microstructures and Textures

MS-13.01.01 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 attentions 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 HREM and electron microdiffraction, are only a less than few nanometers in diameter, and each needle consists of a few nesting cylinders of graphitic carbon sheets. On each graphitic tubule the carbon atom 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,**356**,776(1992)). The latter introduces negative gaussian curvature into a hexagon sheet network and causes various graphitic structures (Mackay & Terrones, 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**,3100(1992)).

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

MS-13.01.02 "GRAPHENE TEXTURES : TUBULES AND WHISKERS RELATED TO FULLERENE CRYSTALLOGRAPHY"

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Since the discovery of the fullerenes with their "master" molecules C60 and C70 , a wealth of scientific research has been devoted to these pure carbon molecules, structures, and textures, so much different from the well-known graphite and diamond-based structures and materials.
The present review will report crystallographic aspects derived by electron microscopy , electron diffraction and also HREM of the growth and structure of graphite tubules and of conical graphite whiskers in relation with the fullerenes.

Crystallography and electron diffraction study of tubules.
During the preparation of C60 and C70 sometimes the soot contains other carbon based products such as graphitic tubules, spheres, onions with possibly fullerene related structural features or textures. S.Iijima [Nature 354 (1991) 56] reported the existence and a study by electron microscopy and high resolution imaging of the tubules which he interpreted as coaxial seamless cylindrical tubes . D. Ugarte [Nature 359 (1992) 707] also reported a HREM-study and growth considerations of tubules and onion textures.

A thorough study by electron diffraction of these tubules enabled further interpretation on the construction and growth characteristics. [X.F. Zhang, X.B. Zhang et al. to appear in J.Crystal Growth (1993)]. From reciprocal space considerations of the tubule texture, diffraction features are predicted and compared with the diffraction patterns obtained under various orientations of the tubule axis with respect to the incident beam. It is found that the tubules consist of a complex succession of concentric tubes of cylindrically bent graphene layers with various but discrete helicities containing also non-helical members. The helicity can be related with the seamless closure of the graphene layers into tubes and a growth mechanism could be proposed. The image features in high resolution in particular concerning the nonhelical tubules could be confirmed by simulation of suitably modeled texture proposals.

Helical graphite whiskers
Conically structured graphite whiskers found under similar conditions of formation as those for fullerene containing soot present unusual features in the electron diffraction patterns taken along the whisker axis. [S.Amelinckx et al. J.Crystal Growth 121 (1992) 543] The fragments of the easily cleaved whiskers have conically shaped ends with an obtuse top and reentrant bottom angle of about 140°. The electron diffraction patterns of disc shaped thin fragments exhibit circular rings of equispaced spots. The spacing is variable but discrete, depending on the type of whisker. A growth mechanism is proposed whereby the initial graphene layer adopts a slitted dome configuration obtained by inserting a fivefold carbon ring in the sixfold network. Successive sheets are rotated with respect to the previous ones over a constant angle, thus realising a helical cone around a "disclination" with a fivefold carbon ring core. This model explains the morphology and the diffraction effects of these columnar graphite crystals. The growth mechanism has a direct relationship to the formation of the "bucky ball" molecules.

MS-13.01.03 HRTEM STUDY OF APERIODIC SOLIDS RELATED TO THE ARCHIMEDIAN SPIRAL

by L.A. Bursill, Peng JuLin and Fan XuDong School of Physics, University of Melbourne Parkville, VIC 3052, Australia.

The concept of spiral lattice was applied to some known mineral structures by Bursill, Inter. J. Mod. Phys. B4, 2197-2216, 1990. These include clino-asbestos, halloysite (a clay) and cylindrite (a sulphide). Further examples of synthetic curved structures, related to normal crystals via conformal transformations, are found in sulphide catalyst particles 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 ELECTRON DIFFUSE SCATTERING.

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$YBa_2Cu_3O_{7-\delta}$ 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-

