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The Electron Microscopic Approach to the Structure and Reactivities of Organic Solids

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Our knowledge of the microstructure of inorganic solids, particularly metals and alloys, has been derived chiefly as a result of the application of the technique of transmission electron microscopy (TEM). Appropriate theoretical methods for interpreting diffraction contrast features have been available since the pioneering studies of Hirsch and co-workers; and gradually over the past two decades increasing attention has been paid to other inorganic, particularly ceramic, materials which have also responded to the well-proven procedures of TEM. Since the early seventies high resolution electron microscopy (HREM) has made its impact in the study of inorganic materials. It has, for example, transformed our ideas on gross non-stoichiometry of metal oxides. It has rendered possible the direct exploration of structure–composition relationships in minerals; and it has thrown much light on the question of non-random disorder and polytypism in layered solids (at the unit cell level). It has also enlarged our understanding of the structure and local composition of various types of heterogeneous catalysts. But what of organic molecular crystals? How much progress has been achieved in similar studies of these solids?

Owing to the ease with which they suffer electron-beam damage, many major difficulties have to be overcome before organic solids in general may be studied electron microscopically using the procedures that have proved so successful for their more robust inorganic analogues. Moreover, organic molecular crystals are structurally and compositionally more complicated, and their crystal symmetries are as a rule low, so that even at the level of interpreting diffraction patterns progress is slow compared to

what may be achieved with simpler inorganic crystals. Nevertheless, by adopting appropriate experimental procedures (e.g. utilization of liquid-helium-cooled-double-tilt-stage microscope and recording techniques[1]) almost all organic solids, including those that melt below room temperature are amenable to modern TEM. Diffraction contrast procedures prove invaluable and most of the information retrieved to date has come from this method. HREM, for exceptional materials (notably the metal-free- and metal-chlorophthalocyanines as well as, recently,[2] anthranthrene and tetracene) has also been of value.

With the prospect that nearly all organic solids are within reach of study by TEM it is appropriate that we review the kind of information which may now be experimentally extracted. One of the great merits of the technique is its ability to yield microdiffraction data which have lead to the discovery of several new phenomena.[3–5] The lecture will illustrate, by reference to both diffraction contrast and high resolution studies, how:

i) the crystal structure of low-temperature or stress-induced phases (e.g. of pyrene and anthracene respectively) may, by joint electron microscopic and computational methods, be established;

ii) coexistent phases and polymorphism may be identified under conditions not hitherto attainable;

iii) structural imperfections, of linear or planar kinds, may be identified and characterized (microtwinning, coincidence lattices and the occurrence of new types of defects with irrational displacement vectors); and

iv) solid-state dimerizations (of anthracene and some of its derivatives) and solid-state polymerizations of diolefines may be monitored.

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