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MICROSCOPIC CHARACTERIZATION OF LAYER STACKING IN PYROLYTIC GRAPHITE

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ABSTRACT

This paper is a review of methods for revealing and describing structural characteristics of vapor deposited carbons which can be observed on surfaces which intersect successively deposited layers. Several features of the layer stacking morphology have important effects on thermal and mechanical properties. Following the analogy of a "stack of wrinkled sheets" suggested by annealing behavior, relative angles of tilt between planes on a layer and gaps or angular deviations between layers in a stack should be important. Certain characteristics analogous to such features in the model can be specified quantitatively from images obtained by normal or polarized light, electron transmission through replicas or thin sections, and displays of the current generated by a scanning electron beam probe.

Polarized light microscopy, although primarily used for classifying deposits qualitatively, has been used for limited quantitative comparisons between structure and properties. A quantitative description of some continuously nucleated deposits is possible in terms of the nuclei dimensions and separation calculated from the shape of hyperbolic boundaries generated during growth.

Rapid characterization of deposits without polarized light can be conducted by simultaneous polishing and etching of sectioned surfaces heated in a flame at 1700°C or above. At such temperatures, the pits caused by impurity catalysis at lower temperatures are not formed; unless contaminated, the flame polished surface is more resistant to subsequent oxidation at temperatures as high as 1000°C. Preferential attack occurs at cracks, including those formed by surface damage during grinding. The growth cones which are nucleated by fibrils ("whiskers") which grow with conical layers are also preferentially etched. At high magnifications, discontinuities between atomic planes, which are not revealed by ion bombardment etching, are etched by the flame. In annealed deposits, parallel crystals having different orientations are attacked at different rates.

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Translucent thin sections perpendicular to the layers which are suitable for electron transmission microscopy may also be obtained by flame polishing. Studies of pyrolytic carbons deposited or annealed at different temperatures show several significant features. Flat, parallel, low-density areas are separated by a distance (100-300 Angstroms) similar to the L_c from electron diffraction line broadening in some deposits. However, greater distances of parallelism (about 1000 Angstroms) are indicated by the length of tilt boundary diffraction fringes. Heat treated or hot worked samples show bands of differing contrast corresponding to graphitized areas or parallel crystals in the stack; dislocations and various defects in annealed deposits are also revealed.