OPTICAL AND ELECTRON MICROSCOPY OF CARBONACEOUS MATERIALS*

by

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ABSTRACT

Methods have been developed for the preparation of carbonaceous materials for optical and electron microscopy. These differ, in some respects, from those known to be employed at other laboratories. Impregnation with an epoxy resin is essential to the preservation of the pore structures and for the study of interfaces. A commercial resin is used which withstands ion bombardment etching well and which also withstands the solvents used in replication for electron microscopy. A different resin, having lower viscosity and a longer pot life impregnates some high density graphites more thoroughly, but has unsatisfactory solvent resistance. A double mounting technique is used for the examination of powders in order to examine particles from all parts of the size spectrum.

Grinding is performed in the usual way, except for the employment of 600-soft as the final step. Alumina abrasives are used for coarse and fine polishing on rotary laps. It has not been found possible to obtain as high a degree of flatness on polycrystalline graphites with vibratory polishing, but this method is satisfactory for pyrocarbons.

Cathodic vacuum etching is used to delineate microstructural features. For optical microscopy, atomic hydrogen ion bombardment is used because it produces a higher degree of optical activity than that found on a polished surface or one etched in other gases tried. For electron microscopy, etching with krypton ions is employed because of the finer detail which is produced. With either gas, a potential drop of about 3 kv is used. The current used with hydrogen is about 4 ma, while that used with krypton is about 1 ma. With hydrogen, a chamber pressure of 30 to 40 millitorr is satisfactory, while a pressure of around 5 millitorr is more suitable with krypton. Etching times commonly vary from 15 minutes to 1 hour, depending on the condition of the sample and the features it is desired to emphasize. An aluminum mask with a grounding wire is used in contact with the specimen because it permits the etching of selected areas, it reduces contamination of the vacuum system by decomposition products of the mounting resin, and because it permits the cathodic vacuum etching of powders dispersed in the epoxy resin.

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For electron microscopy, two-stage, platinum-shadowed carbon replicas are used. An 0.034 mm acetyl cellulose film is used for the first stage of replication for most work. For extremely rough surfaces, an 0.125 mm replicating tape is used to avoid difficulties in stripping the thinner film. The thinner film is used whenever possible, due to the sounder secondary carbon replicas obtained therefrom.

A variety of raw materials and finished products have been examined. Two microstructural features which are common to many of these are termed scaly and lamellar. Evidence is presented to support the belief that these are different aspects of the same structure.

Carbon blacks added to binder in experimental graphites can be identified by means of electron microscopy; this is also an aid in identifying residues from such binders. The term optical domain is introduced and its usefulness is discussed. Structures observed in graphite precipitated from metallic solutions are shown and related to some observed in commercial graphite. Twin-like structures are observed in some graphites deformed at either room or elevated temperature.