HIGH RESOLUTION ELECTRON MICROSCOPY OF SMALL CRYSTALLITE CARBONS

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High resolution electron microscopy has been employed to study microstructural variations in a number of small crystallite (e.g. $<80\text{Å}$) carbons. The types of samples studied include carbon blacks chars, cokes and pyrolized polymers. Differences in crystallite size and orientation among the carbons studied have been resolved by means of diffracted beam electron microscopy. By using tilted illumination, high resolution dark field images may be produced from both the (002) and (10) reflections, the former being most suitable because of its higher intensity and three dimensional nature. Point resolution is below 10A, thus enabling imaging of individual crystallites. In this respect the diffracted beam method is considerably better than conventional bright field electron microscopy and even offers certain advantages over x-ray diffraction methods, e.g. diffracted beam micrographs are capable of showing the size distribution, location and general orientation of the crystallites that are imaged. The study of crystallite orientation is sometimes further simplified by the unusual ability to take high resolution stereo micrographs in dark field. These stereo pairs require no specimen manipulation as in conventional electron microscopy and are very simply produced by small changes in the incident angle of the electron beam. Such angular changes must of course occur within the line breadth of the imaging reflection, if the same crystallites are to be resolved on two different micrographs. Yet, despite this apparent severe limitation, surprisingly good vertical separation of crystallite images is often attainable.

A high percentage of the carbons studied represent carbon black pigments which were manufactured by different means. Specifically, numerous samples produced by the Thermal, Channel, Oil Furnace and Acetylene processes were investigated. Differences in crystallite orientation are often readily discernible among commercial carbon blacks, without resorting to the severe thermal and oxidative treatments that were sometimes required in earlier electron microscope investigations. For example, the previously reported concentric orientation of crystallites in thermal blacks has been quite apparent in particles spanning a size range of a few hundred to several thousand Å in size. Oil furnace blacks, on the other hand, are generally characterized by less orientation with diminishing particle size.

The effects of thermal and oxidative treatments have also been studied for a number of carbon blacks and other type carbons which varied greatly in their initial microstructure. Some of the previous considerations of Franklin regarding graphitizing and non-graphitizing carbons have been reinvestigated using diffracted beam electron microscopy.