

# THE MICROSTRUCTURE OF VAPOR DEPOSITED CARBON FILMS

H.S. SHIM, N.K. AGARWAL, A.D. HAUBOLD  
General Atomic Company  
P.O. Box 81608  
San Diego, California 92138

The carbon film thickness ranging from 500 to 1250 Å were deposited onto cellulose acetate at constant pressure ( $3 \times 10^{-2}$  torr) with bias power ranging from 100 to 500W. Free standing carbon films suitable for transmission electron microscopy including electron diffraction were obtained by dissolving the cellulose acetate away.

The electron diffraction pattern of all carbon films studied revealed the weak and broad diffraction peaks super-imposed on a strong background. Broad peaks indicate that the constituent crystallites are extremely small. Strong background is indicative of the presence of a noncrystalline substance that did not contribute to the diffraction peaks. The positions of the peaks were determined by using an optical densitometer and a polycrystalline aluminum standard specimen. The first band was roughly in the (002) angular position of graphite, the second (100), the third (004), the fourth (110), and the fifth (210). Since there were no (h, k, l) type reflection, it is concluded that the observed bands correspond to (002), (10), and (11) reflection of a turbostratic form of carbon (Table I).

TABLE I

Vapor Deposited Carbon		Graphite (a)	
Observed Lines	(b) $S_0$	(h,k,l)	(b) $S_0$
No. 1	1.84	002	1.84
No. 2	2.97	100	2.96
No. 3	3.80	004	3.67
No. 4	5.30	110	5.12
		200	5.91
No. 5	7.60	210	7.81

(a) From Ref. 1,

(b)  $S_0 = (4\pi \sin \theta) / \lambda$ ;  $\lambda = 0.037 \text{ Å}$

The preferred orientation of the crystallites was studied by observing the change in diffraction pattern as specimen tilted in the microscope. The tilt angle, which is the angle between incident electron beam and specimen normal, was varied from 0 to 75 deg. The basic patterns of the carbon films remained unchanged, indicating that the crystallites in the bulk of the films were randomly oriented and thus satisfying Bragg law for a given reflection over the range of tilt angles. If there were a preferred orientation, segmented ring would have been observed instead of continuous rings. The carbon films are therefore isotropic.

In order to estimate the size of crystallites, the background intensity which decreases with increasing diffraction angle was subtracted from the observed (002) peak intensity of one of the specimen so that the reconstructed peak was symmetrical. From the broadening of (002) peak, the crystallite size in the C-direction was calculated

to be about 8 to  $10 \text{ Å}$ .

Transmission electron micrographs of a typical carbon film revealed a homogeneous structure with no distinctive growth features. The structural homogeneity, as well as the measured density of about only 1.7 to 1.8 g/cc and the strong background in the diffraction pattern, indicates that the carbon films consist of somewhat loosely packed components, probably misaligned, single-layered carbons.

The modulation of peaks of carbon films deposited at low and high bias powers, respectively, was weak and about the same with each other, but it was stronger in carbon films deposited at intermediate bias powers. This is not understood at the present time, because of the complex nature of the deposition process. It is however an indication that the structure (e.g., crystallite size) of vapor deposited carbon can be controlled by a suitable choice of deposition parameters.

In conclusion, the vapor deposited carbons can be formed that is similar to LTI pyrolytic carbons available under the trade name of PyroliteR.\* The LTI carbons are used in medical devices requiring a high degree of thromboresistance<sup>(2,3)</sup>. Since the important structural parameters of the vapor deposited carbons are comparable to those of LTI carbons, it is felt that these carbon films will exhibit the same biocompatibility.

This work was supported in part by NHLI (S.D. Bruck, Program Manager).

\* General Atomic Company, San Diego, California.

- (1) Kakinoki, J., et.al., Acta Cryst. 13, 171(1960)
- (2) Bokros, J.C., LaGrange, L.D., Schoen, F.J., in Chemistry and Physics of Carbon, Vol. 9, 103, Dekker, New York (1972).
- (3) Bokros, J.C., and Akins, R.J., in Proceedings of 4th Buhl International Conference on Materials, p. 243, Carnegie Press, Pittsburgh (1971).