Submitted to Journal of the American Ceramic Society

LBL-6227
Preprint

LATTICE IMAGE OF 'LMSC' GLASSY CARBON

S. Bose, U. Dahmen, R. H. Bragg, and
G. Thomas

September 1977

Prepared for the U. S. Department of Energy
under Contract W-7405-ENG-48

DISCLAIMER

LBL-6227

LATTICE IMAGE OF 'LMSC' GLASSY CARBON

S. Bose, U. Dahmen, R. H. Bragg*, and G. Thomas*

Materials and Molecular Research Division, Lawrence Berkeley Laboratory
and Department of Materials Science and Minerals Engineering,
University of California, Berkeley, California 94720

Carbons made by the controlled pyrolysis of thermosetting resins are non-graphitizing as opposed to the soft carbons that graphitize when heat-treated to or above 2000°C. The structure and properties of the non-graphitizing carbons have been the subject of investigation of a large number of workers. Glassy carbon is a representative of the non-graphitizing class.

High resolution electron microscopy has been used to resolve the question concerning the true structure of the so called glassy carbon. Heidenreich, Hess and Ban[1] were the first to utilize this powerful technique on carbon black. Since then Ban and Hess[2], and Jenkins, Kawamura and Ban[3] used lattice imaging to analyze glassy carbon prepared by a technique developed at Swansea (Great Britain) while Phillips[4] successfully lattice imaged glassy carbon made by Polycarbon of California.

We have been investigating the microstructure and properties of glassy carbon made by the LMSC[5] process and have used lattice image in TEM and x-ray analysis to characterize the structure. The results indicate that the glassy carbon studied is similar to that of previous research[1-3]

Glassy carbon heat-treated at 2700°C for 10 hours was ground to a thickness of 2 mils. A 2 mm disc was then cut ultrasonically. This disc was mounted in an ion-mill using Argon gas for about 100 hours

until it was perforated at the center. The disc was examined on a Philips EM 301 electron microscope operated at 100KV. In the phase contrast mode an aperture was used to allow imaging in the two beam '00.0 - 00.2' situation to occur.

Lattice fringes corresponding to the (00.2) planes of graphite were obtained over large thin areas. Micrographs were taken slightly under focus. The lattice images show fringes but with no preferred orientations indicating the isotropic nature of glassy carbon as shown in Fig. 1. The fringe spacing is 3.4A^{o}. Fringes are continuous usually over 50 A^{o}. The fringe pattern resembles the "Jenkins nightmare" model[3], and there are no definite crystallite boundaries. The layers show extensive bending; stacking disorders are encountered at places. The thickness of each packet of layers and the distance of continuity of the fringes parallel to the layers correspond to the two crystallite dimensions conceived by the x-ray investigators. The dimensions parallel and perpendicular to the stacked layers agree quite well with those determined from x-ray diffraction measurements. Selected area electron diffraction shows the absence of (hk.l) ($h, k \neq 0$) reflections indicating turbostratic nature of the structure. The stacked layers bifurcate at places indicating that pores are enclosed among interweaving layers. These pores have been found to give rise to strong small-angle scattering of x-rays[6].

Thus in conclusion the high resolution electron microscopy has clearly shown that "glassy" carbon is not in fact amorphous but is composed of a complex mixture of small crystallites of large aspect ratio having preferred orientations of (00.l) planes parallel to the long axis of the crystallite.

ACKNOWLEDGEMENT

Continued support of the USERDA through the Materials and Molecular Research Division of the Lawrence Berkeley Laboratory is gratefully acknowledged.

REFERENCES

1. R. H. Heidenreich, W. M. Hess and L. L. Ban, "A Test Object and Criteria for High Resolution Electron Microscopy", J. Appl. Cryst. 1,1, 1-19 (1968).

2. L. L. Ban and W. M. Hess, "Microstructure of Carbons: A High Resolution Microscopy Study", 10th Biennial Conf. on Carbon, Lehigh University, SS99, 161-162 (1971).

3. G. M. Jenkins, K. Kawamura, and L. L. Ban, "Formation and Structure of Polymeric Carbons", Proc. Roy. Soc. (Lond) A327, 501-517 (1972).

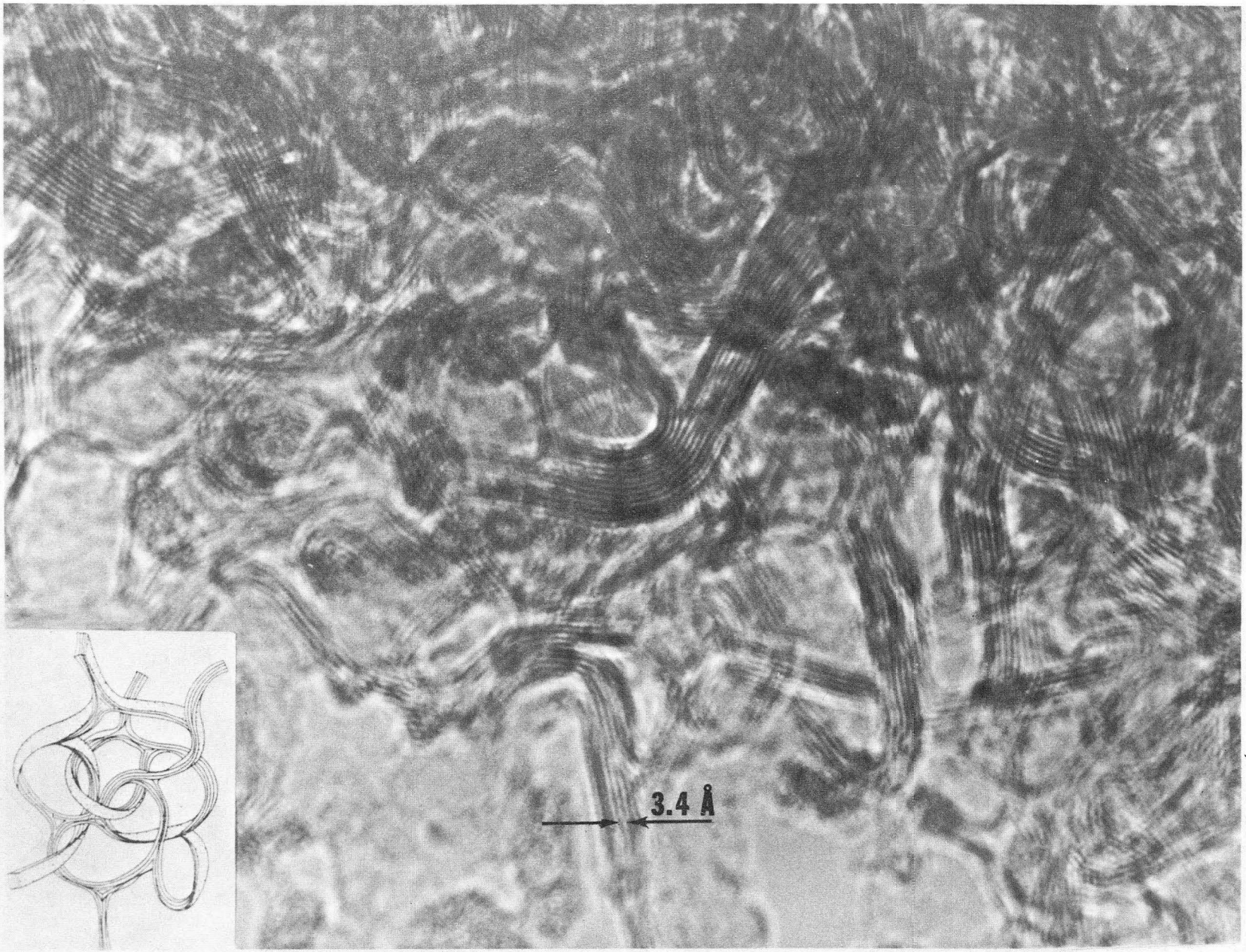
4. V. A. Phillips, "Lattice Structure in 1800°C Processed Vitreous Carbon", Metallography 6, 361-364 (1973).

5. W. Bradshaw, P. Pinoli, G. Watsey, and H. Wigton, 8th Biennial Conf. on Carbon (1967).

6. S. Bose and R. H. Bragg, "Explanation of Unorganized Carbon in Glassy Carbon", Deutsch Keramische Gesellschaft, Carbon '76, 205-207 (1976).

FIGURE CAPTION

Fig. 1. Transmission Electron Micrograph of "glassy" carbon showing 00.1 planes of 3.4A^{o} spacing resolved by lattice imaging techniques 100KV tilted beam illumination. Inset shows the Jenkins model with which the electron micrographs are in excellent agreement.



XBB 778-7454

Fig. 1

TECHNICAL INFORMATION DEPARTMENT
LAWRENCE BERKELEY LABORATORY
UNIVERSITY OF CALIFORNIA
BERKELEY, CALIFORNIA 94720