

X-RAY STUDIES OF GRAPHITIZATION OF GLASSY CARBON

S. Bose, R. Saxena and R. H. Bragg

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

The structure of glassy carbon (GC) can be characterized by the interlayer spacing d_{002} , the crystallite sizes, L_a in the layer planes, L_c normal to the layer planes and the size parameters of the voids enclosed by the graphite like ribbons of the structure. Wide angle x-ray diffraction patterns have been used to determine d_{002} , L_a and L_c while small angle x-ray scattering (SAXS) was used to determine the void parameters. The x-ray diffraction pattern of GC consists of broad peaks near the graphite 002 and hko reflexions. The background intensity is relatively high. In addition there is intense SAXS from the voids. The tail of the SAXS considerably modifies the line profile of (002) reflexion. To determine the structural parameters the background, assumed to be incoherent scattering, was subtracted first. Next, the tail end of the small angle portion which followed a trend $I(2\theta) \propto (\sin\theta)^{-n}$ was subtracted after determining n from small angle region only. Then the Lorentz and polarization corrections were made. Since the interference peaks were broad, a correction for the variation of atomic scattering factor across the peak was also made.

The xray parameters were determined to follow the kinetics of "graphitization" of GC heat treated for various times at temperatures between 1000°C and 2800°C. SAXS data were collected to understand the kinetics of 'void growth'.

The variations of d_{002} , L_a and L_c for isochronal heat treatment are shown in Fig. 1. Three regions can be identified in the variations. Below 1500°C, d_{002} decreases. From 1500°C to 2100°C it remains approximately constant at 3.44Å. Beyond 2100°C, however, d_{002} decreases. L_a , L_c increase within the range of heat treatment.

To determine the activation energy of graphitization, the method of superposition was tried. Fig. 2 shows the variation of L_c as a function of heat treatment time (HTt) at various temperatures (HTT). Fig. 3 shows the composite curve obtained by superposition after translation along the $\ln t$ axis. The curves for 1700°C and 2000°C are mutually superposable but do not overlap the curves for other HTT. This suggests that upto 2000°C there is a slower kinetics. The activation energy derived from curves above 2000°C comes out to be 225 ± 30 K. cal/mole. Attempts to superpose L_a and d_{002} were partially successful.

The kinetics of 'void growth' is represented in Fig. 4. Radius of gyration of the voids determined from SAXS is plotted against HTt for different HTT. It is clear that there is a distinct 'kinetics' associated with the growth of the voids.

REFERENCES

1. R. H. Bragg and M. L. Hammond, Abstracts, Seventh Biennial Conference on Carbon (Cleveland, June 1965); also Carbon 3, 340.
2. Madan Biswal and Robert H. Bragg, Twelfth Biennial Conference on Carbon (Pittsburgh, July 1975), p. 151-3.
3. Fischbach, D. B., Technical Report 32-532, Jet Propulsion Lab., Cal Tech, 1966.

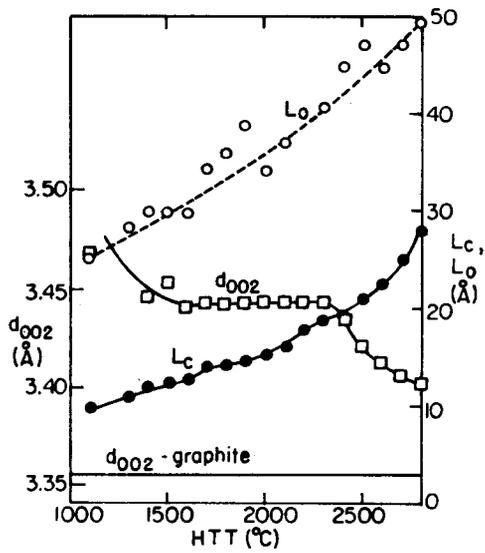


Figure 1

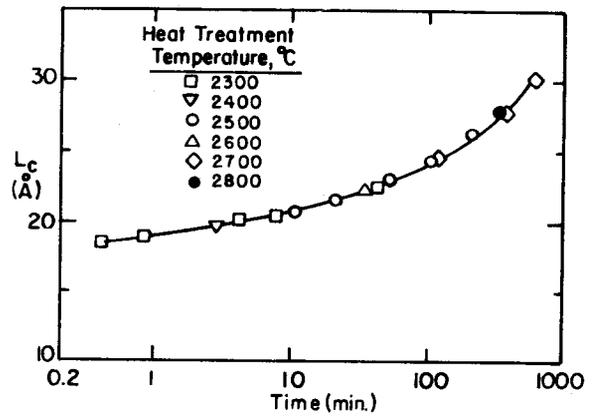


Figure 3

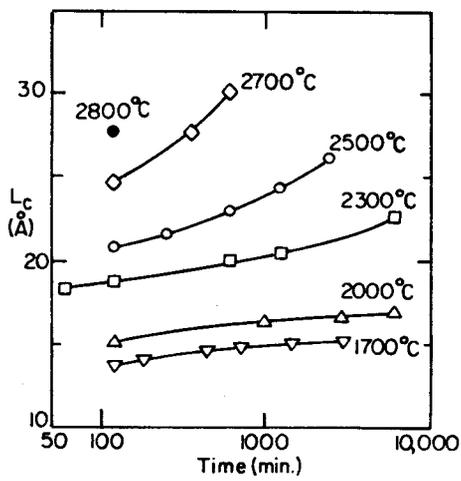
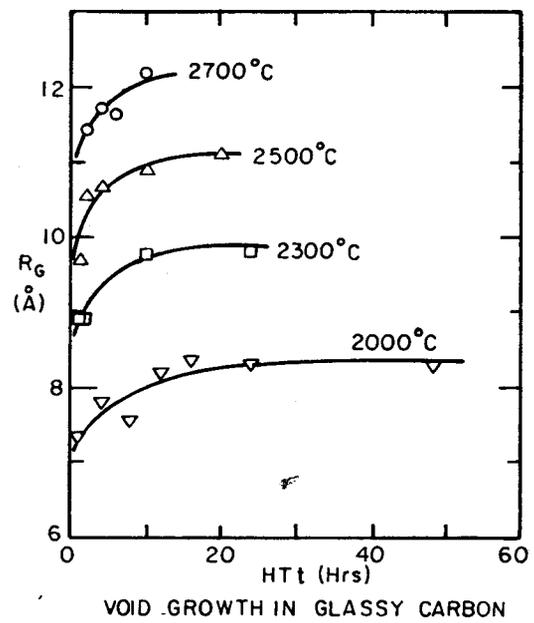


Figure 2



VOID GROWTH IN GLASSY CARBON

Figure 4