CORRECTION FOR COMPTON SCATTERING IN GLASSY-CARBON DIFFRACTION PATTERNS

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ABSTRACT

In a preliminary investigation reported here, it has been found that
the use of Co-Ni balanced filters not only provided better monochromatiza-
tion of CuKα, but is quite suitable for determining the incoherent
(Compton) scattering in two Glassy-Carbon (GC) samples.

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In previous work we have used wide range x-ray diffraction\(^1,2\) or small angle x-ray scattering (SAXS)\(^3\) to characterize the structure of GC. In the latter, the SAXS was measured in transmission and used to calculate the specific surface area, \(S/V\) of samples. Since our measurements showed that at a given temperature the sample volume \(V\) is independent of time, the observed changes in \(S/V\) with HTT and HTt could be used to characterize surface area changes.\(^4\) The relevant equation takes the form

\[
\frac{S}{V} = 4c(1-c) \lim_{h \to 0} [h^3 J(h)] \int_0 \frac{hJ(h)dh}{hJ(h)dh}
\]

or

\[
\frac{S}{V} = \pi c(1-c) \lim_{h \to 0} [h^4 I(h)] \int_0 \frac{h^2 I(h)dh}{h^2 I(h)dh}
\]

depending upon whether the experimental situation employs slit or point\(^5\) collimation. Here, \(c\) = fraction of sample occupied by matter, \(h = 4\pi \sin \theta / \lambda\); \(J(h)\) = intensity for slit collimation; and \(I(h)\) = intensity for point collimation. There has been some uncertainty concerning which approximation is applicable to GC, because instances have been reported wherein the intensity was said to decrease as \(h^{-n}\), where \(n\) might range from 2 to 3.\(^6\) Recent careful measurements have shown that these reports are in error. For the full range of heat treatment temperatures employed in this work, 1000 to 2800°C, the SAXS extends to such large values of \(h\) that our experimental situation always approximates point collimation, for which \(n = 4\).
This result simplifies the correction of our wide range diffraction patterns for SAXS enormously. The corrections for Lorentz-Polarization, atomic scattering factor variations, and distortion due to low specimen absorption are easily made. It appears that the procedures for correcting for Compton scattering that we have used heretofore are adequate for obtaining a fully corrected 002 line profile in GC as shown in Fig. 1. However, we need to determine if the observed narrowing of the widths of x-ray line profiles induced by heat treatment are due to "crystallite" growth or stress relief. To do this, it is necessary that at least three orders of 006 and hk0 reflections be measured in order to separate size and strain components. Since the GC reflections are weak and very diffuse, exact corrections for the large contribution of the Compton component to the total intensity in the regions where the (100), (110) and the (004) reflections occur are mandatory.

In previous work we have assumed that in pyrolytic graphite, PG, the material is of high purity, and sufficiently close to a single crystal that far from a reciprocal lattice point the observed x-ray intensity is due mainly to Compton scattering. Thus if the linear absorption coefficient is µ and the material is t thick, the intensity will be totally caused by Compton radiation, and proportional to \(1/\mu^2\). Thus a separate measurement of background intensity and µ on PG can be used to determine the background level, i.e., the contribution of Compton scattering to the total intensity for a GC sample having the same µ. A paper on this method submitted to Carbon has been returned with the (valid) criticism that this procedure of determining the Compton intensity level should be
justified by comparison with an accepted method. We have decided to utilize a method which is based on the difference of the intensity of a diffracted beam when a filter is placed in the incident beam or in the diffracted beam. Our preliminary estimates of the Compton component obtained in this way differ from that obtained using PG by an order of magnitude. It is felt that the result obtained from PG is more nearly correct, and the low estimate obtained from the differences in absorption are caused by poor monochromatization. We have decided to use balanced filters\textsuperscript{10} to obtain better monochromatization and have just initiated this work.

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References


Figure Caption

Fig. 1 Effect of corrections on the 002 line profile of glassy carbon. (XBL 793-5970).
Fig. 1
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