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K-EMISSION FROM GLASSY CARBON

Based upon X-ray diffraction studies analyzed in terms of intensity and radial distribution functions various models of the structure of glassy carbon have been proposed. Noda and Inagaki\(^1\) interpreted their data in terms of graphitic and disorganized carbon content as evidence for the presence of trigonal and tetrahedral c-c bonds. Kakinoki's\(^2\) model also assumes trigonal and tetrahedral bonds, whereas Furukawa's\(^3\) model contains all kinds of c-c bonds. Other models have also been proposed, and the question is still unsettled.

We have used K-emission to study the bonding character of glassy carbon. K-emission bands are observed when valence band electrons fall into empty K orbitals. The use of K-emission bands to study bonding character is well established.\(^4\) Assuming equal probability of transitions from all parts of the valence band Coulson and Taylor\(^5\) compared their calculated density of states \(N(E)\) with Chalkin's observed K-emission band of graphite. Since then many studies of the graphite K-emission band have been reported. MacFarlane\(^6\) has recently confirmed Coulson and Taylor's prediction of polarizability of \(\pi\) and \(\sigma\) bands in K-band emission of graphite. Both Holliday\(^7\) and MacFarlane analyzed their data as a sum of gaussian and non-gaussian peaks. Holliday concluded that the shift in peak wavelength from diamond to graphite is due to the differences in weights assigned to various gaussian components.

In the work reported here, carbon K-emission bands were measured with a MAC 400S electron microprobe. Second order reflection from a Lead Octadeconate crystal \((2d = 100.5 \text{ Å})\) was used to analyze the X-rays
and the detector was a flow proportional counter using methane gas and 1000 Å polypropylene window. A 15 kV electron beam was used in our experiment. We measured K-emission bands from diamond, pyrolytic graphite (parallel mounted i.e., with c-axis parallel to the electron beam) and three samples of glassy carbon (made by Polycarbon) heat treated at 1000, 1800 and 2800 degrees centigrade for one hour. The data is presented in normalized form in Fig. 1. The shift in peak position from diamond to pyrolytic graphite is clearly seen in the figure. The trend for glassy carbon is as expected, that with higher heat treatment temperature the peak shifts toward the pyrolytic graphite peak.

To facilitate the comparison, we give in Table I peak position, half width, index of asymmetry (defined after Ref. 7 as the ratio of part of the full width at half maximum lying to the long wavelength side of maximum ordinate to that lying on the short wavelength side) and the shift in peak position relative to pyrolytic graphite. Holliday's data on diamond is also given in the table. Our diamond data compares well with Holliday's data. Minor differences of the detail of the emission band may be due to our use of an LOD analyzing crystal as compared to Holliday's use of a grating analyzer.

The peak wavelength of glassy carbon lies in between those of diamond and pyrolytic graphite. This suggests two possibilities: (i) that in glassy carbon the local bonding character around each carbon is intermediate to the two extremes or (ii) that glassy carbon contains both trigonal and tetrahedral c-c bonds. In view of the layered structure observed in high resolution electron microscopy the first possibility is excluded. The shift in peak wavelength of
glassy carbon toward pyrolytic graphite peak with higher heat treatment temperature shows that the amount of tetrahedrally bonded carbon reduces with increasing heat treatment temperature.

The half width of glassy carbon is larger than that of either diamond or pyrolytic graphite and does not change with heat treatment temperature. This also supports the contention that glassy carbon has both diamond and graphite characteristics. The large half width is the result of higher weight assigned to the gaussian component corresponding to the diamond peak in graphite K-emission band. This is better demonstrated in Fig. 2, which shows diamond, pyrolytic graphite and glassy carbon bands superimposed on each other. The index of asymmetry also approaches pyrolytic graphite value with increasing heat treatment temperature. The part of full width at half maximum lying to the long wavelength side of maximum ordinate is approximately constant. This is further evidence that the change in glassy carbon K-emission band with heat treatment temperature is due to reduced weight assigned to the gaussian component corresponding to the diamond peak. Thus reduced amount of tetrahedrally bonded carbon with heat treatment temperature changes the index of asymmetry by reducing part of half width lying to short wavelength side of the maximum ordinate.

We would like to thank Mr. George Georgakopoulos for helping with the measurements. This work was conducted under the auspices of the U. S. Atomic Energy Commission.

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References:


Table I. Comparison of K-emission band parameters for various forms of carbon.

<table>
<thead>
<tr>
<th>Material</th>
<th>Peak Position A</th>
<th>Half Width A (eV)</th>
<th>Index of Asymmetry</th>
<th>Shift in Peak Position from PG Δ(eV)</th>
</tr>
</thead>
<tbody>
<tr>
<td>PG (a)</td>
<td>44.90</td>
<td>1.375 (8.8)</td>
<td>0.75</td>
<td>0</td>
</tr>
<tr>
<td>GC(2700) (b)</td>
<td>44.80</td>
<td>1.5 (9.6)</td>
<td>0.75</td>
<td>0.64</td>
</tr>
<tr>
<td>GC(1800)</td>
<td>44.75</td>
<td>1.5 (9.6)</td>
<td>0.95</td>
<td>0.96</td>
</tr>
<tr>
<td>GC(1000)</td>
<td>44.70</td>
<td>1.5 (9.6)</td>
<td>1.00</td>
<td>1.28</td>
</tr>
<tr>
<td>Diamond</td>
<td>44.45</td>
<td>1.28 (8.0)</td>
<td>1.35</td>
<td>2.90</td>
</tr>
<tr>
<td>Diamond (c)</td>
<td>44.52</td>
<td>(8.1)</td>
<td>1.25</td>
<td>2.10</td>
</tr>
</tbody>
</table>

(a) PG = Pyrolytic Graphite  (c) See Ref. 7
(b) GC = Glassy Carbon
Figure Captions

Fig. 1. Carbon K-emission band, peaks normalized.

1- Diamond  2- Glassy Carbon (1000)
3- Glassy Carbon (1800)  4- Glassy Carbon (2800)
5- Pyrolytic Graphite

Fig. 2. Diamond, Glassy Carbon and Pyrolytic Graphite

K-emission bands superimposed on each other.
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