SURFACE AREAS OF GRAPHITE REGENERATED FROM GRAPHITE OXIDE

by

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INTRODUCTION

Graphite oxide has been known for over one hundred years, and many of its chemical and physical properties have been extensively investigated. A comparatively small amount of work has been undertaken to date, however, studying the thermal decomposition of this carbon compound.

One of the earlier attempts towards elucidating the structure of graphite oxide occurred when Balbiano (1) decomposed a sample of the substance by heat and analyzed the gases for CO, CO₂ and H₂O. His study involved three different types of graphites that were oxidized by two different methods. The purpose of this study was primarily to determine the amount of water tied up by the graphite oxide. The similar results of Kohlschutter and Haenni (12, p. 133) and Selvig and Ratliff (14, p. 129) served to substantiate Balbiano's work.

Much of the inconsistency in the results of the investigations of graphite oxide lies in the difficulty of preparing uniform samples. The substance is very hygroscopic, decomposes readily when heated, and varies
with the nature of graphite used and the method of preparation. It has been found by different investigators (7, p. 562; 10, p. 111) to decompose at temperatures of 150° to 200°C unless the rate of heating was carefully controlled. Kohlschutter and Haenni (12, p. 133) obtained decomposition temperatures varying from 134° to 193°C according to the rapidity of heating. Matuyama (13) was able to heat approximately 0.3-gram samples of graphite oxide at a rate of 42° per hour with a slow rate of decomposition, but when the sample weight was increased 50% an explosion occurred at about 190°C. The rate of 42° per hour was used because it was found to be the maximum rate of heating by which that particular set of samples decomposed without explosion.

Hofmann and coworkers (8) investigated the oxidation of graphite oxide and stated that only H₂O, CO₂ and CO were evolved in the decomposition. Later, however, DeBoer and VanDoorn (6) found that on rapid heating some molecular oxygen is expelled at first. This oxygen is later consumed in reactions to form CO and CO₂.

The black residue from the decomposition is believed to be similar to graphite in its structure, although it still contains some residual oxygen which may be removed by FeSO₄, hydrazine or electrolytic reduction (9). It also can be removed slowly by heating,
in which CO and CO$_2$ are evolved.

DeBoer and VanDoorn (5) oxidized graphites of varying particle sizes by the Staudenmaier method (15). They concluded that all of the different graphites could be oxidized to the constant formula C$_7$H$_4$O$_2$, but found that the samples of larger particle size required a greater number of oxidation steps.

When graphite oxide is heated to the point where explosion takes place, the light, fluffy soot that is produced exhibits the x-ray diffraction pattern of ordinary carbon black. On slower heating they gradually regain the graphite structure by losing step by step the intercalated molecules (13). In some respects these decompositions seem to represent the simplest case of carbonization and graphitization of carbon compounds. Only recently Beckett and Croft (2) reported a slight expansion of the layer planes (based on observations with the electron microscope) to occur in the oxides of graphite.

These investigations mentioned above have been primarily concerned with analyzing the gases evolved from the decomposition, but the black residue that is produced has to a great extent been unquestioned. Therefore it is felt that there is need for a rather comprehensive study of this ash.
Accordingly, a study has been made on a series of graphites of different particle sizes, and the number of oxidation steps has been varied. The surface areas of these ashes were then measured in an attempt to learn about the nature of this peculiar form of graphite.
Sample Preparation

The samples used in this study were prepared from Canadian graphite supplied by the Asbury Graphite Mills, Incorporated, Asbury, New Jersey. The graphite was 99% pure flake and graded 1 through 5 according to size of particles, with number 1 as the coarsest. Grades 1, 3 and 5 were used in this investigation, and no previous treatment was given the graphite before oxidation.

The samples were prepared by a procedure similar to the modified Brodie method (3). Red fuming nitric acid (375 ml) was added to 25 g of graphite in a 1000-ml flask, and the mixture was placed in an ice bath. Potassium chlorate (250 g) was then added at a rate designed to keep the reaction from becoming too violent, roughly a period of about 30 minutes. After the addition of potassium chlorate was completed, the mixture was placed in a water bath and kept at a temperature of from 55-60°C for one hour. During this time nitrogen was bubbled through the mixture at a rate sufficient to keep it well agitated. The nitrogen and residual gases from the reaction were allowed to escape into a hood. Following this, the mixture was diluted with 300 ml of water and centrifuged. After three such wash treatments, the
product was dried at 80°C for a period of 24 hours. At this point one-fifth of the product was removed and the remaining four-fifths was oxidized by the same procedure a second time. The proportions of nitric acid and potassium chlorate are given here for each oxidation step:

Step 1. 25 g graphite, 375 ml HNO₃, 250 g KClO₃
2. 20 g graphite, 300 ml HNO₃, 200 g KClO₃
3. 15 g graphite, 225 ml HNO₃, 150 g KClO₃
4. 10 g graphite, 150 ml HNO₃, 100 g KClO₃
5. 5 g graphite, 75 ml HNO₃, 50 g KClO₃.

Labeling the samples was accomplished by indicating the Canadian graphite number as well as the number of oxidation steps it had undergone. With this notation then, sample C₃B⁵ would describe a sample of Canadian graphite number 3 that had been given 5 oxidation treatments by the modified Brodie method. Grades 1, 3 and 5 were each given from 1 to 5 of these such oxidation steps in this study.

Sample Processing

After the final oxidation the graphite oxide was purified by electrodialyzing it for approximately 48 hours in a Mattson cell using untreated cellophane for membranes. The dialysis was performed with a current
of 50 ma or less and the potential was not allowed to exceed 350 volts. The electrode polarity was reversed periodically to prevent excessive accumulation of material on the membrane at the anode side of the cell. Copper was used for the cathode while platinum was used for the anode. Finally, the sample was again centrifuged and decanted in order to concentrate the suspension following completion of the dialysis.

Thermal decomposition of the samples was carried out in a controlled temperature electric oven. It was possible to attain temperatures up to 350°C, although the samples were adequately decomposed at temperatures below this limit. Approximately 0.4–0.7 g of sample were placed in an 800-ml beaker with a suitable wire screen serving as a cover. The graphite oxide was then heated until combustion was observed to take place; this was possible since the oven was outfitted with a Pyrex window. The sample size indicated above would usually produce from 300–600 ml of light, fluffy carbon residue depending on the type of sample used.

Adsorption Apparatus

The surface areas of the samples were measured; this was done by means of the well known B.E.T. method (4) (Appendix I). Nitrogen was used as the adsorbate, and the
adsorption was measured gravimetrically rather than volumetrically because of the greater simplicity and accuracy of this type of measurement.

A schematic drawing of the adsorption apparatus is shown in Figure 1. The adsorption column consisted of a 5-foot-long 25 mm diameter Pyrex tube, closed at the upper end and sealed into a 4-foot length of 38 mm tubing in order to form a water jacket for temperature control of the springs. The 25 mm tube extended below the jacket about 10 inches and ended in a ball joint. A 12 inch piece of 25 mm tubing, which enclosed the sample, was sealed at this ball joint with Apiezon "W" grease. The system contained three such adsorption columns.

A copper-beryllium spring was suspended from the top of each column. These springs were copper beryllium alloy 25 ASTM 194 obtained from the Beryllium-Copper Supply Company. Hooked to the lower end of the spring was a thin glass rod marked at intervals to serve as reference points when measuring the spring extensions. The sample containers were hooked to the lower end of this marked rod.

The sample containers were small buckets made from 0.0005 inch tin foil. They were 10 mm in diameter and weighed approximately 350 mg. It was found that buckets
Figure I. Gravimetric adsorption apparatus
of this size and weight were necessary to contain the 70-90 mg of the light, fluffy material required for good isotherm studies.

The change in weight of the sample on adsorption was determined by measuring the extension of the spring. This was done with a cathetometer sighted on one of the markings on the above mentioned glass fibre. The amount of deflection was read on a vernier scale on the side of the cathetometer. This scale could be read to ± 0.1 mm, or approximately ± 0.2 mg since the sensitivities of the springs were around 2.0 mg/mm.

The three springs were held at a temperature of 31.8 ± 0.1°C by pumping water from a thermostatted reservoir through the water jackets. The reservoir was heated by a 250-watt heater controlled through an electronic relay by a bimetallic thermoregulator.

The system was evacuated through a dry ice cold trap by a Welch Douseal forepump and a two stage mercury diffusion pump. Under ideal conditions, a pressure as low as $10^{-4}$ mm Hg was obtained, but a pressure as great as $10^{-3}$ mm was considered sufficient as a working pressure. The pressures in this region were measured with a McLeod gauge. Adsorbate gas pressures in the working range of the isotherms were measured by means of a mercury manometer, and the pressures here were read to the
nearest 0.2 mm.

The lower portion of the adsorption column containing the sample was immersed in liquid nitrogen in a Dewar flask. No problem was encountered in maintaining the nitrogen at constant temperature since an isotherm could be completed in a short period of time.

The nitrogen was purified before use by passing it over hot copper turnings prior to admitting it to the storage flask. This served to remove any trace of oxygen that might be present. Any other foreign gases such as water vapors were removed by means of the liquid nitrogen cold trap at the base of the storage flask. A balloon was attached to the purification train to prevent the gas pressure from becoming at any time greater than atmospheric pressure and blowing out the stopcocks. An indication of the amount of gas remaining in the reservoir flask at any given time could be determined by the height of the column in the mercury bubbler.

Procedure

After the graphite oxide had been decomposed in the oven, 60-90 mg portions of the ash were weighed out into the tin buckets. It was not necessary to maintain the samples in any special environment prior to weighing, for they showed no change in weight on being exposed to
the atmosphere. The buckets were then suspended from the marked glass rods hanging from the springs in the adsorption columns. The tubes were sealed and the evacuation of air began.

The pressure was lowered to 1 mm Hg over a period of ½ hour. Then the pressure was lowered to 0.1/1 or less, and outgassing was continued for 2 days to sufficiently remove any gas adsorbed on the sample. At the completion of this period, the sample chambers were submerged in the liquid nitrogen bath and the isotherms were determined.
Discussion and Results

While preparing the samples from the three different graphites, it was noted that the same general color changes occurred during the 5 oxidation treatments. The black graphite was converted to the dark green graphite oxide after the first oxidation, and the product gradually changed through the light green, straw yellow and light brown colors with the completion of the fifth oxidation step. These color changes indicate an increasing oxygen to carbon ratio with each treatment, for it is generally accepted that there is a correlation between color and composition of the graphite oxide. One investigator even goes so far as to give the range of carbon to oxygen ratios present for the various colors of the oxide (11).

The first attempt to decompose the samples was performed by placing them in the oven at room temperature and raising the temperature to 240°C over a period of about 3 hours. This method resulted in the thermal decomposition of about half of the samples. Not one of the five products of Canadian No. 1 graphite was decomposed by this heat treatment, yet it was possible to decompose these same samples by placing them in the oven.
preheated to 300°C. This is consistent with the results of Matuyama (13) who stated that the rate of heating is a determining factor in the thermal decomposition of graphite oxide.

Of the 5 products of Canadian No. 5 graphite, only $C^5_B-1$ would not decompose within the 240°C range. It did ignite and burn at 300°C, however. This would suggest that $C^5_B-1$ was insufficiently oxidized, that is, its oxygen to carbon ratio was too low to be thermally decomposed with the slow rate of heating.

Two different sets of samples were prepared using Canadian No. 3 graphite. The results here were not consistent, for one preparation of $C^3_B-2$ was found to burn within the 240°C range while another preparation of $C^3_B-2$ required a temperature of 300°C for ignition. This same inconsistency was also found with two different preparations of $C^3_B-5$. No explanation can be found for this except that the amounts used may have been a decisive factor. It was observed that of two different portions of the same $C^3_B-4$ sample, one decomposed in the 240°C range while the other would not. Again, the only feasible explanation lies in the quantity of sample used, for the amount used was in 0.4–0.7 g range but was not accurately weighed out.

Since the oven was constructed with a Pyrex
window, it was possible to observe the decomposition of
the samples. Of particular note was the set of Canadian
No. 1 graphite oxide preparations, all of which were
burned in the oven preheated to 300°C. Sample \( C^1_B - 1 \)
expanded to a volume of about 30 times its original size,
then burned slowly from top to bottom over a 5 second
period. Sample \( C^1_B - 2 \) gave similar results, but burned in
one second. Sample \( C^1_B - 3 \) reacted almost spontaneously
while \( C^1_B - 4 \) and \( C^1_B - 5 \) were explosively decomposed. These
reaction rates are evidence of the greater oxygen to
carbon ratio in the samples with a greater number of
oxidation treatments.

Water was used as the adsorbate in the first
attempt to produce an adsorption isotherm on the re-
generated graphite samples, but this resulted in failure
as the water did not measureably adsorb. \( n \)-Hexane was
found to give a good isotherm, yet this proved to be un-
feasible as the area per molecule of the hexane was not
adequately known.

Nitrogen was tried as the adsorbate and gave good
results. The area of the nitrogen molecule is generally
accepted as being 16.2 \( \text{Å}^2 \) and this is the value that was
used in this study. Another advantage of using nitrogen
is that the equilibrium time of about 10 minutes allowed
the entire isotherm to be completed within a 2-3 hour
period; this reduced errors such as air leaking into the system and changes in the temperature of the liquid nitrogen bath. The B.E.T. functions produced by the adsorption isotherms gave good results in the working range of 0.05 to 0.35 relative pressure (Figure 2). (See Appendix II for typical data and calculations.)

The surface areas of the regenerated graphites are shown in Figure 3 and Table 1. The increasing curve produced by Canadian No. 1 graphite can be easily explained. Since each successive oxidation increases the oxygen content of the graphite oxide, and the decomposition process removed this oxygen as $CO_2$, one would expect a continually increasing destruction of the graphite lattice and hence a progressively greater surface area to result.

Both sets of Canadian No. 3 graphite showed a continual increase in surface area except for the second oxidation step. No explanation can be found for this or for the reduced surface area determined for sample $C_5^5$-5. What is more significant, however, is the general increase in surface areas with decreasing particle size of the original graphites. Thus we see that Canadian No. 1 gives lesser surface areas than the corresponding samples of Canadian No. 3 which in turn gives results somewhat below the values obtained for Canadian No. 5.
B.E.T. PLOT of N\textsubscript{2} on \( C_B^3 - 4 \) at -196°C

FIGURE 2
Oxidation Steps

Figure 3. Variation of surface area with oxidation steps
<table>
<thead>
<tr>
<th>Oxidation Steps</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
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<tr>
<td>Canadian Graphite No. 1</td>
<td>78</td>
<td>264</td>
<td>335</td>
<td>355</td>
<td>362</td>
</tr>
<tr>
<td></td>
<td>87</td>
<td>262</td>
<td>332</td>
<td>367</td>
<td>376</td>
</tr>
<tr>
<td>Canadian Graphite No. 3</td>
<td>264</td>
<td>543</td>
<td>368</td>
<td>453</td>
<td>494</td>
</tr>
<tr>
<td></td>
<td>256</td>
<td>543</td>
<td>365</td>
<td>429</td>
<td>491</td>
</tr>
<tr>
<td>Canadian Graphite No. 5</td>
<td>571</td>
<td>446</td>
<td>484</td>
<td>535</td>
<td>544</td>
</tr>
<tr>
<td></td>
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<td></td>
<td>601</td>
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<td>Canadian Graphite No. 5</td>
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<td>545</td>
<td>612</td>
<td>490</td>
</tr>
<tr>
<td></td>
<td>498</td>
<td>566</td>
<td>563</td>
<td>606</td>
<td>487</td>
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</table>
This seems to indicate that the smaller particles contain a greater oxygen to carbon ratio.

Another look at Figure 3 shows that samples \(C^3_B\), 4 and 5 and most of the highly oxidized samples of Canadian No. 3 exhibit areas considerably below the value determined for sample \(C^5_B\). This suggests that most of the oxidation sites on the graphite oxide are located at the surface of the particle, and that even prolonged oxidation does little to penetrate the lattice structure of the graphite. If the interior of the particles contained a higher concentration of oxygen, we would expect greater surface areas to result. The leveling off of the Canadian No. 1 curve also suggests that little interior oxidation is accomplished after the second oxidation.

The temperature at which the graphite oxide is decomposed is an influential factor in determining the area of the regenerated graphite. A sample of \(C^3_B\) that had been given the slow heat treatment up to 240°C gave an area of 490 m\(^2\)/gram while another portion of the same sample produced an area of 610 m\(^2\)/gram when burned in the oven at 300°C. Similar results were noted with sample \(C^5_B\) which showed an increase of approximately 40 m\(^2\)/gram when decomposed at 300°C.

The differing values in surface areas produced
by the two different sets of Canadian No. 3 samples illustrates the difficulty involved in obtaining reproducible samples in a study of this type. Both sets of samples were produced under identical circumstances and no explanation can account for this deviation. More important though, these two curves show the same general shape. One of the $C^3_B$-1 samples was lost shortly after dialysis, consequently no area was recorded.

**Further Work Suggested by This Study**

A further extension of this work should include a comparative study of the surface areas of these samples decomposed at 300°C and at another higher temperature such as 400°C. An investigation of this type would serve to indicate to what extent the decomposition rate affects the destruction of the lattice of the graphite oxide.

Another proposal worthy of consideration would be an electron microscope or x-ray study of the graphite lattice in the decomposed product. This would help in determining to what degree the interior of the graphite particle is oxidized. It might also contribute some information on the general topic of graphitization of carbons.
SUMMARY

The graphite oxide samples in this investigation were prepared from original graphites of three different particle sizes by the modified Brodie method. Each of the different graphites was given from 1 to 5 of these treatments after which the colloidal suspension was cleaned of extraneous salts by electrodialysis. Following this the oxidized samples were decomposed in an electric oven under controlled temperature which produced a light, fluffy graphite residue.

The surface areas of the resulting ash were then measured in a gravimetric system by means of the B.E.T. adsorption isotherm method with nitrogen as the adsorbate. These B.E.T. plots of the data resulted in good straight lines and gave reproducible answers.

The graphite residues from the decomposition process gave surface areas ranging from 90 m²/gram to 650 m²/gram. These values were found to be dependent on the particle size of the original graphite, the number of oxidation treatments, and the rate of heating as well as the amount of sample used in the decomposition process.
BIBLIOGRAPHY


APPENDIX I

B.E.T. Adsorption Theory

The theory of Brunauer, Emmett and Teller (4) is based on the assumption that the same forces that produce condensation are also chiefly responsible for the binding energy of multimolecular adsorption. The heats of adsorption of gas in the second, third, ..., etc., layers are taken to be the same as the heat of liquefaction for the gas. If the adsorption takes place on a free surface, then at $P^o$, the saturation pressure of the gas, an infinite number of layers can build up on the adsorbent. The equation is

$$\frac{P}{W(P^o - P)} = \frac{1}{W_m C} + \frac{(C-1)P}{W_m CP^o}$$

where $W_m$ is the volume of gas adsorbed when the entire adsorbent surface is covered with a complete unimolecular layer and $C$ is a constant equal to

$$a_1b_2/b_1a_2 \cdot e^{(E_1 - E_L)/RT}$$

in which $E_1$ is the heat of adsorption on the first layer, $E_L$ is the heat of condensation, and $a$ and $b$ are rate constants. A plot of $P/W(P^o - P)$ versus $P/P^o$ gives a straight line with a slope of $(C-1)/W_mC$ and an intercept of $1/W_mC$, from which $W_m$ can be calculated.
APPENDIX II

Representative Data Collected for Surface Area Measurements

Data collected for measurement of sample $C_{B}^{3-4}$:

<table>
<thead>
<tr>
<th>Time</th>
<th>Sample Reading (cm)</th>
<th>P Reading (cm Hg)</th>
<th>Wt. Ads. (mg)</th>
<th>W (mg/g)</th>
<th>$P/W(P^o-P)$</th>
<th>$P/P^o$</th>
</tr>
</thead>
<tbody>
<tr>
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<td>75.77</td>
<td>0.00</td>
<td></td>
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<tr>
<td>10:15</td>
<td>75.10</td>
<td>3.40</td>
<td>14.74</td>
<td>138.7</td>
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<td>0.045</td>
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<tr>
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<td>7.70</td>
<td>16.06</td>
<td>151.1</td>
<td>0.000746</td>
<td>0.101</td>
</tr>
<tr>
<td>10:45</td>
<td>74.99</td>
<td>11.50</td>
<td>17.16</td>
<td>161.4</td>
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<td>0.151</td>
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<tr>
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<td>21.34</td>
<td>200.8</td>
<td>0.00215</td>
<td>0.301</td>
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<td>11:45</td>
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<td>26.65</td>
<td>22.88</td>
<td>215.2</td>
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<td>0.351</td>
</tr>
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*Sample Weight = 0.1063 g
*Spring Constant = 22.0 mg/cm

A plot of $P/W(P^o-P)$ versus $P/P^o$ for this data is shown in Figure 2, page 17. The slope was found to be $7.04 \times 10^{-3}$ g/mg and the intercept shows a value of $4 \times 10^{-5}$ g/mg. The weight of adsorbate required to form a monolayer on the surface can be calculated from $W_m = (\text{slope} + \text{intercept})^{-1}$, in this case giving a result of 141.2 mg/g adsorbent. The surface area is then determined as follows:

$$(141.2 \text{ mg/g})(16.2 \text{ Å}^2/\text{molecule})(6.023 \times 10^{23} \text{ molecules/mole})(0.001 \text{ g/mg})(1/28.02 \text{ mole/g})(1 \times 10^{-20} \text{ m}^2/\text{Å}^2) = 492 \text{ m}^2/\text{g}$$

The area of the adsorbed $N_2$ molecule, assuming
close packing, is calculated by the equation

\[ \text{area per molecule} = 4(0.866)(M/4\sqrt{2}AD)^{2/3} = 16.2 \text{ \AA}^2 \]

in which \( M \) is the molecular weight of the adsorbate, \( A \) is the Avogadro number, and \( D \) is the density in a condensed liquid state.