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CRYSTALLOGRAPHIC DATA ON SELECTED ARTIFICIAL GRAPHITES WITH COMMENTS ON THE ROLE OF THE DEGREE OF CRYSTAL DEVELOPMENT IN OXIDATION

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SUMMARY

Crystallographic parameters have been measured by X-ray diffraction on 40 grades of commercially available, nonpyrolytic, artificial graphite (that is, industrially manufactured graphite consisting of filler and binder). The parameters measured are the lattice dimensions (a and d_c), and the crystallite diameter and height. Since artificial graphite is composed of a mixture of two or more graphitic carbons, the measured crystallographic parameters are apparent only and are truly representative of neither the filler nor the binder. However, the measured interlayer spacing of artificial graphite is a good approximation of that of the filler.

Literature data on a number of graphitic carbons have been correlated, relating crystallite diameter and crystallite height to interlayer spacing. Based on the first of these correlations, experimental support is offered for the proposal that the a-dimension of graphitic carbon is invariant. Accordingly, the crystal development of a graphitic carbon is specified by its interlayer spacing alone. On the basis of this result, the crystallographic characteristics of artificial graphite can be adequately described: the measured interlayer spacing of the artificial graphite specifies the crystal development of the filler, and the fact that the binder is less graphitized than the filler qualitatively specifies the crystal development of the binder. For artificial graphites containing more than one type of filler, the binder contacting each type of filler can be reasonably assumed to be less graphitized than the filler, and of the two most common filler carbons in artificial graphite, petroleum coke should be more graphitized than carbon black.

A survey of the literature indicates that the less graphitic a carbon, the less resistant the carbon is to oxidation. For this reason the binder in artificial graphite should be locally more reactive than the filler. Therefore, the aerodynamic erosion resistance of artificial graphite may be largely dependent on the binder, particularly in high-shear environments in which filler particles may be mechanically removed because of preferential oxidation of the binder matrix.

INTRODUCTION

Artificial graphite is a material of considerable interest for aerospace applications because of its desirable structural and thermal properties at high temperatures. Its use is limited, however, because it erodes rapidly in the high-temperature oxidizing environments encountered in some aerospace applications. Furthermore, this erosion rate may differ markedly between different artificial graphites in the same aerodynamic environment. (See, for example, ref. 1.)

Aerodynamic shear, in addition to oxidation, possibly contributes to this high erosion rate, as well as to the observed differences in erosion rate.[1] Therefore certain physical, as well as chemical properties of artificial graphite, acting either individually or in complex interaction with each other, may contribute to or set limits on this erosion rate. The specific properties which are the most important have not yet been established.

One of these properties may be the degree of crystal development. Considerable literature evidence discussed later in this report indicates that the degree of crystal development of a graphitic carbon or artificial graphite[2] has an important effect on its oxidation rate. However, the extent, or even the existence, of such an effect on the total erosion rate of artificial graphite in aerospace environments has not been established. Moreover, detailed crystallographic data on a wide variety of artificial graphites are lacking. Such data provide a necessary foundation for an investigation of the possible effect of crystal development on the erosion rate of artificial graphite under aerospace conditions. The purpose of this report is to present some data of this type and to develop a concept of the crystallographic characteristics of artificial graphite useful for such an investigation.

In this report are presented the results of X-ray crystallographic measurements on 40 selected grades of commercially available, nonpyrolytic, artificial graphite. Correlations of literature data on a number of graphitic carbons are developed relating crystallite diameter and crystallite height to interlayer spacing, and in view of these correlations, the crystallographic measurements on the various artificial graphites are discussed. Also included in this report is a summary of the important literature indicating a dependence of the oxidation rate of graphitic carbons and artificial graphites on the degree of

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[1] "Erosion" is used throughout this report in the broad sense to include all mechanisms of mass removal and is to be carefully distinguished from mass removal due to oxidation alone.

[2] A "graphitic carbon" is defined as a carbon with at least some development of a polycrystalline graphitic structure, whereas "artificial graphite" is defined as the conventional, industrially manufactured graphite composed of filler and binder (that is, composed of two or more graphitic carbons).

their crystal developments. Implications of this dependence are noted regarding the resistance of artificial graphite to erosion caused by aerodynamic shear.

SYMBOLS

a	unit cell dimension in a graphite crystal basal plane layer, angstroms
d_c	interlayer spacing or one-half the graphite crystal unit cell height, angstroms
(hkl)	Miller indices where h, k, and l are integers
$K\alpha$	α doublet of the K series X-radiation of an element
L_a	mean crystallite diameter, angstroms
L_c	mean crystallite height or thickness, angstroms
$L_{(hkl)}$	mean crystallite dimension determined from reflecting plane indicated by Miller indices in subscript, angstroms
p	probability of adjacent graphite layers being disordered
$\beta_{1/2}$	diffraction breadth of an X-ray reflection after correction for experimental broadening, measured at one-half peak intensity as actually indicated by the goniometer, that is, in terms twice those in which the Bragg angle is measured, degrees
Δa	apparent shift in the a-dimension from the true value, angstroms
θ	Bragg angle, degrees
λ	wavelength of X-radiation, angstroms

EXPERIMENTAL MEASUREMENT OF CRYSTALLOGRAPHIC PARAMETERS

The 40 grades of commercially available, nonpyrolytic, artificial graphite investigated in this study are listed in table I. Also listed in this table are the filler material, density, and maximum grain size of each of the grades as furnished by the manufacturers at the time of purchase. All grades were graphitized above 2500° C with the possible exception of AHDG. For all grades coal tar pitch was used as the binder with the exception of AHDG which has a resin binder (ref. 2), and with the possible exception of those grades listed in table I as proprietary.

The crystallographic parameters of interest – interlayer spacing, a-dimension, crystallite height, and crystallite diameter – were determined by conventional X-ray diffraction techniques. All raw X-ray data were generated by Speer Carbon Company Research Laboratory on samples furnished by the National Aeronautics and Space Administration. Three samples of each grade were furnished, all three of which were drawn from the same block of graphite. (In a very few cases, the three samples were drawn from different blocks, but in all cases all three samples were from the same batch.)

Each sample was ground to pass a 200-mesh sieve and admixed with $NaCl$ as an internal standard also ground to pass a 200-mesh sieve. Diffraction data were obtained with a diffractometer employing Ni-filtered Cu (unresolved) radiation. Intensity data were recorded for the (110) and (004) reflections, except for those graphites with too diffuse a (004) reflection, in which case the (002) reflection was recorded. Further details of the experimental procedure are given in reference 3.

The raw data were interpreted by the author. Lattice dimensions, a and d_c, were determined from the peaks of the (110) and $(00l)$ reflections, respectively, by using 1.5418 Å for the wavelength of the Cu Kα radiation. Crystallite dimensions, L_a and L_c, were determined from the half-maximum diffraction breadths of the (110) and $(00l)$ reflections, respectively, by using the Scherrer equation $L_{(hkl)} = \frac{0.89\lambda}{\beta_{1/2} \cos \theta}$. Instrument broadening and doublet separation were corrected by standard methods. (See refs. 4 and 5.)

RESULTS AND DISCUSSION

Presented in the last four columns of table I are the crystallite parameters measured for each of the 40 grades of graphite of this investigation. Each value is an average of the determinations made on the three separate samples. Except where noted, all values of interlayer spacing and crystallite height are based on the (004) lattice reflections. Because of distortion broadening (ref. 6), crystallite heights based on the (004) reflections usually are smaller than crystallite heights based on the (002) reflections. Limited data on artificial graphite in reference 7 indicate the ratio $L_{c(002)}/L_{c(004)}$ to be on the order of $1\frac{1}{2}$ to 2.

Significance of Measured Crystallite Parameters

Artificial graphite is composed of filler and binder carbons intimately mixed and graphitized as one body. Therefore, the observed X-ray lattice reflections from which the crystallite parameters are determined are composites of the lattice reflections of each of the constituent graphitic carbons. Since most artificial graphites contain on the order of only 15 percent binder carbon in the final graphitized body (refs. 8 to 10), the locations of the peaks of the composite reflections largely indicate the locations of the peaks of the filler carbon. (See refs. 8 and 11.) Therefore, the measured lattice dimensions, a and d_c, largely indicate the lattice dimensions of the filler. However, because of the broadening of the lattice reflections due to the multicomponent nature of artificial graphite, the measured crystallite dimensions, L_a and L_c, are smaller than the crystallite dimensions of the filler, and bear little relation to the crystallite dimensions of the binder. (See ref. 11.) Graphitic carbons do not present this difficulty since they are not a mixture of different carbons. Published X-ray diffraction data for graphitic carbons are discussed in the following section.

Crystal Structure of Graphitic Carbons

In reference 12 it was proposed that the crystallite diameter L_a and the interlayer spacing d_c can be correlated for a variety of graphitic carbons by the equation $L_a = 9.5(d_c - 3.354)^{-1}$. However, in developing this correlation, which was formulated directly in terms of degree-of-graphitization data taken from several literature sources, the authors in reference 12 overlooked the fact that different definitions of degree of graphitization were used in several of their data sources. (Although "degree of graphitization" is customarily defined in terms of d_c (degree of graphitization increases as d_c decreases), the proper relation between degree of graphitization and d_c is not completely agreed upon.) The ambiguity in reference 12 can, however, be removed by correlating L_a directly in terms of d_c, and thereby correcting the correlation given in reference 12. Accordingly, direct experimental values of d_c were obtained from references 12 to 14 (the same data sources used by ref. 12), and approximate values of d_c were obtained from reference 15 (also used by ref. 12) by applying the equation developed by the author of reference 15 in an earlier paper (ref. 16) to relate d_c to degree of graphitization. The resulting correlation, which was developed from the data by the method of least squares, is given by the equation $L_a = 7.4(d_c - 3.354)^{-1}$ and is shown in figure 1 along with the correlation from reference 12 for comparison. The data represented are for graphitized polyvinyl chloride coke (refs. 12 and 13), petroleum coke (refs. 12 and 14), coal tar coke (ref. 12), and carbon black (refs. 12 and 15). In view of the severe difficulties encountered in measuring crystallite dimensions with accuracy and precision (refs. 4 and 17), the scatter in the data is not excessive.

These same data sources with the exception of reference 12 and the addition of reference 18 were used to develop a similar correlation between interlayer spacing d_c and crystallite height L_c. This correlation, given by the equation $L_c \approx 32.4(d_c - 3.354)^{-1/2}$, is shown in figure 2. Together, these two correlations permit the crystallite dimensions of a graphitic carbon to be estimated solely from a knowledge of interlayer spacing.

The severe difficulties encountered in accurately measuring crystallite dimensions, as well as possible subtle differences in the experimental techniques of the different data sources used in developing these correlations probably account for much of the scatter in the correlations. It is apparent, however, that systematic increases in L_a and L_c accompany a decrease in d_c, and although it may conceivably be possible to realize small changes in any of these crystallographic parameters without correspondingly changing the others, such changes apparently must be limited. The present correlations should, in this sense, be considered provisional, but are, nevertheless, useful interpretations of existing data.

In order to describe the crystal structure of a graphitic carbon more completely, the lattice parameter a must be known in addition to the interlayer spacing d_c and the crystallite dimensions L_a and L_c. It has been proposed (refs. 19 and 20) that

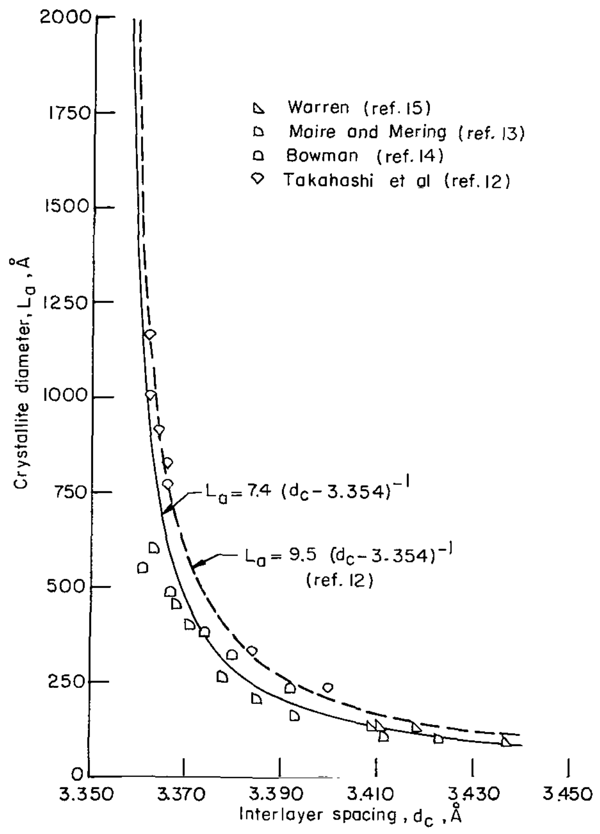

Figure 1.- Correlation of crystallite diameter and interlayer spacing for graphitic carbon.

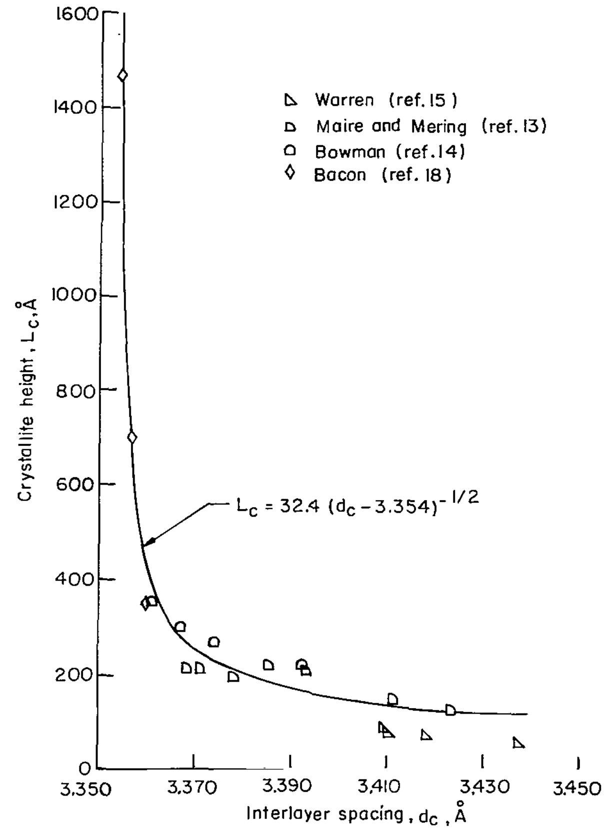

Figure 2.- Correlation of crystallite height and interlayer spacing for graphitic carbon.

customary experimental determinations of this lattice parameter are in error because of the neglect of certain corrections, and further, that the true a-dimension is constant in spite of the fact that the measured values appear to be variable. Support for this proposal can be given by using the developed correlation between L_a and d_c and the lattice dimension data for the artificial graphites of this investigation. A figure presented in reference 20 showing a plot of Δa against p for a family of L_a which was deduced theoretically on the assumption that the true a-dimension is constant, was modified to produce a plot of apparent (experimentally observed) a-dimension against d_c for a family of L_a, as shown in figure 3. This modification entails accepting 2.4614 Å as the true value of the a-dimension (ref. 18), determining d_c from p by the equation $d_c = 3.440 - 0.086\left(1 - p^2\right)$ (ref. 6), and using the relationship $\Delta a = -a \dfrac{0.16\lambda}{L_a \sin\theta}$ (refs. 19 and 21) valid at $d_c = 3.44$ Å for interpolation between and extension of the family of curves presented in reference 20. The previously developed crystallite diameter correlation, $L_a = 7.4\left(d_c - 3.354\right)^{-1}$, was then graphically combined with this modified theoretical family of curves in figure 3 and, as a result, a unique relationship between the apparent a-dimension and d_c was obtained. If the theoretical proposal in reference 20 is correct, that is, if the a-dimension is indeed constant, this unique relationship should correlate experimentally observed a-dimension and d_c data. That this is the case is seen from figure 4 in which apparent a-dimension and d_c data from table I are plotted in comparison with a portion of the derived curve taken from figure 3. The dashed lines

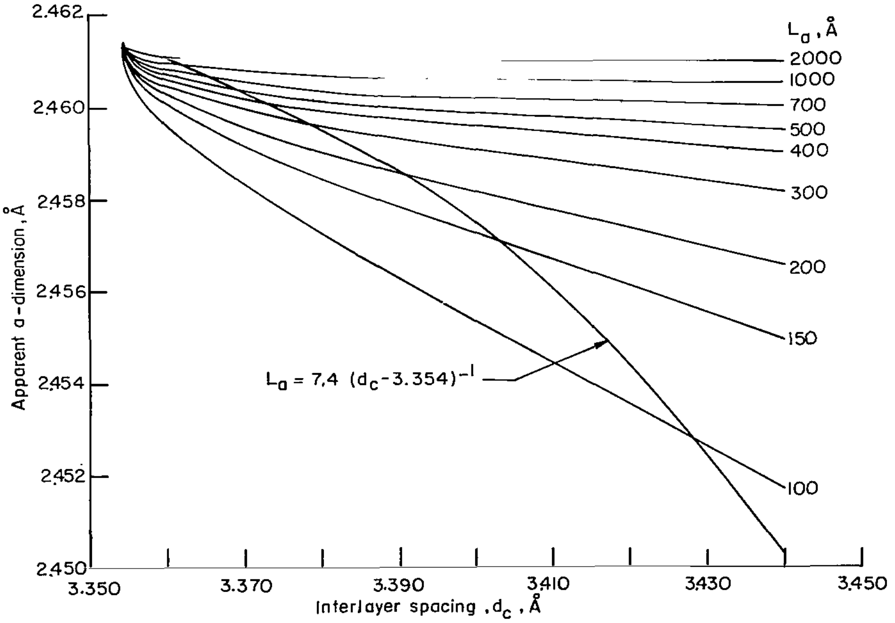

Figure 3.- Modified theoretical family of curves from reference 20 showing the graphical solution with the crystallite diameter correlation.

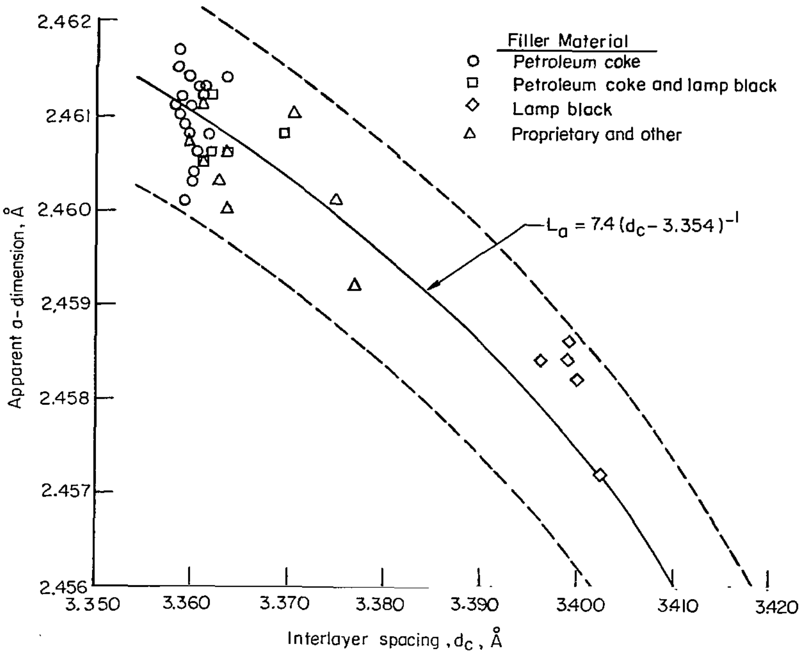

Figure 4.- Experimental data on apparent a-spacing as a function of interlayer spacing for artificial graphite compared with the derived relation between a-spacing and interlayer spacing.

indicate precision error bounds so that if a data point lies between them, the probable (root-mean-square) error bounds of that data point include the solid correlating curve. The success with which the derived curve correlates the data suggests that the true a-dimension is indeed constant, and that 2.4614 Å is a good value.

Crystallographic Characteristics of Artificial Graphite

As discussed previously, the measured crystallite dimensions of artificial graphite do not represent the true crystallite dimensions of either the filler or the binder. Nevertheless, it is common practice to measure such apparent crystallite dimensions, primarily in order to follow the relative degree of graphitization of a body at successive stages in the graphitization process. Furthermore, by using the simplified procedure described in the experimental measurement section, they are easily determined from the same diffraction peaks used to determine the lattice dimensions.

Such apparent crystallite dimensions for the 40 grades of artificial graphite under study are shown in table I. An interesting comparison can be made between these apparent crystallite dimensions and the crystallite dimensions predicted by the correlation curve developed for graphitic carbons. This comparison is shown in figures 5 and 6. Although the data appear to follow the correlation fairly well, this result must be considered fortuitous because of the possible errors involved, that is, because of the

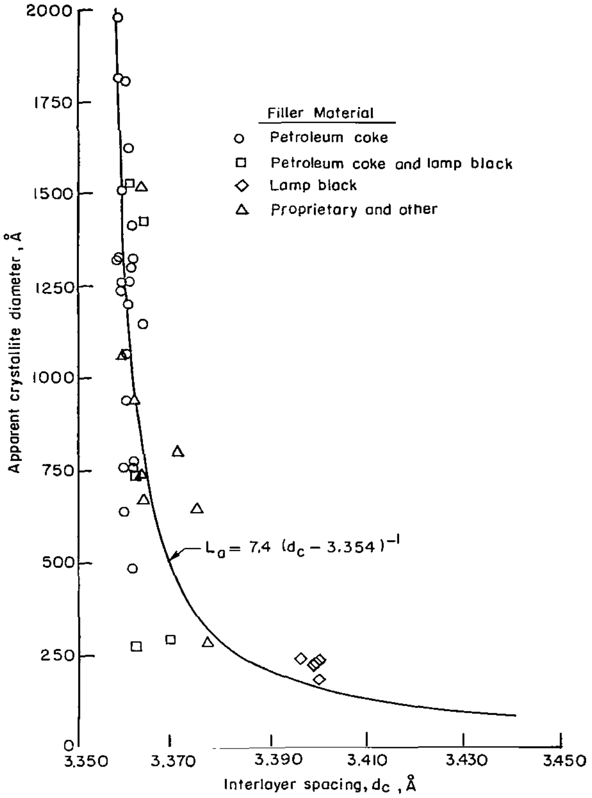

Figure 5.- Experimental data on apparent crystallite diameter as a function of apparent interlayer spacing for artificial graphite compared with the correlation for graphitic carbon.

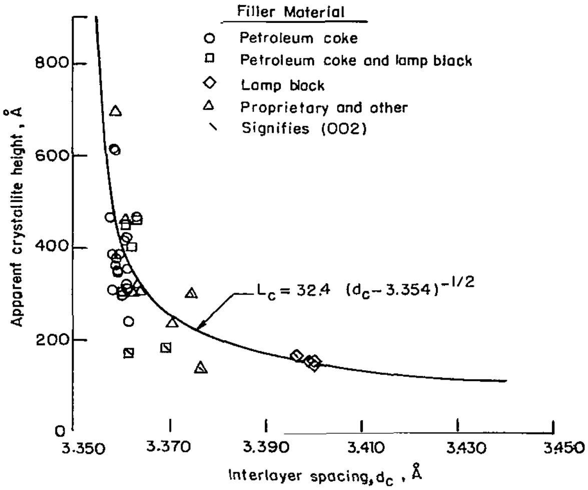

Figure 6.- Experimental data on apparent crystallite height as a function of apparent interlayer spacing for artificial graphite compared with the correlation for graphitic carbon.

approximate method used to determine the crystallite dimensions, and because no fundamental physical significance can be ascribed to such crystallite dimensions since they were calculated from X-ray data taken on what is, in fact, a mixture of graphitic carbons. The misleading nature of these data can be demonstrated from figures 5 and 6 by reference to the data for petroleum coke. These data indicate the possibility of a sizable change in either one of the crystallite dimensions for a constant interlayer spacing. This misleading result arises because, as previously stated, the measured interlayer spacing of the artificial graphite body approximates that of the petroleum coke filler alone, whereas the measured crystallite dimensions of the body (determined from the experimental X-ray line breadths) depend on the binder (both on the percent and on the degree of graphitization) as well as on the filler.

An adequate description of the crystallographic characteristics of artificial graphite can, however, be developed. In view of the foregoing discussion on graphitic carbon, the crystallite diameter and height of graphitic carbon are specified by its interlayer spacing alone, and its a-dimension is a constant. Accordingly, since the measured interlayer

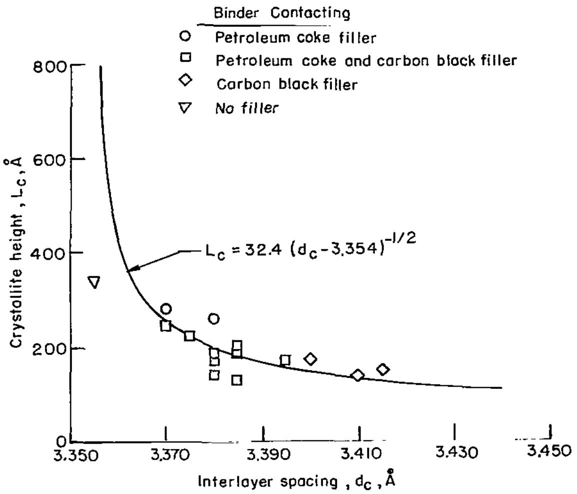

Figure 7.- Data from reference 11 on crystallite height as a function of interlayer spacing for binder pitch in contact with various fillers compared with the correlation for graphitic carbon.

spacing of an artificial graphite body closely approximates the interlayer spacing of its filler, the crystal development of the filler is approximately described solely by the measured interlayer spacing of the artificial graphite body.

The problem remains, then, how to describe the crystal development of the binder. Reference 11 presents experimental results indicating that binder pitch is less graphitizable in the presence of filler than it is alone, and, in addition, that it graphitizes to a lesser degree than the filler, whether the filler be petroleum coke or carbon black. This effect was reasoned (ref. 11) to be due to the binder coating the filler particles with a thin film during coking and thereby geometrically hindering binder crystallite growth.

In order to ascertain whether the crystal structure of binder carbon graphitized in presence of filler bears the previously developed relations between its crystallite parameters, figure 7 was prepared. Crystallite height and interlayer spacing data from reference 11 for binder carbons graphitized in the presence of different fillers are plotted in comparison with the previously developed correlation relating these parameters. The trend of these data suggests that binder pitch, even though graphitizing to a lesser degree in the presence of filler than when alone, bears the same relations between its crystallite parameters as other graphitic carbons. Although the single data point for binder pitch graphitized alone deviates from this trend, the reported interlayer spacing for this one point may be too small as suggested by other data on graphitized pitch cited in reference 11 and also data from reference 12.

Some artificial graphites contain more than one type of filler material. For such cases the measured interlayer spacing of the graphite body is likely to be unsatisfactory as an indication of the crystal structure of any one of the various fillers, unless it is known that one filler definitely predominates. However, of the two most common filler carbons in artificial graphite, petroleum coke should be more graphitized than carbon black. (See, for example, refs. 11 and 12.) In addition, binder carbon in contact with each type of filler may be reasonably assumed to be locally less graphitized than the filler it contacts.

DEGREE OF CRYSTAL DEVELOPMENT AND OXIDATION RATE

That the oxidation rate of polycrystalline graphitic carbon in air or oxygen decreases with increasing degree of graphitization is well established. (See refs. 22 to 25.) This decrease in oxidation rate with increasing degree of graphitization has also been shown to occur for artificial graphite (ref. 26). Although the precise structural factors producing this decrease are difficult to isolate (ref. 22), there is experimental evidence that both more perfectly ordered layers (ref. 23) and larger crystallite sizes (ref. 22) contribute to a lower oxidation rate.

It is also well established that the edges of a crystallite are more reactive than its basal planes (refs. 27 to 33) because edge atoms of a crystallite have unpaired valence electrons available for reaction, whereas layer atoms have their electronic structures completed (refs. 27 and 28). As a consequence, it is to be expected that large crystallites, having fewer exposed edge atoms per unit mass than smaller crystallites, should be less reactive than smaller crystallites. In addition, because of the nonuniformity of crystal reactivity, the preferential crystallite orientation in artificial graphite can be an important factor affecting oxidation rate.

The implications of this relation between crystal development and oxidation to the oxidation rate of artificial graphite are evident. Since the degree of graphitization of binder in contact with filler is less than that of the filler, the binder should be locally more reactive to oxygen than the filler. It is therefore possible that the binder in an artificial graphite is a weak link in determining the aerodynamic erosion resistance of the artificial graphite, particularly in high-shear environments in which filler particles may be mechanically removed from the body because of preferential oxidation of the binder matrix.

CONCLUSIONS

Crystallographic data have been measured by X-ray diffraction on 40 grades of commercially available, nonpyrolytic, artificial graphite. As a result of these measurements and an analysis and correlation of literature data on graphitic carbons, the following conclusions are drawn:

1. The measured interlayer spacings d_c of artificial graphite are close measures of the interlayer spacings of the filler carbon. The measured crystallite dimensions L_a and L_c are representative of neither the filler nor the binder.

2. Correlations of literature data for graphitic carbons have been developed relating the crystallite dimensions L_a and L_c to the interlayer spacing d_c. Experimental

support has been given to the proposal that the a-dimension is a constant (a good value is 2.4614 Å). Accordingly, the crystal development of a graphitic carbon is described by its interlayer spacing alone.

3. The crystallographic characteristics of artificial graphite can be adequately described by the fact that its measured interlayer spacing closely approximates that of its filler (and thereby specifies the crystal structure of the filler), and the fact that its binder is less graphitized than its filler. For artificial graphites containing more than one type of filler, the binder contacting each type of filler can be reasonably assumed to be less graphitized than the filler, and of the two most common filler carbons in artificial graphite, petroleum coke should be more graphitized than carbon black.

4. The aerodynamic erosion resistance of artificial graphite may be largely dependent on the binder. This may be particularly true in high-shear aerodynamic environments in which filler particles may be mechanically removed because of preferential oxidation of the binder matrix. This condition can occur because binder is less graphitized than filler and therefore should be more reactive to oxygen than the filler.

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National Aeronautics and Space Administration,
Langley Station, Hampton, Va., March 20, 1968,
129-03-12-05-23.

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TABLE I.- GRAPHITE GRADES AND RESULTS OF CRYSTALLITE MEASUREMENTS

Grade (a)	Filler (b)	Approximate bulk density, g/cm^3	Maximum grain size		Interlayer spacing, d_c, Å	Apparent a-dimension, a, Å	Apparent crystallite diameter, Å	Apparent crystallite height, Å
			in.	mm				
AHDG	[c]AG	[d]1.90	[d]0.033	0.84	3.361	2.461	1300	420
ME11	PC; LB, AG	1.65	.003	.076	3.364	2.460	670	300
ME14	PC; LB, AG	1.78	.003	.076	3.363	2.460	740	300
ME15	LB; PC, AG	1.77	.007	.18	[e]3.379	2.459	280	[e]140
ME18	PC; LB, AG	1.65	.003	.076	3.364	2.461	1510	320
H-205	Proprietary	1.80	.016	.41	3.370	2.461	800	240
H-205-85	Proprietary	1.82	.006	.15	[e]3.375	2.460	650	[e]300
MHLM	[c]PC	1.79	.033	.84	3.358	2.462	1980	610
MHLM-85	[c]PC	1.83	.033	.84	3.359	2.461	1240	610
2BE	PC	1.50	.006	.15	3.361	2.461	1260	410
2D8D	LB	1.50	.007	.18	[e]3.403	2.457	180	[e]140
2D9B	LB, PC	1.62	.007	.18	[e]3.369	2.461	290	[e]180
W119	PC, NG	1.70	.010	.25	3.361	2.461	940	460
L-56	LB	1.62	.006	.15	[e]3.399	2.459	220	[e]150
[f]L-56-GP	LB	1.62	.006	.15	[e]3.396	2.458	240	[e]160
P-3W	PC	1.60	.006	.15	3.361	2.461	750	290
[f]P-3W-GP	PC	1.60	.006	.15	3.360	2.461	1200	300
E-24	LB	1.53	.005	.13	[e]3.399	2.458	220	[e]150
3499	PC	1.68	.003	.076	3.359	2.460	1510	340
3499-S	PC	1.63	.003	.076	3.360	2.460	940	370
39-RL	PC	1.64	.003	.076	3.360	2.461	1800	340
4007	PC	1.70	.008	.20	3.359	2.461	1260	380
8827	PC	1.77	.003	.076	3.358	2.462	1320	380
9-RL	PC	1.68	.003	.076	3.359	2.461	1810	310
9050	PC	1.80	.003	.076	3.360	2.461	1620	290
L1	PC	1.59	.006	.15	3.364	2.461	1150	460
L31	LB	1.66	.006	.15	[e]3.400	2.458	230	[e]150
331	PC	1.76	.003	.076	3.362	2.461	480	240
AGSX	[c]PC	[d]1.67	[d].016	.41	3.359	2.461	1060	100
ATJ	PC	1.74	.006	.15	3.361	2.461	770	350
[f]ATJ-GP	PC	1.72	.006	.15	3.360	2.461	760	390
ATJS	PC	1.83	.006	.15	3.361	2.461	1320	310
[f]ATJS-GP	PC	1.83	.006	.15	3.361	2.461	1410	320
ATL	PC	1.78	.030	.76	3.360	2.461	640	360
[f]ATL-GP	PC	1.89	.030	.76	3.360	2.460	1060	380
CDA	PC, LB	1.61	.006	.15	3.361	2.461	1520	450
CDG	PC, LB	1.49	.016	.41	3.364	2.461	1420	460
[f]CDG-GP	PC, LB	1.47	.016	.41	3.362	2.461	740	400
CMB	PC, LB	1.75	.003	.076	[e]3.362	2.461	270	[e]170
PGR	PC	1.68	.030	.76	3.358	2.461	1320	460

[a] All grades are molded except AHDG and AGSX, which are extruded.

[b] Filler materials are: AG, artificial graphite; LB, lampblack; NG, natural graphite; PC, petroleum coke.

[c] From reference 2.

[d] From reference 1.

[e] Based on (002) reflection.

[f] Gas-purified version of base grade denoted by GP.

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