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CRYSTALLITE ORIENTATION IN MOLDED GRAPHITES

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16. Abstract

An extensive investigation of the orientation of crystallites in molded artificial graphite has shown that the crystallites in such graphites are usually, but not always, distributed symmetrically about a single axis. The symmetry axis has been found to differ frequently from the forming axis, contrary to what has generally been assumed. Furthermore, the direction of the symmetry axis and the degree of orientation have been found to vary somewhat within a given graphite block.

A two-parameter equation has been found to be suitable for describing the distribution of crystallites about the symmetry axis. Specification of the direction of the symmetry axis involves two additional parameters. A technique for evaluating these parameters from experimental data is presented.

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SUMMARY

An extensive investigation of the orientation of crystallites in molded artificial graphites has shown that the crystallites in such graphites are usually, but not always, distributed symmetrically about a single axis. The symmetry axis has been found to differ frequently from the forming axis, contrary to what has generally been assumed. Furthermore, the direction of the symmetry axis and the degree of orientation have been found to vary somewhat within a given graphite block.

A two-parameter equation, originally proposed by Pappis et al. (in High Temperature Materials, Vol. II, Interscience Publ., 1963), has been found to be suitable for describing the distribution of crystallites about the symmetry axis. Specification of the direction of the symmetry axis involves two additional parameters. A technique for evaluating these parameters from experimental data obtained by the Bacon method (J. Appl. Chem. (London), vol. 6, 1956, pp. 477-481) is presented.

INTRODUCTION

Virtually all nonpyrolytic artificial graphites are polycrystalline, and most have their crystallites preferentially oriented in some fashion. Crystallite orientation in artificial graphites results from an interaction between the structure of the crystallites and the methods by which graphite bodies are generally manufactured. An extensive treatment of graphite structure and manufacture is given by Nightingale (ref. 1); Harris, Miller, and Craik (ref. 2) discuss the effect of these factors on crystallite orientation.

Because many of the properties of graphite crystallites are anisotropic (i.e., directionally dependent), preferential crystallite orientation results in anisotropy of the properties of most bulk graphites. The degree of anisotropy of a graphite determines its suitability for many applications. For example, a spacecraft heat shield ideally should have a

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high thermal conductivity parallel to the surface in order to distribute the heat load over its entire area but a low thermal conductivity perpendicular to the surface to insulate the spacecraft. Thus, a graphite intended for use as a heat shield should be highly anisotropic. On the other hand, such a graphite might be unsuitable as a moderator in a nuclear reactor because of mechanical problems associated with nonuniform expansion of the graphite when subjected to neutron irradiation. The degree of anisotropy of a given property is related to the degree of crystallite orientation within the graphite (ref. 3), and consequently, the degree of orientation is, itself, an important property. Unfortunately, no simple method for completely and unambiguously describing the degree of orientation within a graphite is presently available. The purpose of this study was to develop such a method.

The study was pursued both theoretically and experimentally. The principal experimental technique employed was the classic transmission method of Bacon (ref. 3), but some data were taken by the back-reflection method of Ali, Fitzer, and Ragoss (ref. 4). Both methods are described in detail in subsequent sections of this report, as is the theoretical technique developed in this study. For convenience, this study was limited to molded graphites, but the method of describing orientation which is developed should be applicable to extruded graphites as well.

SYMBOLS

A,B	parameters in equations (4) and (17)
a,b	parameters in equations (18) and (19)
D	film density
I	relative number of crystallites per unit solid angle
$\hat{i},\hat{j},\hat{k}$	unit vectors parallel to x-, y-, and z-coordinates, respectively
l,m,n	direction cosines, $\cos\alpha$, $\cos\beta$, and $\cos\gamma$, respectively
M	parameter in equations (3), (4), and (17)
$\hat{N}$	unit vector normal to basal planes of a crystallite
P	forming axis

$\hat{P}$	unit vector parallel to forming axis
$\hat{S}$	unit vector parallel to symmetry axis
x,y,z	coordinates
α,β,γ	direction angles referred to x-, y-, and z-coordinates, respectively, deg
δ	angle between forming and symmetry axes
θ	angle between incident X-ray beam and crystallite basal planes
ξ	angle on diffraction image
σ	square root of variance
ϕ	orientation angle

Subscripts:

calc	calculated
exp	experimental
N	basal plane normal
S	symmetry axis

The basal planes (fig. 1) of graphite are frequently designated in X-ray diffraction work as (002) planes, the numbers in parentheses being Miller indices. Both designations are used interchangeably in this report.

The orientation of a crystallite is usually expressed in terms of an imaginary ray normal to the basal planes of the crystallite rather than in terms of the basal planes themselves. This convention will be used throughout this report.

SURVEY OF PREVIOUS WORK

The earliest systematic study of the orientation of crystallites in graphite was reported by G. E. Bacon (ref. 3). The experimental arrangement for the method used by

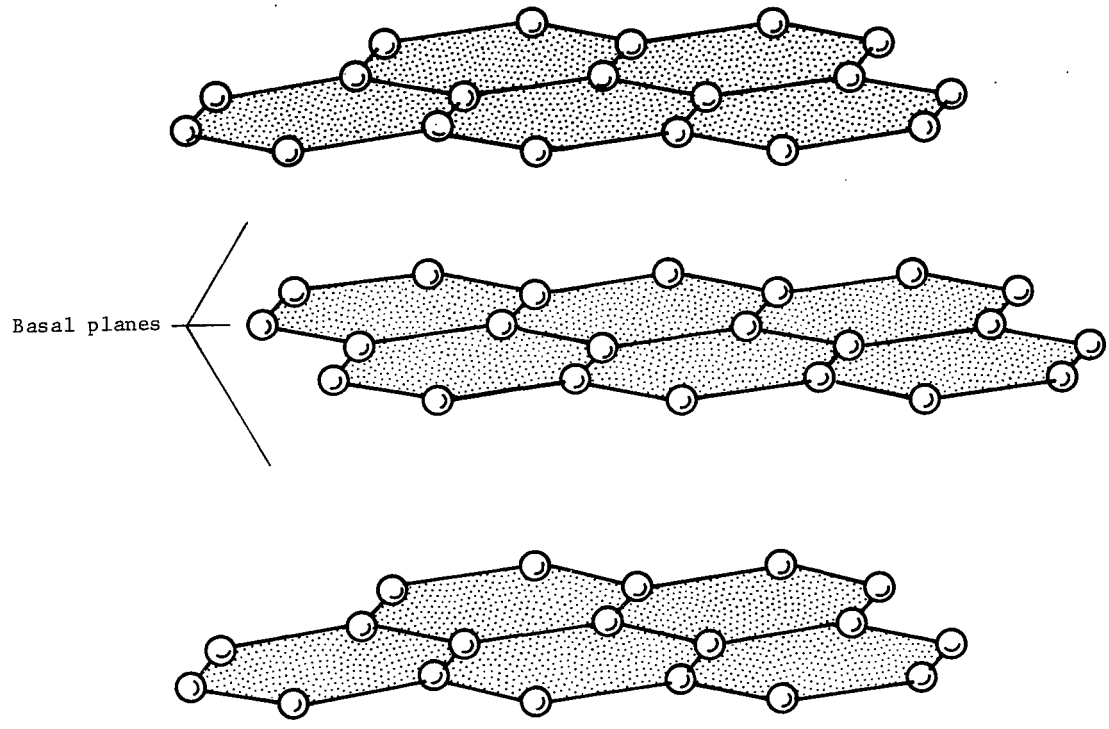


Figure 1.- Crystal structure of graphite.

Bacon is illustrated in figure 2. The incident X-ray beam x is horizontal; the X-rays are unfiltered CuK radiation. The graphite specimens are thin flat plates approximately 2 by 1 by 0.1 cm, cut with their 1-cm edges parallel to the forming axis P. Each specimen is alined so that its 2-cm edges are horizontal and perpendicular to the incident X-ray beam, and its 1-cm edges are tilted from the vertical z by 13°. This tilt is necessary so that crystallites with orientations of 0° to 13° with respect to the forming axis can be detected, as discussed in the section "Theoretical Analysis."

The graphite specimens are thin enough to allow transmission of a significant fraction of both the diffracted and undiffracted components of the X-ray beam. The diffracted X-rays form a cone with a half-angle of 26°, which is twice the Bragg angle for the (002) reflection obtained with CuK radiation. When the diffracted X-rays strike the photographic film (which is perpendicular to the undiffracted X-ray beam and, thus, to the axis of the cone), they form a circular image of varying density on the film. The density of the image at any angle ξ is proportional to the number of crystallites at some related orientation within the specimen. The film density is determined at each angle of interest with a microdensitometer.

The main theoretical problem of the Bacon method is the determination of the relationship between angles on the film image and the orientation of crystallites within the graphite specimen. Bacon assumed that the crystallites in artificial graphites are

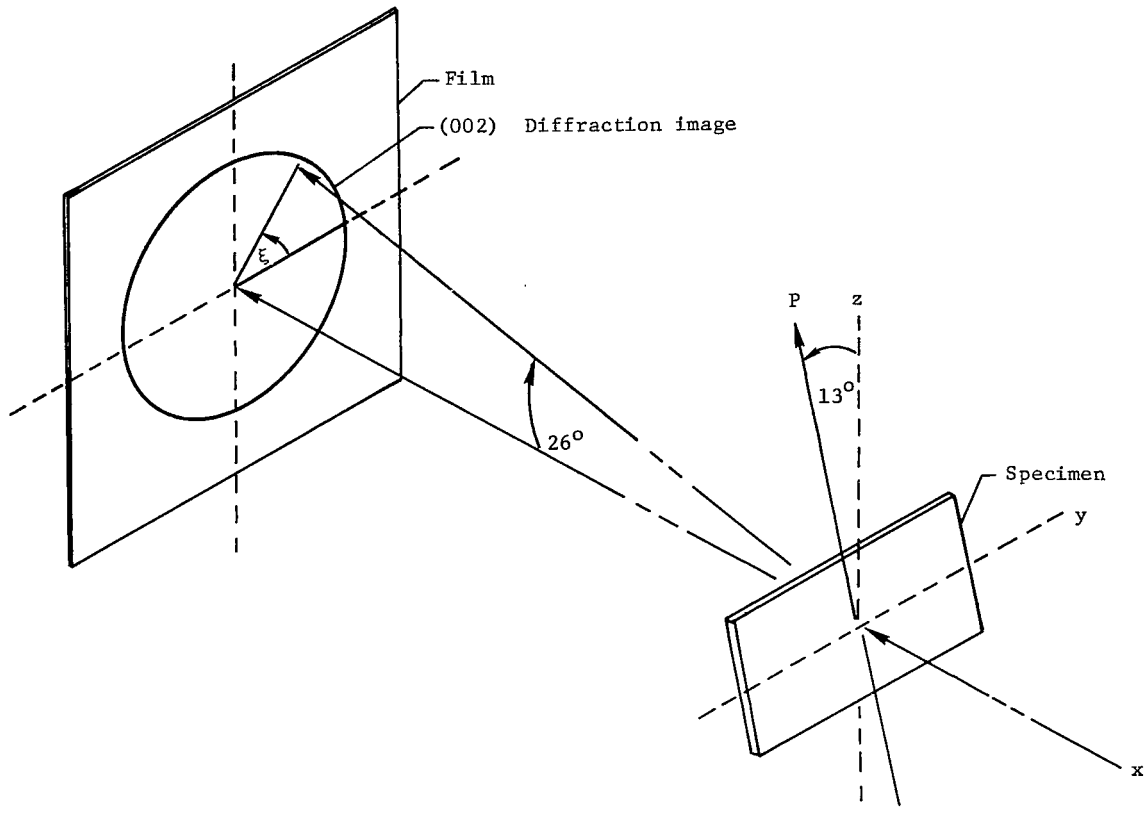


Figure 2.- Experimental arrangement for method of Bacon.

symmetrically distributed about the forming axis. Such a distribution would mean that the only parameter necessary to describe the orientation of the crystallites is the angle ϕ which their normals make with the forming axis. On the basis of this assumption, Bacon derived the following equation relating ϕ and ξ:

$$\cos \phi = \cos^2 77^\circ - \sin^2 77^\circ \sin \xi \qquad (1)$$

For convenience in expressing the angular distribution of crystallites, Bacon defined an orientation function $I(\phi)$ as the relative number of crystallites per unit solid angle about the orientation angle ϕ. This function is usually normalized so that $I(\phi) = 1$ when $\phi = 0^\circ$. In practice, $I(\phi)$ is taken as the relative diffraction intensity which, for the Bacon method, is assumed to be equal to $D(\phi)/D(0)$, where $D(\phi)$ and $D(0)$ are the film densities at ϕ and 0°, respectively.

The Bacon method of determining crystallite orientation has the advantage that with one specimen and one exposure it furnishes a continuous quantitative mapping of $I(\phi)$. The principal disadvantage of the method is that it does not yield diffraction intensities directly and immediately, but requires the intermediate steps of film processing and

microdensitometry. These steps are not only inconvenient but also constitute possible sources of error.

Ali, Fitzer, and Ragoss (ref. 4) contend that a plot of $I(\phi)$ as a function of ϕ in polar coordinates will always be elliptical within experimental error. On the basis of this contention, they have proposed that intensity measurements need be made at only two angles, 0° and 90°, since the value of $I(\phi)$ for any intermediate angle can be calculated from the polar equation of an ellipse,

$$I(\phi) = \frac{I(0)I(90)}{\left[I(0)^2\sin^2\phi + I(90)^2\cos^2\phi\right]^{1/2}} \tag{2}$$

The experimental procedure proposed in reference 4 is as follows: Two flat plates, or disks, are cut so that one is parallel and one is perpendicular to the forming axis of the graphite to be studied. Each specimen is mounted in the flat specimen holder of an X-ray diffractometer and, by use of a counter-goniometer, the back-reflected (002) peak of each is determined and recorded on a strip chart recorder. The value of $I(90)$ is taken as the peak height of the specimen parallel to the forming axis ($\phi = 90^{\circ}$) divided by the peak height of the perpendicular specimen ($\phi = 0^{\circ}$). The value of $I(0)$ is taken as 1. If intermediate angles are to be investigated, a separate specimen must be cut for each.

The method of reference 4 is quite simple if only two specimens need be cut and studied. However, this will be the case only if the distribution of crystallites in the graphite to be studied is, in fact, both symmetrical and elliptical about the forming axis. Unfortunately, Ali, Fitzer, and Ragoss present only limited experimental substantiation of their assumption and they give no quantitative results.

Harris, Miller, and Craik (ref. 2) present polar plots of $I(\phi)$ as a function of ϕ for four graphites, and in no case is the distribution elliptical. However, all four graphites were extruded rather than molded, and three of them were specially prepared in the laboratory rather than commercially manufactured. These results, therefore, do not rule out the possibility that molded commercial graphites possess elliptical distributions. It should be noted that if the assumption in reference 4 of a symmetrical elliptical distribution is correct, the normalized value of $I(90)$ constitutes a single parameter which, in conjunction with equation (2), provides a complete specification of the spatial distribution of graphite crystallites.

An alternative one-parameter equation that has been proposed by several investigators (refs. 3, 5, 6, and 7)[1] is

[1]Actually, Bacon proposed the form $I(\phi) = \sin^M\phi$ which was intended to be applicable to extruded graphites.

$$I(\phi) = \cos^{M}\phi \tag{3}$$

Equation (3) is fairly representative of pyrolytic graphites, but it usually is not applicable to molded graphites because they generally have some crystallites with their normals at 90^{o} to the forming axis. Equation (3) assumes that $I(90) = 0$.

Pappis et al. (ref. 8) have proposed a variation of equation (3) that overcomes this problem

$$I(\phi) = A \cos^{M}\phi + B \tag{4}$$

Unfortunately, these investigators also fail to present any quantitative substantiation of their equation with experimental data. The equation is still of considerable interest, however, and should be compared with the ellipse proposed in reference 4 to see if either is clearly superior to the other. Equation (4) can be regarded as a two-parameter equation since, by proper normalization of $I(\phi)$, $A + B = 1$.

Several investigators (refs. 3, 9, 10, and 11) have proposed other orientation parameters which attempt to specify the degree of crystallite orientation within a graphite with a single number. Such numbers, although useful for some purposes, do not describe the spatial distribution of crystallites. Therefore, they will not be further discussed herein.

The assumption of a symmetrical distribution of crystallites about the forming axis is either stated or implied in most papers on graphite orientation. Cavin (ref. 12), however, has recently reported experimental results which contradict this assumption. The experimental technique used by Cavin employed a Schultz preferred-orientation apparatus (ref. 13), which makes possible the determination of the diffraction intensity not only as a function of the inclination angle ϕ with regard to the forming axis but also as a function of the azimuthal angle lying in a plane perpendicular to the forming axis. In his investigation, Cavin observed a shift of the symmetry axis from the forming axis by as much as 12^{o}.

THEORETICAL ANALYSIS

In this section the implications of assuming that the distribution of crystallites in molded graphites is symmetrical about an axis which is not necessarily coincident with the forming axis are considered. Furthermore, a technique is devised by which (1) the spatial distribution of crystallites in a given graphite can be completely determined from a properly obtained Bacon film image, and (2) the distribution so determined can be completely and unambiguously expressed with a minimum number of parameters.

Assume that the graphite specimen shown in figure 2 has a symmetry axis with some unspecified direction, and let $\hat{S}$ be a unit vector coincident with the symmetry axis. In the coordinate system shown in figure 2,

$$\hat{S} = \cos \alpha_S \hat{i} + \cos \beta_S \hat{j} + \cos \gamma_S \hat{k} = l_S \hat{i} + m_S \hat{j} + n_S \hat{k} \tag{5}$$

where α_S, β_S, and γ_S are the angles which the symmetry axis makes with the x-, y-, and z-coordinates, and l_S, m_S, and n_S are the corresponding direction cosines.

Let $\hat{N}$ be a unit vector normal to the basal planes of some crystallite of interest. In terms of the direction angles α_N, β_N, and γ_N and the direction cosines l_N, m_N, and n_N,

$$\hat{N} = \cos \alpha_N \hat{i} + \cos \beta_N \hat{j} + \cos \gamma_N \hat{k} = l_N \hat{i} + m_N \hat{j} + n_N \hat{k} \tag{6}$$

The scalar product of the unit vectors $\hat{N}$ and $\hat{S}$ is by definition equal to the cosine of the angle between them. But the angle between $\hat{N}$ and $\hat{S}$ is the orientation angle ϕ. Thus,

$$\hat{N} \cdot \hat{S} = l_N l_S + m_N m_S + n_N n_S = \cos \phi \tag{7}$$

From the properties of direction cosines,

$$l_N^2 + m_N^2 + n_N^2 = 1 \tag{8}$$

$$l_S^2 + m_S^2 + n_S^2 = 1 \tag{9}$$

Solving equation (8) for m_N and equation (9) for n_S results in

$$m_N = \pm \sqrt{1 - l_N^2 - n_N^2} \tag{10}$$

$$n_S = \pm \sqrt{1 - l_S^2 - m_S^2} \tag{11}$$

If it is assumed that the vector $\hat{S}$ always has an upward component, equation (11) is positive only.

For (002) diffraction to occur, the angle between the basal planes and the incident X-ray beam must be the Bragg angle for the radiation used. The Bragg angle for (002) diffraction with CuKα radiation is approximately 13°. If the angle between the incident X-ray beam and the basal planes is 13°, the angle of the basal plane normals must be either 77° or 103°, depending on whether the x-component of the normal is positive or negative. Requiring it to be positive yields

$$\alpha_N = 77^\circ \tag{12}$$

It can be shown from consideration of spherical coordinates that

$$n_N = \sin 77^\circ \sin \xi \tag{13}$$

If equations (10), (11), (12), and (13) are combined with equation (7), the following general equation relating ϕ and ξ is obtained:

$$\cos \phi = l_S \cos 77^\circ \pm m_S \sin 77^\circ \sqrt{1 - \sin^2 \xi} + \sqrt{1 - l_S^2 - m_S^2} \sin 77^\circ \sin \xi \qquad (14)$$

The second term on the right-hand side of this equation is negative when $90^\circ < \xi < 270^\circ$, otherwise it is positive. This results from the fact that, for the diffracted beam to strike the left half of the film, the y-component of the crystallite normal vector must be negative.

Note that equation (14) contains two unknown parameters l_S and m_S, the direction cosines of the symmetry axis with respect to the x- and y-coordinates. Bacon, in assuming that the symmetry axis is coincident with the forming axis, in effect assumed that $\alpha_S = 103^\circ$ and $\beta_S = 90^\circ$. This is equivalent to the assumption that $l_S = -\cos 77^\circ$ and $m_S = 0$. If these values of l_S and m_S are inserted into equation (14), the result is

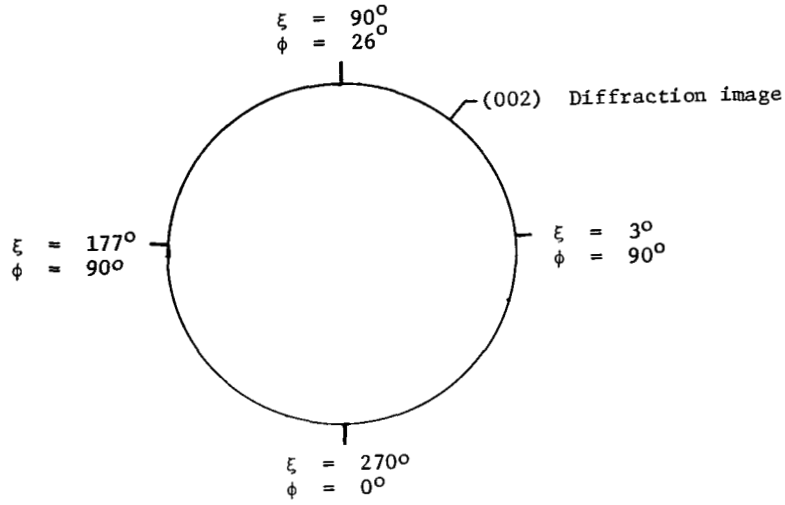
$$\cos \phi = -\cos^2 77^\circ + \sin^2 77^\circ \sin \xi \qquad (15)$$

Equation (15) is essentially the equation derived by Bacon; it differs from Bacon's equation only in the sign of the terms on the right. Since only values of ϕ between 0° and 90° are of interest, only positive values of $\cos \phi$ need be considered. Therefore, equation (15) may be rewritten as

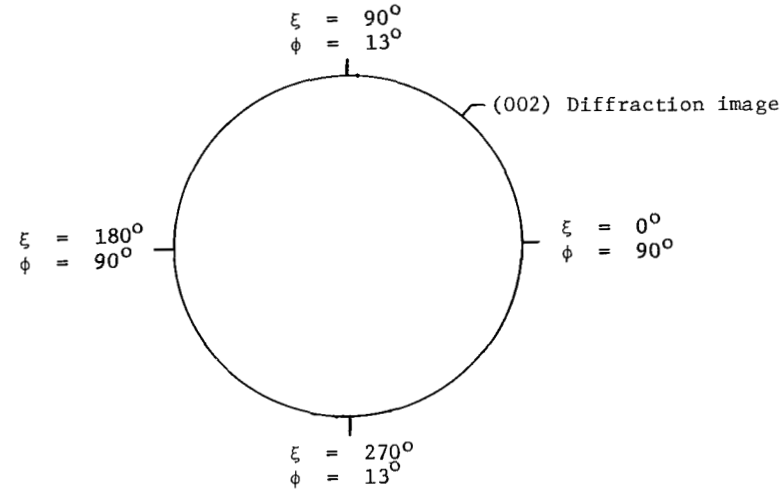
$$\cos \phi = \left| \cos^2 77^\circ - \sin^2 77^\circ \sin \xi \right| \qquad (16)$$

Equation (16) results in all values of ϕ from 0° to 90° being represented on the film image, as is indicated schematically in figure 3(a). Many values of ϕ are represented at several points on the film. Figure 3(b) is a schematic representation of the limiting values of ϕ and ξ if the specimens are alined vertically rather than tilted by 13°, as specified by Bacon. These values result from applying the condition that $\alpha_S = 90^\circ$, and therefore $l_S = 0$, to equation (14). It is obvious from figure 3(b) that crystallites with orientation angles between 0° and 13°, would not be detected if the specimens were alined vertically and the forming axis was coincident with the orientation symmetry axis.

If Bacon's assumption of the coincidence of the symmetry and forming axes is incorrect, two difficulties arise: (1) the values of l_S and m_S are not known a priori and (2) not all inclinations ϕ are necessarily represented on a given film image, even with the prescribed 13° tilt applied to the specimens. These difficulties can be overcome, at least in principle, if a valid closed-form expression relating I and ϕ is known and if sufficient experimental data are available. The procedure involved will be illustrated by considering equation (4). Note that ϕ appears only through its cosine. Equation (14) gives $\cos \phi$ as a function of the parameters l_S and m_S and the variable ξ. If



(a) $\cos \phi = \left|\cos^2 77^\circ - \sin^2 77^\circ \sin \xi\right|$.



(b) $\cos \phi = \left|\sin^2 77^\circ \sin \xi\right|$.

Figure 3.- Limiting values of ξ and ϕ.

equations (4) and (14) are combined,

$$I = A\left(l_S \cos 77^\circ \pm m_S \sin 77^\circ \sqrt{1 - \sin^2 \xi} + \sqrt{1 - l_S^2 - m_S^2} \sin 77^\circ \sin \xi\right)^M + B \qquad (17)$$

Equation (17) contains five unspecified parameters A, B, M, l_S, and m_S as well as the variables I and ξ. Values of the five parameters can, in principle, be determined if five pairs of experimental values of I and ξ are available. However, if more than five pairs of values of I and ξ are available, a statistically preferred set of values of the parameters can be obtained by use of the method of least squares. A least-squares

method for solving nonlinear equations such as equation (17) is given in reference 14. This method was used in this investigation.

Equation (2) can also be combined with equation (14) to yield an equation similar to equation (17). However, for generality, equation (2) is first rewritten as

$$I = \frac{ab}{\left[a^2 + (b^2 - a^2)\cos^2\phi\right]^{1/2}} \tag{18}$$

In this equation the parameters $I(0)$ and $I(90)$ have been replaced by a and b, respectively, since there is no justification for attaching more significance to experimental values measured at 0° and 90° than to values measured at other angles. When equations (14) and (18) are combined, the result is

$$I = \frac{ab}{\left[a^2 + (b^2 - a^2)\left(l_S \cos 77^\circ \pm m_S \sin 77^\circ \sqrt{1 - \sin^2\xi} + \sqrt{1 - l_S^2 - m_S^2} \sin 77^\circ \sin \xi\right)^2\right]^{1/2}} \tag{19}$$

Equation (19) can be solved by the same least-squares method applied to equation (17) to yield the best values of a, b, l_S, and m_S.

Once the parameters in either equation (17) or equation (19) have been determined, the value of I at any angle ϕ can be calculated (by using eq. (14) to relate ϕ and ξ). Thus, it is not necessary that a particular angle be studied experimentally or even that it be represented on the film image.

The number of parameters actually specified need be only four in the case of equation (17) or three for equation (19), since by proper normalization, $A + B = 1$ and $a = 1$. Equations (17) and (19) can be compared by applying each to several sets of experimental data and comparing their variances.

The angle by which the symmetry axis is displaced from the forming axis can be obtained by first representing the forming axis by the unit vector $\hat{P}$, where

$$\hat{P} = \cos 103^\circ \hat{i} + \cos 13^\circ \hat{k} = -\cos 77^\circ \hat{i} + \cos 13^\circ \hat{k} \tag{20}$$

and then taking the scalar product of $\hat{S}$ and $\hat{P}$

$$\hat{S} \cdot \hat{P} = -l_S \cos 77^\circ + \sqrt{1 - l_S^2 - m_S^2} \cos 13^\circ = \cos \delta \tag{21}$$

The arc cosine of $\cos \delta$ is the desired displacement angle δ.

EXPERIMENTAL TECHNIQUES

The methods of Bacon (ref. 3) and Ali, Fitzer, and Ragoss (ref. 4) were employed in this study. (For convenience, these methods are referred to hereinafter as the Bacon method and the Ali method.) The vast majority of the data were taken by using the Bacon method, but a few checks were made with the Ali method because of its simplicity. Each of these methods is outlined in the section "Survey of Previous Work." Details of the methods as employed in this study are presented in this section.

The X-ray instrument utilized for both the Bacon and Ali methods was a General Electric XRD-5 diffractometer with various accessories. The X-rays were nickel-filtered CuKα radiation. The peak tube voltage was 50 kV.

The experimental apparatus used for the Bacon method was a transmission Laue camera with a 0.5-mm-diameter (0.020-inch) pinhole collimator and a 10- by 12-cm (4- by 5-inch) film cassette. Ten grades of graphite were investigated by the Bacon method. Three specimens of each grade except ATJ and 2D8D were studied; only two specimens of these grades were available. The grades studied are listed in table 1 along with certain of their properties.

The specimens, which were 2.5 by 1 by 0.1 cm, were mounted in a specially constructed holder which permitted them to be oscillated horizontally in a plane normal to the incident X-ray beam. The oscillation increased the number of grains irradiated. The period of oscillation was 1 minute, and the amplitude was 1.9 cm. The speed of oscillation was kept constant so that all grains irradiated were exposed for the same length of time. The exposure time was 1 hour. Specimens of grade CDG, which is somewhat coarsely grained, were exposed in two steps of 1/2 hour each. After the first 1/2 hour, the specimens were raised in the holder so that more grains would be exposed.

The film used was Kodak Industrial X-ray Film Type M. The films were individually processed with Kodak Liquid X-ray Developer and Replenisher and Kodak Liquid X-ray Fixer and Replenisher. The manufacturer's processing instructions were followed throughout.

The density of the processed film was determined with a Joyce-Loebl double-beam recording microdensitometer. The films were mounted on a special rotary state with angular markings to 0.1°. The stage was then rotated to each desired value of ξ, and radial scans of the circular diffraction image were made. The result of each scan was a trace, on ruled paper, of the density as a function of $\tan 2\theta$.

Bacon stated in his paper that the integrated density (the area under the diffraction peaks) is proportional to $I(\phi)$. Ali, Fitzer, and Ragoss (ref. 4) and Guentert (ref. 5) contend that the peak height and peak area are proportional to each other and to $I(\phi)$ and,

furthermore, that the height is preferable to the area since it is easier to measure. Actually, the height and area are not always proportional to each other, as is shown in table 2, in which the ratio of the normalized peak height to peak area is listed for the two specimens of grade ATJ. It can be seen that the ratio is not unity and, in fact, is not even constant. The variations are too large and systematic to be due entirely to experimental error.

Neither the peak height nor the peak area is a completely satisfactory measure of $I(\phi)$ for several reasons. As previously noted, the abscissa of the diffraction peaks obtained from Bacon films is $\tan 2\phi$ rather than 2θ. Furthermore, the size and shape of a diffraction peak are influenced by other factors in addition to the number of crystallites causing diffraction (see, for example, ref. 15). These factors include the Lorentz factor and instrument broadening effects. They are complicated by the fact that molded graphite is not a homogeneous material; it has at least two phases, binder and filler, and more than two phases if two or more fillers are used. In view of the preceding problems, it was believed that a comparison of the results obtained by use of peak height and peak area data would be of interest. Therefore, both were measured in this investigation.

The 28 films obtained from the 10 grades of graphite studied were scanned in 10° increments of ξ from 0° through 350°. In addition, one grade (2BE) was reread every 2.5° of ξ. The heights of the diffraction curves were obtained by subtracting the average background height from the average height of the peak crests, both of which were read directly from the ruled paper.

Three grades of graphite (ATJ, 2D8D, and CMB) were investigated by the Ali method. The approximate dimensions of the specimens used were 7 by 2.5 by 0.8 cm. For each specimen, the value of 2θ was varied continuously over about a 5° range encompassing the Bragg value. The variation was extensive enough to encompass the entire peak as well as some background on either side. The peak area was determined with a planimeter, and the height was taken as the peak deflection minus the average of the background deflections.

The peak heights measured in this study have an uncertainty of about ± 1 to 2 percent except when a dust speck or film blemish caused a spurious deflection at or near the peak crest. In such cases the uncertainty is estimated to be about ± 5 percent. The uncertainty in peak areas is considered to be greater than in peak heights because of the increased effect of uncertainties in the base line. Errors in determining the base line have a linear effect on the uncertainty of the peak heights but a much greater effect on the uncertainty of the peak areas because of the divergence of the diffraction peak at its base. Dust specks and film blemishes are also troublesome when areas are being determined. All such spurious deflections were faired through before the areas were determined, but

some additional uncertainty was introduced. Overall, the planimeter integrations are estimated to be uncertain to about ±2 to 3 percent.

Many factors contribute to the uncertainty in the angular orientation of the specimens, but the major source of uncertainty unquestionably was the cutting process. The specimens for the Bacon method were cut in two steps: First, 2.5-cm cubes were sawed from the billets as received at the Langley Research Center. The cubes were cut with an angular accuracy of about 2° or better, and the pressing direction was clearly marked. Second, the specimens were cut from the cubes by a contractor. No estimate of angular uncertainty was furnished by this contractor. A value of 2° will be assumed, since the final cutting of the specimens should not have been more inaccurate than the sawing of the cubes. All other sources of error contributed less than 1°. Thus, the total angular error in the Bacon specimens should be less than 5°. The specimens for the Ali method were cut in one step at the Langley Research Center; their total angular uncertainty is no more than 2°.

RESULTS AND DISCUSSION

The data from the 28 Bacon-type diffraction films were reduced by use of equations (17) and (19). The least-squares values of the various parameters contained in these equations were determined for each set of data by using the computer programs and subprograms listed in reference 14. The values of the parameters which were computed are presented in table 3. The values of the parameters a and A are not presented in table 3 since the data and results were normalized so that $a = 1$ and $A = 1 - B$. Although equations (17) and (19) were solved in terms of the direction cosines l_S and m_S, the direction angles α_S and β_S are easier to visualize; therefore, these angles rather than the direction cosines are presented in table 3. Also presented in this table are the values of δ the angle between the symmetry and forming axes and σ^2 the variance of the data.

Three important observations can readily be made from the results presented in table 3. (1) The values of the parameters obtained by use of peak heights to represent $I(\phi)$ generally do not agree with the values based on peak areas, even within experimental uncertainty. (2) The values of the parameters computed by using equation (17) frequently do not agree with the values obtained by using equation (19). (3) The values of δ, in many cases, are too large to be accounted for by experimental error. Each of these observations and others related to them will be discussed in this section.

The fact that the peak height and peak area data generally do not yield the same values for the various orientation parameters conflicts with the assumption in references 4 and 5, that they are equivalent measures of $I(\phi)$. Although this investigation

did not attempt to resolve the question of which is theoretically the better measure of $I(\phi)$, the peak height data have been shown to be more consistent. For example, every one of the 56 cases listed in table 3 which involve peak height data converged to a valid solution; whereas 17 of the 56 cases involving peak area data failed to converge.[2] Also, the average variance of the cases involving peak heights is only 0.81 of the average for the cases involving areas.[3]

Similar comparisons can be used to show that equation (17) yields more consistent results than does equation (19). All but four cases involving equation (17) converged, but 13 cases involving equation (19) did not converge. The average variance obtained with equation (17) is only 0.31 of the average variance obtained with equation (19). Furthermore, the individual variance for equation (17) is smaller in every case except two, for which it is equal to that obtained with equation (19).

It is clear that the most consistent results are those obtained by use of equation (17) and peak height data. These results will be referred to exclusively throughout the remainder of this section.

Perhaps the most significant observation to be made from table 3 is that in well over half of the cases, the value of δ is greater than 5°, which is the estimated maximum angular error. In several cases, δ is more than 10°. Thus, significant angular differences between the forming axis and the experimentally determined symmetry axis exist in many of the specimens investigated. This observation corroborates the finding of Cavin (ref. 12) and contradicts the assumption of Bacon (ref. 3) and others.

The importance of experimentally determining the symmetry axis and using it, rather than the forming axis, as the reference axis for specifying the orientation angle ϕ is illustrated in figure 4. In this figure the peak height data on film 201 are plotted in two ways. In figure 4(a), ϕ is referred to the forming axis; in figure 4(b), ϕ is referred to the computed symmetry axis. It is obvious that the data points are much less scattered when ϕ is referred to the symmetry axis.

It is noteworthy that δ is generally not constant among the three specimens of each grade of graphite. Angular differences of 5° or more exist among the specimens of 6 of the 10 grades studied. It appears that the symmetry axis of a graphite body does not necessarily have the same direction at all points within the body. Furthermore, the degree of orientation was also found to vary throughout a graphite body, since the parameters b, B, and M generally vary somewhat among the specimens within a given grade.

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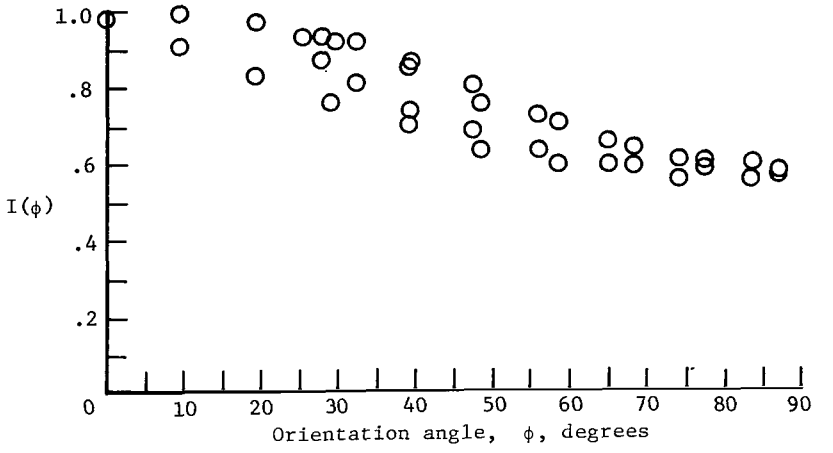
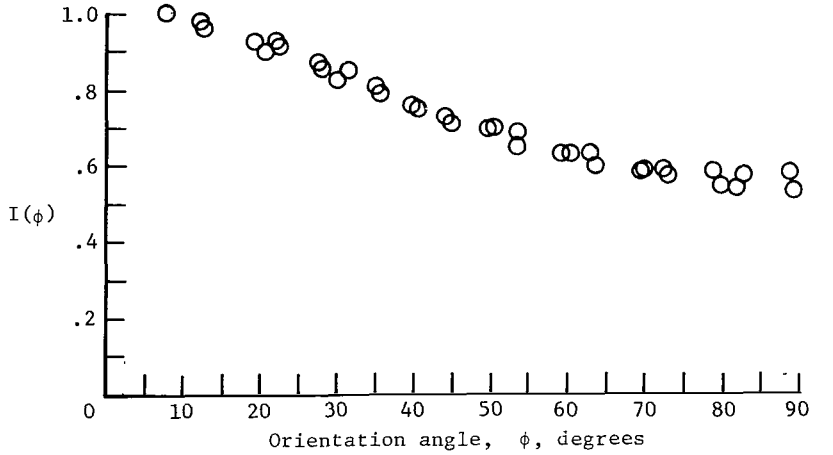
[2]Failure of the computer program to converge to a valid solution is frequently caused by either too high imprecision in the data or by failure of the equation used to describe the data adequately.

[3]The average variances mentioned here and in the next paragraph do not include the variances of specimens 1 and 3 of grade 2BE, which are believed to be atypical. This point will be discussed subsequently in this section.



(a) $\cos \phi = \cos^2 77^\circ - \sin^2 77^\circ \sin \xi$.



(b) $\cos \phi = l_S \cos 77^\circ \pm m_S \sin 77^\circ \sqrt{1 - \sin^2 \xi}$
$+ \sqrt{1 - l_S^2 - m_S^2} \sin 77^\circ \sin \xi$.

Figure 4.- $I(\phi)$ plotted against ϕ for graphite grade CDG.

It is not known why the direction of the symmetry axis and the degree of orientation vary within a graphite body, but the answer probably involves an uneven distribution of forces within the body during the forming process. Unfortunately, the positions of the specimens within the body were not noted, and thus, no patterns regarding the variations can be ascertained.

Two of the specimens of grade 2BE behaved quite differently from the other specimens tested in that the variances associated with them were abnormally high. Furthermore, the deviations between the experimental and calculated values of $I(\phi)$ (based on the least-squares parameters) were not random, as shown in table 4. The three films

for this grade were reread at intervals of 2.5^{o} in ξ to minimize the effects of errors in the individual data points. The data were reduced in toto and also by using only every fourth data point so that the increment of ξ was 10^{o}. Only equation (17) was used. The results are presented in table 5. It can be seen that no significant change in the variance or the values of the various orientation parameters results from considering the additional data points.[4] Apparently, the distribution of crystallites in specimens 1 and 3 of grade 2BE is not truly symmetrical about any axis. Specimen 2 from the same block of graphite does, however, exhibit a symmetry axis. The cause of this behavior is probably related to the cause of the variation of the direction of the symmetry axis but is not presently known. In any event, it appears that the results for a sample taken at one location in a block of commercial graphite do not necessarily apply at other locations within the block.

It should be clear from the preceding discussion that the Ali method cannot be expected to give more than a rough approximation of crystallite orientation, since this method involves two fairly large samples which must necessarily be cut from different locations, and also since the method depends upon the assumption of an elliptical distribution of crystallites, which has been found to be not generally correct. A comparison of Ali method measurements of $I(90)/I(0)$ on grades ATJ, 2D8D, and CMB with the values of B[5] for these grades is given in table 6. As expected, the results do not agree very well.

CONCLUSIONS

The results of this investigation lead to the following conclusions:

1. Most molded artificial graphites have an axis about which their crystallites are symmetrically oriented. However, a symmetry axis does not necessarily exists in all such graphites, as indicated in this investigation by grade 2BE.

2. The symmetry axis of a molded graphite does not necessarily correspond to the forming axis and, in fact, frequently does not.

3. The degree of crystallite orientation, the direction of the symmetry axis, and, in fact, whether or not a symmetry axis even exists can vary from one location to another within a graphite body.

4. The equation $I = A \cos^{M}\phi + B$ is capable of describing the angular distribution of crystallites about the symmetry axis within the limits of experimental error. In this

[4]Comparison of the values in this table with those in table 3(b) illustrates the reproducibility of the technique used.

[5]The orientation parameter B is essentially the least-squares estimate of the true value of $I(90)/I(0)$.

equation, I is the relative number of crystallites per unit solid angle, A, B, and M are unspecified parameters, and ϕ is the orientation angle.

5. The angle ξ on a Bacon-type diffraction film is related to the orientation angle ϕ through the equation

$$\cos \phi = l_S \cos 77^\circ \pm m_S \sin 77^\circ \sqrt{1 - \sin^2 \xi} + \sqrt{1 - l_S^2 - m_S^2} \sin 77^\circ \sin \xi$$

where l_S and m_S are the direction cosines of the symmetry axis to which ϕ is referred.

6. The parameters B, M, l_S, and m_S constitute a set of parameters capable of completely specifying the orientation of crystallites in molded artificial graphites.

7. The height of a diffraction peak is not necessarily proportional to the area under the peak. Consequently, values of the orientation parameters based on peak height data frequently do not agree with the values obtained with peak area data. The precision of peak height data is generally greater than that of peak area data for the techniques employed in this study.

Langley Research Center,
National Aeronautics and Space Administration,
Hampton, Va., March 16, 1971.

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TABLE I.- SOME PROPERTIES OF GRAPHITE GRADES STUDIED

Grade	Filler	Density, $g\text{-}cm^{-3}$	Maximum grain size, mm
ATJ	PC[a]	1.74	0.15
ATJ (G.P.)[b]	PC	1.72	.15
2BE	PC	1.40	.15
9-RL	PC	1.68	.08
3499-S	PC	1.63	.08
4007	PC	1.70	.20
L31	LB[c]	1.66	.15
2D8D	LB	1.40	.18
CDG	PC and LB	1.49	.41
CMB	PC and LB	1.79	.08

[a]Petroleum coke.

[b]Gas purified.

[c]Lampblack.

TABLE 2.- RATIO OF NORMALIZED PEAK AREA TO PEAK HEIGHT FOR GRADE ATJ

ξ, deg	$I(\phi)_{area}/I(\phi)_{height}$	
	Specimen 1	Specimen 2
0	1.06	1.05
10	1.02	1.03
20	1.02	1.02
30	.99	.99
40	.98	.96
50	.92	.92
60	.93	.87
70	.86	.86
80	.88	.88
90	.88	.88
100	.89	.88
110	.91	.87
120	.92	.90
130	.96	.93
140	.99	.94
150	1.02	.99
160	1.09	1.04
170	1.09	1.01
180	1.11	.99
190	1.09	1.00
200	1.07	1.04
210	1.01	1.02
220	1.01	.99
230	1.00	.96
240	.96	.98
250	1.00	.94
260	.98	.95
270	.96	.93
280	.99	.94
290	.97	.96
300	1.00	.98
310	1.01	.97
320	1.05	1.04
330	1.05	1.04
340	1.10	1.05
350	1.10	1.06

TABLE 3.- VALUES OF ORIENTATION PARAMETERS

FROM LEAST-SQUARES METHOD

(a) Peak area data reduced with equation (17)

Grade	Specimen	B	M	α_S, deg	β_S, deg	δ, deg	σ^2
ATJ	1	0.52	2.8	114	91	11	2.1×10^{-4}
ATJ	2	.52	2.6	122	92	19	1.1
ATJ (G.P.)[a]	1	.42	3.1	122	89	19	2.2
ATJ (G.P.)[a]	2	.44	2.9	118	90	15	2.7
ATJ (G.P.)[a]	3	.42	3.1	114	91	11	3.6
2BE	1	.35	3.0	111	88	8	19.5
2BE	2	.31	3.3	103	84	6	1.7
2BE	3	.33	3.2	101	91	3	8.1
9-RL	1	.52	2.7	106	91	3	2.4
9-RL	2	.50	2.6	120	89	17	2.2
9-RL	3	.52	2.5	122	87	19	2.0
3499-S	1	.59	2.3	117	87	15	2.6
3499-S	2	.52	2.5	108	88	5	2.4
3499-S	3	.52	2.6	111	93	8	2.9
4007	1	.50	2.9	110	100	12	2.2
4007	2	.51	2.5	116	78	19	3.0
4007	3	.43	2.8	112	90	9	2.7
L31	1	(b)	(b)	(b)	(b)	(b)	(b)
L31	2	.81	2.6	149	95	46	1.9
L31	3	(b)	(b)	(b)	(b)	(b)	(b)
2D8D	1	(b)	(b)	(b)	(b)	(b)	(b)
2D8D	2	(b)	(b)	(b)	(b)	(b)	(b)
CDG	1	.61	3.0	107	78	13	3.1
CDG	2	.60	2.5	107	102	12	2.7
CDG	3	.59	2.6	128	87	25	2.6
CMB	1	.66	2.2	124	94	22	3.6
CMB	2	.67	2.1	120	88	17	6.3
CMB	3	.65	2.3	117	90	14	9.4

[a]Gas purified.

[b]No solution.

TABLE 3.- VALUES OF ORIENTATION PARAMETERS

FROM LEAST-SQUARES METHOD – Continued

(b) Peak height data reduced with equation (17)

Grade	Specimen	B	M	α_S, deg	β_S, deg	δ, deg	σ^2
ATJ	1	0.48	2.6	103	90	1	1.1×10^{-4}
ATJ	2	.51	2.7	109	91	6	.7
ATJ (G.P.)[a]	1	.41	3.0	111	89	8	1.8
ATJ (G.P.)[a]	2	.43	3.0	108	90	5	2.6
ATJ (G.P.)[a]	3	.38	3.0	107	90	4	1.6
2BE	1	.34	3.0	104	88	2	21.5
2BE	2	.29	3.4	98	84	8	2.0
2BE	3	.31	3.2	95	91	8	7.8
9-RL	1	.48	2.6	97	90	6	1.5
9-RL	2	.49	2.5	109	88	7	1.1
9-RL	3	.51	2.6	108	87	7	1.1
3499-S	1	.56	2.5	101	87	4	2.7
3499-S	2	.50	2.7	97	87	7	1.7
3499-S	3	.48	2.7	101	91	2	.6
4007	1	.46	3.0	102	98	8	1.8
4007	2	.48	2.6	106	79	12	1.3
4007	3	.41	3.1	101	89	3	1.5
L31	1	.86	2.2	109	86	8	1.3
L31	2	.87	1.9	102	91	2	1.1
L31	3	.86	1.7	115	87	12	1.4
2D8D	1	.83	1.9	116	88	13	1.8
2D8D	2	.86	1.7	113	90	10	1.7
CDG	1	.57	2.5	99	80	11	1.2
CDG	2	.57	2.9	96	100	12	1.1
CDG	3	.64	2.3	110	86	9	3.3
CMB	1	.67	2.4	108	94	7	2.4
CMB	2	.63	2.4	107	88	4	1.4
CMB	3	.62	2.7	105	90	2	1.8

[a]Gas purified.

TABLE 3.- VALUES OF ORIENTATION PARAMETERS

FROM LEAST-SQUARES METHOD – Continued

(c) Peak area data reduced with equation (19)

Grade	Specimen	b	α_S, deg	β_S, deg	δ, deg	$\sigma 2$
ATJ		0.51	109	91	7	4.2×10^{-4}
ATJ	2	(b)	(b)	(b)	(b)	(b)
ATJ (G.P.)[a]	1	(b)	(b)	(b)	(b)	(b)
ATJ (G.P.)[a]	2	(b)	(b)	(b)	(b)	(b)
ATJ (G.P.)[a]	3	.40	108	91	7	17.1
2BE	1	.37	104	88	2	35.9
2BE	2	.31	96	84	9	21.6
2BE	3	.32	95	91	8	25.1
9-RL	1	.51	101	91	2	6.0
9-RL	2	(b)	(b)	(b)	(b)	(b)
9-RL	3	(b)	(b)	(b)	(b)	(b)
3499-S	1	.59	112	87	10	5.4
3499-S	2	.52	102	88	2	6.8
3499-S	3	.52	105	93	4	8.3
4007	1	.50	105	100	11	6.7
4007	2	.52	111	77	16	7.0
4007	3	.44	105	90	2	12.1
L31	1	(b)	(b)	(b)	(b)	(b)
L31	2	(b)	(b)	(b)	(b)	(b)
L31	3	(b)	(b)	(b)	(b)	(b)
2D8D	1	(b)	(b)	(b)	(b)	(b)
2D8D	2	(b)	(b)	(b)	(b)	(b)
CDG	1	.60	105	77	13	3.7
CDG	2	.60	103	102	12	4.6
CDG	3	(b)	(b)	(b)	(b)	(b)
CMB	1	(b)	(b)	(b)	(b)	(b)
CMB	2	.67	115	87	13	6.7
CMB	3	(b)	(b)	(b)	(b)	(b)

[a]Gas purified.

[b]No solution.

TABLE 3.- VALUES OF ORIENTATION PARAMETERS

FROM LEAST-SQUARES METHOD – Concluded

(d) Peak height data reduced with equation (19)

Grade	Specimen	b	α_S, deg	β_S, deg	δ, deg	σ^2
ATJ	1	0.48	98	90	5	8.1×10^{-4}
ATJ	2	.51	104	91	1	5.6
ATJ (G.P.)[a]	1	.42	105	90	2	11.0
ATJ (G.P.)[a]	2	.43	103	90	0	10.6
ATJ (G.P.)[a]	3	.39	100	91	3	15.6
2BE	1	.35	98	88	6	38.1
2BE	2	.28	94	83	11	21.6
2BE	3	.30	92	91	11	25.3
9-RL	1	.47	94	90	9	8.6
9-RL	2	.49	103	88	3	8.5
9-RL	3	.51	103	87	4	6.4
3499-S	1	.55	98	87	6	6.2
3499-S	2	.49	94	87	9	6.2
3499-S	3	.48	97	91	6	7.6
4007	1	.45	98	98	10	6.8
4007	2	.49	100	78	12	8.1
4007	3	.40	96	88	7	11.4
L31	1	.86	108	86	7	1.3
L31	2	.87	101	91	2	1.1
L31	3	.87	111	88	9	1.5
2080	1	.83	112	88	9	1.9
2080	2	.87	110	91	7	1.8
CDG	1	.56	96	79	13	4.5
CDG	2	.55	94	100	13	2.1
CDG	3	.64	107	86	6	4.3
CMB	1	.67	105	94	5	2.8
CMB	2	.63	103	88	2	3.0
CMB	3	.61	102	90	1	2.7

[a]Gas purified.

TABLE 4.- PERCENT DEVIATION OF PEAK HEIGHT DATA FOR GRADE 2BE

ξ, deg	$100\frac{I(\phi)_{calc} - I(\phi)_{exp}}{I(\phi)_{exp}}$ (Values of $I(\phi)_{calc}$ are based on eq. (17))		
	Specimen 1	Specimen 2	Specimen 3
0	16.6	5.6	13.2
10	13.0	2.3	10.4
20	10.1	-1.0	5.1
30	9.2	-.7	5.2
40	12.2	1.5	3.7
50	12.2	4.0	7.1
60	9.9	1.2	5.2
70	8.9	-.5	5.0
80	5.3	.2	2.5
90	-.3	-.4	-1.0
100	-5.2	1.0	-2.2
110	-7.2	-1.6	-3.6
120	-10.1	-1.1	-4.4
130	-10.8	-3.6	-3.6
140	-11.4	-6.2	-4.4
150	-11.8	-5.3	-9.3
160	-11.7	2.5	-7.0
170	-7.3	6.9	-1.8
180	-5.4	1.9	.1
190	-9.9	-1.3	-6.2
200	-8.7	-5.0	-10.9
210	-10.3	-2.6	-10.5
220	-6.4	-.9	-6.1
230	-4.2	.2	-4.4
240	-3.9	1.8	-1.3
250	-3.7	-1.3	-2.0
260	-2.5	-1.9	-1.4
270	-1.0	-1.5	-.2
280	2.3	1.0	.5
290	4.5	3.7	-.1
300	7.0	4.3	6.5
310	8.8	0	7.7
320	8.9	-3.3	5.6
330	8.7	-2.5	1.9
340	8.2	2.9	5.9
350	14.1	3.0	6.8

TABLE 5.- ORIENTATION PARAMETERS FOR GRADE 2BE FROM PEAK HEIGHT DATA AND EQUATION (17)

Specimen	$\Delta\xi$, deg	B	M	α_S, deg	β_S, deg	δ, deg	σ^2
1	2.5	0.34	2.9	105	87	3	18.6×10^{-4}
1	10.0	.34	2.9	105	88	3	20.7
2	2.5	.29	3.4	97	84	8	2.0
2	10.0	.30	3.4	97	84	8	2.1
3	2.5	.31	3.2	95	90	8	6.2
3	10.0	.31	3.2	95	90	8	7.4

TABLE 6.- COMPARISON OF RESULTS OF ALI AND BACON METHODS

Grade	I(90)/I(0) (Ali, ref. 4)	B [a] (Bacon, ref. 3)
	Peak area data	
ATJ	0.56	0.52
2D8D	.96	(b)
CMB	.68	.66
	Peak height data	
ATJ	0.57	0.50
2D8D	.99	.85
CMB	.52	.64

[a] Average least-squares solution to equation (17) for all specimens of the grade.

[b] No solution.

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