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Variances in the Measurement of Thermal Diffusivity on Coarse-Weave Carbon-Carbon Composites in Terms of Fiber-Fraction Involvement

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A geometrical analysis was performed on the sensitivity of the fiber-fraction parameter applicable to the sighting area of a detector for thermal diffusivity measurements using the flash technique on a coarse-weave C-C composite. The percentage variation in the detector viewing-area fiber fraction was examined as a function of viewing diameter for the axial and radial cases of a proprietary material, billet 304. Suggestions are made for optimization of the viewing diameter for thermal diffusivity measurements. The uncertainty limits caused by small displacements of the spot center or by uncertainties in the spot diameter are derived for viewing diameters of 0.1 and 0.5 in. Selected thermal diffusivity data obtained from room temperature to 1300 K using a 0.1 in. diameter spot on three different types of proprietary coarse-weave carbon-carbon composites (100, 200, and 300 series) are presented. From the analysis of these data, it is concluded that the thermal diffusivity shows a strong dependence upon fiber fraction at room temperature and that this dependence decreases considerably at higher temperatures. Typical thermal diffusivity results obtained in the radial direction for billet 206 as a function of temperature using both 0.10 and 0.37 in. diameter spots are analyzed in view of the conclusions drawn in the geometric analysis study. Finally, suggestions are made concerning the characterization necessary in reporting data on the thermal diffusivity of coarse-weave carbon-carbon composites.

KEY WORDS: carbon-carbon; composites; thermal diffusivity.

1. INTRODUCTION

For the measurement of the thermal diffusivity on coarse-weave C-C composites, the matter of infrared (IR) detector viewing on the back face of

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the sample is an important one. It is expected that this viewing area should be representative of the bulk material. Since the viewing area is circular in shape and the coarse-weave structure has a rectangular geometry, a significant problem arises when representing the actual fiber fraction of the composite within a small circular area. One would like to know, for instance, the number of unit cells which must be included within a circular viewing area in order to represent the fiber fraction of the bulk composite material with reasonable accuracy. A 0.1 in. spot diameter is used for thermal diffusivity measurements at some laboratories, while others customarily view most of the rear face of a sample, typically 0.5 or 0.75 in. in diameter. The supposition is that, if one views the entire rear face, one includes more unit cells and will have a better representation of the material. The present efforts attempt to study the extent of the uncertainties which arise when representing the fiber fraction (f) in a circular viewing area as compared to the fiber fraction of the composite, referred to here as f-unit cell.

The fact that uncertainties in f do result when specimen viewing is restricted can be easily demonstrated through Fig. 1. This figure shows a representative billet cross-section in the radial direction, with circles of 0.1 and 0.5 in. drawn to represent viewing areas with different center locations. It can be seen from Fig. 1 that, when a 0.1 in. circle is drawn centered on a fiber bundle, the circle includes only one bundle, but when the circle is centered on the transverse-bundle overlap, almost no fibers are included. Thus the fiber fraction within the circular viewing area is a strong function of diameter and location of the spot on the unit-cell face. Figure 1 also depicts two circles of 0.5 in. diameter drawn at two different locations to illustrate this point further. When a 0.5 in. circle is drawn centered on a fiber bundle, it includes 21 full bundles, but when centered on the transverse-bundle overlap, it includes 16 full bundles plus 8 fractional ones for an equivalent area of 17.86 full bundles. The fiber fraction, defined as the area occupied by fiber ends per unit spot area is 0.1371 in the first case and 0.1166 in the second. Thus it is illustrated that even if one uses a larger diameter, f in the viewing area is still a function of location. Another aspect of the variation in f is that due to small uncertainties in spot diameter, which also can be illustrated through Fig. 1. Consider the 0.5 in. diameter spot located at the center of a fiber bundle. Now, if one allows $\pm 5\%$ variation in diameter, it is seen that a 5% increase in diameter does not include any additional fiber area, but that the increase in diameter does have the net effect of decreasing f in the circular viewing area. On the other hand, a 5% decrease in diameter will reduce the fiber area appreciably more than 5%. In the case of the 0.5 in. diameter spot centered on a transverse-

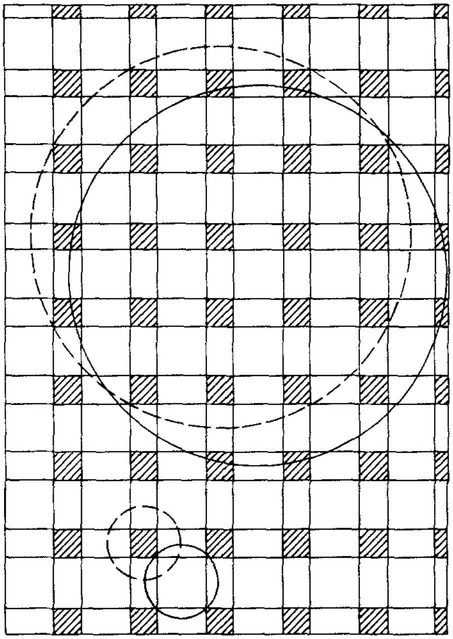

Fig. 1. Illustration of effect of area displacement (0.1 and 0.5 in. in diameter). The dashed line circle is centered on the fiber-bundle end: 0.1 in. circle includes 1 bundle; 0.5 in. circle includes 21 bundles. The solid line circle is centered on the transverse-bundle overlap: 0.1 in. circle includes no bundle; 0.5 in. circle includes 16 bundles + 8 fractional bundles (≡ to 1.86 bundles ~2bundles) for a total of 18 bundles.

bundle overlap, diameter variations can have an even larger effect on f for a circular viewing area. The matter is further complicated if one considers the contributions of fiber-bundle bulging and unit-cell distortion in these calculations. Thus it is essential to evaluate the effects of spot size, spot location, and small changes in each of these, in order to determine f in situations involving circular viewing areas. A geometric analysis of the fiber-fraction parameter for billet 304 was therefore undertaken, the results of which are presented below.

The purpose of this work is, in broad terms, to evaluate the effect of fiber fraction on thermal diffusivity measurement results. To pursue this goal, thermal diffusivity data [1] were obtained for similarly woven billets having different unit-cell dimensions. The thermal diffusivity was measured by the flash diffusivity technique, using a 0.1 in. spot ir detector to measure temperature response, over the temperature range from room temperature to 1300 K. Included with some of the data are f values obtained by microscopic examination of the specimen viewing area [1]. Due to limitations of the available information, the present evaluation is limited only to the qualitative aspects of the thermal diffusivity and fiber-fraction relation, and no quantitative conclusions are drawn.

An attempt is made to verify the results of the geometrical analysis using experimental data. Observed thermal diffusivity versus temperature values were obtained using 0.1 and 0.37 in. spot diameters for radial direction specimens from inside and outside diameter locations of billet 206, for which variations in f amounting to a factor of two had been observed. An evaluation of these data is carried out, utilizing the results of the geometric analysis. Observations are presented to draw attention to questions concerning those conditions under which the effects of changes in spot size may be experimentally observed. Finally, qualitative conclusions are presented on the effect of fiber fraction upon the thermal diffusivity, and recommendations are given for the appropriate characterization of measurement conditions in reporting thermal diffusivity data.

2. GEOMETRICAL ANALYSIS

The goal of the geometrical analysis for a coarse-weave structure is to determine the range of fiber fraction in a circular viewing area for the spot diameter under consideration. This range is obtained in terms of the f_{max} and f_{min}, which are observed by moving the spot center across the face of a unit cell.

There are two distinct aspects of the geometrical analysis. First is the identification of the extremal points on the face of the unit cell. For instance, consider a case where a spot of some diameter is displaced across the face of the unit cell under observation. One can realize that f in the viewing area is going to change as the position of the center of the spot is changed. The positions at which the extrema (maxima or minima) in f occur must be known. A detailed evaluation of this problem was carried out for simple unit cells and for the unit cells of billet 304. A detailed note on this study is in preparation [2].

The second part of the analysis consists of a calculation of f at the extremal points for circular viewing areas of different diameters. Also included are calculations for the effects upon f resulting from minor (5 to 10%) changes in spot size or shifts in the location of the spot center.

The unit-cell nomenclature and dimensions for billet 304 [3] used in this study are presented in Fig. 2. The figure shows axial and radial direction views with extremal points 1 through 4 marked on them. Point 1 represents the center of a fiber bundle, point 3 represents the center of the

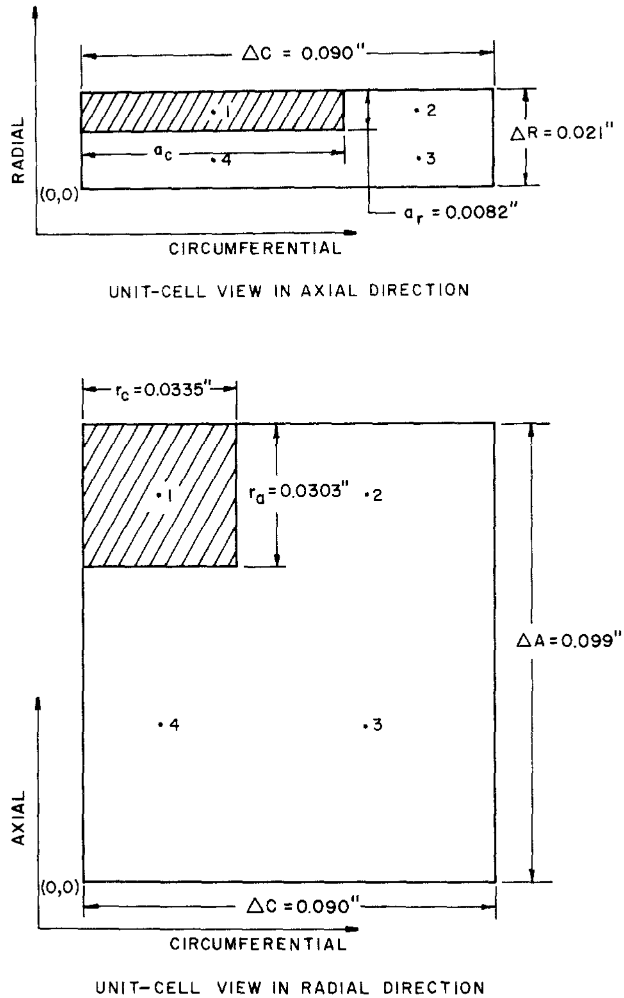

Fig. 2. Unit-cell views for billet 304 (IF section).

transverse-bundle overlap, and points 2 and 4 are centers of the remaining regions, alternately transverse faces or interstitial matrix pockets.

A computer program was developed to carry out the calculations required for this analysis. This program uses the measured dimensions of the unit cell and the location of the viewing-spot center as input and calculates f for a circular viewing area at diameters of 0.025 to 1 in. The program also calculates f as a function of ± 5 and $\pm 10\%$ changes in

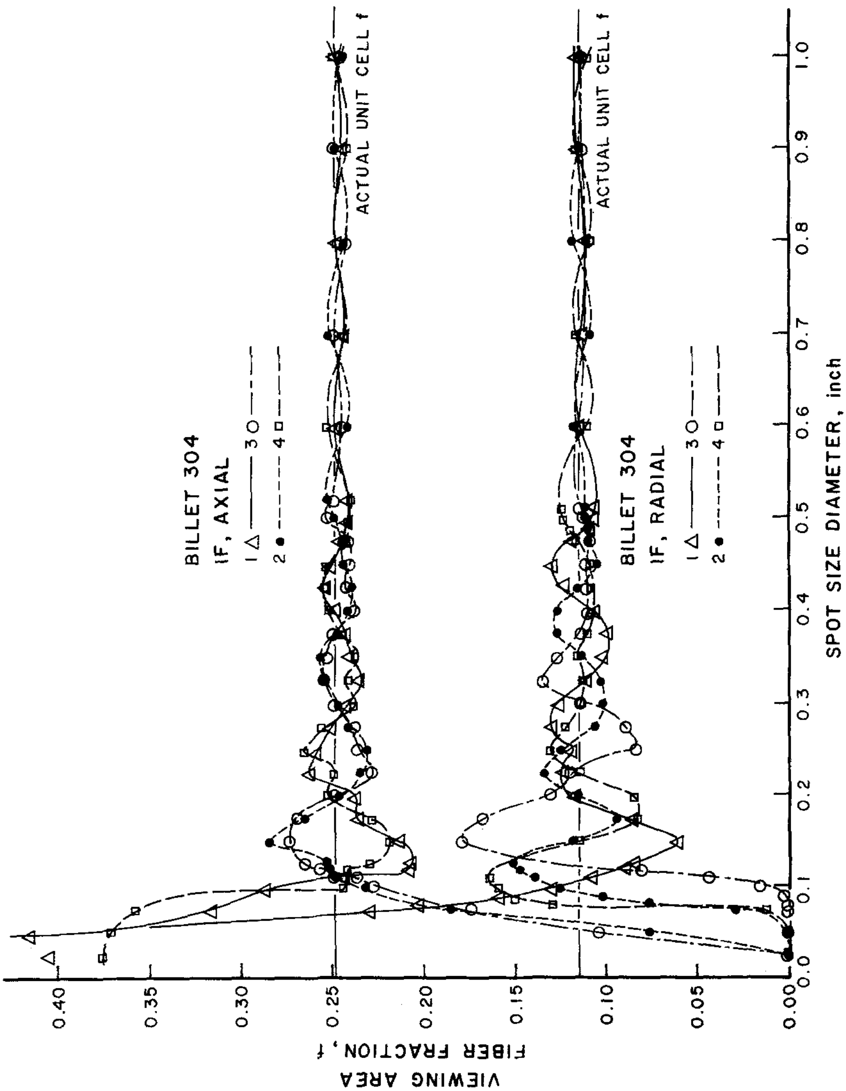

Fig. 3. Variation of viewing-area fiber fraction with spot diameter.

viewing diameter and spot center displacements along the x and y directions, with both calculations being carried out for viewing areas of 0.1 and 0.5 in. diameter.

The calculations on billet 304 were completed for IF (inside forward), IA (inside aft), and OA (outside aft) sections. The results of IF axial and radial directions are presented in Fig. 3. The results for the other sections followed similar patterns. The curves in Fig. 3 show f versus spot diameter with the extremal points 1 to 4 as spot centers. The oscillations in the curves are due to the periodic nature of the weave structure. All curves eventually attenuate, but spot diameters of more than 1 in. are required for attenuation to the f-unit cell value. Also, these curves attenuate out of phase, which gives them an asymmetric appearance (see Fig. 3). The asymmetry in the oscillations means that an average over a large number of measurements would still not reproduce the unit-cell results. Since the uncertainty limits of f are very large, the procedure of testing the same sample with different spot locations and interpolating the results to obtain a result corresponding to the bulk f value is not feasible.

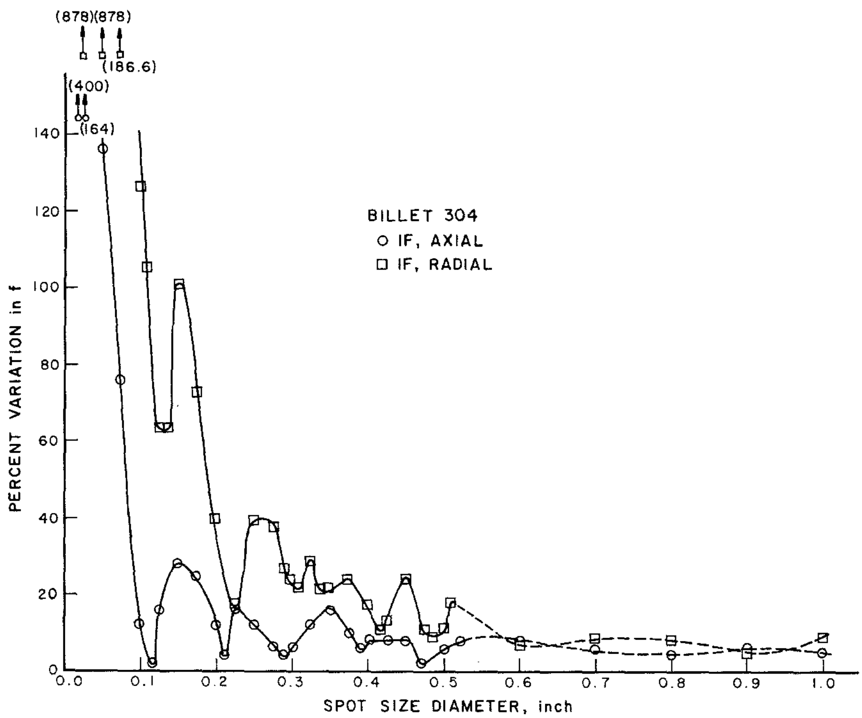

Fig. 4. Percent variation in f as a function of spot diameter.

It is of interest to know the extent of the deviations in the viewing-area fiber fraction f. The percent variation in f is defined as follows:

$$\text{percent variation in } f = \frac{f_{\max} \text{ in viewing area} - f_{\min} \text{ in viewing area}}{f\text{-unit cell}} \times 100.$$

Figure 4 shows that possible variations in viewing-area fiber fraction f are quite large at 0.1 in. spot diameter. Even at 0.5 in. diameter, these variations are of the order of 10%, and one has to use diameters of 0.6 in. and above in order to be within 10%. Thus it appears from Figs. 3 and 4 that the choice of viewing most of the sample is desirable, and consequently, that large diameter specimens should be used. The situation is

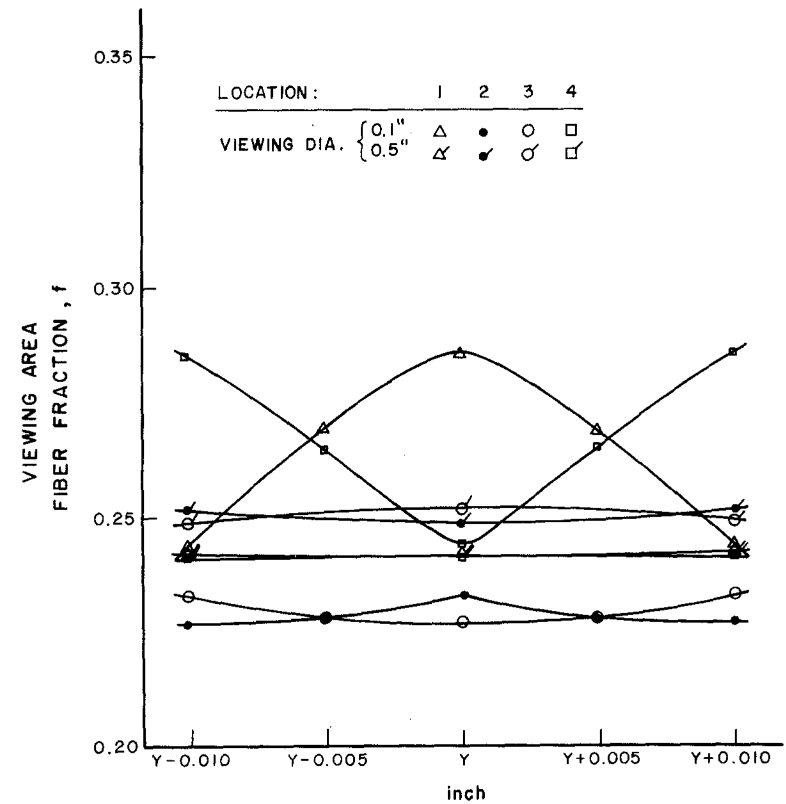

Fig. 5. Viewing-area fiber-fraction variation due to displacement of spot center along y axis for billet 304, IF axial.

worse for the OA section (results are not presented here), as the weave becomes more coarse in this case.

The variations in f due to minor (5 to 10%) variations from the 0.1 and 0.5 in. spot diameters were investigated. For the radial direction, a 5 to 10% variation in a 0.1 in. diameter can cause variations in viewing area f of more than 100%, depending upon spot location. These variations become the order of 5% in the case of a 0.5 in. viewing diameter. For the axial case, the results are very similar.

Typical variations of f due to displacement of the spot center along the x and y directions by ± 0.005 and ± 0.010 in. are represented in Fig. 5 for the axial case. It is noted that slopes of the curves are zero for the points 1 to 4. This confirms that points 1 to 4 are extremal points. It is also seen that the effect of displacement of the center may be minor for some locations but significant for others. In the worst cases, this effect can cause deviations of up to 10 to 20% in viewing area f. Similar situations are observed for the radial case.

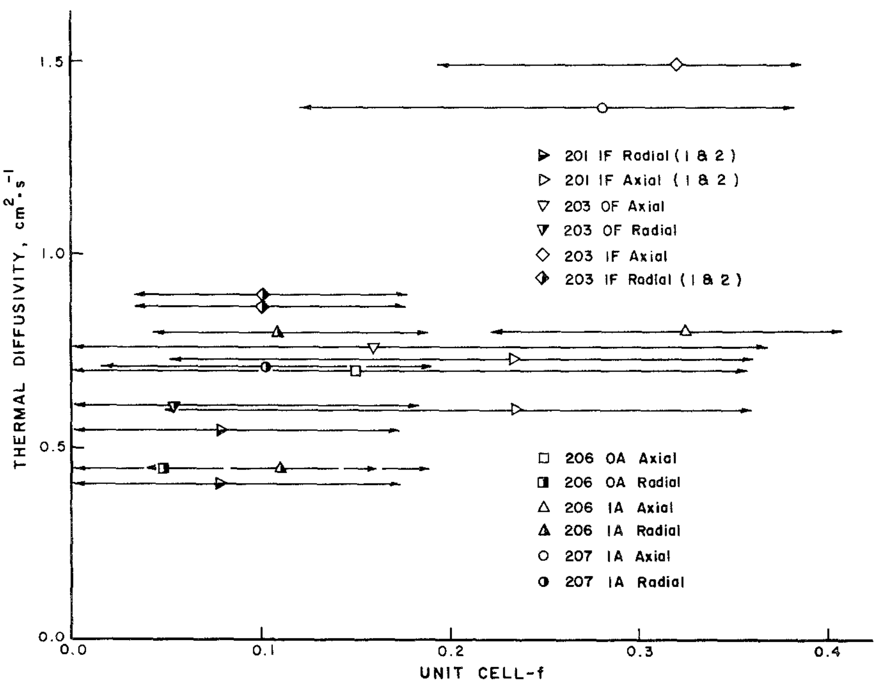

Fig. 6. Room temperature thermal diffusivity versus unit-cell f showing uncertainty limits in f for 200 series billets.

3. RELATION OF FIBER FRACTION TO THERMAL DIFFUSIVITY

In this analysis it is assumed that the values of f to be associated with reported values of the thermal diffusivity are the measured values for unit-cell f. The plot of room temperature diffusivity values versus unit-cell f (Fig. 6) uses bars to express the theoretical band of possible f values corresponding to each thermal diffusivity value. In view of the extent of the uncertainties in f shown in Fig. 6, it is very difficult to draw any conclusion concerning the dependence of the thermal diffusivity at room temperature upon f. Figure 7 shows the thermal diffusivity versus fiber fraction (unit-cell f) for temperatures of 100, 500, and 1000°C. This figure indicates that the thermal diffusivity versus unit-cell f behavior at higher temperatures can be approximated with straight lines. One can easily see that as the temperature increases, the slope of these lines decreases, implying that $d\alpha/df$ (where α is the thermal diffusivity) decreases as the temperature increases. This behavior was also noted for other billets as shown, for example, in Fig. 8 for 100 series billets.

Figure 9 shows that the thermal conductivity values presented for an

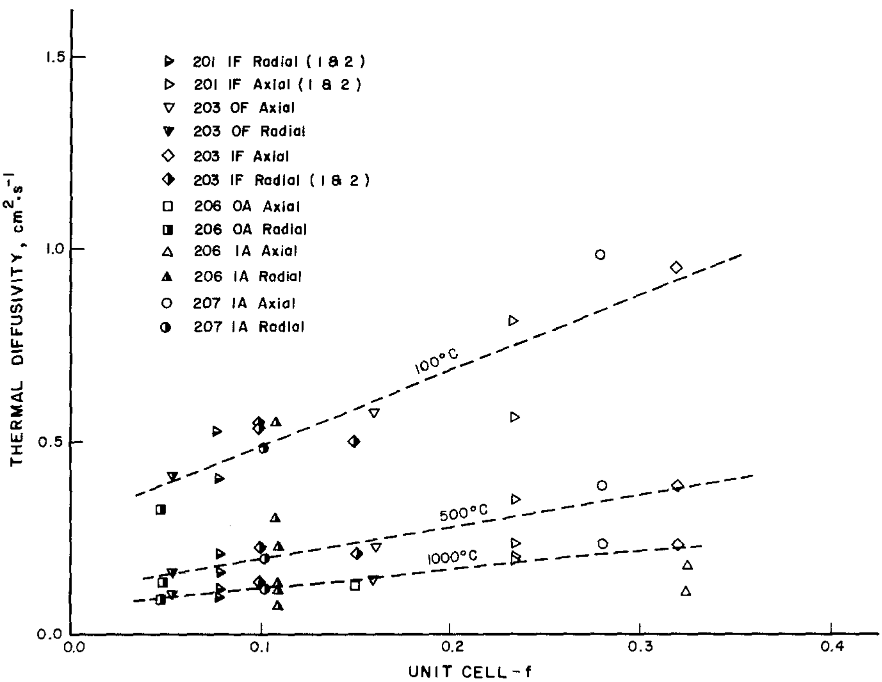

Fig. 7. Thermal diffusivity versus unit-cell f at elevated temperatures for 200 series billets.

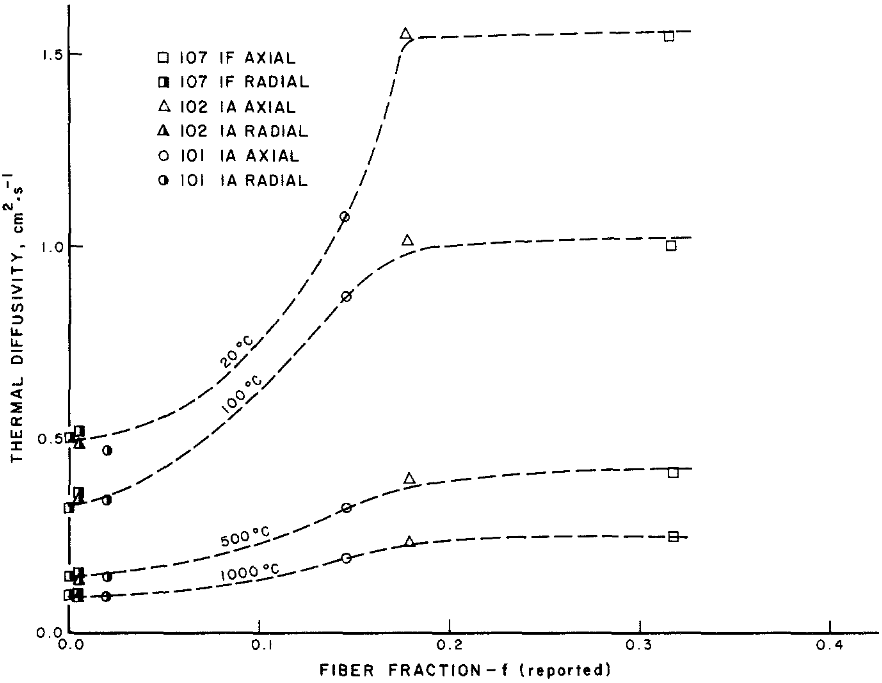

Fig. 8. Thermal diffusivity versus reported *f* from room temperature to 1000°C for 100 series billets.

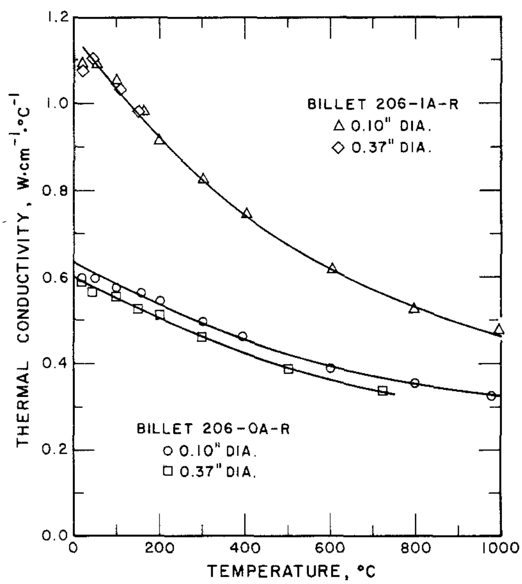

Fig. 9. Derived thermal conductivity versus temperature for billet 206 in radial direction using 0.10 and 0.37 in. spot diameters.

Table I. Values of f at Different Locations in Billet 206 for 0.10 and 0.37 in. Diameter Viewing Spots

Location	Unit cell f	Spot location	f During measurement 0.10	f During measurement 0.37	% Difference[a] 0.10	% Difference[a] 0.37
IA-R	0.1087	1	0.0941	0.1016	− 13.43	− 6.53
Inside diameter		2	0.1883	0.0968	73.23	− 10.95
after section		3	0.0386	0.1240	− 64.49	14.06
Radial direction		4	0.0921	0.1078	− 15.27	− 0.83
OA-R	0.048	1	0.08195	0.0365	70.73	− 23.96
Outside diameter		2	0.1630	0.0417	239.58	− 13.13
after section		3	0.0000	0.0525	− 100.00	9.38
Radial direction		4	0.0000	0.0579	− 100.00	20.63

[a] % Difference = $\dfrac{f_{\text{measurement}} - f_{\text{unit cell}}}{f_{\text{unit cell}}} \times 100.$

OA section at lower temperatures are approximately half those for the companion IA section, for which the unit-cell f is also halved (see Table I and Fig. 7). However, the diffusivity values resulting from increasing the spot diameter from 0.10 to 0.37 in. did not appreciably change for the IA section, while for the OA section, the diffusivity values decreased by only 5–10%. Since the potential for changing the fiber fraction in the viewing area upon increasing the spot diameter from 0.10 to 0.37 in. is large (Figs. 3 and 4), these results appear to be contradictory. Consequently, the f values at the four extremal points for the 0.10 and 0.37 in. diameter viewing spots were calculated. The results are shown in Table I. By examining this table, we see that there are locations at which changing the spot diameter from 0.10 to 0.37 in. would result in almost no change in f, and there are other locations in which the change in f would be huge. Since we do not know the exact location of the viewing spot, it is obvious that we could choose locations which would explain these results. Another important fact is that this spot-variation study was done on radial samples, for which the fiber fraction is about 0.05 to 0.10. Referring to Fig. 8, it can be seen that $d\alpha/df$ approaches 0 as f approaches 0. Therefore, a relatively large change in f would cause a smaller change in α in this case.

4. CONCLUSIONS

The following conclusions can be drawn from the geometrical analysis of a composite having a coarse-weave structure and from the qualitative analysis of C-C composite billets of series 100, 200, and 300:

1. The spot size of the viewing area is an important variable in the flash thermal diffusivity measuring technique. It is advisable to use most of the sample face as the viewing area for IR detection. In this discussion, by optimum diameter we mean those diameters for which the viewing area fiber fraction f differs from unit-cell f by no more than an acceptable amount. From the results presented here, it is concluded that viewing diameters of 0.6 in. or greater are required in order for f to be within $\pm 10\%$ of unit-cell f for the IF section of billet 304.
2. For the 0.1 in. spot diameter case, the percentage variation in f as based upon extremal values can be very high, as much as 125% for the radial case and 25% for the axial case. A 5–10% uncertainty in diameter causes up to 20% variation in the viewing area f. A similar imprecision in location could cause up to 10 to 15% uncertainty.
3. An examination of the thermal diffusivity versus fiber fraction reveals that the thermal diffusivity depends upon the fiber fraction but that this dependence becomes weaker as the temperature is increased.
4. The change in the thermal diffusivity with respect to a change in the fiber fraction approaches zero for values of f in the range from 0.0 to 0.1 for all billets. In certain instances, one may not observe a dependence of α on spot diameter, as experienced in the case of billet 206 IA radial samples, which have an f range wherein $d\alpha/df$ is small.
5. The results of the analysis show that the value of f appropriate to the thermal diffusivity measurement may be significantly different from the unit-cell value. Consequently, it is necessary to have a fiber-fraction value applicable to the measurement before the thermal diffusivity to fiber-fraction correlation can be examined quantitatively.
6. The value of f in the viewing area, while converging to unit-cell f for large diameters, may still be significantly different from the value planned in the manufacture of the billet.
7. By using a smaller spot size (as pointed out in item 2 above for a 0.1 in. spot), one incorporates larger uncertainty limits in f. Thus data generated under these conditions cannot be used quantitatively to evaluate the dependence of the thermal diffusivity upon fiber fraction, as each reported fiber-fraction value carries these wide uncertainty limits. Also, due to asymmetry in the oscillations observed in the curves of f versus the viewing diameter, one should not expect

that large numbers of measurements could resolve the uncertainty involved in the measurements.

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