THERMAL DIFFUSIVITY MEASUREMENTS IN CARBON-CARBON COMPOSITES

D. Michele Heath and William P. Winfree
NASA
Langley Research Center
Hampton, VA  23665

INTRODUCTION

In recent years, carbon-carbon composite materials have come into widespread use in aerospace industries. These materials are particularly attractive for high temperature applications due to their thermal and mechanical behavior. Few quantitative measurements, however, have been made to characterize these materials. One problem encountered with carbon-carbon composites is porosity. Materials engineers have determined that degree of porosity is correlated to inter-laminar shear strength in carbon-carbon composites. Since repetition of the carbon-carbon processing cycle reduces porosity, a technique for assessing porosity between processing cycles that is non-contacting and does not contaminate the material would be of value. A material property which is related to density and therefore to porosity, is thermal diffusivity. Thermal diffusivity is easily measured non-contactingly and remotely with infrared techniques and is therefore an attractive candidate measurement for assessing porosity between processing cycles of carbon-carbon composites.

In this work, a non-contacting measurement technique for measuring thermal diffusivity which utilizes a Nd-YAG laser as a thermal wave source and an IR camera detector is described. Thermal diffusivity is extracted from the phase delay between the thermal wave source and a spatially offset detection region. Results are given for three orthogonal directions to indicate the variation of diffusivity with structural anisotropy of the material. Diffusivity measurements taken in a linear pattern over the sample surface are presented to illustrate the effect of local variations in the fiber direction due to fiber weave in the composite lay-up. In addition, results are given for diffusivity measurements of a series of samples which have undergone varying numbers of repetitions of the processing cycle, producing a range of porosity levels. A strong correlation of diffusivity in the through ply direction with the bulk density, and therefore with the level of porosity of the composite material was shown.

EXPERIMENT

By measuring the temperature of carbon-carbon samples as a function of time, at the source of heating and comparing this function to a similar measurement made simultaneously over a region spatially offset...
from the source, the phase delay, and thus the thermal diffusivity of the samples was found. Two experimental configurations were used to determine the diffusivity in directions in the plane of the sample face and perpendicular to the sample face, i.e. through the sample thickness. Figure 1 is a schematic diagram of the experimental configuration used for determining diffusivity in the plane of the sample. A Nd-YAG laser operating at 1.06 \textmu m was used as a thermal point source. The source was pulsed at 1 Hz. for in plane measurements. The detector was a video format infrared camera, sensitive to 8-12 \textmu m radiation. The temperature images obtained were digitized and stored on disk for the analysis processing described below. For in plane measurements, the detector was positioned on the front face side of the sample, i.e. on the same side that heating occurred. A spatial profile of the point source is shown in figure 2, obtained by taking a cross section of one image frame. Vertical cross sections of the temperature images correspond to heat flow in the cross fiber direction, while horizontal cross sections correspond to flow in the fiber direction. Monitoring the temperature at a point 2 cm from the source yields the curve shown in figure 3.

When considering flow in the through ply direction, the experimental configuration shown in figure 4 was used. In this configuration, the camera was positioned on the side of the sample opposite to the side that heating occurred. A beam splitter was used to direct a portion of the laser beam to the back side of the sample to be used as a timing reference. Due to the low diffusivity in the through ply direction, a lower frequency heating pulse, .08 Hz, was used. In all other aspects,
the experimental technique for the through ply measurement was identical to that for the in plane measurements.

THEORY

Frequency domain analysis using the phase delay between a thermal stimulus and response is a common approach to measuring thermal diffusivity in solids. For this variation, a harmonically varying thermal point source is used and the phase lag, measured as a function of distance from the source, is used to calculate the diffusivity of the material. Carslaw and Jaeger show in [1] that for a two dimensional case with periodic heat source, the temperature profile is given by a modified Bessel function. When the distance from the source, $x$, is sufficiently large, the phase of this thermal wave is approximately linear. The phase versus $x$ curve, then, will approach a linear function for $x$ greater than some critical value $x_c$. The slope of this line is proportional to the thermal diffusivity of the material by the following equation:

$$k = \frac{\pi F}{m^2}$$

(1)

where $k$=thermal diffusivity
$F$= source frequency
$m$= slope of phase versus position curve.

The phase of the thermal wave at a given point is found by numerically calculating the Fourier transform of the temporal history of temperature at that point. The inverse tangent of the imaginary divided by the real components of the Fourier transform gives the phase of the thermal wave at the given point. Repeating this calculation for each point on the scan line and referencing this phase to that of the phase at the source position, results in the phase versus $x$ curve. Figure 5 depicts a phase versus $x$ curve for one sample, measuring $x$ in the plane of the sample face, parallel to the fiber direction. It can be seen that the phase approaches 0 at $x=1.1$ cm, corresponding to the location of the source, and increases with distance from the source. For values of $x$ greater than approximately 1.15 cm, the phase function is roughly linear, until distance from the source becomes so great that the signal is lost to noise. A segment of the curve in the linear region, along with its best linear fit, is given in figure 6. It is shown that the linear approximation is reasonable for the segment of the curve where $x$ is between 0.02 and 0.25 cm from the source, for this sample and at the given frequency of 1 Hz. Using the slope of the linear fit then, the diffusivity is calculated from equation 1.
RESULTS

The technique described above was used to determine the diffusivity of carbon-carbon coupons which were fabricated to give a range of porosity levels. The coupons were fabricated in a two dimensional orthogonal weave configuration, with a 0°-90° ply lay-up. Figure 7 depicts the fiber weave-layup configuration used. A series of 7 samples was measured, each of which had undergone a different number of densification cycles during processing. During these densification cycles, the samples are immersed in a phenolic resin and then pyrolized. The resin penetrates the pores and upon pyrolysis, is reduced to carbon residue, which fills the pores. By varying the number of densification cycles, samples were produced with a range of porosity levels. The level of porosity was reflected in the measured bulk density of the sample, which increased when densification was repeated. The 7 samples used, spanned a range of densities from 1.435 to 1.645 gm/cm³.

Measurements were made on the coupons in the through ply, or z direction. These results are shown in figure 8 which plots diffusivity against density, with the discrete points representing measured data and
the line representing the best linear fit to these points. A definite upward trend is seen as density increases, with a functional dependence of diffusivity with density that is roughly linear.

Measurements were also made in the plane of the sample face, both parallel and perpendicular to the fibers. These results are shown in figures 9 and 10 respectively. As the figures indicate, no consistent trend is seen over the range of densities measured. The values measured for the cross fiber direction, however, were consistently lower than the values found in the fiber direction, as expected.

To further illustrate the fiber orientation effect, a linear source was used which extended over the fiber weave on the sample surface, so that in some areas the measurement represented diffusivity parallel to the fibers, and in other areas represented diffusivity perpendicular to the fibers. The results of these measurements for one sample are shown in figure 11. Diffusivity is plotted here as a function of position across the sample. The positions between 0.15 to 0.4 cm, and 1.15 to 1.4 cm correspond to cross woven fiber bundles, so that measurements here represent diffusivity in the cross fiber direction. Again, the expected increase in diffusivity in the direction of the fibers is seen. A change of approximately 40 percent is shown for this sample.
Figure 9. Diffusivity Versus Density
In Fiber Direction

Figure 10. Diffusivity Versus Density
Cross-fiber direction

Figure 11. Diffusivity Versus Position
DISCUSSION

The measurement results presented indicate that standard thermal measurement techniques are applicable to characterizing carbon-carbon composites. The thermal diffusivity values obtained in each of the three orthogonal directions fell within the range of values quoted in the literature. Since the fibers are the prime thermal carriers, the fiber direction is expected to have the highest diffusivity, with the cross-fiber and through-ply directions following. This relationship was observed in these results. Further, the thermal wave phase detection technique, when used in the through-ply configuration, was shown to be sensitive to the level of density changes normally encountered in carbon-carbon processing. Through-ply measurements are expected to be more strongly affected by density changes due to the greater contribution of the matrix properties in this direction. Since the diffusivity in the in-plane directions are highly affected by the fibers, which are not changed by repeated densification of the composite, this may explain the absence of any correlation between diffusivity and density for the in-plane results. In the through-ply direction, a strong correlation was observed, with a roughly linear functional dependence. Care must be taken when measuring carbon-carbon materials, to account for fiber orientation, since this effect was shown to be larger than the effect of the small density changes of interest. In the through-ply direction, however, this effect is minimized, and the technique can be used to monitor carbon-carbon materials between processing cycles.

REFERENCES