Microscopic Thermal Diffusivity Mapping Using an Infrared Camera

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ABSTRACT

Standard flash thermal diffusivity measurements utilize a single-point infrared detector to measure the average temperature rise of the sample surface after a heat pulse. The averaging of infrared radiation over the sample surface could smear out the microscopic thermal diffusivity variations in some specimens, especially in fiber-reinforced composite materials. A high-speed, high-sensitivity infrared camera was employed in this study of composite materials. With a special microscope attachment, the spatial resolution of the camera can reach 5.4 μm . The images can then be processed to generate microscopic thermal diffusivity maps of the material.

SRM 1462 stainless steel was tested to evaluate the accuracy of the system. Thermal diffusivity micrographs of carbon-carbon composites and SCS-6/borosilicate glass were generated. Thermal diffusivity values of the carbon fiber bundles parallel to the heat flow were found to be higher than the matrix material. A thermal coupling effect between SCS-6 fiber and matrix was observed. The thermal coupling and measured thermal diffusivity value of the fiber were also dependent upon the thickness of the specimen.

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INTRODUCTION

Composites and other inhomogeneous materials present a problem for traditional thermal diffusivity measurements, i.e. the local variations in diffusivity cannot be detected. The infrared detector used in the standard flash diffusivity technique[1] is a single point detector which responds to the average thermal radiation from the specimen surface. It cannot distinguish high or low diffusivity regions in the sample. The basic assumptions for the flash technique are that the specimen is homogeneous and that one-dimensional heat flow occurs. These assumptions are not valid in the case of composites. Computer modeling was used to predict the effective thermal diffusivity of composites [2,3]. Thermal diffusivity mapping has recently been used to overcome the problem of averaging thermal radiation from the surface [4-6]. With improved spatial resolution and digital image processing capability, it is possible to generate thermal diffusivity maps with microscopic resolution.

Experimentally determining the thermal diffusivity is very important to the modeling of heat transfer and the understanding of thermal properties in composite materials. The effects of sample thickness, fiber volume fraction, and interface quality between the fibers and matrix can be determined by the thermal diffusivity mapping technique. These factors are closely related to each other in affecting the measured thermal diffusivity.

A detailed study of thermal diffusivity mapping is presented in this paper. The effects of local heating and coupling between fiber and matrix were investigated. A homogeneous standard material, i.e. SRM 1462 stainless steel, was studied and the effect of averaging was observed by systematically increasing the pixel numbers in an Area of Interest (AOI). Thermal diffusivity of composites was obtained by the same technique. Local variations were observed in carbon-carbon and SCS-6/borosilicate composites. When a single fiber in borosilicate matrix was studied, strong thermal coupling effects and thickness dependency were observed.

EXPERIMENTS

A high-speed, high-sensitivity infrared camera, Amber Galileo, was used in this study. The IR camera utilized a 256 x 256 InSb Focal Plane detector array. The snap-shot mode of the camera allows all pixels to capture thermal radiation from a surface simultaneously. In the sequence mode, the camera can take 145 images per

Figure 1. Experimental setup for microscopic thermal diffusivity mapping

second upon triggering. Detailed descriptions of the camera can be found in other references [4,5].

Figure 1 shows the experimental setup for thermal diffusivity mapping. The IR camera was not originally equipped for microscopic studies. A special sample holder/manipulator was used to get better images. To avoid vibration caused by firing of the flash lamp, the sample holder was placed on a separate foundation from the lamp.

Samples for thermal diffusivity mapping were cut into small discs with varying thickness. A high power xenon flash lamp with four flash bulbs was used to provide the heat pulses. The IR camera was positioned to monitor the back surface of the sample. The trigger signal came from a LED diode at the vicinity of the flash lamp. The imaging software collected up to 250 images at a preset frequency. Three sets of samples were used in this study:

- 1) Bulk SRM 1462 stainless steel.
- 2) 4 directional (4D) carbon-carbon composite.
- 3) A single Textron SCS-6 fiber in borosilicate glass matrix.

RESULTS AND DISCUSSION

The SRM 1462 stainless steel sample was tested using a microscope lens with $5.4\text{ }\mu\text{m}$ resolution. One hundred images were taken after a heat pulse. In the image analysis software, ImageDeskTM, an area of interest (AOI) was chosen, and a time profile of the AOI was plotted. Figure 2 shows the Temperature vs. Time curves of four AOIs with decreasing number of pixels in the box, from 100×100 to 10×10 pixels. The halfrise time values of the four curves are shown in Table 1. Thermal diffusivity was calculated from Parker's equation [1]. All the thermal diffusivity maps were calculated using the same equation. This calculation was selected because of its simplicity and the emphasis on relative values in diffusivity mapping. A single point IR detector was used to check the accuracy of the IR camera. The average halfrise time measured from the four AOIs and the single point detector showed basically the same value. The size of the AOI has no effect on the diffusivity values. Temperatures within the field of view of the microscope lens, i.e., $1.4\text{ mm} \times 1.4\text{ mm}$, were considered uniform.

Figure 2. Thermal diffusivity curves of four AOIs of SRM 1462 stainless steel.

Table 1: Halfrise time and thermal diffusivity measurements.

AOI Size (pixels)	Halfrise Time (seconds)	Thermal Diffusivity (cm ² /s)
100 x 100	0.225	0.0406
50 x 50	0.226	0.0406
25 x 25	0.226	0.0406
10 x 10	0.225	0.0406
IR Detector	0.225	0.0406

A 4D carbon-carbon composite similar to previous tests [3,4] was used. The volume percentage of fiber bundles in the direction parallel to the heat flow was 30%. The fiber bundles had diameters about 1.9 mm. The density of the 4D carbon-carbon composite was 2.02 g/cm³. Because the diameter of the fiber bundle was larger than the field-of-view of the microscope lens, another lens with 25 μ m to 35 μ m resolution was used. Figure 3 is the thermal diffusivity surface map of a fiber bundle and its surrounding matrix.

Figure 3. Thermal diffusivity map of a fiber bundle in a 4D carbon-carbon composite.

Thermal diffusivity of the fiber bundle was $1.50 \text{ cm}^2/\text{s}$, which was about 20% higher than the matrix value, $1.20 \text{ cm}^2/\text{s}$. The high volume fraction of fiber bundles, 30%, in the direction parallel to heat flow made it easier to obtain the diffusivity map. The coupling effect was shown as a gradual increase of diffusivity from the matrix to the center of the fiber bundle.

In most Continuous Fiber Ceramic Composite (CFCC) materials, fibers are not bundled together and the fiber diameters are much smaller. The spatial resolution of the camera was limited by the IR diffraction wavelength, $3\text{-}5 \text{ }\mu\text{m}$. The Textron SCS-6 fiber which has a large diameter of $140 \text{ }\mu\text{m}$ was selected. It has a carbon core and SiC coated outer surface by the CVD technique. A 6 mm diameter borosilicate glass rod with one SCS-6 fiber in the center was made for this study. Five samples with thickness varying from 0.667 mm to 9.305 mm were prepared. Figure 4 shows two curves obtained from two AOIs in the center of the fiber and in the matrix from the 2.024 mm thick sample. Instead of seeing two distinctly different curves with a fast and a slow response, the two curves looked identical. Calculations showed their thermal diffusivity values were actually the same. In other words, the thermal diffusivity of the SCS-6 fiber was the same as the borosilicate glass. The test was

Figure 4. Thermal diffusivity curves of fiber and matrix with 2.034 mm thickness.

repeated three times. It was clear the low measured diffusivity of the SCS-6 fiber was real. For thicker samples, 3.403 mm and 9.034 mm, the same effect was observed. The fiber seemed to be non-existent. The effective thermal conductivity of a composite can be calculated using the following equation [7]:

$$K_{\text{effective}} = V_{\text{fiber}} K_{\text{fiber}} + V_{\text{matrix}} K_{\text{matrix}} \quad (1)$$

where V_{fiber} and V_{matrix} are volume fractions of the fiber and the matrix. In the case of the carbon-carbon composite, the fiber bundle volume fraction was 30%. The effect of fiber bundles was very significant. For the single SCS-6 fiber in borosilicate glass matrix, the fiber volume fraction was 0.048%. Equation (1) predicts the effect of fiber on the effective thermal conductivity is negligible. In a well-coupled low thermal conductivity matrix, the fiber itself cannot conduct heat without being affected by the surrounding material. Thermal coupling between the fiber and the glass matrix prevented the fiber temperature getting higher during heat transfer. In fact, the higher temperature of the fiber caused lateral heat conduction into the matrix. The result was that the fiber showed exactly the same thermal diffusivity as the glass matrix.

Although the volume fraction is not changed by varying thickness, the heat conduction caused by coupling may be different for thinner samples. Figures 5(a), 5(b), 5(c) show three thermal diffusivity maps of varying thickness of 2.024 mm, 1.140 mm and 0.667 mm. In Figure 5(a), thermal diffusivity of the fiber and the matrix are almost the same, $0.0085 \text{ cm}^2/\text{s}$. The same flat maps were obtained for samples 3.470 mm and 9.305 mm thick. When the sample thickness was reduced to 1.140 mm, the center of the fiber started to have higher diffusivity, $0.012 \text{ cm}^2/\text{s}$, as shown in Figure 5(b). The matrix thermal diffusivity was $0.0090 \text{ cm}^2/\text{s}$. Further reducing the thickness to 0.667 mm made the fiber stand out nicely from the matrix. However, the thermal diffusivity value of the fiber, $0.025 \text{ cm}^2/\text{s}$, was far less than for the individual SCS-6 fiber. The effect of specimen thickness played an important role when the fiber diameter to specimen thickness ratio was larger than 10%. This result does not mean the intrinsic thermal diffusivity of the fiber changes with sample thickness or volume fraction. It indicates the measured thermal diffusivity values were significantly lower than the intrinsic value because of the coupling effect.

Originally, the idea of using a high thermal conductivity fiber in a low thermal conductivity matrix was to show how the fiber conducts heat differently from the

Figure 5(a) Thermal diffusivity of a 2.034 mm thick sample.

Figure 5(b) Thermal diffusivity map of a 1.140 mm thick sample.

Figure 5(c). Thermal diffusivity map of a 0.667 mm thick sample.

matrix. The experimental results show that even a high conductivity fiber is insignificant in heat conduction in a low conductivity matrix when the volume fraction is small and the sample is considerably thick. This effect has never been shown experimentally. For a single fiber, equation (1) holds not only for effective thermal conductivity of the composite but also reflects the measured thermal conductivity of the fiber and the matrix.

Since the microscopic diffusivity maps can provide detailed thermal properties of the test specimen rather than an averaged value, heat conduction models based on the observed thermal diffusivity values could be developed. Designers could be more confident using the thermal transport property data to calculate the temperature and heat conduction of components in field applications.

CONCLUSIONS

Thermal diffusivity maps of composite materials were generated on a microscopic scale. For large fiber volumes, the diffusivity map shows significant differences between the fiber and the matrix. For small fiber volumes, the effect of coupling is dominant. For the first time, the thermal diffusivity of a SCS-6 fiber in a borosilicate glass matrix is determined experimentally. Only when the fiber diameter

vs. sample thickness ratio is greater than 10%, does the fiber start to stand out from the matrix in a thermal diffusivity map.

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