THE DEPENDENCE OF THE CHANGE IN THE COEFFICIENT OF THERMAL EXPANSION OF GRAPHITE FIBER REINFORCED POLYIMIDE IM7-K3B ON MICROCRACKING DUE TO THERMAL CYCLING

Melissa C. Stewart
CraigW. Ohlhorst

Research and Technology Group
Materials Division
Environmental Interactions Branch

ABSTRACT

Composite IM7-K3B was subjected to a simulated high speed aircraft thermal environment to determine the effects of microcracking on the change in CTE . IM7-K3B is a graphite fiber reinforced polyimide laminate, manufactured by Dupont. The lay-up for the material was $[0,90]_{3s}$. The specimens were placed in a laser-interferometric dilatometer to obtain thermal expansion measurements and were then repeatedly cycled between -65°F and 350°F up to 1000 cycles. After cycling they were scanned for microcracks at a magnification of 400x . The material was expected not to crack and to have a near zero CTE. Some microcracking did occur in all specimens and extensive microcracking occurred in one specimen. Further testing is required to determine how closely the CTE and microcracking are related.

INTRODUCTION

Researchers are currently investigating polymer matrix composites as a material for use in the narrow dimensional confines of supersonic aircraft. These composites show many desirable traits such as high stiffness and small changes in the coefficient of thermal expansion.
This study reports thermal expansion data for a graphite fiber reinforced polyimide (IM7-K3B) and examines the dependence of the change in CTE of the composite on microcracking. This microcracking is due to thermal cycling and has been shown to significantly alter physical properties of a composite, sometimes seriously affecting its dimensional stability [1].

EXPERIMENTAL PROCEDURE

Eight specimens of IM7-K3B, produced and manufactured by Dupont, were selected for thermal expansion testing: 1 $[0]_{12}$, 1 [90]12, 1 [0]24, 1 [90]24 and 4 [0/90]3s lay-ups. This report covers data from the initial testing of the four 12-ply [0/90]3s symmetric crossplies.

The specimens were 7.5cm(3 in.) x 2.5cm(1 in.) with one polished edge. Each specimen had twelve 0.0127cm (0.005 in) plies. Each specimen was roughly 0.1524 cm(0.06 in) thick. A 2.54 cm(1 in.) section in the middle of the polished side of each specimen was examined for microcracking using an optical microscope at a magnification of 400x at 0, 25, 100, 500 and 1000 thermal cycles. The specimens were measured for the axial CTE at the same intervals.

The thermal expansion measurements were obtained using a laser-interferometric dilatometer at NASA Langley Research Center. This setup has a 1 microstrain resolution and measures the expansion of the specimen with respect to a NIST quartz standard [2]. The temperature range that the IM7/K3B specimens were exposed to was -54 °C (-65 °F) to 121 °C (250 °F).

A small scale thermal cycling chamber at NASA Langley Research Center was used to simulate the high speed aircraft thermal environment. One thermal cycle consisted of three steps. The first step started at 27 °C (80 °F) and rose to 177 °C (350 °F). The second dropped from 177 °C (350 °F) to -54 °C(-65 °F) and the final step brought the temperature back up to 27°C (80 °F.) The specimens were in direct contact with liquid Nitrogen during the cooling step.

RESULTS

Although testing is incomplete, initial data shows recognizable trends. Figures 1 through 4 display the CTE verses the number of cycles for each specimen. In each case the slope appears to be slightly negative, which indicates that the material shrinks as the number of thermal cycles increases. Specimen #2 is currently the only specimen of the four that has been tested for CTE after 1000 cycles, so the upward turn after 500 cycles shown in Figure 2 may be characteristic of the material. Comparison with CTE data from the other three specimens will reveal more conclusive results.

CTE data at 1000 cycles is currently unavailable for specimens 2,3 and 4.

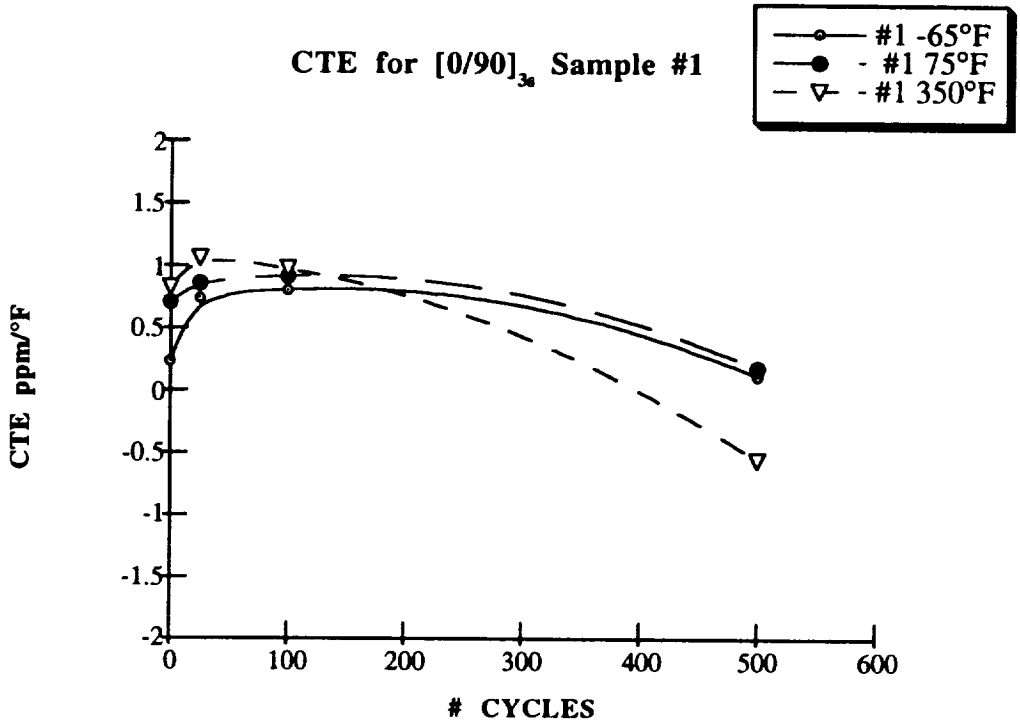

Figure 1 High temperature CTE data this specimen at 500 cycles was extrapolated. This may account for the deviation of the CTE at 350°F

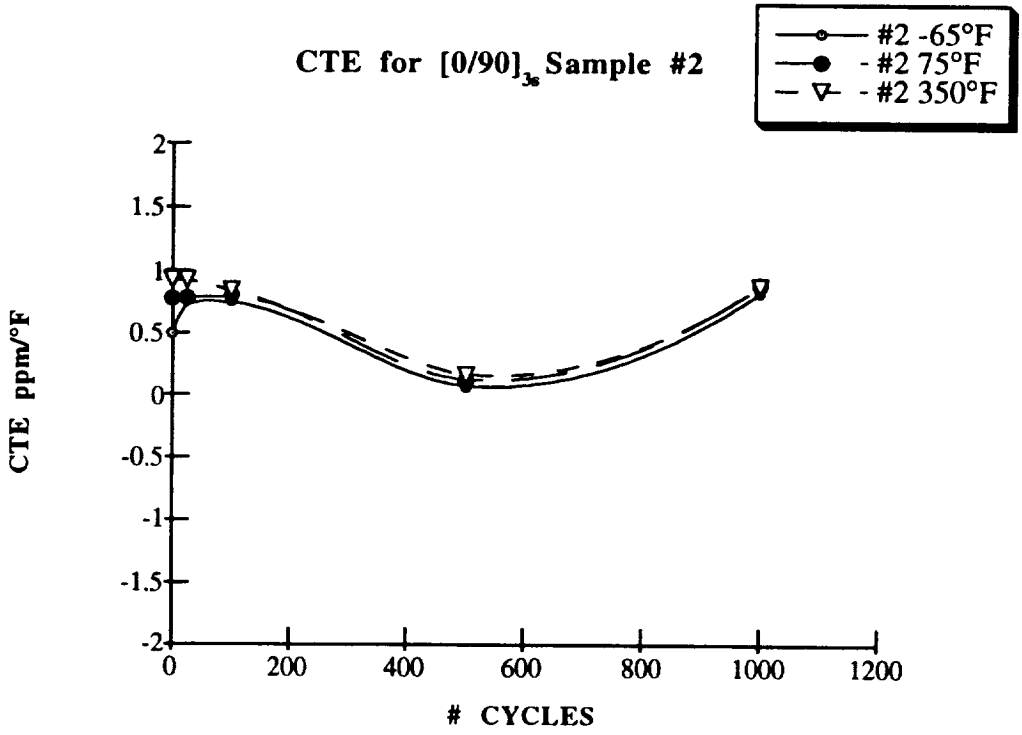

Figure 2 Notice upward turn after 500 cycles.

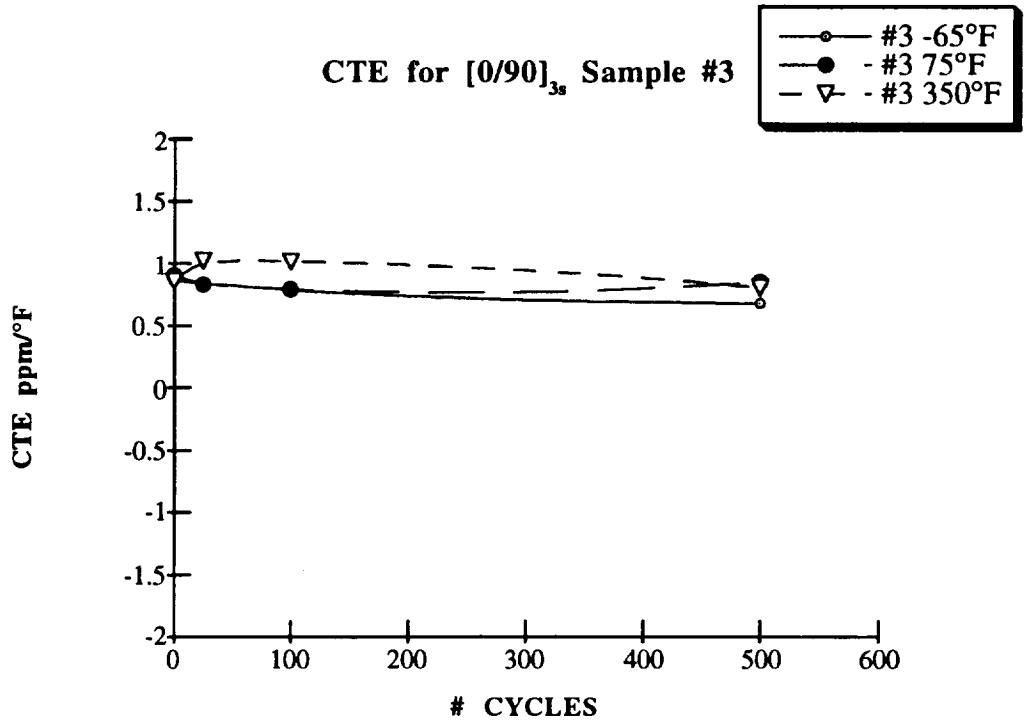

Figure 3

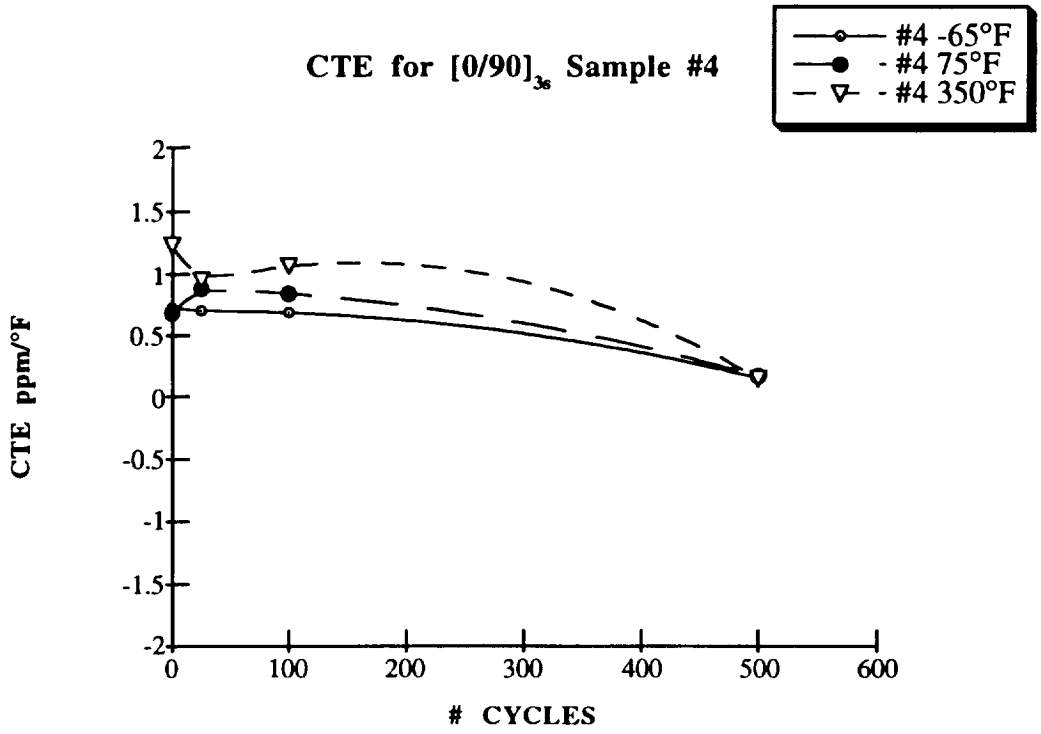

Figure 4

Figures 5 and 6 show the development of microcracks with increasing numbers of thermal cycles. Only data from the 90° plies is reported because microcracking occurs parallel to the 0° plies and cannot be seen by scanning the side of a specimen. The microcracking behavior of specimens 2, 3 and 4 is fairly uniform and appears to reach a limit of approximately 15 cracks/in. At this time only samples 2 and 4 have completed 1000 thermal cycles. Sample 3 is expected to behave similarly, but sample 1 shows radical differences in both the number of microcracks and the rate of microcracking. Possible explanations for this unexpected behavior include damage during fabrication, nonuniform thickness, and possible temperature surges during testing. All of these can contribute to microcracking.

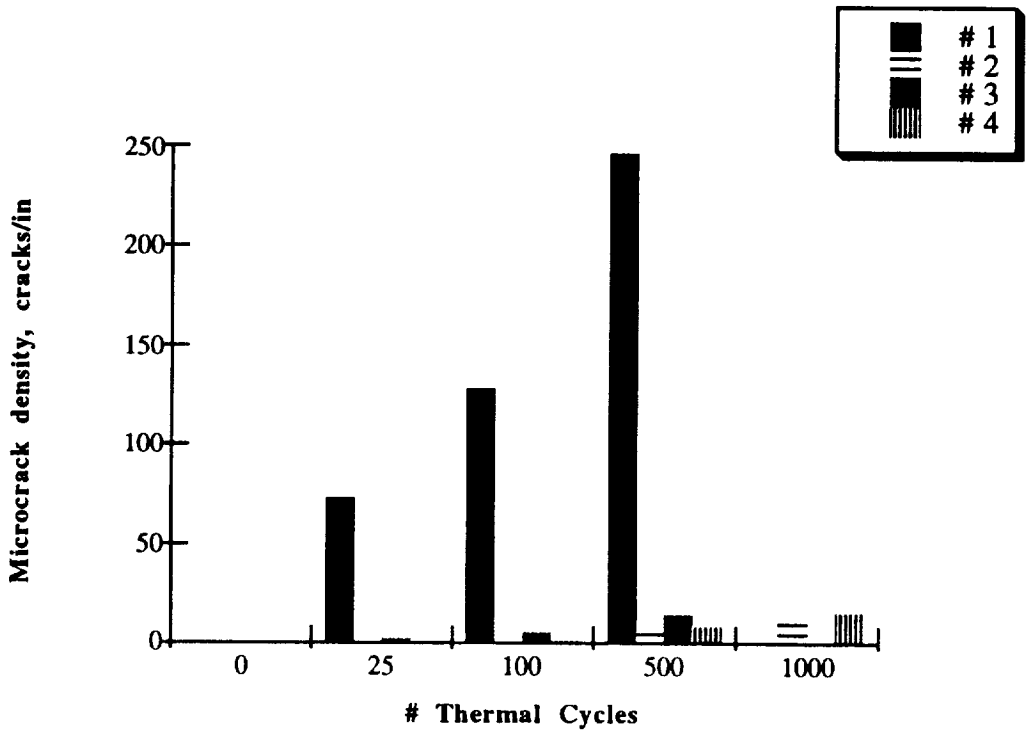

Figure 5 Sample #1 shows a large deviation from the behavior of the other specimens. Possible explanations for the behavior are damage during manufacturing and nonuniform specimen thickness.

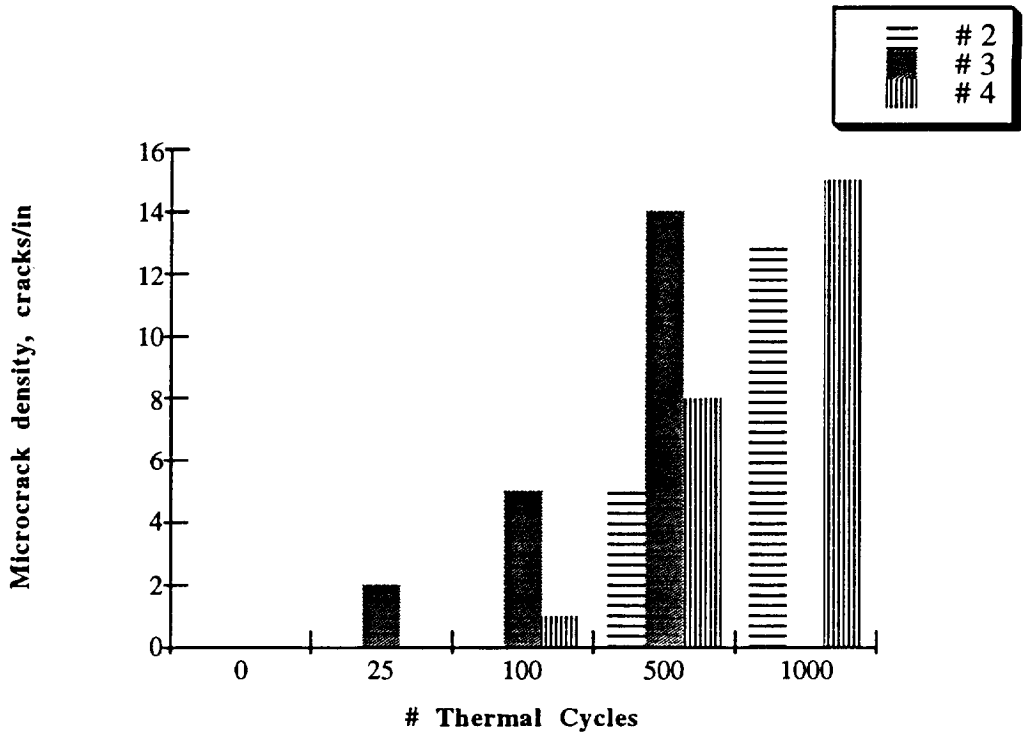

Figure 6

With a few exceptions, Figures 7 and 8 show that the average CTE appears to fluctuate around 0.9 ppm/°F.

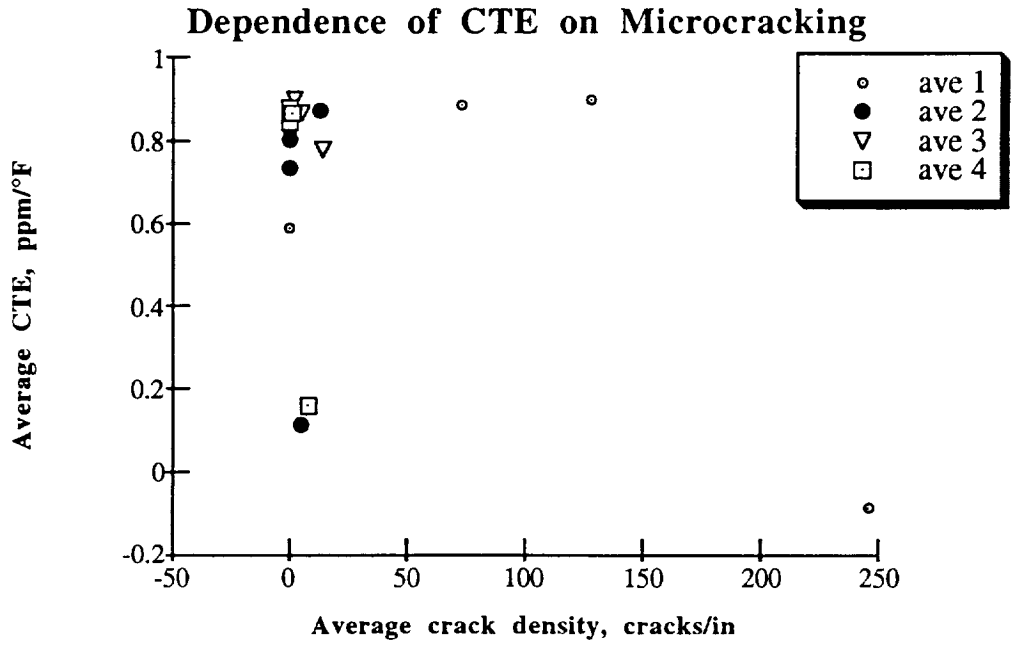

Figure 7

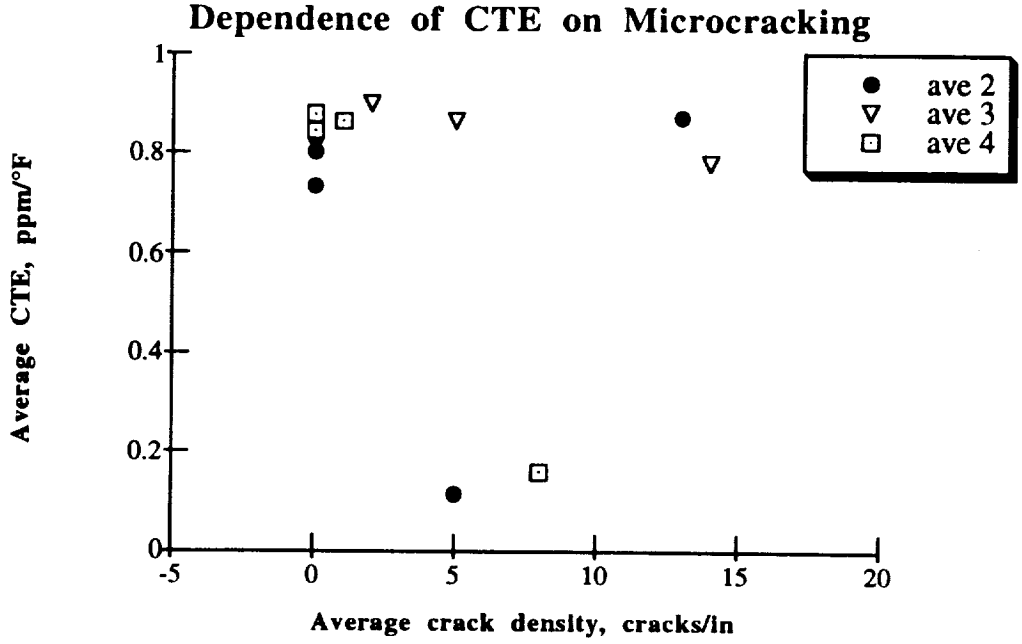

Figure 8

CONCLUSIONS

The thermal expansion of four specimens of IM7-K3B up to 500 thermal cycles has been measured and compared. Additional tests are needed to acquire microcracking and CTE data for 1000 cycles.

In each of the four specimens, the CTE decreased initially as the number of thermal cycles increased. Specimen #2 showed and upward turn after 500 cycles, but no information is currently available for specimens 1,3 and 4.

All specimens began with no visible microcracking, however microcracking did occur in all specimens at some point. The particularly high rate of cracking in specimen #4 should be investigated further at some point. Possible areas to investigate are the manufacturing and cutting process, the location of the specimen in the original panel and the uniformity of the ply thickness.

REFERENCES

1. B. Knouff, S.S. Tompkins, N. Jayaraman, "The Effect of Microcracking Due to Thermal Cycling on the Coefficient of Thermal Expansion of Graphite Fiber Reinforced Epoxy-Cyanate Matrix Laminates", 25th International SAMPE Technical Conference, Volume 25, Advanced Materials: Expanding the Horizons, October 26-28, 1993, Philadelphia, PA pp:610-621.

2. S.S. Tompkins, D.E. Bowles, and W.R. Kennedy, "A Laser-Interferometric Dilatometer for Thermal-Expansion Measurements of Composites", Experimental Mechanics 26, (1), 1, (1986)