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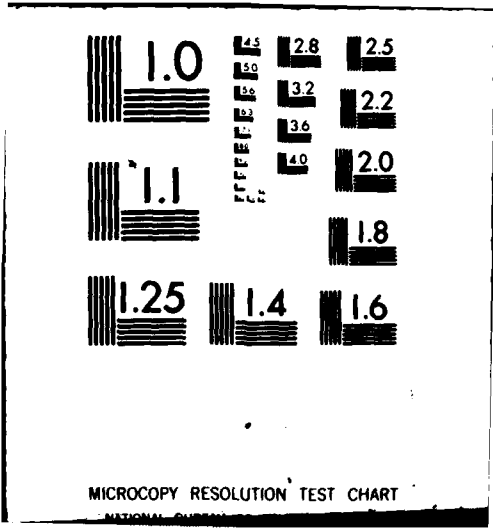
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THERMAL FATIGUE BEHAVIOR OF FP ALUMINA/MAGNESIUM COMPOSITES

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Introduction

Composites of magnesium alloys reinforced with FP-Alumina are being considered for aerospace applications because of their high specific strength and modulus, and because of their relative ease of fabrication. Some of these applications require repeated high temperature exposure of the composite material for extended periods of time. In a recent study, Bhatt, et al. (1) have evaluated the effects of isothermal and cyclic exposure on the room temperature mechanical properties of unidirectionally reinforced FP-Al₂O₃/EZ33 magnesium composites. Results of this study indicate no significant loss in the room temperature axial tensile strength and dynamic flexural modulus of composites thermally cycled between 50° and 250°C or of composites isothermally heated at 350°C for up to 150 hours from the strength and modulus data for the as-fabricated composites. In contrast, thermal cycling between 50° and 300°C caused considerable loss in both room temperature strength and modulus. The major causes of strength degradation were attributed to softening and cracking of the matrix and debonding of the fibers from the matrix.

The objective of this investigation was to study the effects of matrix composition on thermal fatigue behavior of FP-Al₂O₃/magnesium composites as determined by tensile strength and modulus measurements after cyclic exposure.

In this study, 55 volume percent fiber unidirectionally aligned composites were exposed in air to temperatures of either 250° or 350°C for up to 3000 cycles. Isothermal exposure at 350°C was used as a baseline to evaluate the additional effect of cycling. Metallographic and fractographic studies were made on untreated and thermally cycled composite material in an attempt toward understanding the failure mechanisms involved.

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Experimental

The FP-Al₂O₃/QH21A Mg composites used in this study were fabricated by the Dupont Pioneering Research Laboratory using molten metal infiltration techniques. The nominal FP-Al₂O₃ fiber content used was 55 volume percent. The fibers were aligned unidirectionally in a QH21A magnesium alloy matrix having a composition of 2 to 3% Ag, 0.6 to 1% Th, 0.6 to 1.5% rare earths, 0.4 to 1% Zr, and bal. Mg. Plates of 0.25 cm thickness were cast and cut into specimens 12.7 cm long and 1.25 cm wide. The fiber orientation was either parallel to the specimen length (0°) for axial testing or perpendicular to the specimen length (90°) for transverse testing. Thermal cycling was done by alternately dipping a frame supporting six specimens into a hot (250° or 350°C) fluidized sand bath and then into a cold bath that equilibrated near 50°C. Each complete thermal cycle lasted for six minutes. Typical time-temperature profiles of composite specimens cycled to 250° or 350°C are shown in Figure 1. The time at temperature during each cycle was approximately two minutes.

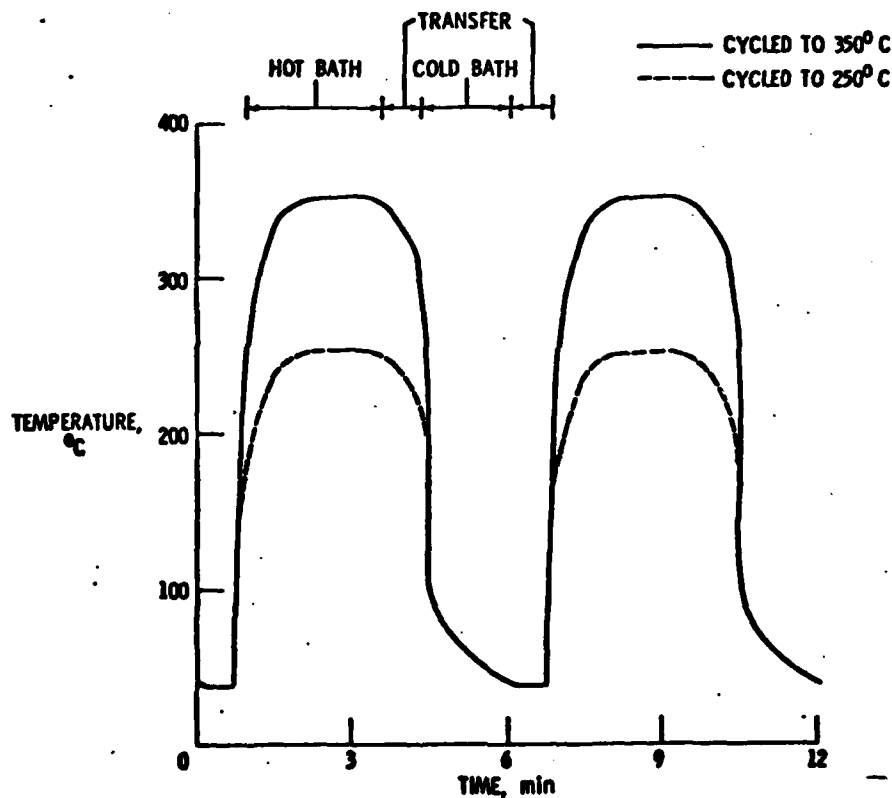


Figure 1 Typical thermal cycle temperature-time profiles.

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Similar composite specimens were also isothermally heated at 350°C in a sand bath for periods up to 100 hours, which corresponds to a time equivalent to the time at temperature for 3000 cycle experiments.

After cycling to a predetermined number of cycles or isothermal heating to set time periods, the specimens were removed from the bath and aluminum doublers were adhesively bonded to the specimen ends.

Tensile testing was done in an Instron testing machine equipped with wedge-type grips. The specimens were pulled to failure at a constant cross head speed of 0.126 cm/min.

A flexural modulus test was used for measuring the dynamic modulus of the composite specimens. The test method and the equations used for calculating dynamic modulus of the composites are described in Reference 1.

Results and Discussions

The room temperature flexural moduli of the 55 fiber volume percent FP-Al₂O₃/QH21A Mg composites cycled to 250° or 350°C to a maximum of 3000 cycles are shown in Figure 2. The flexural moduli of untreated composites are also

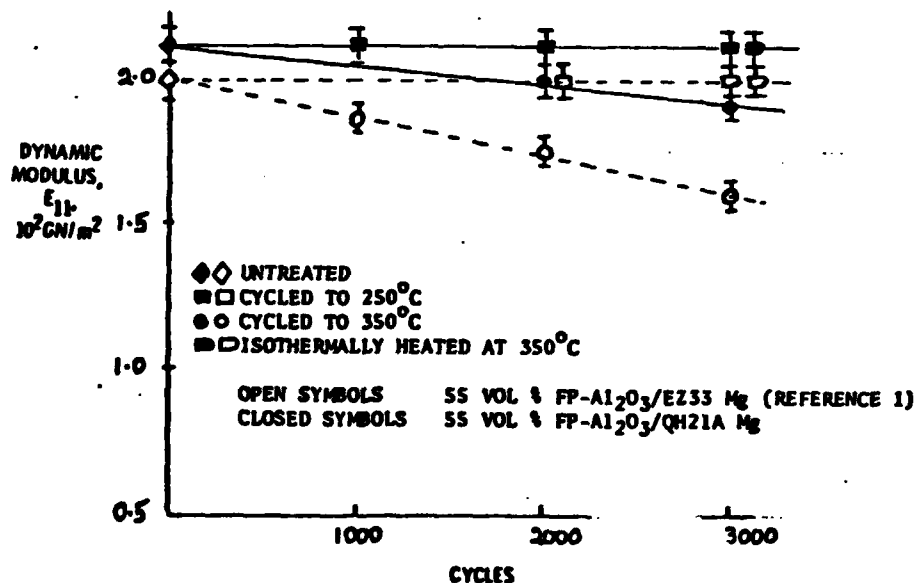


Figure 2 Room temperature dynamic moduli of FP-Al₂O₃/magnesium composites after cycling to 250°C or 350°C for indicated number of cycles. Dynamic moduli of composites isothermally heated at 350°C for 100 hours are shown. Dynamic moduli of untreated composites are shown at zero cycles.

shown in Figure 2 for baseline comparison. The data points represent the range and average value for at least three determinations. The averaged data of Bhatt, et al. (1) for 55 fiber volume percent $FPAl_2O_3/EZ33$ Mg composites thermally cycled to 250° or $350^\circ C$ for up to 3000 cycles are shown in this figure for comparison. The modulus values for 55 fiber volume composites thermally cycled to $250^\circ C$ show no significant change from the modulus values of as-fabricated unheated composites. However, composites, thermally cycled to $350^\circ C$ show a loss in flexural modulus proportional to the number of thermal cycles. Similar behavior was also observed in thermally cycled $FP-Al_2O_3/EZ33$ Mg composites. After 3000 cycles to $350^\circ C$, 55 fiber volume composites degraded to near 80 percent of the modulus values of unheated composite specimens. Also shown in Figure 2 is the modulus values for similar composite specimens isothermally treated at $350^\circ C$ for 1000 hours, a time equivalent to the cumulative time at temperature for the 3000-cycle test. These specimens did not show any loss in flexural modulus even after 100 hours of exposure. In the cyclic tests, while the specimen length and weight remained nearly the same, the width and thickness of the specimen increased continuously with cycling. In isothermally heated composites, however, no dimensional changes were measured even after 100 hours of exposure of $350^\circ C$. The losses in the dynamic modulus of the cycled composites were also found to be proportional to the width and thickness changes.

The room temperature axial tensile strengths of the 55 volume percent composites cycled to 250° or $350^\circ C$ to a maximum of 3000 cycles are shown in Figure 3. Again, the data points indicate the range and the average

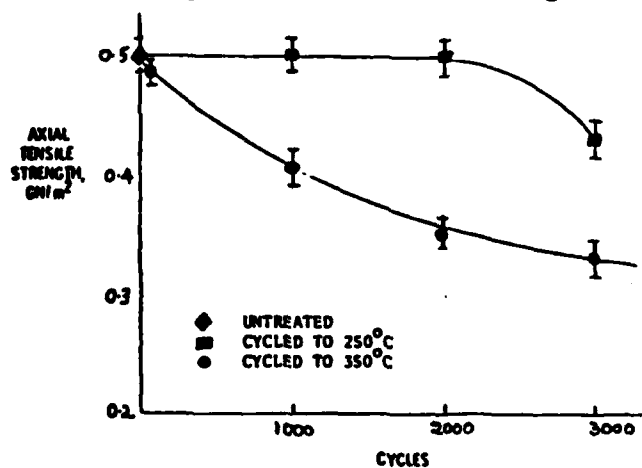


Figure 3 Room temperature axial tensile strengths of 55 volume percent $FP-Al_2O_3/QH21A$ Mg composites after cycling to $250^\circ C$ or $350^\circ C$ for indicated number of cycles. Strengths of untreated composites are shown at zero cycles.

value for typically three tests. The axial strength data for the 55 fiber volume percent composites without thermal treatment are also shown in Figure 3 for a baseline comparison. The strength data, as seen in Figure 3 for the 55 fiber volume percent composite cycled to 250°C shown no appreciable degradation from the unheated composite strength of 0.50 GN/M² after 2000 cycles. Additional cycling to 3000 cycles resulted in a loss of strength to 0.435 GN/m². Similar composites cycled to 350°C showed a gradual loss of strength from the baseline value of 0.50 GN/M² after 3000 cycles.

The baseline axial strength data and strength data measured after 1,000, 2000, and 3000 cycles from Figure 3 are replotted in Figure 4 against maximum cycle temperature to better illustrate the temperature dependence of the strength degradation. The average strength data of Bhatt, et al. (1) for 55 fiber volume percent FP-Al₂O₃/EZ33 Mg composites in as-received and thermally cycled to 250° or 350°C for up to 3000 cycles are also shown in the figure for comparison. Clearly from Figure 4, no significant strength loss occurred for the 55 fiber volume percent composites when cycled to 250°C, however, a rapid strength loss occurs. This loss appears

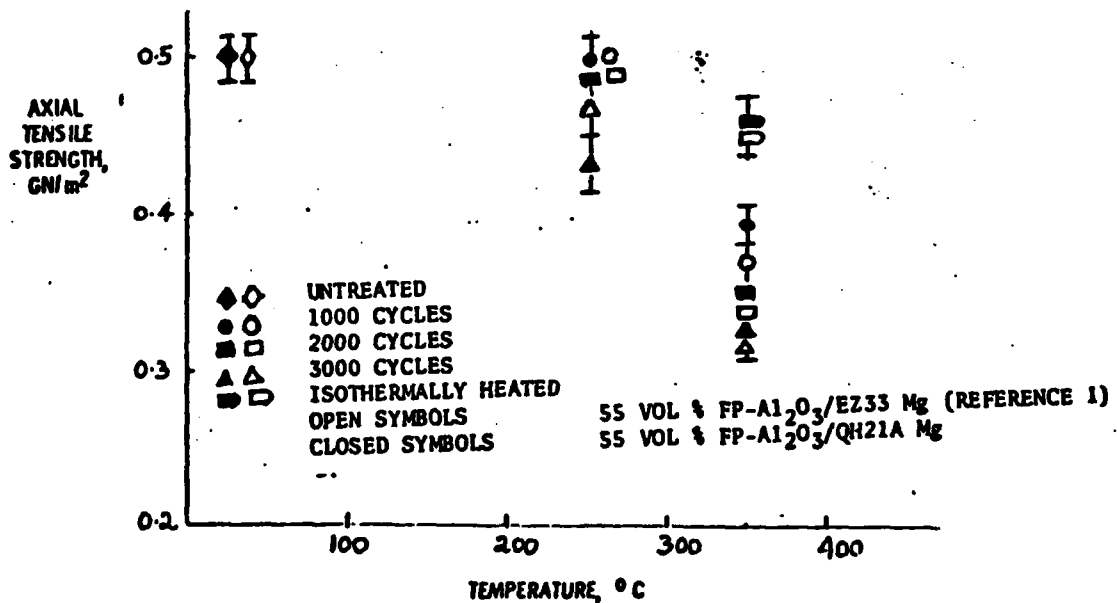


Figure 4 Room temperature axial tensile strengths of FP-Al₂O₃/Magnesium composites cycled 1000, 2000, and 3000 times to indicated temperatures. Strengths of similar composites isothermally heated at 350°C for 100 hours are also shown.

to be more a function of cycle temperature than number of cycles. Also shown in Figure 4 is the range and average value of all the strength data of similar composite specimens which have been isothermally heated at 350°C for 100 hours a time equivalent to the cumulative time-at-temperature for specimens cycled 3000 times. The 55 fiber volume percent composite specimens isothermally heated at 350°C show a 10 percent loss of strength to a value of 0.45 GN/m². Strength degradation obtained after isothermally or cyclically heated composites of FP-Al₂O₃/EZ33 Mg or FP-Al₂O₃/QH21A Mg was similar. In all cases, however, the strength values of isothermally heated specimens were equal to or higher than the strength values for thermally cycled composite specimens.

The greater degradation observed for cyclically heated composites is seen as evidence of a mechanism involving more than a simple thermally activated process. If a single thermally activated process were operating, one would expect similar behavior for specimens which were cyclically heated or isothermally heated for an equivalent time at temperature. A likely candidate for this additional mechanism is one which involves the generation of large matrix stresses due to the differences between the thermal expansions of the fiber and matrix during heating. The maximum thermally induced matrix stress, σ_m , in fiber composites can be obtained from the equation derived by Piggott(2).

$$\sigma_m = \frac{3 (\alpha_m - \alpha_f) (\Delta T) V_f E_m E_f}{E_c + E_f (1 + \gamma_m V_m) - \gamma_f E_m}$$

where α is the thermal expansion coefficient, V is the fiber volume fraction, E is the elastic modulus, γ is Poisson's ratio, and ΔT is the cyclic temperature range. The subscripts m, f, and c refer to the matrix, fiber, and composite, respectively. For calculating σ_m , the measured value of $E_c = 2.1$ GN/m² was used along with $\Delta T = 200^\circ$ or 300°C , $\alpha_m = 25.4 \mu/\text{cm}/\text{cm}/^\circ\text{C}$, $\alpha_f = 5.7 \mu/\text{cm}/\text{cm}/^\circ\text{C}$, $E_f = 379$ GN/m² and $E_m = 44.8$ GN/m²(3), and $\gamma_f = 0.2$, $\gamma_m = 0.33$ (3,4). The maximum matrix thermal stresses, σ_m , calculated for 55 fiber volume percent composites are shown in Table 1.

Table I

$(T_2 - T_1) = \Delta T, ^\circ\text{C}$	$\sigma_m, \text{GN/m}^2$ 55 Vol Percent
200	0.174
300	0.262

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These stresses are similar to the room temperature yield stress value of 0.17 GN/m^2 for the QH21A magnesium alloy. (3) In the thermally cycled composite therefore plastic deformation will occur during each cycle. The cumulative effect of such repeated deformation has been observed to produce voids in the matrix in highly constrained regions where the matrix deformation cannot be reversed. (5,6) Evidence of similar void growth and matrix cracking in this composite is seen in Figure 5 which shows microphotographs of the 55 volume percent FP- Al_2O_3 /QH21A Mg composite before thermal exposure and after cyclic heating to 350°C . The voids here appear to extend to the fiber/matrix interfaces. The presence of voids or cracks at the interface will result in a loss of fiber/matrix bonding.

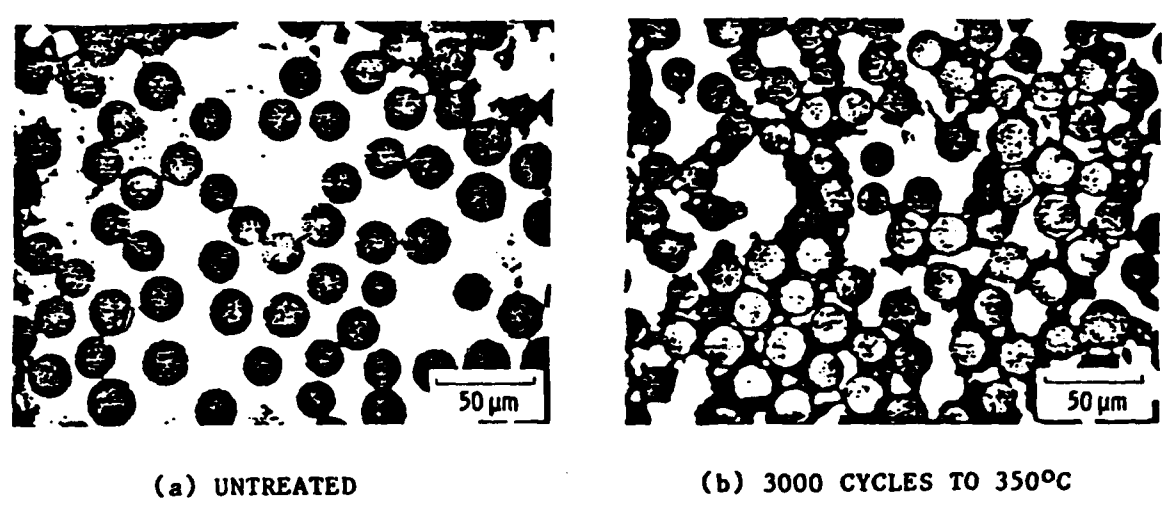


Figure 5 Photomicrograph of untreated and thermally cycled composite show effect of cycling to 350°C for indicated number of cycles.

Whether the loss of axial strength results from fiber/matrix debonding or from a weakening of the fiber or the matrix cannot be determined from these micrographs. However, some insight may be obtained from the results of transverse strength of the composite. Figure 6 shows the transverse strength data for 55 fiber volume percent composites thermally cycled to 350°C . It is obvious from this figure that there is no appreciable strength degradation from the untreated composite strength of 0.185 GN/m^2 even after as many as 3000 cycles. Also shown in Figure 6 are the transverse strength data for similar composites isothermally heated at 350°C for 100 hours. As with the data for cycled composites, no appreciable loss in strength was observed at 350°C . This indicates that matrix strength loss may not be a major factor for the strength degradation of thermally cycled composites.

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To obtain further understanding about the cause of strength degradation, fracture surfaces of thermally cycled composites were examined. Typical fracture surfaces of the untreated and thermally cycled composites for 1000 and 3000 cycles are shown in Figure 7. This figure indicates fiber debonding and pull-out in thermally cycled composites. Since isothermally heated composites showed no voids or fiber pull-out, we associate the gradual strength decrease with the number of cycles as shown in Figure 3 with debonding of the fiber and the matrix due to growth voids at the fiber matrix interface.

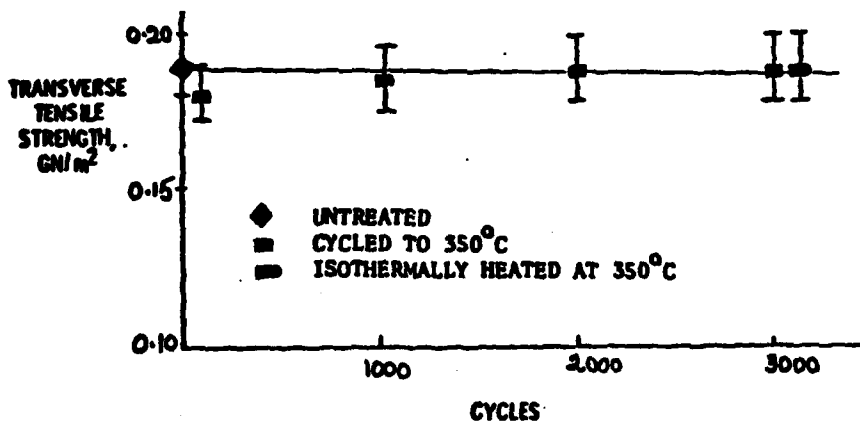


Figure 6 Room temperature transverse tensile strengths of FP- Al_2O_3 /QH21A Mg composites after cycling to 350°C for indicated number of cycles. Strengths of similar composites isothermally heated at 350°C for 100 hours are shown. Strengths of untreated composites are shown at zero cycles.

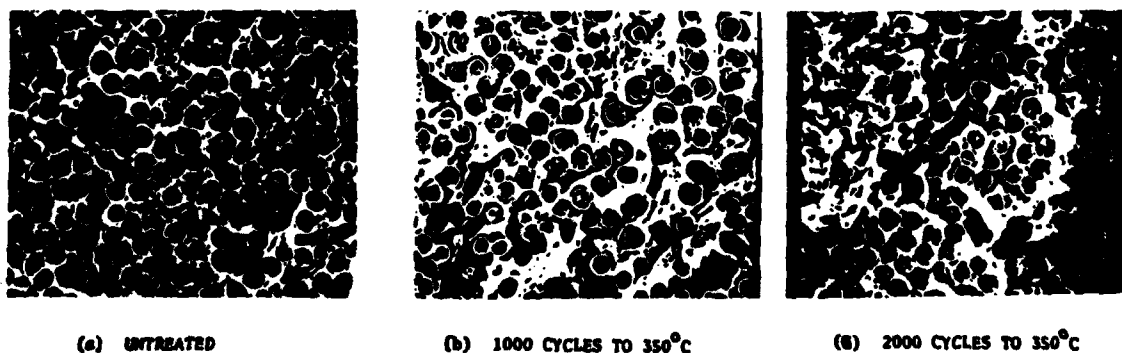


Figure 7 Fracture surface of untreated and thermally cycled FP- Al_2O_3 /QH21A Mg composites stressed in axial direction showing effect of cycling to 350°C for indicated number of cycles.

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Summary

→ The effects of cyclic and isothermal thermal exposure on the axial and transverse tensile strength and axial moduli of 55 volume percent FP-Al₂O₃/QH21A Mg composites have been evaluated to understand the cause of thermally induced strength degradation and to help determine the limiting use conditions for these composites. Specific findings are as follows:

(1) Thermal cycling of FP-Al₂O₃/QH21A Mg composites to 250°C for 2000 cycles did not cause any appreciable room temperature strength or modulus loss compared with baseline data for untreated composites. In contrast, composites thermally cycled to 350°C showed considerable loss in both axial strength and modulus. No appreciable loss in room temperature transverse tensile strength of composites was measured after thermal cycling.

(2) Measurement of the transverse strength and fractographic analysis of thermally cycled composites indicated interface void formation and matrix cracking and fiber debonding as prime contributors to observed strength and modulus losses. These results are consistent with degradation mechanism based on thermal-induced stresses in the matrix.

(3) No appreciable loss in the axial dynamic modulus of these composites was observed after isothermal exposure. Small strength losses observed in isothermally heated composites were attributed to matrix softening.

(4) The high temperature mechanical properties were not measured for the composites in this study. This would be required to properly design using FP-Al₂O₃/QH21A composites. However, aside from the usual matrix softening at higher temperatures, this study indicates that we would not expect additional axial strength or modulus degradation resulting from either isothermal or cyclic exposure below 2000 cycles or 250°C.

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