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MODULUS, STRENGTH AND THERMAL EXPOSURE STUDIES OF FP-Al_2O_3/ALUMINUM AND FP-Al_2O_3/MAGNESIUM COMPOSITES

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ABSTRACT

Several approaches were used to improve the mechanical properties of FP-Al_2O_3 fiber reinforced composites prepared by liquid metal infiltration techniques. The first approach was the incorporation of a strengthening addition, magnesium, to the aluminum-lithium matrix alloy usually selected for these composites because of its good wetting characteristics. This ternary composite, FP-Al_2O_3/Al-(2-3)Li-(3-5)Mg, showed improved transverse strength compared with FP-Al_2O_3/Al-(2-3)Li composites. The lower axial strengths found for the FP-Al_2O_3/Al-(2-3)Li-(3-5)Mg composites were attributed to fabrication-related defects. The second approach was the use of Ti/B coated FP-Al_2O_3 fibers in the composites. This coating is readily wet by molten aluminum and permitted the use of more conventional aluminum alloys in the composites. However, the anticipated improvements in the axial and transverse strengths were not obtained due to poor bonding between the fiber coating and the matrix. A third approach studied to improve the strengths of FP-Al_2O_3 reinforced composites was the use of magnesium alloys as matrix materials. While these alloys wet fibers satisfactorily, the result indicated that the magnesium alloy composites used offered no axial strength or modulus advantage over FP-Al_2O_3/Al-(2-3)Li composites.

Isothermal exposure at 350° C for 120 hours produced no significant loss in the room temperature axial strength in both FP-Al_2O_3/Aluminum and FP-Al_2O_3/Magnesium composites. In contrast, isothermal exposure at 450° or 482° C for 120 hours resulted in significant loss in the room temperature axial strength. This strength loss was attributed primarily to matrix softening.

INTRODUCTION

Composite materials composed of light metals reinforced with alumina fibers offer great potential where high specific strength or stiffness, high temperature capability and low comparative costs are required. Using the Dupont fiber, FP-Alumina, composites with aluminum and magnesium matrices have been prepared by low cost liquid infiltration methods.(1,2,3). However, composite strength properties consistant with the fiber and matrix strength properties have generally not been obtained. For the aluminum matrix composites this has been attributed to poor wetting and bonding between the fiber and the matrix. To improve the wetting two general approaches have been suggested; (1) modify the matrix alloy by the addition of small amounts of active metals such as lithium or calcium which promote chemical reaction between the fiber and the matrix; or (2) coat the alumina fiber with a metal or ceramic which is wet by the aluminum alloy. Of the two approaches, only the addition of lithium has received significant attention to date. While lithium did improve the fiber/matrix bonding, the room temperature transverse strength of this composite was still low. In addition, studies have shown that lithium concentration and time and temperature of the process must be closely controlled to prevent excessive fiber reaction and strength degradation of the fiber.(1,2) Part of the effort of this study, therefore, was to evaluate the transverse strengthening effect of the addition of ternary additive, magnesium, to the aluminum - lithium matrix alloy. The influence of the magnesium addition on the strength degradation of the composite due to thermal exposure was also studied.

The second approach to the improvement of the wetting of FP-Alumina by molten aluminum - the use of fiber coatings - has not been previously reported. However, Material Concepts Incorporated has recently developed a titanium/boron (Ti/B) fiber coating(4) which is wet by molten aluminum. A second objective of this study, then, was to evaluate the tensile strength properties of aluminum matrix composites reinforced with Ti/B coated alumina fibers. Several commercially significant aluminum alloy matrices were used to determine possible matrix composition dependences.

Finally, it is known that molten magnesium shows good wetting of the FP-Alumina fiber without special treatments(3). But, despite their potential for even higher specific strength, FP-Al_2O_3/Magnesium composites have received much less attention than the aluminum matrix composites. The third objective of this study, then, was to determine the tensile strength and stiffness properties of pure magnesium and two magnesium alloys (EZ33 and QH21A) reinforced with FP-Alumina. The effect of thermal exposure on the room temperature mechanical properties was also determined. The results for the magnesium matrix composites were compared with those for the aluminum matrix composites.

EXPERIMENTAL PROCEDURE

Materials

The lithium and magnesium doped aluminum matrix composites were fabricated by the Dupont Pioneering Research Laboratory using liquid metal infiltration techniques. The stated lithium content of the matrix was between 2-3 weight percent, comparable to that for the binary lithium - aluminum matrix used in reference 1, and the magnesium content was between 3-5 weight percent. Two fiber volume fractions were studied, 35 volume percent and 55 volume percent.

The Ti/B coated FP-Alumina fiber composites were prepared by Material Concepts Incorporated (MCI), again using liquid metal infiltration techniques. The aluminum based matrix alloys used were 1100, A201, 2024, and Al-(3-5)Mg. The nominal compositions of these alloys are given in Table I. The fiber content of all the coated fiber composites used in this study was 25 volume percent. The Ti/B coating was achieved by simultaneous chemical vapor deposition of titanium and boron onto the FP-Alumina fiber. The exact composition of the coating is unknown.(4)

The FP-Al_2O_3/Magnesium composites were also fabricated by Dupont using liquid metal infiltration techniques. The magnesium matrix alloys selected for study were QH21A, EZ33, and commercially pure magnesium. The nominal compositions of these alloys are also given in Table I. Two fiber volume fractions were used, 35 and 55 volume percent.

Tensile specimens were cut from the cast plates to the dimensions of 1.27cm (0.5 inch) by 12.7cm (5.0 inch) and 0.25cm (0.1 inch) thick. Both axial (0°) and transverse (90°) tensile specimens were prepared.

Testing Procedures

Tensile testing at room temperature was accomplished in a Instron machine using conventional wedge grips and a constant crosshead speed of 0.05cm/min (0.02in/min). Aluminum doublers, 3.8cm (1.5 inch) long were adhesively bonded to each side of the test specimen in the gripping area. This insured specimen failure in the 5.1cm (2.0 inch) test section. The axial strains in the specimens were measured with a standard "clip on" strain gauge. The elastic moduli were calculated from the stress strain curves obtained in the tensile tests.

The diamond pyramid hardness (D.P.H.) was measured in a Vickers harness testing machine using a 25gm load.

Isothermal exposures at 350°C and 450°C or 482°C for periods up to 120 hrs were accomplished by heating the specimens in an evacuated and sealed glass tubes in a furnace.

RESULTS AND DISCUSSION

FP-Alumina/Lithium Doped Aluminum Composites

The room temperature axial and transverse elastic moduli and strengths for 35 and 55 fiber volume percent FP-Al_2O_3/Al-(2-3)Li-(3-5)Mg composites are shown in Figures 1 and 2. Each data point indicates the average value and range for typically three tests. The averaged data of Champion et. al (1) for 35 and 55 fiber volume percent FP-Al_2O_3/Al-(2-3)Li composites are shown in these figures to provide a reference against which we may evaluate the effect of the ternary component, magnesium. As seen in Figure 1, the axial and transverse moduli for the FP-Al_2O_3/ Al-(2-3)Li-(3-5)Mg composites are comparable with the moduli found by Champion et al.(1) for the FP-Al_2O_3/Al-(2-3)Li composites.

In contrast to the elastic modulus results, the room temperature axial strength data for FP-Al_2O_3/Al-(2-3)Li-(3-5)Mg composites, shown in Figure 2, were found to be low compared with the data of Champion et al.(1) The averaged strength values for the 35 and 55 fiber volume percent FP-Al_2O_3/ Al-(2-3)Li-(3-5)Mg composites were 0.265 GN/m^2 (38.5 KSI) and 0.496 GN/m^2 (72 KSI), respectively. However, the strength value of 0.496 GN/m^2 (72 KSI) for the 55 fiber volume percent composite was in fact 80 percent better than the strength value of 0.275 GN/m^2 (40 KSI) for the unreinforced alloy reported by Sanders et al.(5) On the other hand, the 35 fiber volume percent composite showed no strength improvement with reinforcement.

In contrast, the room temperature axial tensile strength data for FP-Al_2O_3/Al-(2-3)Li-(3-5)Mg composites, shown in Figure 2, were found to be low compared with the data of Champion et al.(1). The 55 fiber volume percent FP-Al_2O_3/Al-(2-3)Li-(3-5)Mg composite showed a 75 percent increase in axial strength over 0.275 GN/m^2 (40 KSI) strength found by Sanders et al.(5) for the unreinforced alloy. However, the 35 fiber volume percent composite showed no strength improvement with reinforcement.

The transverse strengths for both the 35 and 55 fiber volume percent composites were higher than those for the binary matrix composites of Champion et al.(1) and were comparable to the strengths of unreinforced Al-(2-3)Li-(3-5)Mg alloy.(5) This is usually indicative of satisfactory bonding. In the case of poor or inadequate bonding, the transverse strength for the composite will fall below that for the unreinforced matrix and will decrease with increasing fiber volume fraction. Photomicrographs of composites sectioned transverse to the fiber axis (Fig. 3) show good consolidation and no apparent signs of inadequate bonding.

Photomicrographs of specimens sectioned parallel to the fiber axis reveal two fabrication related defects which may contribute to the axial strength loss. Figure 4 shows two different regions of the same plane section of the 55 fiber volume percent composites. For this composite over one third of the fibers are obviously misaligned. This defect will decrease the axial strength in proportion to the number and degree of misalignment of the off axis fibers. For the 35 volume percent, composite in addition to misaligned fibers, a second process defect is observed, poorly infiltrated regions resulting in matrix voids. This is seen in Figure 5, where the voids appear both at the fiber/matrix interfaces and throughout the bulk matrix. Prior studies(1,2,3) have indicated that such poor infiltration can lead to composite strength loss, such a strength loss, in fact, explain the exceptionally low axial strength of the 35 volume percent composite relative to the 55 volume percent composite which was relatively free of these voids.

The effects of isothermal heating on the room temperatures axial and transverse strength of 55 volume percent FP-Al$_2$O$_3$/Al-(2-3)Li-(3-5)Mg composites at 350^{o}C or 482^{o}C for up to 120 hours are shown in Figure 6. The axial tensile strength is seen to degrade slowly with time at both temperatures. After heating for 120 hours at 482^{o}C the axial strength has degraded by 0.07 GN/m^2 (10 KSI).

The transverse strength for this composite also degrades with time at both temperatures. Again, the higher temperature exposure resulted in a greater strength loss. In contrast to the axial data, however, the transverse strength loss is rapid during the first 60 hours then remains relatively constant to 120 hours, the longest time treated. The maximum strength loss at 350^{o}C is about 0.05 GN/m^2 (7KSI) and 0.14 GN/m^2 (20KSI) at 482^{o}C. Also shown in Figure 6 is the transverse strength data of Kim, et. al (6) for FP-Al$_2$O$_3$/Al-(2-3)Li composites isothermally heated at 500^{o}C for times to 120 hours. They attributed the observed strength loss of 0.05 GN/m^2 (7KSI) in 30 hours of isothermal exposure to matrix softening based on a good correlation with matrix hardness measurements. Our measurement of the matrix hardness versus exposure time for the 55 volume percent FP-Al$_2$O$_3$/Al-(2-3)Li-(3-5)Mg composites shown in Figure 7. The good correlation with the transverse strength again suggests that matrix softening is primarily responsible for the observed strength loss. Additional evidence of matrix softening in the thermally treated composite is found in SEM micrographs of the fracture surfaces of failed specimens. Figure 8 shows a typical transverse fracture surface of the composite in (a) as-fabricated condition and (b) after heat treatment for 10 hours at 482^{o}C and (c) after heat treatment for 50 hours at 482^{o}C. After heat treatment, the matrix is seen to fail in a much more ductile manner than in the as-fabricated condition. In addition, there is increasing tendency for void formation in the matrix with time during thermal exposure at this temperature.

Also evident in these micrographs is increased fiber splitting with time at temperature indicating a possible fiber weakening. Additional evidence of fiber degradation was obtained from the elemental Mg X-ray micrographs. The elemental Mg X-ray micrographs of the as-fabricated and thermally exposed specimens are shown in Figure 9. This figure indicates extensive segregation of magnesium to the fiber/matrix interface during thermal exposure supporting fiber degradation argument.

The observed linear axial strength loss with increasing time at temperature is indicative of strength loss due to something more than that of matrix softening alone. The observed fiber degradation and void growth are likely causes.

Ti/B Coated FP-Al_2O_3/Aluminum Composites

The room temperature axial and transverse elastic moduli and tensile strengths of several aluminum matrix composites reinforced with 25 volume percent, Ti/B coated, FP Alumina fibers are shown in Figures 1 and 2. Again the range and average value for typically three tests are indicated for each data point. The axial modulus values for 25 volume percent, Ti/B coated FP-Al_2O_3/Aluminum composites were again seen to be comparable with an extrapolation of data of Champion et al.(1) The transverse modulus was not measured due to the poor surface condition of the composite specimen which precluded the use of the strain gauge.

The room temperature axial and transverse strength data for the Ti/B coated FP-Al_2O_3/Aluminum composites shown in Figure 2 were found to be low compared with the data of Champion et al.(1) The transverse strengths of these composites were also much lower than the strength of the unreinforced matrix, indicating poor bonding between the fiber and the matrix. Confirmation of poor bonding was obtained from SEM photographs of the transverse fracture surfaces. Figure 10 for the Ti/B coated FP-Al_2O_3/Al-(3-5)Mg composite is typical of this group. This figure shows extensive debonding between the fiber and the matrix. Poor bonding could also contribute to the low axial strengths of this group of composites. For the limiting case of no bonding between the fibers and the matrix, the axial load would be carried essentially by a bundle of noninteracting fibers. Methods available for calculating this "bundle" strength from the distribution of strengths of individual fibers usually predict composite strengths no greater than 2/3 of the strength of a well bonded composite.(7,8) The measured axial strengths for the Ti/B coated FP-Al_2O_3 reinforced composites fall slightly above 2/3 of the extrapolated data of Champion et al.(1)

FP-Al_2O_3/Magnesium Composites

The room temperature elastic moduli and tensile strengths of three FP-Al_2O_3/Magnesium composites as a function of volume fraction of the fiber are shown in Figures 11 and 12. Figure 11 shows that both the axial and transverse moduli of FP-Al_2O_3/Magnesium composites are lower than those for the FP-Al_2O_3/Aluminum composites of similar fiber content, (Figure 1), due to differences in the elastic moduli of the aluminum 68.96 GN/m^2 (10 x 10^3 KSI) and magnesium alloys 44.82 GN/m^2 (6.5 x 10^3 KSI).

The axial strengths of all magnesium alloy composites studied shown in Figure 12 were similar to or higher than corresponding data for the FP-Al_2O_3/Al-(2-3)Li-(3-5)Mg composites of Figure 2. The transverse strengths of all the magnesium composites except the 35 volume percent FP-Al_2O_3/QH21A Mg composites were equal to or higher than those for the unreinforced matrix alloys, indicative of reasonably good bonding in these composites. Optical micrographs of these magnesium composite confirm good bonding between the fiber and the matrix, Figure 13.

The effects of isothermal exposure at 350^{o}C and 450^{o}C on the room temperature axial and transverse tensile strengths of the 55 volume percent FP-Al_2O_3/Magnesium composites are shown in Figure 14 and 15. The axial tensile strengths of EZ 33Mg and QH21Mg matrix composites were not affected by exposure at 350^{o}C but were seen to degrade uniformly with time at 450^{o}C. After 120 hours of exposure, the axial tensile strength decreased by about 0.08 GN/m^2 (12KSI). The commercially pure magnesium matrix composite showed essentially no strength loss after 120 hours of exposure at either temperature. The transverse tensile strengths for all three composites degraded with thermal exposure. Again the strength losses were greater for the QH21A Mg and EZ33Mg matrix composites and reached a maximum strength loss of 0.1GN/m^2 (14KSI) after 1 hour of exposure at either temperature. No further loss in transverse strength was observed up to 120 hours. For the pure magnesium matrix composite the strength loss was 0.025 GN/m^2 (4KSI) and 0.075 GN/m^2 (12KSI) after an hour exposure at 350^{o}C and 450^{o}C respectively. The diamond pyramid hardness test results on the composite matrix, shown in Figure 16, indicate a similar decrease in matrix hardness after thermal exposure. The loss in matrix hardness correlates well with the loss in transverse strength for all magnesium alloy composites except the 55 volume percent FP-Al_2O_3/pure magnesium composite exposed at 482^{o}C. For this material the loss in transverse strength is much more than the loss in matrix hardness. As with the aluminum matrix composites, the matrix softening which occurred with thermal treatment appeared to be responsible for the observed loss of transverse strength and at least part of the axial strength loss.

CONCLUSION

Three approaches to improve the transverse and axial strengths of FP-Al_2O_3/Aluminum composites prepared by liquid metal infiltration were studied. The first approach was the addition of magnesium to an aluminum-lithium alloy previously found to promote wetting. This ternary aluminum alloy composite, while apparently poorly fabricated in this study, did show improved transverse strength compared with the binary matrix composite. The somewhat lower axial strengths found for the FP-Al_2O_3/Al-(3-5)Mg-(2-3)Li composite were likely due to fabrication defects, which the author believes can be eliminated by better processing.

The improved transverse strength coupled with adequate modulus and axial strength, then, attest to the success of this approach.

Studies of the effects of thermal exposures on axial and transverse strength indicate a general softening of the matrix at temperatures below 350^{o}C. This softening, which maximizes after about one hour, markedly reduces the transverse strength of the composite and contributes to an axial strength loss. The axial strength loss did not stabilize as did the transverse strength loss. The failure of the axial strength loss to reach a limiting value was taken as evidence of fiber degradation. This was also indicated by fracture studies and interfacial chemical analyses.

The second approach to improve wetting was the use of a Ti/B coated FP-Al_2O_3 fiber in the composites. This approach eliminated the need for the lithium addition to the aluminum alloy and so three conventional alloys were used. The coated fiber composites, however, all showed poor transverse strengths attributed to bond failure. This lack of bonding also reduced the axial strengths. The composites were considered unsatisfactory and no thermal exposure studies were made.

A third approach to improve liquid metal infiltrated, FP-Al_2O_3 reinforced composites was the use of magnesium alloys as matrix materials. Molten magnesium alloys were known to wet Al_2O_3 satisfactorily and the light magnesium alloys offer promise of even greater specific strengths than the aluminum matrix composites. The results of this study indicate that the magnesium alloys composites used offered no axial strength advantage over the aluminum composites. The transverse strengths found for the magnesium composites were all lower than that for the ternary aluminum composite. The effect of thermal exposure was generally similar to that found for the aluminum composites.

In conclusion, then, the ternary aluminum matrix composite and at least the QH21A Mg matrix composite offer a combination of axial and transverse strength combined with excellent stiffness suitable for many stiffness critical applications. Prolonged use at temperatures in excess of 300°C should be avoided.

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TABLE I. - COMPOSITION OF THE MATRIX ALLOYS

Alloy designation	Composition
1100	99% + Al
A201	4% Cu, 0.7% Ag, 0.35% Mg, 0.35% Mn, 0.10% Si, 0.25% Ti, 0.15% Fe, 94% Al
2024	4.5% Cu, 1.5% Mg, 0.6% Mn, 93.4% Al
EZ33	3.2% rare earths, 2.5% Zn, 0.75% Zr, 0.1% Cu, 93.65% Mg
QH21A	0.1% rare earths, 2.5% Ag, 1% Th, 0.7% Zr, 0.2% Zn, 0.1% Cu, 95.4% Mg

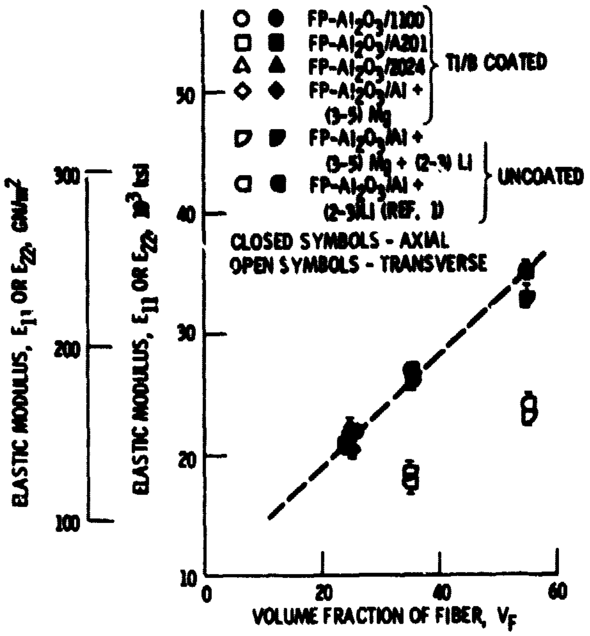


Figure 1. - Axial and transverse elastic moduli of FP-Al_2O_3/aluminum composite as a function of fiber content.

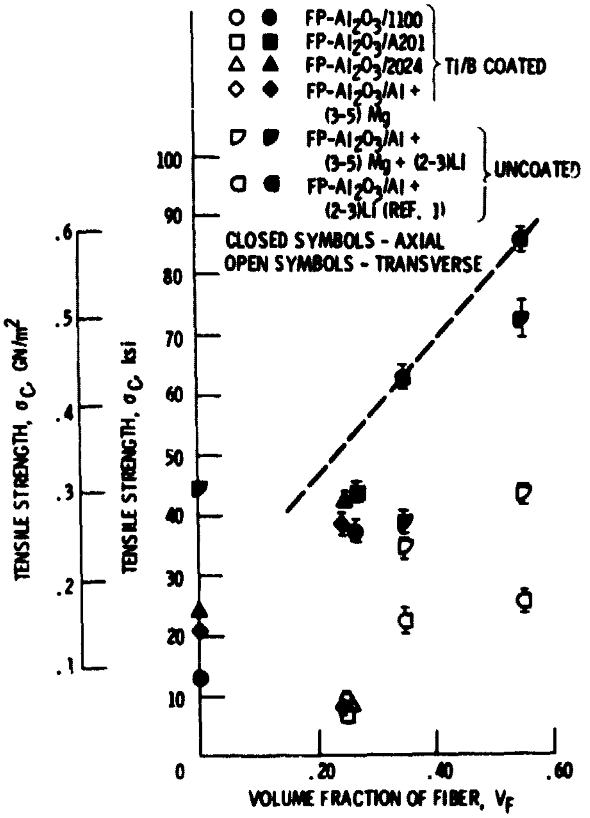


Figure 2. - Axial and transverse tensile strength of FP-Al_2O_3/aluminum composites as a function of fiber content.

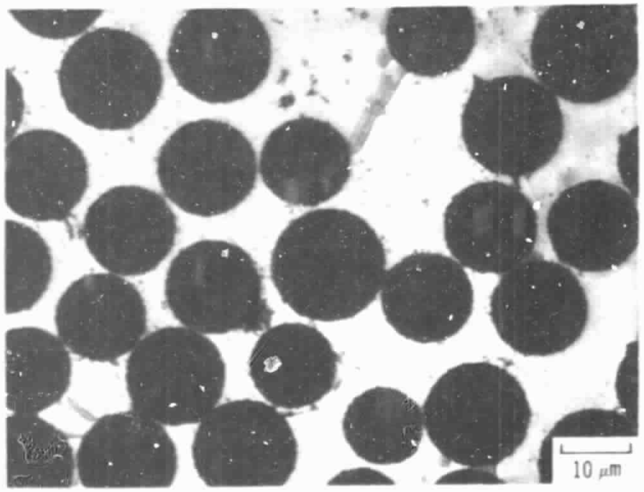


Figure 3. - Photomicrograph of as-received 55 vol % FP-Al_2O_3/Al-(2-3) Li-(3-5)Mg specimen.

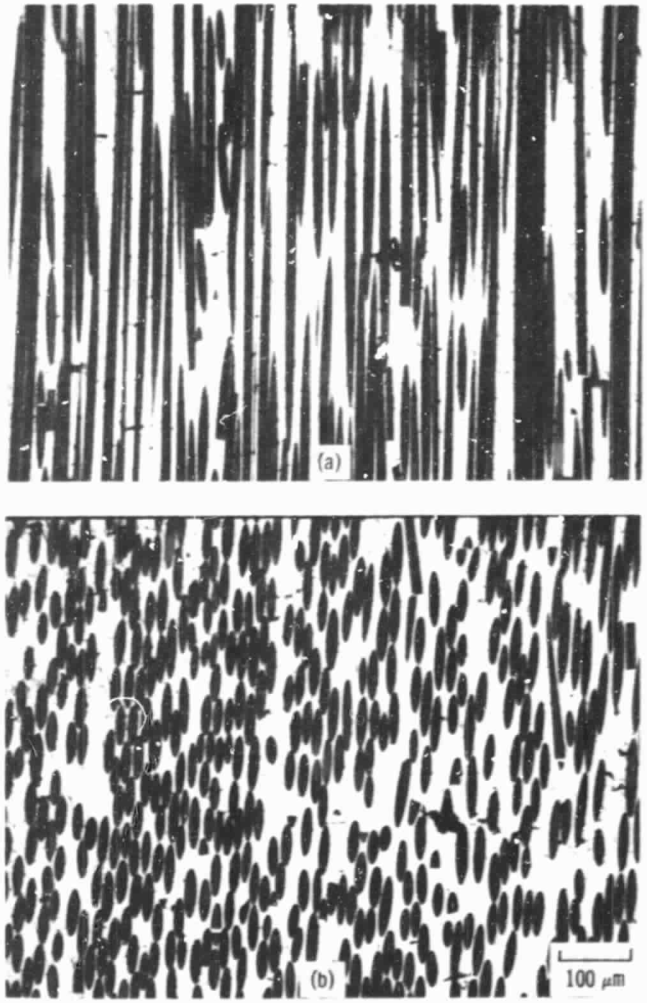


Figure 4. - Photomicrographs of as-received 55 vol % FP-Al_2O_3/Al-(2-3)Li-(3-5)Mg specimen from two different regions of the same planar section.

Figure 5. - Photomicrograph of an as-received 35 vol % FP-Al_2O_3/Al-(2-3)Li-(3-5)Mg specimen showing poorly infiltrated regions and matrix voids.

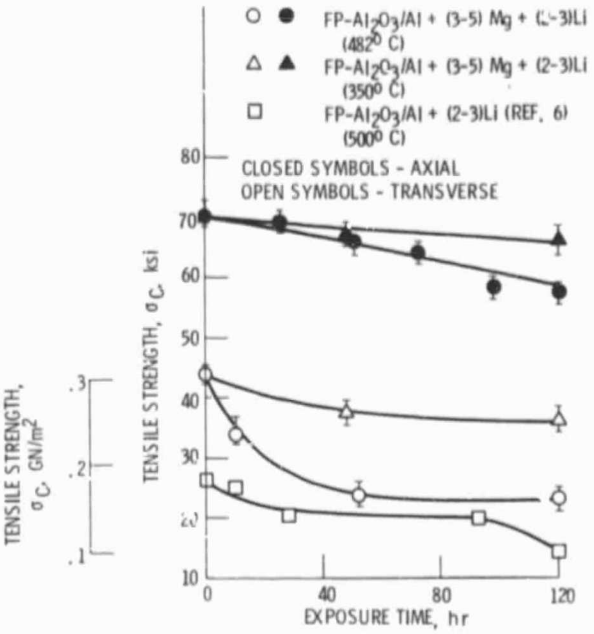


Figure 6. - Effect of isothermal exposure on room temperature axial and transverse tensile strengths of 55 vol% FP-Al_2O_3/aluminum composites as a function of exposure time.

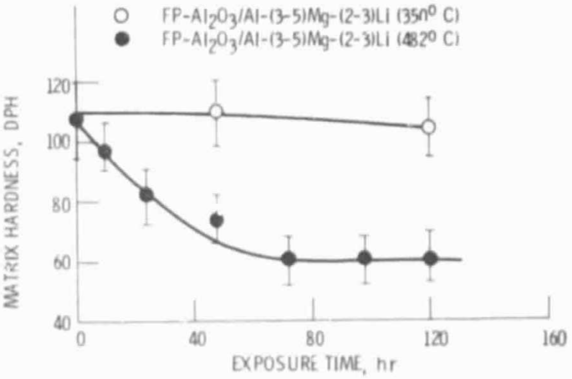
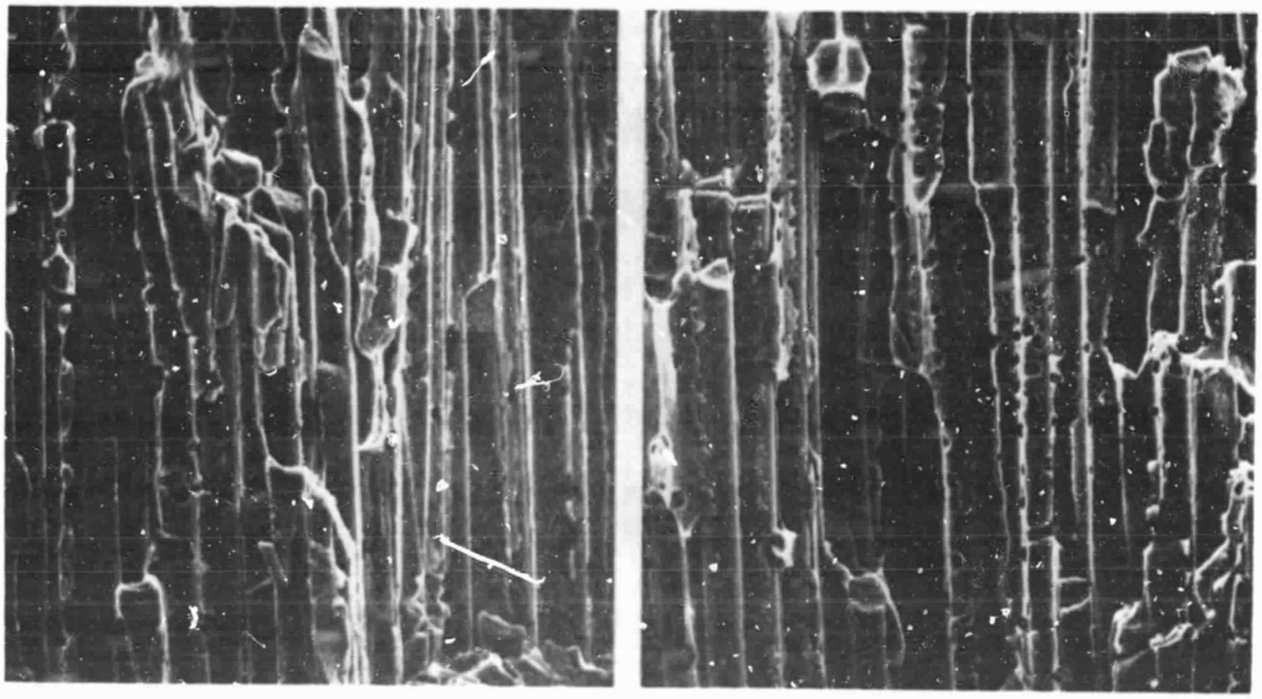
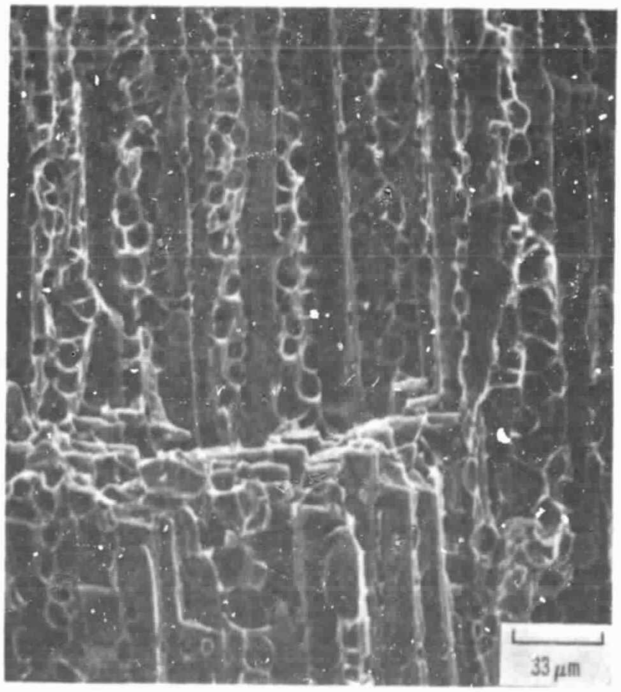


Figure 7. - Effect of isothermal exposure on room temperature matrix hardness of 55 vol % FP-Al_2O_3/Al-(3-5)Mg-(2-3)Li as a function of exposure time.



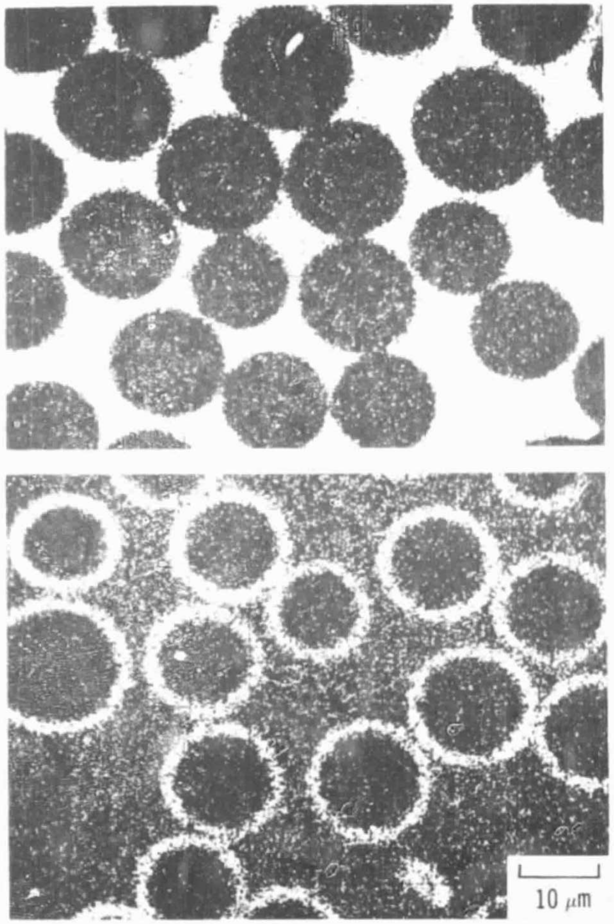
(a) AS-FABRICATED.

(b) ISOTHERMALLY HEATED AT 482° C FOR 10 HOURS.



(c) ISOTHERMALLY HEATED AT 482° C FOR 52 HOURS.

Figure 8. - Fracture surface of 55 vol % FP-Al_3O_3/Al-(2-3)Li-(3-5)Mg stressed in transverse direction.



(a) AS-FABRICATED CONDITION.

(b) ISOTHERMALLY HEATED AT 482° C FOR 52 HOURS.

Figure 9. - Elemental Mg X-ray maps showing the effect of thermal exposure on the relative distribution of magnesium around fibers in FP-Al_2O_3/Al-(2-3)Li-(3-5)Mg composite.

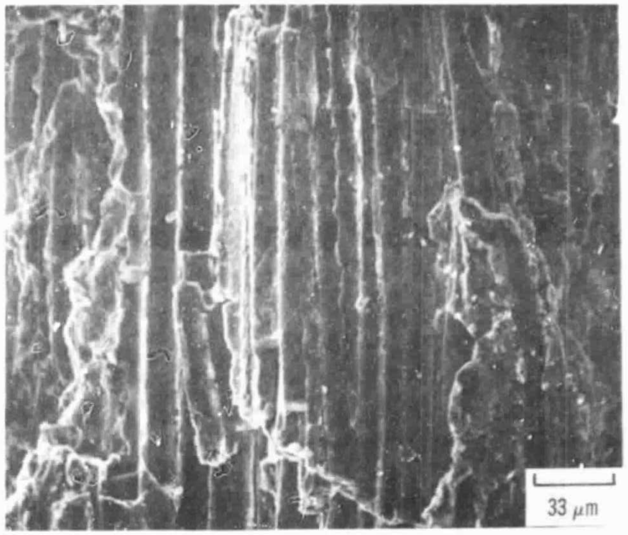


Figure 10. - Fracture surface of as-received Ti/B coated FP-Al_2O_3/Al-(3-5)Mg composite stressed in the transverse direction.

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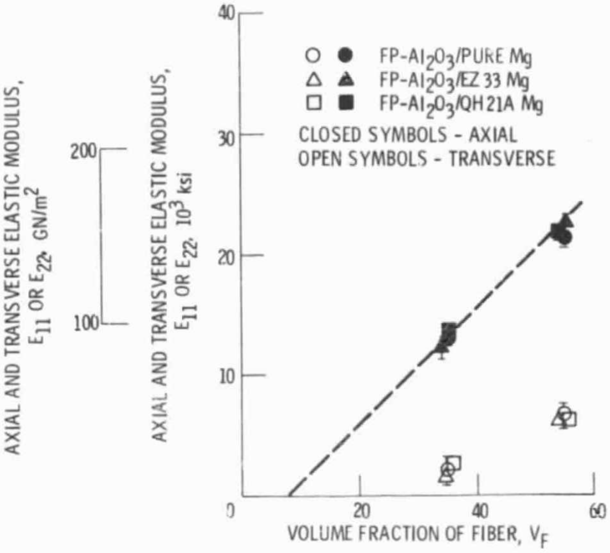

Figure 11. - Axial and transverse elastic moduli of FP-Al_2O_3/magnesium composites as a function of fiber content.

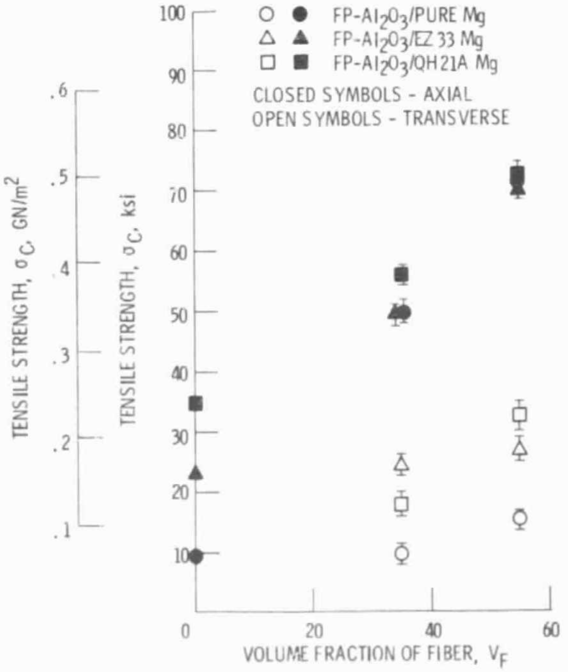

Figure 12. - Axial and transverse tensile strengths of FP-Al_2O_3/magnesium composites as a function of fiber content.

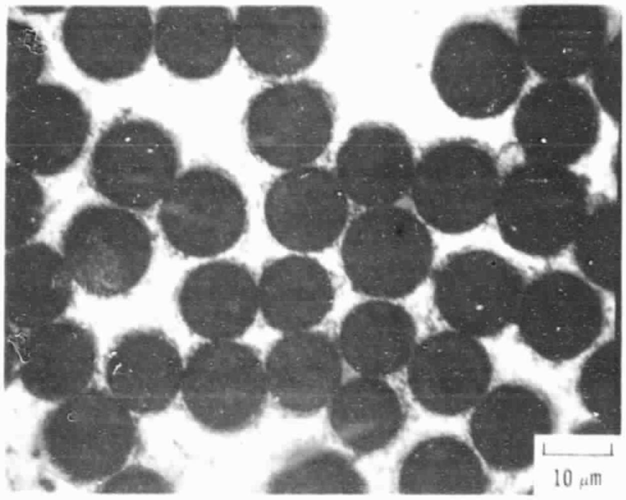

Figure 13. - Photomicrograph of as-received FP-Al_2O_3/Mg composite specimen.

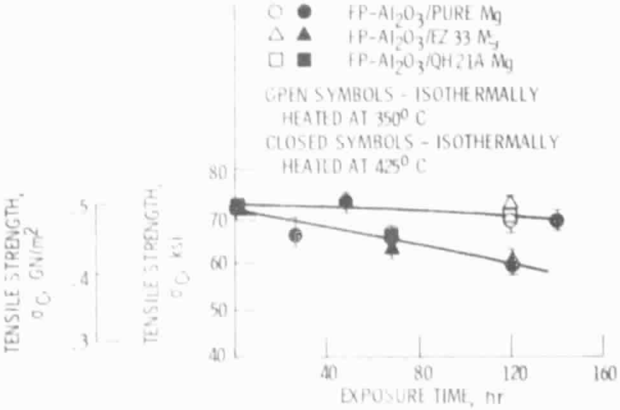

Figure 14. - Effect of thermal exposure on room temperature axial tensile strength of 55 vol % FP-Al_2O_3/magnesium composites as a function of exposure time.

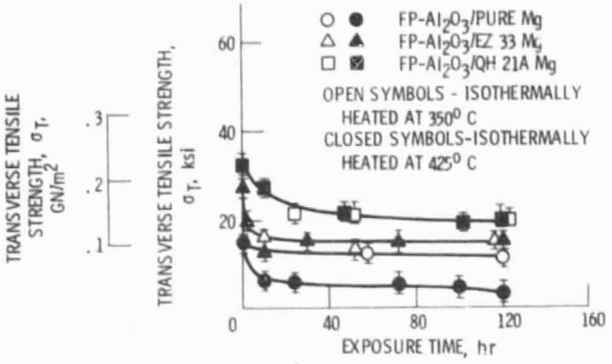

Figure 15. - Effect of thermal exposure on room temperature transverse tensile strength of 55 vol % FP-Al_2O_3/magnesium composites as a function of exposure time.

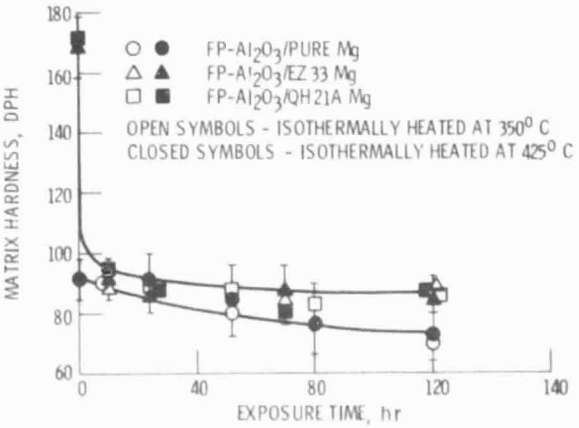

Figure 16. - Effect of isothermal exposure on the matrix hardness of 55 vol % FP-Al_2O_3/magnesium composites as a function of exposure time.