

The Effect of Processing Parameters on the Tensile Properties of Alumina Fiber Reinforced Magnesium

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The effect of processing variables on the tensile properties of commercially pure (CP) magnesium reinforced with FP alumina fibers has been studied. Composite plates were cast at 698 °C, 755 °C, and 810 °C, with a five-minute hold period prior to solidification. The properties of the composites were compared to those of both conventionally cast CP magnesium and ZE41A (Mg-4.25Zn-0.5Zr-1.25RE) composites. The tensile properties were found to be independent of casting temperature in the range 698 to 810 °C, although the five-minute hold period significantly decreased the axial properties of the CP magnesium. The results indicated that, during processing, the time at temperature has a more pronounced effect on tensile strength than the actual casting temperature. Increasing casting temperature was found to increase the MgO fiber-matrix reaction zone to a size comparable to that observed in conventionally processed ZE41A material. However, the strengths of the CP magnesium composites cast at the three different temperatures were significantly lower than that of ZE41A, indicating that the interfacial strengthening observed in ZE41A is not due to larger MgO reaction zone particles. A possible explanation for the interfacial strengthening in ZE41A is the presence of zinc and aluminum in low concentrations, which act to enhance the bonding between the reaction zone and the metallic matrix.

I. INTRODUCTION

A previous study of the fatigue and tensile behavior of unidirectional FP Al₂O₃ fiber-reinforced metal matrix composites with either a commercially pure (CP) magnesium or a ZE41A magnesium alloy (Mg-4.25Zn-0.5Zr-1.25RE) matrix has revealed a dramatic strengthening effect accompanying the alloy additions.¹ The alloy additions were found to improve the off-axis properties but decrease the axial properties, due to both an increase in the matrix and interfacial strengths and a decrease in the fiber strength. The differing mechanical behavior was accompanied by a differing off-axis failure mode between the two materials. In the CPMg material, off-axis failure occurred parallel to the fiber orientation in all orientations studied (22.5, 45, and 90 deg).² In the stronger ZE41A material off-axis failure was a combination of failure across and along the fibers, up to an orientation of 45 deg. The differing failure modes and higher off-axis strength indicated an improved interfacial strength in the ZE41A. It was originally hypothesized that the alloying elements in ZE41A did not directly contribute to the improved interfacial strength, since Auger electron spectroscopy (AES) revealed that there was no segregation of alloying or impurity elements to the fiber-matrix interface region. Transmission electron microscopy (TEM) showed that the fiber-matrix reaction zone in both CPMg and ZE41A was composed of MgO particles, although the MgO particles in the ZE41A were approximately twice as large as those found in the CPMg. Based on these results, it was suggested that the interfacial strengthening in the ZE41A was due to the increased size of the reaction zone and/or the change in the size and spacing of the MgO crystals, and not to any change in the chemical nature of the reaction zone. It was postulated that this larger reaction zone resulted from

a longer contact time with the highly reactive liquid matrix during processing, due to differences in the solidification behavior of the two matrices.³

The implications from this previous work were that, by properly modifying the casting conditions, improved properties could be obtained for CPMg without the expense of alloying element additions. It has been shown that, by heating CPMg samples above the melting point of the matrix, the MgO precipitate size increased with increasing contact time between the molten matrix and the Al₂O₃ fibers.³ There was also an increase in the amount of Mg matrix adhesion to the fibers with increasing contact time. However, whether there was any improvement in interfacial strength was not determined.

This paper reports the results of a continued study of the effect of processing variables on the tensile properties of FP Al₂O₃ fiber reinforced CPMg. E. I. DuPont de Nemours and Co. changed their normal processing conditions in order to cast composite plates at 698 °C, 755 °C, and 810 °C, with a 5-minute hold period before quenching in each case. Axial and off-axis tensile properties of material from each plate were measured, and the microstructure of the composites was characterized with conventional light microscopy, TEM, and AES. These data, together with earlier reported data, provide the basis for determining whether processing conditions can be exploited for improving the properties of the CPMg metal matrix composite.

II. EXPERIMENTAL

Composite plates of CPMg reinforced with 35 vol pct continuous, unidirectional, 20 μm diameter FP Al₂O₃ fibers were utilized in this program. These plates, which were approximately 15.2 cm square and 1.27 cm thick, were manufactured by E. I. DuPont de Nemours and Co. by a liquid metal vacuum infiltration technique. Identical plates had been used in the previous study.^{1,2,3} Although actual

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Manuscript submitted July 1, 1986.

details of this technique are regarded as proprietary by DuPont, an overview description of the process is available in the literature.^{4,5} DuPont agreed to change their normally employed casting conditions in order to vary both the casting temperature and the contact time between the fibers and the molten matrix. Consequently, three plates were cast at 698 °C, 755 °C, and 810 °C, each with a hold period of 5 minutes before quenching.

Tensile specimens, 15.2 cm long with 1.27 cm × 0.25 cm rectangular cross sections, were cut from each plate. The specimens were machined so that the Al₂O₃ fibers were oriented at 0, 45, and 90 deg to the tensile axis. Aluminum tabs, 2.5 cm long, were bonded with epoxy to both sides of the specimens' ends for gripping purposes. Tensile tests were then performed at room temperature in a screw-driven Instron testing machine. The tests were performed under displacement control at a strain rate of $2.1 \times 10^{-4} \text{ s}^{-1}$. Both axial strain and load were monitored continuously during testing, the strain being determined from strain gages attached to the opposite faces of the specimens. Three specimens of each material condition (casting temperature and fiber orientation) were tested in order to determine meaningful properties. The fracture surfaces of tensile specimens from each cast plate and at each fiber orientation were characterized by scanning electron microscopy.

The microstructure of the composites, including the constituents and morphology of the fiber reaction zone, was characterized with conventional light microscopy, AES and TEM. The AES was performed in order to determine the location of the fracture path for the off-axis specimens which failed parallel to the fibers, and to determine whether this fracture path was influenced by the processing conditions received by the composite plates. By analyzing spectra obtained from AES, it was possible to determine whether the fracture path was at the fiber-reaction zone boundary, the reaction zone-matrix boundary, through the matrix, or some combination of these. Standard notched Auger fracture specimens with dimensions 3.5 × 3.5 × 30 mm were prepared with the fibers oriented perpendicular to the longitudinal direction. The specimens were fractured *in situ* parallel to the fiber direction, and Auger analysis was performed immediately following fracture. Analyses were performed on a number of fibers and troughs for each heat treatment. The beam conditions included a primary electron beam voltage of 3 kV, beam current of 0.5 to 1.5×10^{-7} A, and a modulation voltage of 2 V peak-to-peak.

Thin foils from each cast plate were prepared for TEM by various steps of mechanical thinning and ion milling. Small 3 mm diameter discs were trepanned from material that had been ground to approximately 50 to 75 μm thick. These discs were then mechanically thinned further in a Gatan Dimple Grinder to a final thickness of 20 to 30 μm using 0.25 μm diamond paste. The foils were then thinned to perforation in an ion mill operating at 3.5 kV and 1 mA gun voltage and current, respectively. The thin foils were examined in a Phillips 301 scanning transmission electron microscope. The structure of the fiber-matrix reaction zone was examined at high magnification and the size of the interface particles was measured. Microdiffraction patterns were obtained by limiting the specimen area illuminated with a focused second condenser aperture.⁶ A 5 μm aperture was used, which yielded an illuminated area of approximately

130 nm in diameter due to the demagnification of 38 times obtained in the microscope. Diffraction patterns were taken at multiple orientations so that the interface particles could be unambiguously identified.

III. RESULTS

A. Tensile Behavior

The results of the tensile tests are presented in Table I and Figures 1 and 2. It is apparent from these data that the fiber orientation affected the tensile behavior in a similar manner to that previously observed for fiber reinforced CPMg, ZE41A, and Al3Li metal matrix composites.^{1,2,7,8} The ultimate tensile strength (UTS) and elastic modulus (E) were found to decrease as the misorientation angle between the fibers and loading axis increased. The strength was reduced by approximately a factor of two for each 45 deg change in fiber orientation. The elastic modulus was substantially higher at zero degrees than for both of the off-axis orientations (45 deg and 90 deg). The strain to failure (ϵ_f), on the other hand, was a maximum at the 45 deg orientation, exhibiting a sharp increase from the axial (0 deg) orientation. The strain to failure was a minimum in the transverse (90 deg) orientation.

It is seen from the results that there was little effect of casting temperature on the tensile properties for the three plates studied. As expected, the elastic modulus was particularly insensitive to the casting temperature employed in composite fabrication. The strengths of the materials cast at 698 °C and 755 °C were almost identical, whereas the strength of the material cast at 810 °C was somewhat lower, especially for the off-axis specimens.

Previous tensile data² for CPMg reinforced with 35 vol pct FP Al₂O₃ fibers are also included on Figures 1 and 2 for comparison to the present data; these original data were from a plate cast according to DuPont's normal time-temperature process parameters. Although the exact processing parameters were not made available by DuPont, it is known that the plate was cast at a temperature equal to or

Table I. Tensile Properties of 35 Vol Pct Al₂O₃ Fiber Reinforced CP Magnesium

Casting Temp., °C	Orientation Degrees	UTS MPa	E GPa	ϵ_f Pct
698	0 (axial)	277	157	0.20
	45	123	95	0.36
	90 (transverse)	63	88	0.11
755	0	277	159	0.19
	45	139	93	0.50
	90	68	83	0.13
810	0	270	159	0.18
	45	107	96	0.55
	90	46	84	0.08
Conventionally cast plate*	0	383	149	0.26
	45	108	90	0.45
	90	104	85	0.42

All values except where noted are the averages of triplicate tests.

*Conditions not known but estimated to be approximately 750 °C with no hold period. Data from duplicate tests.

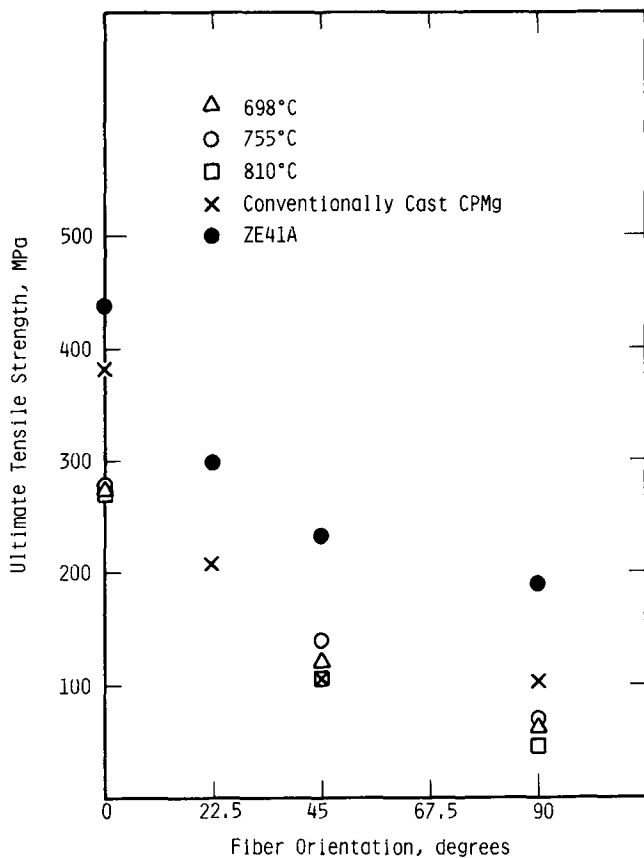


Fig. 1—Effect of fiber orientation and casting temperature on ultimate tensile strength of commercially pure magnesium with 35 vol pct fibers. Data for conventionally cast CPMg and ZE41A from Refs. 1 and 2 are also included.

less than 750 °C, with essentially no hold period prior to quenching.⁹ This material exhibited a very similar orientation dependence to the materials studied in the present program, with the exception that the properties of the 45 deg and 90 deg material were very similar. It is seen that the axial properties of the original plate were significantly better than any of the plates cast with a 5-minute hold period prior to quenching. Thus, the data suggest that the 5-minute hold period was sufficient to cause reaction with the fibers which degraded the fiber strength by approximately 25 pct. At a fiber orientation of 45 deg, the composite strength was fairly insensitive to either casting temperature or hold period, although the strain to failure was quite variable. At a 90 deg fiber orientation, the strengths of the plates cast with an extended hold at 698 °C, 755 °C, and 810 °C were all less than the conventionally cast plate.

B. Fractography

Fractographic examination of the tensile specimens indicated that, at any given orientation, the fracture morphology was essentially the same for each of the three casting temperatures studied. Failure of the axial specimens occurred at 90 deg to the applied stress, that is, through the fibers (Figure 3). In some locations a small amount of fiber pull-out was observed (Figure 4). Failure of the 45 deg specimens occurred primarily along the fibers at approximately 45 deg to the applied stress, although small steps observed

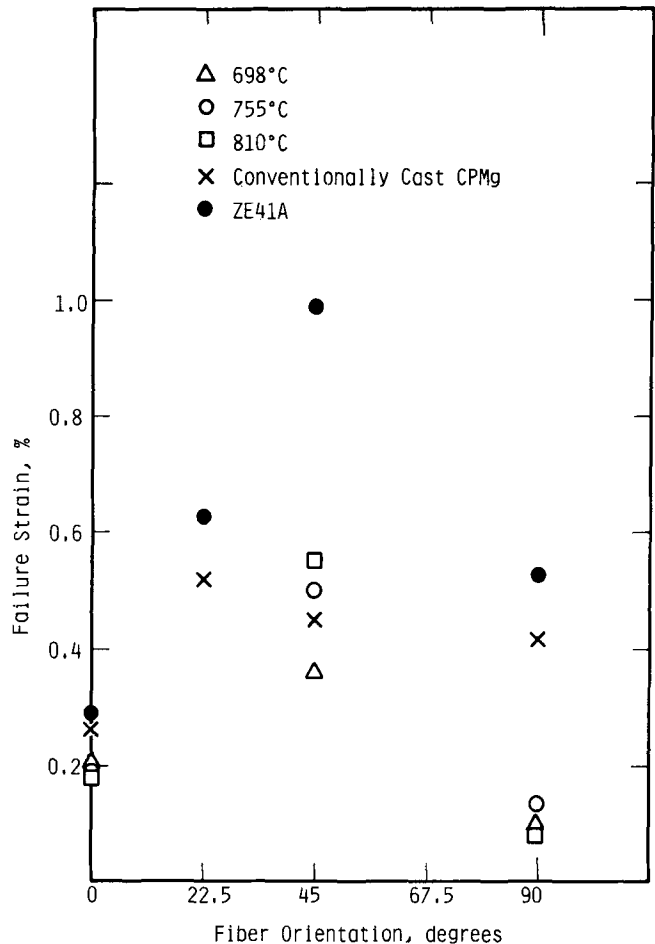


Fig. 2—Effect of fiber orientation and casting temperature on failure strain for commercially pure magnesium with 35 vol pct fibers. Data for conventionally cast CPMg and ZE41A from Refs. 1 and 2 are also included.

on the fracture surface indicated some failure across the fibers. Comparison of typical fractographs from the 698 °C, 755 °C, and 810 °C cast material with the 45 deg fiber orientation showed that failure occurred primarily by interfacial failure, together with a small amount of matrix failure. There were no significant differences in fracture morphology as a function of casting temperature. Failure of the 90 deg specimens occurred normal to the tensile axis and along the fiber-matrix interface, with a fracture morphology that was very similar to that observed in the 45 deg specimens (Figure 5).

High magnification fractographs of fiber surfaces in the three materials studied are shown in Figure 6. The micrographs show the fiber-matrix reaction zone, and although there is a wide variation in particle size at any given casting temperature, it is clear that the average particle size increased with increasing casting temperature.

C. Auger Spectroscopy

The results of Auger analyses performed on specimens with a 90 deg fiber orientation fractured *in situ* in the scanning Auger microprobe are shown in Figures 7 and 8. Secondary electron micrographs taken with the Auger electron microscope of the fracture surfaces of each of the three composite plates studied are shown in Figures 7(a)

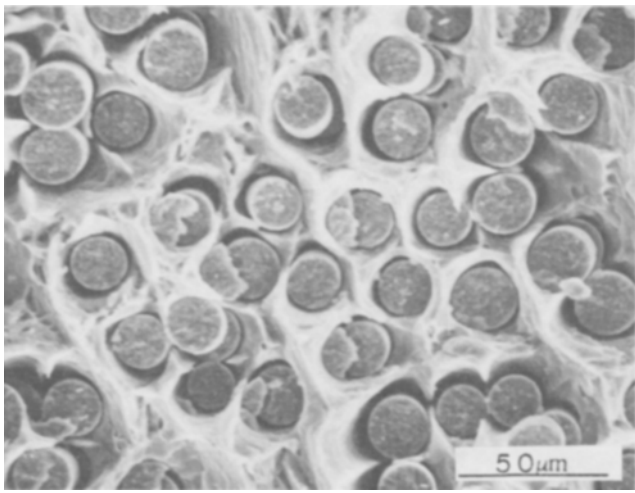


Fig. 3—Scanning electron micrograph of axial tensile failure in Al_2O_3 fiber reinforced CPMg cast at 698°C . Micrograph is typical of failure observed in 755°C and 810°C materials as well.

through (d). Corresponding Auger spectra from various features of these fracture surfaces are shown in Figures 8(a) through (c).

The secondary electron micrographs, Figures 7(a) through (c), show that all three specimens fractured cleanly along the fiber-matrix interface. The fibers (designated by the letter “A”) and troughs (designated by the letter “B”) are clearly evident, and their respective surfaces are seen at higher magnification in Figure 5. The only apparent difference among the three plates cast at the various temperatures was the amount of the lighter-appearing phase (designated by the letter “C”) which adhered to the fibers. The amount of this lighter phase covering the fibers increased with decreasing casting temperature, especially from 810°C to 755°C . As can be seen in Figure 7(d), this lighter phase appears to be a real interfacial layer, and not solely a result of charging of the expected oxide interfacial layer. The clear

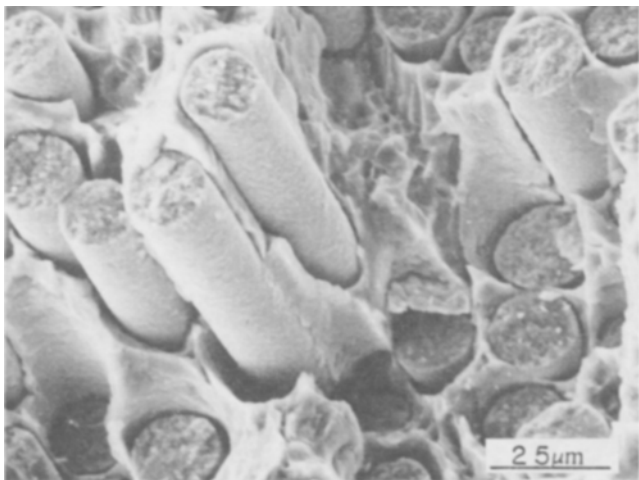
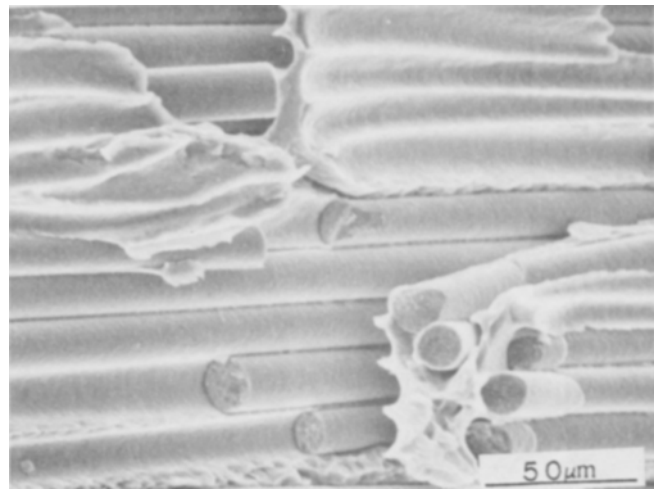
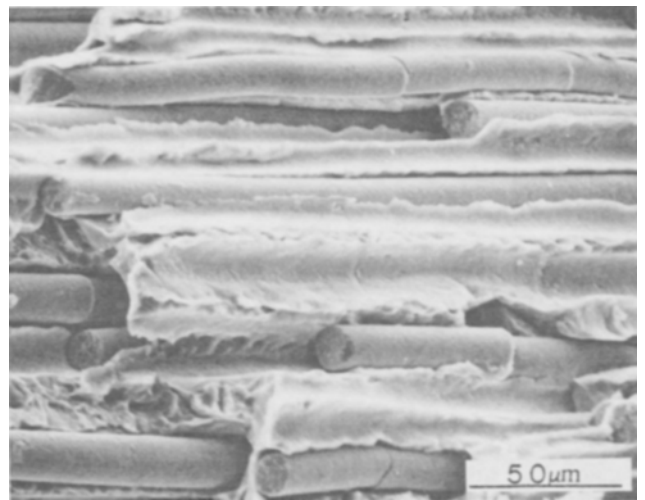


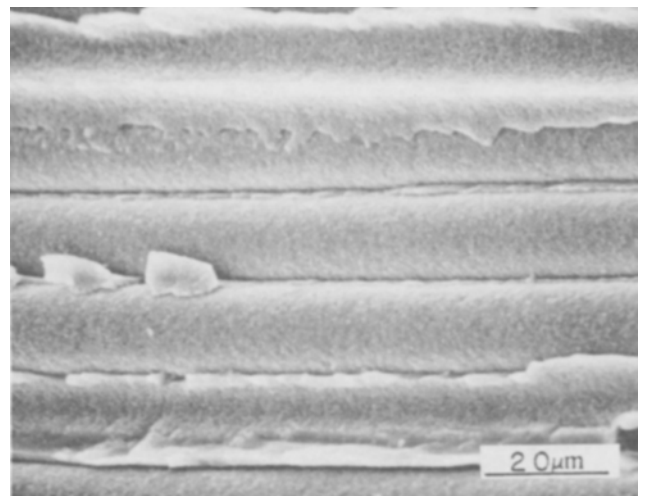
Fig. 4—Scanning electron micrograph of axial tensile failure in CPMg cast at 755°C showing small amount of fiber pull-out.



(a)

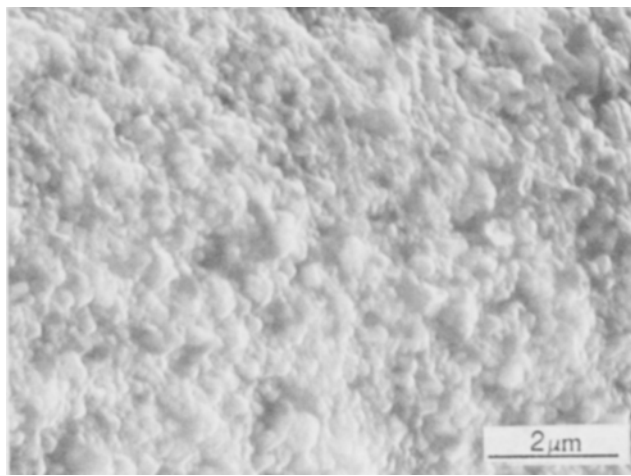


(b)

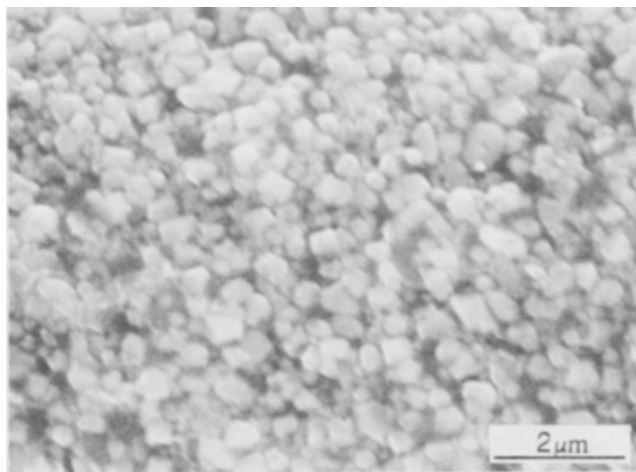


(c)

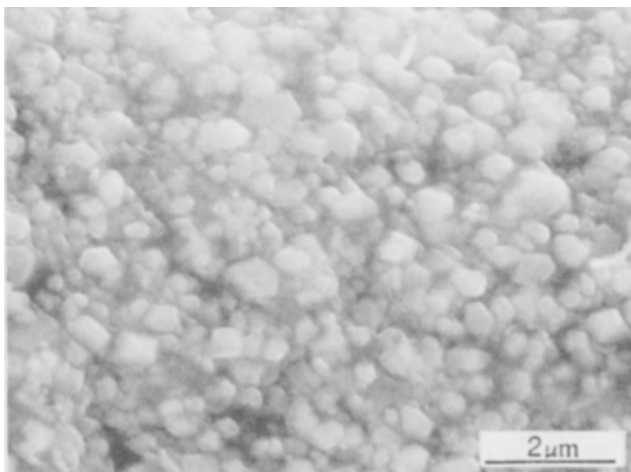
Fig. 5—Scanning electron micrographs showing typical transverse tensile failure in Al_2O_3 fiber reinforced CPMg cast at (a) 698°C , (b) 755°C , and (c) 810°C . Micrographs are typical of failures observed in 45° orientation material as well.



(a)



(b)



(c)

Fig. 6—Scanning electron micrographs of Al_2O_3 fiber surfaces in CPMg cast at (a) 698 °C, (b) 755 °C, and (c) 810 °C.

evidence of the lighter-appearing layer in the Auger micrographs, and the difficulty in observing it in the previous micrographs (Figure 5), is probably due to the change in contrast resulting from the low primary beam voltage used in obtaining the Auger micrographs and spectra, 3 keV, as opposed to that used to obtain micrographs in the scanning electron microscope, 30 keV.

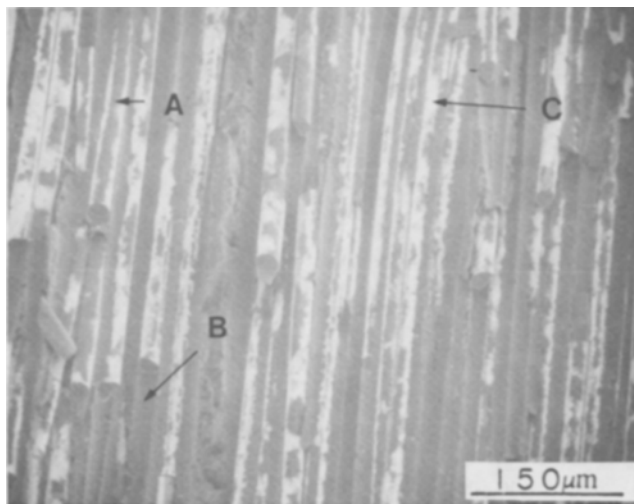
The chemistry of the various interfaces was analyzed by examining the higher energy Auger spectra of magnesium as was done in the previous investigation.⁷ Examination of the fine structure and peak positions of these spectra and comparison with standard reference spectra for magnesium (Mg) and magnesium oxide (MgO)¹⁰ indicate that the fiber surfaces were made up of pure MgO while the corresponding troughs were elemental Mg. The spectra from the lighter-appearing phase showed a mixture of the MgO and Mg spectra; some evidence of the fine structure for elemental Mg can be seen in Figure 8(c), while the main elemental peak has been shifted to lower energy from 1186 eV. The degree of this mixing varied between locations on a particular area of the lighter-appearing phase, but the amount of mixing did not vary to any great extent among the three processing treatments.

D. Electron Microscopy

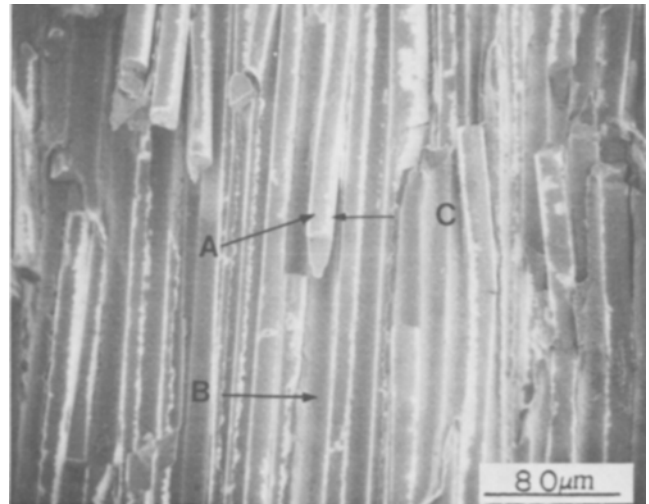
Transmission electron microscopy indicated the presence of a submicron fiber-matrix reaction zone for each of the three casting temperatures. Microdiffraction of the reaction zones identified each of them as being composed of MgO particles. The reaction zones contained a wide variation in MgO particle size, thus resulting in a very irregular interface between the MgO reaction zone and the metallic matrix. Typical micrographs of the reaction zones formed under the three different processing conditions are presented in Figure 9. Micrographs of typical reaction zones in conventionally processed CPMg and ZE41A have been included in Figure 9 for comparison purposes. The average MgO particle size increased with increasing casting temperature, changing from 0.16 μm at 698 °C to 0.30 μm at 755 °C, and finally to 0.37 μm at 810 °C. Thus, the 698 °C average MgO particle size was equivalent to the 0.14 μm average particle size of the conventionally processed material,² while the 755 °C and 810 °C particle sizes were significantly larger than the conventionally pressed CPMg.

IV. DISCUSSION

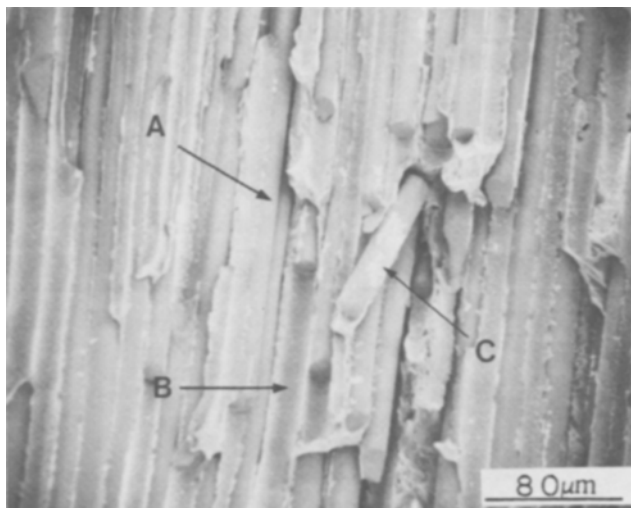
The SEM and TEM results indicate that the inclusion of a five-minute hold at 755 or 810 °C prior to solidification was effective in generating larger reaction zone particles. The average MgO particle sizes obtained with the five-minute hold at these temperatures (0.30 μm at 755 °C and 0.37 μm at 810 °C) were significantly larger than the average particle size in conventionally processed CPMg (0.14 μm) and at least as large as the average particle size in conventionally processed ZE41A (0.24 μm). The coarse reaction zone morphology closely resembled that of the conventionally processed ZE41A; that is, the fiber surface of the CPMg cast at the higher temperatures (755 and



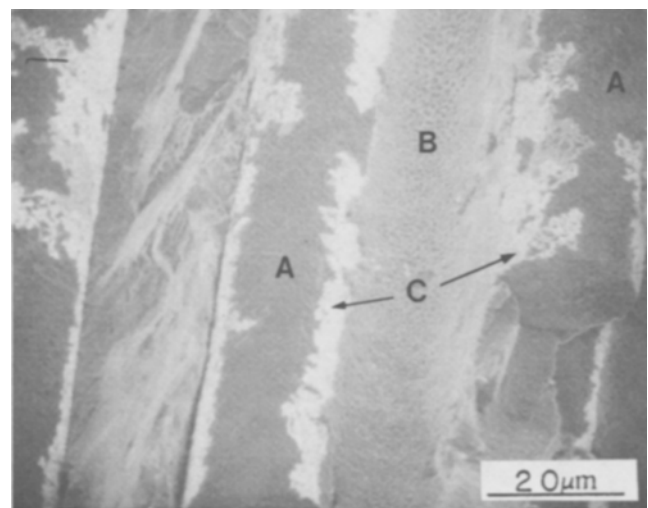
(a)



(b)



(c)



(d)

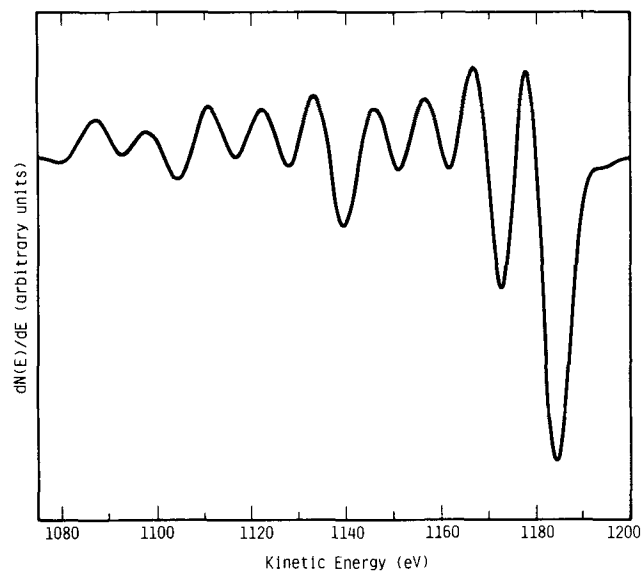
Fig. 7—Secondary electron micrographs taken in the Auger electron microscope of the fracture surfaces of specimens with 90 deg orientation fractured *in situ*. (a) Material cast at 698 °C. (b) and (d) Material cast at 755 °C. (c) Material cast at 810 °C. A—fiber, B—trough, C—lighter-appearing phase.

810 °C) were very similar to the fiber surfaces of the ZE41A material. On the other hand, a five-minute hold period at 698 °C produced a mean reaction zone particle size and fiber surface appearance similar to that observed in the conventionally processed CPMg.

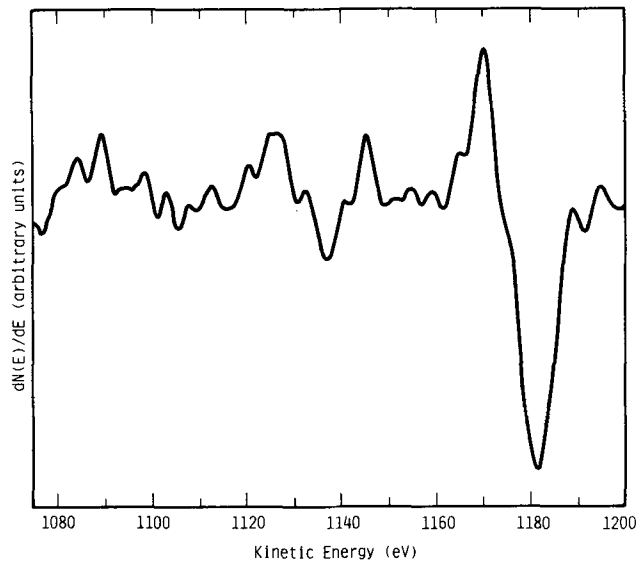
The size of the reaction zone, which is diffusionally controlled, is dependent on both the actual casting temperature and the time at temperature. Although the exact casting conditions for the conventionally processed CPMg were not known, the material was cast at an approximate temperature of 750 °C, with essentially no hold period prior to quenching. Thus, the large increase in reaction zone size observed for the material cast at 755 °C (0.30 μm compared to 0.14 μm) was due solely to the extended five-minute hold period. The material cast at 698 °C with a five-minute hold

demonstrated that the casting temperature was equally as important as the hold period in controlling the reaction zone size. The results showed that the expected large increase in reaction zone size due to the hold period was negated by the lower temperature, resulting in a reaction zone size comparable to that in the conventionally processed CPMg. The material cast at 810 °C with a five-minute hold would be expected to reflect the effects of both an increased temperature and extended hold. Since the reaction zone was only slightly larger than that observed in the 755 °C cast material (0.37 μm compared to 0.3 μm), this indicates that the growth of the reaction zone was size limited, as would be expected for a diffusion controlled process.

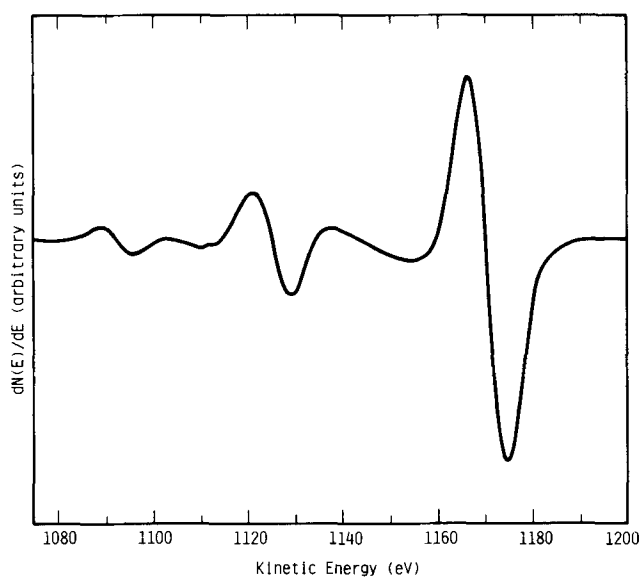
As expected, the increased reaction zone thickness resulted in a decrease in the axial strength, as compared to the



(a)



(c)



(b)

Fig. 8—Typical high energy Auger spectra for magnesium taken from the fracture surface features shown in Fig. 7. (a) Fiber surface, (b) trough, and (c) lighter-appearing phase.

conventionally processed CPMg. This reduction in fiber strength is thought to be caused by the enhanced reaction between the fibers and molten matrix which degrades the fiber properties. However, the axial properties of the material cast at 698 °C were also degraded as compared to those of the conventionally processed CPMg, even though the reaction zone sizes were equivalent. This indicates that the size of the reaction zone cannot simply be used as a true measure of the reaction that occurs to degrade the fiber strength.

It is also evident from the tensile results obtained in this study that the increase in transverse strength that was anticipated with increasing reaction zone size never materialized. Instead, the transverse strengths of the specially cast materials were relatively independent of the casting temperature

and reaction zone size, and, additionally, fell below the transverse strength of the conventionally processed ZE41A material.

The fractography and the Auger results also support the conclusion that the increased particle size obtained during the extended hold period at 755 and 810 °C did not result in a strengthened interface. There was no discernible difference between the fracture morphology of the conventionally cast CPMg material and the CPMg material produced with the extended hold periods. Furthermore, the Auger results demonstrated that interfacial failure in all of the CPMg materials occurred along the interface between the metallic magnesium of the matrix and the magnesium oxide of the reaction zone with little, or no, metallic magnesium adhering to the reaction zone surface. The Auger results also

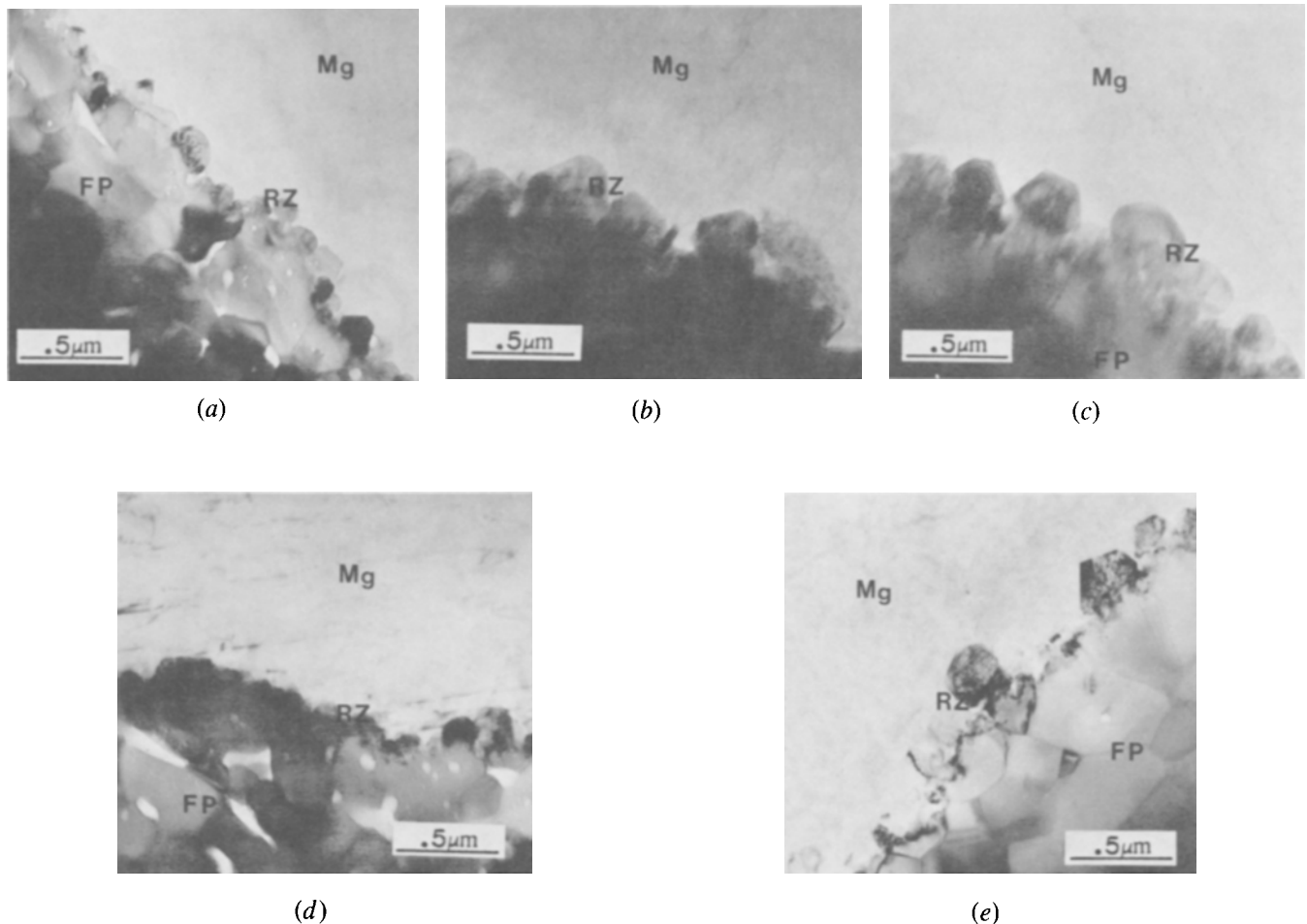


Fig. 9—Transmission electron micrographs of the fiber/matrix reaction zones in CPMg cast with a five-minute hold at (a) 698 °C, (b) 755 °C, and (c) 810 °C; (d) conventionally cast CPMg, and (e) conventionally cast ZE41A. The various constituents are indicated by Mg (matrix), RZ (reaction zone product) and FP (Al_2O_3 fiber).

failed to identify the presence of any interfacial segregation which might explain the apparent loss in interfacial strength as a result of the revised casting process.

As mentioned in the Introduction, previous studies of the fatigue and tensile behavior of unidirectional Al_2O_3 fiber reinforced composites with either a commercially pure magnesium matrix or a ZE41A magnesium alloy matrix revealed a dramatic shift in the off-axis failure mode and an increase in off-axis strength with a change from the CPMg matrix to the ZE41A matrix.¹ The difference in off-axis behavior between the two materials was attributed to increases in interfacial and matrix strengths with a subsequent decrease in fiber strength in the ZE41A matrix composite. Based on the results of detailed microstructural and chemical characterizations of the fiber-matrix interface regions in these materials, it was concluded that the interfacial reaction zone particles were MgO in both composites and that segregation of impurity or alloying elements to the interfacial region had not occurred in either material. The major difference observed between the interfacial reaction zones was that the MgO particles in the ZE41A matrix material were consistently about twice as large as those in the CPMg matrix material. If the interfacial strengthening was due solely to the morphology of the reaction zone, then it should be

possible to improve dramatically the off-axis strength of CPMg matrix material through proper alteration of the processing conditions.

The present results do not support the above hypothesis. Although the extended hold period at 755 and 810 °C prior to solidification was effective in developing enlarged MgO reaction zone particles, a reduction in off-axis strength resulted, rather than the expected increase in strength. The relative independence of the tensile strength on casting temperature also implies that the MgO particle morphology plays, at most, a minor role in determining the interfacial strength. Since these results refute the earlier hypothesis, it is necessary to consider alternate strengthening mechanisms, the most reasonable of which is strengthening due to the effect of the alloy additions in ZE41A (zinc, zirconium, and rare earths) on the bonding between the magnesium oxide reaction zone and the magnesium matrix. Bonding between the magnesium oxide reaction zone and the metallic matrix is of critical interest since it is along this interface that failure occurs.¹ Although previous Auger results for the ZE41A material indicated an absence of segregation at the interface between the reaction zone and the metallic matrix, there was evidence of small quantities of zinc and aluminum in both the reaction zone and the metallic

matrix around the reaction zone.¹ The source of the zinc was the magnesium alloy matrix and the source of the aluminum was the alumina (Al₂O₃) fibers. Neither zinc nor aluminum was observed in the Auger investigations of interfacial failures in the conventionally cast CPMg material¹ or the materials cast with an extended hold period in this study. Thus, one possible explanation for the interfacial strengthening in ZE41A is that the presence of zinc and/or aluminum, even in low concentrations, acts to increase the bonding between magnesium oxide and metallic magnesium. Although the presence of zinc was not expected in these materials with a commercially pure Mg matrix, the presence of aluminum in the reaction zone and matrix around the reaction zone could still be anticipated. Since aluminum was not observed, the incorporation of aluminum in the reaction zone region appears to be dependent on the presence of the alloying elements (specifically zinc) in the ZE41A material.

V. CONCLUSIONS

The following conclusions can be drawn from the results of the present investigation:

1. The tensile properties of alumina fiber reinforced CPMg cast at 698 °C, 755 °C, and 810 °C with a five-minute hold period prior to solidification were independent of casting temperature. The reduction in tensile properties observed for off-axis loading was due to a weak fiber-matrix interface, and was related to a change in fracture mode from flat fracture across the fibers in the axial condition to failure along the fiber-matrix interface in the off-axis condition. This interfacial delamination occurred along the interface between the magnesium matrix and the magnesium oxide reaction zone.
2. The five-minute hold period prior to solidification significantly decreased the axial properties when compared to conventionally cast CPMg. However, the properties were found to be independent of casting temperature, although the casting temperature significantly affected the reaction zone size. The hold time and casting temperature used during conventional processing appear to have been optimized.
3. The size of the magnesium oxide reaction zone was found to increase with increasing casting temperature.

However, as this did not result in improved off-axis strength, the interfacial strengthening observed in ZE41A was not due to the larger magnesium oxide reaction zone particles, as earlier hypothesized.

4. Auger spectra for the specially cast CPMg materials and the conventionally cast CPMg and ZE41A indicate that the interfacial strengthening in ZE41A may have been due to the presence of zinc and/or aluminum, which may, even in low concentrations, act to increase the bond strength between magnesium oxide and metallic magnesium.

ACKNOWLEDGMENTS

The authors are grateful for the support of this work by the Army Research Office under Contract No. DAAG29-85-K-0050. The assistance of Messrs. R. L. Tuck and H. G. Saldana in specimen preparation, testing, and thin foil preparation is greatly appreciated. Helpful discussions with G. R. Leverant are also acknowledged.

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