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# 알루미나 장섬유 강화 복합금속재의 피로균열성장거동

# Fatigue Crack Growth Behavior of A Continuous Alumina Fiber Reinforced Metal Matrix Composite Materials

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요 지

장섬유 강화 마그네슘 복합제(FP/ZE41A)의 피로 균열 성장 거동에 대한 열처리 효과를 규명한 것으로 TEM관측에 의해 알루미나 섬유와 마그네슘 복합 매트릭스간의 상호 접면을 완화시키기 위하여 풀림을 실시하였다.

피로 균열 성장 방향에 수직한 섬유와 평행한 섬유들에 대한 피로 균열 성장 거동에 관한 실험을 실시한 바, 피로 균열 성장 방향에 수직한 시험편의 경우 열처리를 실시한 시험편은 잔류 응력을 제거시키지 않은 시험편에 비해 피로 균열 성장에 대한 더 많은 저항성을 갖고 있음을 알수 있었다.

그러나, 이에 반해 피로 균열 성장 방향에 평행한 시험편의 경우는 잔류 응력을 제거시키지 않은 시험편이 열처리를 실시한 시험편에 비해 더 많은 피로 균열 성장 저항성을 내포하고 있다는 피로 균열 성장 거동에 대한 차이점을 발견할 수 있었다.

피로 파괴 표면에 대한 연성 파열과 섬유 박리를 SEM관찰한 결과 열처리는 피로균열 성장 거동에서 지적된 바와 같이 섬유와 매트릭스 상호면의 강도를 약화시킨다는 것을 알 수 있었다.

Abstract

**The effects of heat treatment on fatigue crack growth behavior were studied in continuously reinfored, magnesium-based composite (FP/ZE41A). Following an earlier TEM investigation, specimens were thermally aged to modify the interfacial zone between the alumina fibers and mg alloy matrix. The fatigue crack growth experiments were conducted with specimens having the fiber orientation normal to the crack growth direction(longitudinal)and also specimens with the fibers oriented parallel to the crack growth direction (transverse). A comparision of the fatigue crack growth behavior indicates that aged longitudinal specimens are more resistant to fatigue crack growth than as-fabricated longitudinal specimens. Conversely, as-fabricated transverse specimens are more resistant to fatigue crack growth than aged transverse specimens. SEM observations of fiber pullout and ductile**

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**tearing on the fatigue fracture surfaces indicate that the aging weakens the strength of the fiber/matrix interface, giving rise to the observed fatigue crack growth behavior.**

## Introduction

Magnesium and magnesium alloys are among the lightest candidate materials for practical use as the matrix phase in metal matrix composites (MMCs). When compared to other currently available structural materials, Mg is a very attractive because of its unique combination of low density and excellent machinability[1-3]. Although Mg alloys do not compare favorably with Al alloys in terms of absolute strength, their low density(35% lower than that of Al) makes them competitive in terms of strength/density values. With the development of MMC technology, it has become possible to combine the properties of Mg matrices(ductility and toughness)with ceramic properties(high strength and high modulus), leading to greater strength in shear and compression and higher service temperature capabilities[4]. Interest in MMCs for aerospace, automotive and other structural applications has increased over the last five years as a result of availability of relatively inexpensive reinforcements and the development of various processing routes which result in reproducible microstructures and properties[5].

The use of metal-matrix composites in major load bearing components and structures requiers a capability for providing adequate resistance to crack growth under fluctuating loading conditions. The development of this capability can be achieved through a better understanding of the relationship between microstructure and mechanical properties. Within the complex microstructures of MMCs, the interface between the reinforcement phase and the matrix phase is typically the least understood feature. The relatively high sensitivity of the reinforcement/matrix interface to stress state, alloying, deformation and heat treatment presents a challenge to those attempting to optimize the fatigue properties of these materials. Thus, it is important that the effects of these processing variables on fatigue behavior are studied to achieve a better understanding of the mechanismes that control fatigue damage in MMCs.

Studies of tensile and fatigue properties for fiber reinforced Mg alloys are reported elsewhere [3-6]. In these investigations, mechanical behavior is compared for different alloy compositions, fiber volume fractions and fiber orientations. In a more recent study, McMinn *et al.*[6] varied the casting temperatures as well as the contact time between alumina fibers and a molten magnesium matrix to investigate the effects of processing variables on the tensile properties of the composite. They found that increasing casting temperature resulted in an increase in size of both the interface reaction zone and MgO particles within this zone. Mechanical testing revealed that this microstructural modification did not result in improved off-axis strength. Moreover, it was found that a five-minute hold period prior to solidification significantly decreased the strength of the composite.

Taking an alternate approach, Chin modified the interfacial reaction zone in alumina fiber reinforced ZE41A Mg alloy by thermal aging[7]. This composite is particularly attractive because the alloy additions to Mg give rise to superior fiber/matrix interfacial strength and mechanical properties as compared to alumina reinforced pure magnesium[4]. Chin's TEM observations[7] indicate that aging at 450℃ for 95 hours results in several microstructural changes : (1) the interfacial zone incressed from 0.25 $\mu$m to $1-2$ $\mu$m in width, (2) the size of MgO interfacial particles decreased, (3) polygonization of the dislocation structure in the magnesium matrix occurred and (4) rod-like MgZn precipitates dissolved while the number and size of spherical precipitates, probably MgZn, increased. It was suggested that the apparent decrease in MgO particle size may increase the interfacial fracture strength since the stress concentrations associated with these particles would be reduced.

This paper presents preliminary experimental results from a study of the effects of that treatment on fatigue crack growth in the continuous fiber reinforced Mg alloy, FP/ZE41A. Emphasis is placed on the role of the fiber/matrix interface in facilitating crack growth. Microstructural observations are combined with data from the fatigue crack growth experiments. The long term goal of the research is to develop a better understanding of fatigue crack growth mechanisms in this and other metal matrix composites.

## Procedures

The material selected for the present study was polycrystalline $\alpha-$alumina fiber (DuPont FP) reinforced magnesium cast plates, 15.24cm $\times$ 15.24cm $\times$ 1.27cm. The material was fabricated at E.I.DuPont de Nemours by infiltrating FP fibers with a magnesium casting alloy (ZE41A) at 35% fiber volume fraction. The nominal composition of th casting alloy was Mg $-3.8-4.4$ Zn$-0.1-0.3$ Zr$-0.4-0.9$ rare earths(in wt.%)[5]. The materials were obtained as a courtesy of Mr. Ernest Chin at the Army Materials Technology Laboratories.

Two conditions were selected for the present investigation : as-fabricatied by pressure infiltration casting and thermally aged. The second condition involved aging the as-fabricated material for 95 hours at 450℃ in a Lindberg vacuum furnace. This is the same heat treatment performed by Chin in his TEM study of the effects of thermal aging on the microstructure of FP/ZE41A[7]. In order to avoid extensive oxidation of the materials during thermal exposure, the furnace was pumped down to a vacuum level of 80 torr and flushed with argon gas; this procedure was repeated five times.

Single edge-notched(SEM) specimens with a thickness of 2.54mm and a width of 12.7mm were machined from both the as-fabricated and aged material. The fibers were oriented parallel to the loading direction for half of the specimens(longitudinal) and oriented perpendicular to the loading direction for the other half(transverse). These two fiber orientations were chosen to investigate fatigue crack growth along as well as through the reinforcing fibers in both as-fabricated and heat treated specimens. Before testing, silicon carbide polishing paper was bonded to both ends of the specimens for gripping purposes. A photograph of an SEN specimen with fibers oriented longitudinally is shown in Figure 1.

The crack length was measured using both a traveling optical microscope, during periodic interruptions of the tests, and a direct current (dc) potential drop technique, for in-situ crack monitoring. In the present investigation, a crack length resolution of about 0.05mm was achieved with the potential drop technique for the FP/ZE41A composite using 20 amps. In order to minimize specimen heating and electric current drift errors, each potential dorp measurement was made by applying the constant current along the length of a specimen for only one and a half seconds. During this period, the voltage between points on either side of the crack was measured to determine the crack length. The positions of the crack voltage probes are indicated by dimples on the specimen surface in

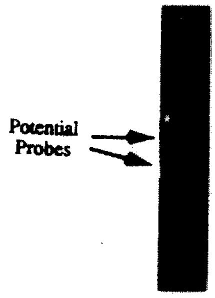



Figure 1. The single edge notch(SEN) specimen geometry used in the present investigation. This specimen has the longitudinal fiber orientation (normal to the crack growth direction).

Figure 1. Difficulties in spot welding the Mg alloy made it necessary to machine these dimples on the specimens so that the crack potential probes could be attached mechanically at precise locations. Prior to the utilization of the potential drop technique, it was necessary to acquire calibration data by correlation optically measured crack length values with crack potential measurements. Different calibration correlation curves were obtained for the two different fiber orientations used in this investigation. This difference is apparently due to the effect of the alumina fibers on the electric current flow through the specimens.

The experiments were performed using an MTS 810 servo-hydraulic test system equipped with precision aligned hydraulic collet grips interfaced to a computer based data acquisition and control system. The specimens were all tested under a load ratio, R, of 0.1 at a frequency of 15 Hz. Before testing, specimens of the same fiber orientation were pre-cracked under identical cyclic loading conditions. The samples with the longitudinal fiber orientation were tested under constant load amplitudes ranging from 400 to 2400 N and the specimens with the transverse orientation were tested under load amplitudes ranging from 280 to 400 N. As-fabricated specimens and aged specimens were tested under the same loading conditions to examine the effect of thermal aging on the fatigue crack growth behavior of the aluminar fiber reinforced Mg alloy.

The fatigue fracture surfaces of the crack growth specimens were examined using SEM techniques. To ensure adequate conductivity, the specimens were sputtered with Au-40 wt.% Pd before examination in the scanning electron microscope.

## Results and Discussion

### Mechanical Testing

A typical plot of crack length versus cycle number for an as-cast longitudinal specimen and an aged longitudinal specimen tested under identical loading conditions is illustrated in Figure 2. The fatigue crack growth rate, the slope of this data, is always greater for the as-cast specimen resulting in a decrease in fatigue life by over a factor of 2. This indicates that the aged composite has a greater resistance to fatigue crack growth through the fibers. Both specimens fractured catastrophically once the crack length reached about 8.5mm.

Figure 3 shows a typical plot of crack length versus cycle number for transverse as-fabricated and aged specimens under the same load range. The data in this plot illustrate that the resistance to fatigue crack growth parallel to the fiber direction is greater for the as-fabricated material. We note that the crack growth rate temporarily decelerates for the as-fabricated material corresponding to crack lengths of approximately 7.5mm and 9.5mm. Preliminary observations suggest that these temporary reductions in the crack growth rate result from defects in the fiber structure in regions near the crack tip. Further work is currently underway to examine the cause of this non-uniform

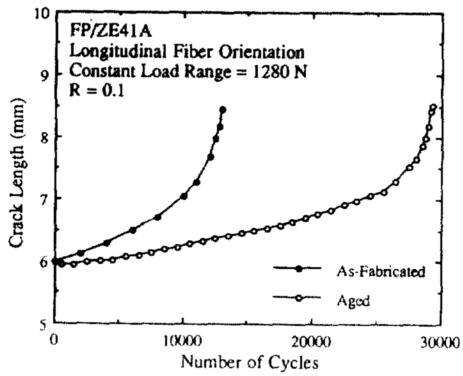



Figure 2. Crack length versus cycle number for FP/ZE41A alumina fiber reinforced Mg alloy with the fibers oriented longitudinally(parallel to the applied stress). Data are shown for both the as-fabricated and aged conditions.

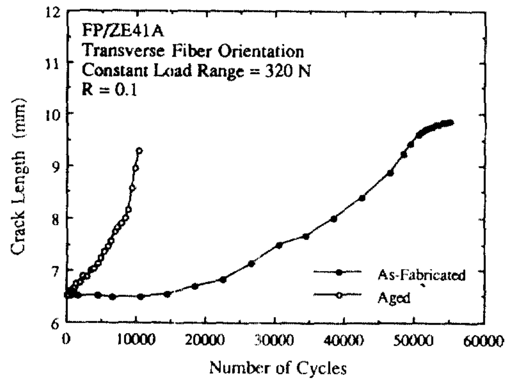



Figure 3. Crack length versus cycle number for FP/ZE41A alumina fiber reinforced Mg alloy with the fibers oriented transverse to the applied stress. Data are shown for both the as-fabricated and aged specimens.

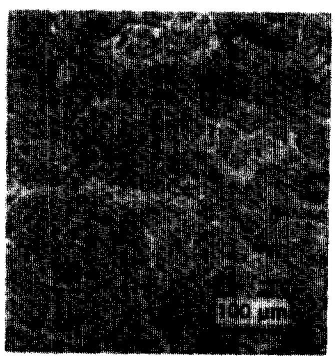



Figure 4. Fatigue fracture surface of an as-fabricated specimen with the fibers oriented normal to the crack plane(parallel to the applied stress). The region shown in this micrograph is approximately 7.5mm from the notched edge of the specimen.

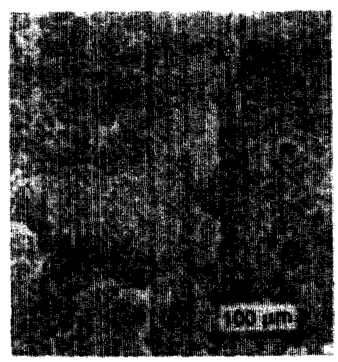



Figure 5. Fatigue fracture surface of an aged specimen with the firbers oriented normal to the crack plane(parallel to the applied stress). The region shown in this micrograph is approximately 7.5mm from the notched edge of the specimen.

crack growth behavior.

## Fractographic Observations

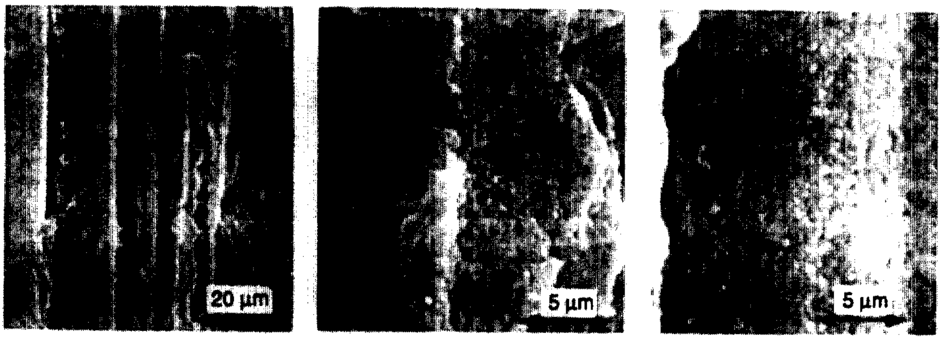
Figure 4 shows a typical SEM micrograph of the fracture surface of the longitudinal as fabricated specimen tested under a load range of 1280 N. The direction of crack propagation was from the top to the bottom of this micrograph. We note that very little fiber pull-out occurred suggesting that the interfacial shear strength was sufficient to inhibit debonding between the fiber and the matrix during crack growth through the fiber. Agglomerates of fine particulate matter can also be seem on the fracture surface. This debris, which could not be removed by ultrasonic cleaning, appears to be associated with the fatigue crack growth process since it was not observed on the part of the fracture surface that corresponds to final unstable fracture. We note that it is difficult to distinguish many of the fibers from the matrix in Figure 4. This observation also indicates that very little interfacial deformation and debonding occurred during crack growth.

A representative micrograph of the surface of the aged longitudinal specimen, that was also tested under a load range of 1280 N, is shown in Figure 5. In contrast to figure 4, almost all of the fibers are distinguished from the matrix as a result of localized shear and debonding at the fiber/matrix interfaces. This, in addition to the more prevalent fiber pullout, indicates that the interface between the fiber and the matrix is weaker than that for the as-fabricated specimen tested under the same loading conditions. We also note the presence of debris observed on the fracture surface of the as-fabricated specimen is less noticeable on the fracture surface of the aged specimen.

The fracture surface of the transverse as-fabricated specimen tested under a load of 320 is shown in Figure 6.a. Fatigue cracking in this specimen occurred by a combination of delamination of the fiber/matrix interface and crack propagation through the matrix. This combination of fatigue cracking mechanisms in off-axis FP/ZE14A was also observed by Hack *et al*[3]. Figure 6.b is a micrograph at higher magnification of a region corresponding to matrix material (labeled "b" in Figure 6.a). The area reminiscent of brittle cleavage on the left corresponds to fatigue crack propagation through the Mg alloy. The region on the right, which is resembles a ductile rupture surface, is a result of the crack propagation along the fiber/matrix interface. It appears from the interfacial fracture surface that microcracks formed by decohesion at the fiber/matrix interface. This was then followed by ductile shear failure of the matrix ligaments that remain bonded to the fiber. This mechanism is further supported in Figure 6.c which shows an exposed fiber surface, labeled "c" in Figure 6.a, at higher magnification. It appears in this figure that the surface of the fiber is made up of alumina grain facets(alumina grain size$=0.5\mu$m)surrounded by shear ridges of alloy material.

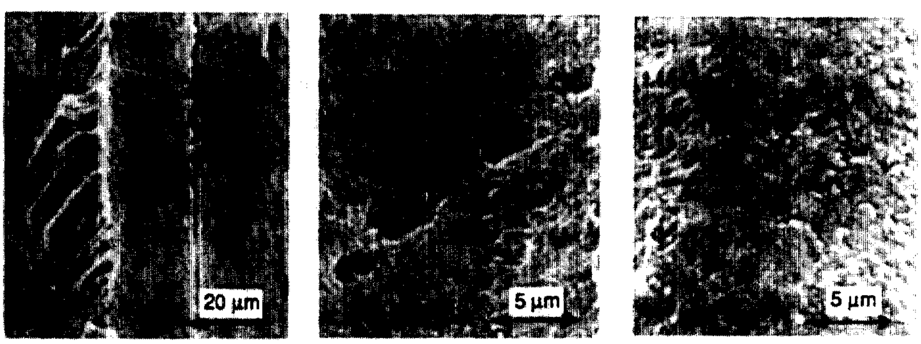
Figure 7.a shows the fracture surface of the aged transverse specimen tested under a load range of 320 N. It can be seen here that the crack growth also occurs by a combination of interfacial and matrix cracking for this material condition. However, the regions resulting from interfacial cracking are quite different from that observed in Figure 6. We note, for example, that large faceted dimples are left in the matrix, as indicated at higher magnification in Figure 7.b. It also appears that the interface failed in a more brittle manner with less plastic deformation. A micrograph of an exposed fiber at higher magnification is shown in Figure 7.c. In contrast to Figure 6.c, the alumina grain facets of the fiber are not visible on the fracture surface suggestion that the crack did not grow just along the alumina fiber surface, rather it seems to have propagated through the interfacial reaction zone, which becomes larger during aging[7]. We also note the presence of particulates, on the order of 500nm in diameter, on the fracture surface in Figure 7.c. Their size and density indicate they are probably spherical MgZn precipitates that are reported to form during this aging treatment[7]. Since these particles are potential sights for microvoid nucleation, it is reasonable that their excessive presence substantially weakens the tensile strength of the fiber/matrix interfacial reaction zone.

The crack growth data in Figure 3 and the observation of ductile tearing in Figures 6.b and 6.c indicates that more plastic strain energy is




a b c

Figures 6.a, b and c. Fatigue fracture surface of an as-fabricated transverse specimen showing the predominant fiber/matrix interface cracking with relatively minor matrix cracking. The region in Figure 6.b is the result of matrix cracking, labeled "b", and ductile rupture of the matrix material, where a fiber pulled away at the interface, labeled "c". Figure 6.c shows the surface of a fiber at a magnification revealing the alumina grain structure and remaining ligaments of Mg alloy. The region of the fracture surface shown in these micrographs is approximately 7.5mm from the notched edge of the specimen.




a b c

Figures 7.a, b and c. Fatigue fracture surface of an aged transverse specimen indication fiber/matrix interface cracking and matrix cracking. The area in Figure 7.b is where a fiber away at the fiber/matrix interface. Figure 7.c shows the surface of a fiber which has a different appearance compared to that observed for the as-fabricated specimen(Figure 6.c). The region of the fracture surface shown in these micrographs is approximately 7.5mm from the notched edge of the specimen.

dissipated during fatigue crack propagation in as-fabricated transverse specimens. This results in a reduced driving force for crack growth as compared to that for the aged transverse specimens tested under the same conditions. Conversely, the results indicate that the weaker interfaces resulting from thermal aging give rise to slower crack growth in longitudinal specimens due to the additional energy dissipated by interface debonding and fiber pullout[8, 9].

## Conclusions

Fatigue crack growth specimens of a continuous alumina fiber reinforced Mg alloy were tested in the as-fabricated and thermally aged condition. Experiments with the fiber orientation normal to the crack plane(longitudinal) indicate that the crack growth resistance is greater for the aged material resulting in an increase in fatigue life of abou a factor of 2. Conversely, fatigue crack growth tests with the fiber orientation parallel to the crack plane(transverse)indicate that the resistance to fatigue crack growth along the fibers is reduced by the thermal aging treatment.

Observations of the fracture surfaces of longitudinal specimens reveal that more debonding and fiber pullout occurs in the aged specimens. This observation suggests that, although the fiber/matrix interface is weaker, additional energy is dissipated for crack growth after thermal aging. This additional work requirement is consisten, with the observed difference in fatigue crack growth rates for the aged and as-fabricated longitudinal specimens.

SEM observations on the fatigue fracture surface of transverse specimens indicate that, for this fiber orientation, the fatigue cracks grow primarily along the fiber/matrix interface and, to a lesser degree, through the matrix. For the as-cast condition, cracking along the fiber/matrix interface occurred by microcracking at or very near alumina grain facets accompanied by extensive shearing of the remaining Mg alloy ligaments. For the aged condition, interface delamination appears to be dominated by the presence of closely spaced precipitate particles in the reaction zone. The difference in fracture surface morphology suggests that more strain energy is dissipated by crack growth in the as-fabricated transverse material. This finding is supported by the observed difference in fatigue crack growth rates for the aged and as-fabricated transverse specimens.

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