High Temperature Properties of AZ91D Magnesium Alloy Composite Reinforced with Short Alumina Fiber and Mg$_2$Si Particle

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Short alumina fiber- and in situ Mg$_2$Si particle reinforced magnesium alloy composites were fabricated by squeeze casting. The in situ Mg$_2$Si particles were formed during the infiltration with the melt into the preforms consisting of the fibers having Si particles attached to their surfaces. The microstructure of the composites and their tensile strength and Young’s modulus in the range from 293 K to 523 K were investigated. The effect of the Mg$_2$Si particles on the tensile properties was examined by comparison with the properties of the fiber-reinforced composite without the Mg$_2$Si particles. Fine Mg$_2$Si particles with a grain size of approximately 5 μm were formed due to the rapid solidification in the permanent mold. The dispersion of the Mg$_2$Si having a high Young’s modulus was found to be effective for improving Young’s modulus of the fiber-reinforced magnesium alloy composite. The experimental value of the composite was in good agreement with the calculated value based on a previously proposed model. The tensile strength of the composite with 18 vol% fibers and Mg$_2$Si particles was higher than that of the conventional fiber-reinforced composite at both room temperature and high temperature. Examination of the fracture surfaces indicated that stress transmission between the fiber and the matrix became easy due to the particles, which reduced the fiber-to-fiber contact.

1. Introduction

The application of magnesium (Mg) alloys has recently increased in electrical instruments, mobile instruments and automobiles in which their lightweight is an advantage. Although heat-resistant Mg alloys containing alloying elements have been developed, the application of the Mg alloy in parts that require high-temperature strength is still limited due to their poor strength at high temperature. The reinforcement of the Mg alloy with ceramics fibers is one of the possible techniques to improve the high temperature strength. We investigated the relationship between the alumina fiber volume fraction and high temperature strength of the fiber-reinforced AZ91D Mg alloy composite fabricated by squeeze casting, and then clarified that the tensile strength of the composite at 523 K showed a maximum at 18 vol% fiber. The strength at 523 K was 160 MPa, which was 1.8 times higher than the AZ91D Mg alloy (90 MPa). In order to obtain a composite having a higher strength at high temperature, we fabricated the composite reinforced with alumina fibers and in situ formed fine Mg$_2$Si particles. In this process, Mg$_2$Si is formed by the reaction expressed by the following eq. (1) during the Mg melt infiltration into the fiber preform containing pure silicon (Si) particles.

\[2\text{Mg} + \text{Si} \rightarrow \text{Mg}_2\text{Si}\]

Mg$_2$Si is a heat-resistant intermetallic compound. However, there are few reports about the strength properties of the composite reinforced with short alumina fibers and in situ Mg$_2$Si particles. Considering the practical application of the composite, the clarification of not only the tensile strength, but also the properties in the non-destructive region, that is, the rigidity is very important.

In the present study, the tensile strength and Young’s modulus of the alumina fiber- and Mg$_2$Si particle reinforced AZ91D Mg composites fabricated by squeeze casting were investigated. In addition, the effect of the Mg$_2$Si particles on the tensile properties at high temperature was examined by comparison with the properties of the fiber-reinforced composite without the Mg$_2$Si particles.

2. Experimental Procedure

The AZ91D Mg alloy with the chemical composition shown in Table 1 was used as the matrix metal. Short alumina fibers (Saffil, ICI) were used as a reinforcement. The chemical composition and properties of the alumina fiber are shown in Table 2. Pure Si particles (99.9 mass% Si) were used as the starting material to form Mg$_2$Si by the reaction with Mg in the alloy melt. Figure 1 is a SEM micrograph and size distribution of the Si particles, showing that their average size is 5 μm. The typical properties of the Mg$_2$Si are shown in Table 3. The preforms were fabricated as follows. First, 1 mass% polyvinyl alcohol as the organic binder, 2 mass%
Al₂O₃ sol as the inorganic binder and Si particles were added to distilled water to form a slurry. Second, the alumina fibers were soaked in the slurry in order to allow attachment of the Si particles and binders, followed by dewatering and forming of the cylindrical preform (55 mm diameter, 30 mm height). The preforms were dried at 373 K for 3 hours, and then sintered at 1173 K for one hour. Each fiber volume fraction in the composite was set to 0, 7, 13, 18, 23 and 33 vol%. Each fiber volume fraction in the preform was set lower than the fiber volume fraction of the composite by 2–3 vol%, because we confirmed that the fiber volume fraction was increased by 2–3 vol% due to the contraction during the melt infiltration.¹) For the alumina fiber reinforced AZ91D alloy composite, the composite with 18 vol% fiber was the strongest at high temperature.¹) Therefore, the fiber volume fraction of the composite with Mg₂Si was set to 18 vol% in the present study. The volume fraction of the Si particles was set to 3.2 vol%.

The composite was fabricated by squeeze casting. The preform was horizontally placed in the mold, and 200 g of the AZ91D alloy melt (1003 K) was poured into the mold (673 K). Pressure (40 MPa) was quickly applied and maintained until the solidification was complete. Although the AZ91D alloy can be strengthened by the heat treatment, we confirmed that the high temperature strength of the fiber-reinforced AZ91D alloy composite without heat treatment was higher that that of the heat-treated composite.¹) Therefore, the heat treatment was not performed in the present study. The microstructures of the composite and the Mg₂Si particles extracted from the composite by 30% nitric acid were observed. Tensile test specimens were cut parallel to the planar direction, with a gage length of 10 mm, a cross sectional width of 6 mm and a thickness of 3 mm. The test temperatures ranged from room temperature (293 K) to 523 K. Stress-strain curves were obtained by attaching the strain gage to the specimen. Young’s modulus was obtained from the gradient in the elastic region of the stress-strain curve.

### Results

#### 3.1 Microstructure of composite

Figure 3 shows the microstructures of the parallel section of the composites. Alumina fibers appeared black in the microstructure and are oriented in random configurations. A granular phase, dispersed in the matrix, was also observed in the composite fabricated using the preform containing Si particles (arrows in Fig. 3(d)). As a result of electron probe X-ray microanalysis and X-ray diffractometry, it can be concluded that the granular phase is Mg₂Si. The area fraction of the Mg₂Si particles in the composite measured by an image analyzer using 10 fields in the optical micrographs of

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**Table 3** Properties of Mg₂Si.¹)

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Melting point (K)</td>
<td>1358</td>
</tr>
<tr>
<td>Density (Mg/m³)</td>
<td>2.0</td>
</tr>
<tr>
<td>Elastic Modulus (GPa)</td>
<td>120</td>
</tr>
<tr>
<td>Hardness (HV)</td>
<td>460</td>
</tr>
</tbody>
</table>

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Fig. 2 SEM micrographs of preforms used to fabricate 18% fiber-reinforced composite (a) preform without particles and (b) preform with Si particles.

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Fig. 3 Microstructure of composites.
400 \times (280 \mu m \times 200 \mu m) was 12\%. In contrast, the volume fraction of the Mg_2Si particles in the composite calculated from the weight of the AZ91D alloy and Si particles in the preform was 12 vol\% when all of the Si particles react with the alloy melt. These results indicate that all of the Si particles in the preform reacted with Mg to form the Mg_2Si, and the Mg_2Si particles were dispersed in the matrix. The composite with 18 vol\% short alumina fibers and 12 vol\% Mg_2Si particles was termed “composite with fiber and Mg_2Si”.

Subsequently, Mg_2Si particles extracted from the composite by 30\% nitric acid were observed by scanning electron microscopy (SEM). The ferret diameter and the size distribution of the Mg_2Si particles were measured using 10 fields in the SEM micrographs of 1000 \times (110 \mu m \times 80 \mu m). Figure 4 shows the SEM micrograph and the size distribution of the Mg_2Si particles. In the Fig. 4, extracted fibers were also observed. Many Mg_2Si existed as independent particles, and the average size of the particles was 5 \mu m. The formation of the fine particles was probably due to the rapid infiltration and rapid solidification in the permanent mold.

### 3.2 Tensile properties

Figure 5 shows stress-strain curves of the AZ91D alloy and composites tested at room temperature (293 K) and 523 K. At 293 K, the tensile resistance to deformation increased as V_f increased (Fig. 5(a)). At 523 K, however, the composite with 18 vol\% fibers and 12 vol\% Mg_2Si (composite with fiber and Mg_2Si) showed the highest resistance as shown in Fig. 5(b).

![Fig. 4 SEM micrograph and size distribution of Mg_2Si particles extracted from composite.](image)

![Fig. 5 Stress-strain curves of unreinforced alloys and composites at (a) 293 K and (b) 523 K.](image)

![Fig. 6 Effect of temperature on Young’s modulus of unreinforced alloy and composites.](image)

![Fig. 7 Tensile strength of the AZ91D alloy and composites.](image)

Figure 6 shows Young’s modulus of the AZ91D alloy and composites at various temperatures. Young’s modulus increased as V_f increased at every measured temperature. Young’s modulus decreased as the temperature increased, and this tendency was pronounced at the V_f of 33 vol\%. Young’s modulus of the composite with fibers and Mg_2Si was greater than that of the composite with 23 vol\% fibers at every temperature, and equivalent to that of the composite with 33 vol\% fibers at 523 K. Based on these results, the dispersion of the Mg_2Si particles having the Young’s modulus of 120 GPa was found to be effective for improving the Young’s modulus of the short alumina fiber-reinforced Mg alloy composite.

Figure 7 shows the tensile strength of the AZ91D alloy and composites. At every temperature, the tensile strength of the composites was higher than that of the AZ91D alloy. The
tensile strength increased as $V_f$ increased up to 423 K, above which the strength of the composite with 33 vol% fibers sharply decreased as the temperature increased. The tensile strength of the composite with 18 vol% fibers was approximately 160 MPa at 523 K. The strength of the composite with fibers and Mg$_2$Si was approximately 20 MPa higher than that of the composite with 18 vol% fibers at every temperature. At 523 K, the strength of the composite with fibers and Mg$_2$Si was 180 MPa, which was higher than that of the heat-resistant aluminum alloy (150 MPa). 6)

Figure 8 shows the effect of the reinforcement volume fraction on the tensile strength of the composites. At 523 K, the tensile strength of the composite increased as $V_f$ increased up to 18 vol%, above which it decreased. The tensile strength of the composite with fibers and Mg$_2$Si was higher than that with 18 vol% fibers, showing that the dispersion of the fine Mg$_2$Si particles improved the high-temperature strength of the composite.

Based on these results, it was found that the composite having a superior strength and rigidity at high temperature can be obtained by properly adjusting the $V_f$ and dispersing the fine Mg$_2$Si particles.

4. Discussion

SEM micrographs of the fracture surfaces of the composites after tensile test at 293 K and 523 K are shown in Fig. 9. At 293 K, many fibers were fractured without pullout
on the fracture surface in all the composites, suggesting that the interfacial bond between the fibers and matrix was strong. Some bunches of fibers were observed in the fiber-reinforced composites (circles in Figs. 9(a) and (b)). In contrast, few bunches of fibers were observed on the fracture surface of the composite with fibers and Mg₂Si (Fig. 9(c)). At 523 K, the fracture surfaces of the composite with 18 vol% fibers and that with Mg₂Si was similar to the surface at 293 K (Figs. 9(d) and (f)), while many bunches of fibers, a smooth fiber surface without adhesion of the matrix, and grooves left in the matrix were seen on the fracture surface of the composite with 33 vol% fibers (circles in Fig. 9(e)).

Based on these results, the effect of the fibers and Mg₂Si particles on Young’s modulus and the tensile strength are discussed. Few bunches of fibers were observed on the fracture surface of the composite with fibers and Mg₂Si at every temperature. Some reports[7,8] indicated that introducing the fine ceramic particles into the matrix of the continuous fiber-reinforced aluminum alloy composite improved the strength of the composite because the stress transmission between the fiber and matrix became easy due to the particles, which reduced the fiber-to-fiber contact and resultant stress concentration. In the present study, as well as for the continuous fiber-reinforced composite, the stress transmission between the fiber and the matrix would become easy due to the Mg₂Si particles, which reduced the fiber-to-fiber contact. This would be why the tensile strength of the composite with fibers and Mg₂Si was higher than that of the composite without Mg₂Si. In contrast, many bunches, and a smooth surface of the fiber and grooves left in the matrix were seen on the fracture surface of the 33 vol% fiber composite at high temperature. This suggests that the fracture mainly occurred at the fiber-matrix interfaces, as reported by Akbulut et al.[9] and Shinkawa et al.[10]. An increase in Vf would result in an increase in the fiber touching. The fracture surface of the 33 vol% fiber composite reveals that little stress was transferred to the fibers, leading to the result that the fracture was initiated at the fiber touching points and then propagated along the interface between the fibers and matrix. In this case, the strengthening effect is difficult to obtain.

Subsequently, to consider Young’s modulus of the composite obtained in the present study, the experimental values were compared with the theoretical values calculated using the model, which was previously proposed. Akbulut et al.[11] calculated Young’s modulus of the short fiber-reinforced composites (Eₗ) using the following eq. (2), which was originally introduced by Nielsen and Chen.

\[
E_c = \frac{3}{8}E_L + \frac{5}{8}E_T
\]

where E_L is Young’s modulus parallel to the fibers and E_T is Young’s modulus perpendicular to the fibers. E_L and E_T are obtained from the following eq. (3) and (4).

\[
E_L = E_f V_f + E_m(1 - V_f)
\]

\[
E_T = \frac{E_f E_m}{E_f(1 - V_f) + V_f E_m}
\]

where E_f is Young’s modulus of the fiber (300 GPa in the present study). E_m is Young’s modulus of the matrix. The experimental value of the AZ91D alloy was used for E_m of the composite without Mg₂Si. Since the 12 vol% Mg₂Si particles were dispersed in the composite with the fibers and Mg₂Si, Young’s modulus of the Mg₂Si particle-dispersed AZ91D alloy must be obtained to use these equations. Yi et al.[12] introduced the following eq. (5) to estimate Young’s modulus of the in situ TiB₂ particle-dispersed aluminum alloy composite, and reported that the estimated values are in good agreement with the experimental values.

\[
E_{cp} = \frac{E_m[(E_p - E_m)(V_p/k)^{2/3} + E_m]}{(E_p - E_m)(V_p/k)^{2/3} - (E_p - E_m)V_p + E_m}
\]

where E_{cp} is Young’s modulus of the particle-dispersed composite. E_p is Young’s modulus of the particle, V_p is the particle volume fraction, and k is a coefficient to characterize the effect of the shape of the particles. In the present study, E_p was 120 GPa[5] and k was set to 1; the particle is assumed to be spherical. Young’s modulus of the Mg₂Si particle-dispersed AZ91D alloy calculated by eq. (5) was substituted into eqs. (3) and (4) to obtain E_L and E_T (E_{cp} is equal to E_m in this case), and then the E_L and E_T values were substituted into eq. (2) to obtain Young’s modulus of the composite with the fibers and Mg₂Si.

Figure 10 shows a comparison between the experimental and calculated Young’s modulus results of the composites. At 293 K, the experimental values of every composite are in good agreement with the calculated values. At 523 K, the
experimental value of the composite with 33 vol% fibers was smaller than the calculated value, indicating that the stress transmission from the matrix to the fiber was not enough because the fracture at the fiber-matrix interfaces occurred by elastic deformation at high temperature. The experimental values of the other composites were in good agreement with the calculated values; Young’s modulus of the composite can be improved by dispersing the Mg$_2$Si particles under the good stress transmission between the fiber and the matrix.

5. Conclusions

A short alumina fiber- and in situ Mg$_2$Si particle reinforced Mg alloy composite was fabricated by squeeze casting, and its Young’s modulus and tensile strength in the range from room temperature (293 K) to high temperature (523 K) were investigated. The following results were obtained.

1) By squeeze casting in a permanent mold, the Mg$_2$Si particles with the grain size of approximately 5 µm were formed and dispersed in the matrix of the composite.

2) Young’s modulus was improved by reinforcing with the fibers at every measured temperature, but the increase in $\nu_f$ decreased the improvement effect in Young’s modulus at high temperature. When the volume fraction is high, the stress transmission from the matrix to the fiber would not be sufficient because the fracture at the fiber-matrix interfaces occurred due to the elastic deformation at high temperature. Young’s modulus of the composite with fibers and Mg$_2$Si was greater than that of the composite with 23 vol% fibers at every temperature, and equivalent to that of the composite with 33 vol% fibers at 523 K. Therefore, it can be concluded that the dispersion of the Mg$_2$Si was effective for improving Young’s modulus of the fiber-reinforced composite.

3) The tensile strength increased as $\nu_f$ increased up to 423 K. Above 423 K, the tensile strength of the composite increased as $\nu_f$ increased up to 18 vol%, above which it decreased. The dispersion of the fine Mg$_2$Si particles improved the high-temperature strength of the composite. This would be due to the decrease in the fiber-to-fiber contact by dispersing the Mg$_2$Si particles. The fiber distribution without the contact provides a good stress transmission between the fiber and the matrix.

REFERENCES