Influence of Interfacial Reaction on Wear Resistance of Aluminum Alloy/\textit{SiC} Composites Fabricated by Low Pressure Infiltration Process

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Hybrid-MMCs have been fabricated by a low pressure infiltration method, using aluminum alloy as matrix, SiC fiber and SiC particle as reinforcements. Influences of orientation of fibers, interfacial reaction and matrix property on the wear resistance of Hybrid-MMCs have been investigated. The 3D-distribution of SiC fibers in the Hybrid-MMC are effective to prevent SiC particles from drooping out during the wear test. Thin reaction products at the interface between matrix and SiC fiber or SiC particle strengthen the interface bond, which results in an increase in the wear resistance of the Hybrid-MMC. The wear resistance can be improved by the formation of interfacial reaction layer and an age-hardening of the matrix. Volume expansion of the matrix accompanied by the age-hardening of the matrix increases the interface bond strength. The strengthened interface bond protects the reinforcements from dropping out and results in a superior wear resistance of the MMC.

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1. Introduction

In recent years, application of Al-base Metal Matrix Composite (MMC) has been expanded, because the MMC can improve fuel efficiency and performance of the automobiles by its superior properties, e.g., light weight and high strength. As for the fabrication method of the MMC, the casting process has advantages in formability and productivity and has been extensively studied. Among many kinds of casting processes, pressure infiltration process$^{1-2}$ is widely used for the fabrication of MMC containing a high percentage of reinforcement. In the conventional pressure infiltration method, the alloy melt is forced to infiltrate into the preform of the reinforcement with high pressure ($\approx 100$ MPa$^3$) and brittle fibers tend to be broken into short fibers by the high pressure, which reduces an efficiency of the reinforcements in strengthening the matrix. In our previous works$^{4-6}$ we developed a low pressure infiltration (LPI) process by which MMC can be produced at much lower pressure (0.2$-0.3$ MPa) as compared with the conventional high pressure infiltration method. In the LPI process, metal particles (Al particles) are added into the preform as an infiltration promoter to lower the critical infiltration pressure to attain a perfect infiltration$^{4-7}$.

As mentioned above, the Al-base MMC is being used for automotive parts, e.g. brake and cylinder block. In these applications, the MMC is required to have high wear resistance. It is well known that the wear characteristic of the MMC is affected by the size and distribution of reinforcements, in particular, the orientation of fibers in MMC. Rahimian et al. reported that the wear of Al/Al$_2$O$_3$ composite reduces with increasing the reinforcement particle size from 3 $\mu$m to 48 $\mu$m$^9$. On the other hand, inter-particle distance reduces with an increase in particle size, which caused an increase in the wear of MMCs. It is, therefore, suggested that an optimal particle size is several tens $\mu$m. The importance of the orientation of fibers on the wearing surface has been pointed out$^{9-12}$. For example, Fukunaga and Sakai reported that the wear rate decreases in the order transverse (highest), longitudinal, normal and random (lowest).$^9$ It has been also found in our recent work$^{13}$ that alumina fibers with two-dimensional (2D) distribution are easily scratched off from the wear surface by abrasion, but fibers having three-dimensional (3D) distribution in alumina fiber/particle reinforced Hybrid-MMC prevent the reinforcements from dropping out during the wear test and the Hybrid-MMC exhibits a superior wear resistance.

Besides the size and distribution of reinforcements, the interfacial bond strength between the matrix and reinforcements and the hardness of matrix should affect the wear resistance of the MMC in which the interfacial reaction and age hardening of matrix occur. In the present study, SiC fiber and particle have been employed as the reinforcements of the Al-base MMC and Super duralumin has been employed as the matrix in addition to Al-Cu, Al-Mg and Al-Si alloys. Since the hardness of SiC is higher than that of alumina, the MMC reinforced with SiC is expected to have superior wear resistance. However, SiC will react easily with Al alloy melt at high temperature to form a reaction layer at the interface. Hence, we have investigated the influence of the interfacial reaction between the matrix and reinforcements and the age hardening of matrix on the wear resistance of the SiC fiber/particle reinforced Al-base MMC.

2. Experimental Procedure

2.1 Fabrication of MMC

SiC short fibers, 0.5 mm in length, 14 $\mu$m in diameter and SiC particles (30$-50$ $\mu$m) were used as reinforcements. Aluminum particles ($\approx 20$ $\mu$m) were added as a promoter of infiltration. SiC and Aluminum particles were mixed in a planetary ball mill for 6h. Then, these particles and polyethylene glycol (PEG) 2 g were mixed with the SiC fibers in ethanol. The slurry was poured into a quartz tube and pressed into a cylindrical tube with 10 mm in height and 15 mm in diameter. After holding at room temperature for one
day, the organic binder (PEG) was burned up by heating at 773 K for 1 h in air to obtain a preform.

From the pressure balance at an advancing melt front, infiltration pressure required to overcome the pressure opposing infiltration is given by the sum of a capillary pressure and a pressure drop caused by a friction between alloy melt and reinforcement. In this work, metal particles as infiltration promoter are dissolved by a heat from an alloy melt and act as a void in the infiltration process, which results in a reduction of infiltration pressure. On the basis of the calculation of critical infiltration pressure where the replacement fraction of voids with aluminum particles is taken into consideration, the infiltration pressure of 0.3 MPa (the pressure is different from the ambient pressure; 0.2 MPa) was used in this work.

As shown in Fig. 1, coarse alumina beads (about 1 mm in diameter) and the preform were put in a quartz tube (inner diameter of 15 mm) with a small hole at the bottom to leak the air inside the tube. The inner surface of quartz tube was coated with boron nitride for protecting a reaction between the quartz tube and the alloy melt. Then, the matrix alloy was placed on the preform. In the present work, Al-4 mass% Cu, Al-4 mass% Mg, Al-12 mass% Si alloys and Super duralumin (Al-4.5 mass% Cu-1.5 mass% Mg, 2024) were employed as a matrix. After melting each matrix alloy at 1173 K by high frequency induction heating, the alloy melt was infiltrated into the preform by applying an argon gas pressure of 0.3 MPa on the melt surface. Pressing was stopped when a small amount of the alloy melt flowed out through the nozzle.

Super duralumin-base MMCs were subjected to an aging treatment at 473 K for 12 h.

2.2 Microstructure observation and Vickers hardness measurement

Microstructures of the MMC samples were observed on the perpendicular planes and parallel ones to the infiltration direction using optical microscope and scanning electron microscope (SEM). The interfacial reaction was observed using Transmission Electron Microscope (TEM). Vickers hardness of the matrix was measured using AKASHI MVK-H1 hardness tester with 9.8 N load and given as an average of more than ten indentations.

2.3 Wear test

Wear tests were performed against No. 240 SiC (particle size ~ 100 µm) abrasive paper at room temperature under various loads of 19.6 N and 29.4 N, wear speed of 1.6 m·s⁻¹ and dry condition. The pin-on-belt type wear tester using a rotating belt-shape abrasive paper was employed. Weight loss was measured after every 100 m run and an enough rest time was taken between the wear tests to suppress the temperature increase. The reproducibility of wear rate (wear loss vs. wear distance) was examined. The experimental error was within 5%. The wear surfaces of the samples were examined using SEM.

3. Results and Discussion

3.1 Orientation of fibers

At first, the orientation of fibers in MMCs was investigated. Figure 2 shows the microstructures on the perpendicular and parallel plane to the infiltration direction in the SiC fiber-reinforced Al-Mg base MMC (FRMMC) which includes 20 vol% SiC fiber and the Al-Mg base Hybrid-MMC which includes 12.5 vol% SiC fiber and 7.5 vol% SiC particle. Fibers in the FRMMC are needle-shaped at the perpendicular plane and small globular-shape at the parallel plane, indicating that the fibers have 2D-distribution in the FRMMC. On the other hand, the fibers in the Hybrid-MMC are needle-shaped or globular-shaped at both perpendicular and parallel planes and the particles are tetragonal-like shaped. Thus, the fibers are oriented randomly (3D-distribution) in the Hybrid-MMC. In order to verify the 2D-distribution of the fibers in the FRMMC and the 3D-distribution of the fibers in the Hybrid-MMC, the inclined angle β of fiber axis from infiltration direction was measured.
for about 600 fibers in the FRMMC and the Hybrid-MMC. In Fig. 3, the distribution of $\beta$ in the FRMMC shows a peak in the range of $\beta = 80^\circ$~$90^\circ$, indicating that the fibers are oriented two dimensionally on the plane perpendicular to the infiltration direction. On the other hand, the orientation of fibers shows a broad peak at $\beta = 30^\circ$~$80^\circ$ in the Hybrid-MMC, indicating that fibers in the Hybrid-MMC have a random 3D-distribution. Wear loss of the Al-Mg alloy matrix FRMMC and the Hybrid-MMC under an applied load of 19.6 N is shown in Fig. 4. The wear loss of the Hybrid-MMC is smaller than that of FRMMC. As discussed in our previous work, the reason why the Hybrid-MMC exhibits a smaller wear loss is that SiC fibers having 3D-distribution are effective to prevent the SiC particles from dropping out during the wear test.

3.2 Influence of interfacial reaction on wear resistance

Microstructures on the perpendicular plane to the infiltration direction in as-cast Hybrid-MMCs with Al-Mg and Al-Si alloy matrices are shown in Fig. 5. It can be seen in the figure that the fibers exhibit 3D-distribution because the shapes of fibers is similar to those in Fig. 2. Wear losses of the MMCs with Al-Cu, Al-Mg and Al-Si alloy matrices are shown in Fig. 6. As seen in the figure, the wear rates (wear loss vs. wear distance) of the MMCs are reduced for Al-Si matrix, Al-Mg matrix and Al-Cu matrix in order. On the other hand, Vickers hardness of Al-Cu, Al-Mg and Al-Si matrix alloys is 48, 57 and 67 (±5) HV0.1, respectively. Thus, the matrix hardness does not correspond to the wear rate. This is possibly attributed to an effect of interfacial reaction between fibers and matrix.

It has been reported that a reaction occurs between Al-Cu alloy melt and SiC as follows:\textsuperscript{5,17}

$$3\text{SiC} (S) + 4\text{Al} (L) \rightarrow \text{Al}_4\text{C}_3 (S) + 3\text{Si} (L) \quad (1)$$

In our previous work,\textsuperscript{5} we revealed an existence of the reaction product, $\text{Al}_4\text{C}_3$, which is formed surrounding the SiC particle, as shown in Fig. 7. The interfaces between SiC/Al$_4$C$_3$ and Al$_4$C$_3$/\alpha-Al matrix are smooth and no interface dislocation is seen, which suggests that the coherent interfaces and the reaction layer strengthen the interfacial bond.

In the Al-Mg/SiC system, Al-Mg alloy melt reacts with SiC particle as follows:\textsuperscript{18}

$$\text{SiC} (S) + 2\text{Mg} (L) \rightarrow \text{Mg}_2\text{Si} (S) + \text{C} (L) \quad (2)$$

Microstructure near the interface between Al-Mg matrix and SiC fiber is shown in Fig. 8. Non-smooth interface indicates a feature of interface reaction in Fig. 8(a). The TEM image of the interface and the selected diffraction pattern of the reaction phase are given in Fig. 8(b) and Fig. 8(c), respectively. The reaction phase was identified as $\text{Mg}_2\text{Si},$

![Fig. 3 Distribution of inclined angle $\beta$ of SiC fibers to the infiltration direction in the Al-Mg alloy matrix FRMMC and Hybrid-MMC.](image)

![Fig. 4 Wear loss of Al-Mg alloy matrix, FRMMC and Hybrid MMC.](image)

![Fig. 5 Microstructures of Hybrid-MMCs with (a) Al-Mg and (b) Al-Si alloy matrices.](image)
which is in good agreement with the reaction product in the eq. (2). The interface products in Fig. 7 and 8 enhance the interface bond strength which results in smaller wear rates in Fig. 6. On the other hand, no traces of interfacial reaction is seen in the Al-Si/SiC system as shown in Fig. 9, where the SiC fiber has a smooth surface as in the as-received condition. It is, therefore, concluded from the observation of the interfaces in the three Hybrid-MMCs that a thin reaction product at the interface between matrix and SiC fiber/SiC particle strengthens the interfacial bond.

3.3 Influence of age-hardening of matrix on wear resistance

Fibers on the wear surface are broken and fallen out by scratching with SiC abrasive paper during the wear test. Wang and Hutchings have reported the critical load \( P_c \) at which fiber fracture occurs in a MMC subjected to abrasive wear,\(^{19}\) given by

\[
P_c = \frac{1}{2}d^2(3\pi\sigma_f H_m)^{1/2}
\]

where \( d \) is diameter of the fiber, \( \sigma_f \) is the fracture strength of the fiber and \( H_m \) is the hardness of the matrix. As indicated from the eq. (3), the critical load \( P_c \) increases with an increase in the matrix hardness \( H_m \), i.e., the hardened matrix can protect fiber fracture. Age-hardening is a typical treatment to harden matrix, and is expected to raise the interfacial bond strength due to the volume expansion of the matrix.  

![Fig. 6 Wear loss of Al-Si MMC, Al-Mg MMC and Al-Cu MMC.](Image)

![Fig. 7 Microstructure near the interface between Al-Cu matrix and SiC fiber. (Reproduced from Ref. 5)).](Image)

![Fig. 8 Microstructures near the interface between Al-Mg matrix and SiC fiber. (a) SEM image, (b) bright field image, (c) diffraction pattern of the reaction product (Mg\textsubscript{2}Si).](Image)

![Fig. 9 Microstructures near the interface between Al-Si matrix and SiC fiber.](Image)
order to elucidate the influence of the age-hardening of matrix on the wear resistance, A2024 Super duralumin, which is a typical age-hardening alloy, was employed as matrix in this work. The Super duralumin-base MMCs were subjected to different thermal history on the infiltration process, i.e., (a) melting at 1073 K and rapid cooling to obtain a thin reaction layer and (b) melting at 1173 K and slow cooling to obtain a thick one. Figure 10 shows the microstructures of Al-4.5 mass% Cu-1.5 mass%Mg (A2024) MMC fabricated at different infiltration temperatures. The inserts show enlarged images near a SiC fiber. In the enlarged image of the rapid cooling specimen, θ phase (light color) is seen in contact with the SiC fiber, while, in the slow cooling specimen, a dark phase is seen which surrounds the SiC fiber. Figure 11 shows SEM images at higher magnifications. A reaction layer (R.L.) is clearly seen in both specimens. The R.L. consists of two phases, Mg2Si and Al4C3 and is in accord with the reaction products in the eqs. (1) and (2).

The wear loss of the MMCs with A2024 matrix is shown in Fig. 12. The wear rate of the rapid cooling specimen with a
thin reaction layer is smaller than that of the slow cooling specimen with a thick one, which indicates that a thinner reaction layer is preferable due to the intrinsic brittle nature of the reaction layer which consists of the brittle phases. In Fig. 13, a characteristic of dropped-out SiC fiber is seen in the slow cooling specimen while relatively smooth surface is seen in the rapid cooling specimen. The observation of the worn surfaces supports the result of wear curves in Fig. 12.

Here the influence of age-hardening of the matrix on the wear resistance is examined. The rapid cooling specimen was subjected to the peak-aged heat-treatment at 473 K for 12 h. The MMC with the peak-aged matrix shows a wear rate smaller than the wear rate before the peak-aged heat-treatment. Lee et al. reported that SiC particle reinforced MMC with the peak-aged A2014 (so-called Duralumin) matrix exhibits the best abrasive wear resistance. They explained that a compressive stress is generated on the SiC particles due to the volume expansion of age-hardened matrix and the low wear rate is attributable to the compressive stress acting on the particles. In the present work, a similar compressive stress may be generated and enhances the interfacial bond strength, which results in a superior wear resistance in the MMC with the peak-aged matrix. It is, therefore, concluded that the wear resistance can be improved by age-hardening of the matrix and both the effects of interfacial reaction layer and age-hardening of the matrix increase the interfacial bond strength and the high interfacial bond strength protects the reinforcements from dropping out.

4. Conclusion

Influences of the orientation of fibers, interfacial reaction and matrix property on the wear resistance of Hybrid-MMCs have been investigated. The results obtained are summarized as follows.

1. 3D-distribution of fibers in the Hybrid-MMC is effective to prevent SiC particles from dropping out during the wear test.
2. A thin reaction product at the interface between matrix and SiC fiber or SiC particle strengthens the interfacial bond.
3. Wear resistance can be improved further by an age-hardening of the matrix. Both the effects of interfacial reaction layer and age-hardening of the matrix increase the interfacial bond strength and the high interfacial bond strength protects the reinforcements from dropping out.

REFERENCES