

# Aging and wear resistant characterization of Al-5Mg-X(Si, Cu, Ti)/SiC<sub>p</sub> composites produced by pressureless infiltration method

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The effects of SiC particle size and alloy elements such as Si, Cu and Ti on the response to aging treatment and wear resistance in Al-5Mg-X(Si,Cu,Ti)/SiC<sub>p</sub> composites fabricated by pressureless infiltration method have been investigated by hardness tester, scanning electron microscope (SEM), X-ray diffractometer (XRD), differential scanning calorimetry (DSC) and wear tester. The Al-5Mg-0.3Si-0.1Cu-0.1Ti/SiC<sub>p</sub> composites had better wear resistance property among Al-5Mg-X(Si,Cu)/SiC<sub>p</sub> composites. The wear resistance property of all the composites was enhanced after aging at 170°C for 8 hrs due to precipitates of β'(Mg<sub>2</sub>Si) phase. The wear resistance property of the composite as-fabricated with 50 μm size of SiC particle is superior to that of the composite as-fabricated with 100 μm SiC size of particle. In Al-5%Mg alloy aged at 170°C for 8 hrs, the frictional seizure appeared more than abrading speed of 1.90 m/s, but in Al-5Mg-(Si,Cu,Ti)/SiC<sub>p</sub> composites aged at 170°C for 8 hrs, the frictional seizure was not found at abrading speed ranging from 0.5 m/s to 4.3 m/s.

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## 1. Introduction

In recent year, much attentions have been paid into developing an effective fabrication process to make metal matrix composites (MMCs). MMCs that have a homogeneous distribution of ceramics and a volume of ceramic fractions are made by the pressureless infiltration fabrication process [1–4]. Therefore, it can be expected that these composites will be applied for many advanced applications, including aerospace, automobile parts and electronic packaging etc. [5]. However, despite their potential advantage, the processing difficulties and economic constraints have been limiting commercialization of MMCs. So far, considerable efforts have been expended to solve those problems mentioned in many papers [1–3]. It is well known that aging treatment is an effective method to increase the strength and wear resistant properties in aluminum alloys. But few studies have investigated the effect of aging treatment on the resultant microstructure. It is considered that Si and Cu elements are effective elements for aging in Al-Mg alloy, while Ti forms TiC particle by an *in situ* reaction between SiC<sub>p</sub> and Ti in Al-Ti melt. This paper reports a study of the wear resistance property of MMCs after aging treatment of Al-Mg/SiC<sub>p</sub> composites containing Si, Cu and Ti.

## 2. Experimental procedure

In this work, SiC particle of 50 to 100 μm, high purity Al particle and Al-Mg alloys were used to make

specimens. Mixed materials with SiC<sub>p</sub> and aluminum particles were placed into an alumina vessel and a designated Al alloy was placed on the SiC<sub>p</sub> bed. The aluminum vessel was heated to 950°C under the nitrogen gas at 10 cc/min flow rate, and held at the soaking temperature for 5 hrs, and then allowed to cool to 550°C in furnace in order to prevent the oxidation of composites. And samples were taken out from the furnace and immediately cooled to the ambient temperature. Table I shows the chemical composition of alloy and occupied area percentage (%) of SiC particles in composite. Microstructure was observed by OM and SEM. The phases formed within melt during soaking at 950°C were analyzed by the EDX and XRD. Hardness was measured by the micro-Vickers tester, using 1 kg load for matrix and the Rockwell hardness tester, using 100 kg load for composite. The wear resistance was measured by the OAT-U type disc wear tester under the abrasive conditions (dry abrasive conditions; load 12.6 kg, length 400 m, abrasive speed 0.59, 1.09, 1.90 and 4.30 m/s).

All composites were homogenized at 560°C for 24 hrs, and then were solution heat treated at 550°C for 1 hr, subsequently quenched into ice water. But specimens of Al-5%Mg alloy were homogenized at 480°C for 24 hrs, and were solution heat treated at 450°C in salt bath for 0.5 hrs and then quenched into ice water. The composite and Al-5Mg alloy were then aged at 170°C for various aging times. DuPont 910 calorimeter was carried out in order to analyze the response to

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TABLE I Chemical compositions of composites (wt%) and occupied area percentage of SiC particles

| Alloy No.                      | Alloy design   | Area percentage of SiC particle (%) |
|--------------------------------|--|-------------------------------------|
| R0                             | Al-5Mg alloy   |                                     |
| SiC particle 100 $\mu\text{m}$ |  |                                     |
| A0                             | Al-5Mg/SiCp (100 $\mu\text{m}$ ) composite             | 51.8                                |
| A1                             | Al-5Mg-0.3Si/SiCp (100 $\mu\text{m}$ ) composite       | 50.5                                |
| A2                             | Al-5Mg-0.3Si-0.1Cu/SiCp (100 $\mu\text{m}$ ) composite | 51.5                                |
| A3                             | Al-5Mg-0.3Si-0.1Cu-0.1Ti/SiCp (100 $\mu\text{m}$ )     | 51.5                                |
| SiC particle 50 $\mu\text{m}$  |  |                                     |
| B0                             | Al-5Mg/SiCp (50 $\mu\text{m}$ ) composite              | 58.8                                |
| B1                             | Al-5Mg-0.3Si/SiCp (50 $\mu\text{m}$ ) composite        | 55.9                                |
| B2                             | Al-5Mg-0.3Si-0.1Cu/SiCp (50 $\mu\text{m}$ ) composite  | 56.8                                |
| B3                             | Al-5Mg-0.3Si-0.1Cu-0.1Ti/SiCp (50 $\mu\text{m}$ )      | 57.9                                |

aging treatment of specimens. The samples were first heated to 500°C at the heating rate of 10°C/min.

### 3. Results and discussion

#### 3.1. Microstructural observation

Infiltrating depth into SiCp bed entirely depends on the Mg content in aluminum alloys and a flow rate of nitrogen gas. Therefore, in this study, Al-5%Mg alloy (R0) was used as the reference alloy and Al-5%Mg-X alloy melt penetrated into SiCp bed under the atmosphere of nitrogen gas. This result was shown in Fig. 1.

Fig. 1 shows the scanning electron micrographs of composites (A3, B3). All Al-5Mg-X (Si,Cu,Ti) alloys melt penetrated successfully the SiCp bed in the case of either single and complex addition of Si, Cu, and Ti element. The occupied area percentage (%) of SiCp in composite reinforced with 50  $\mu\text{m}$  size of SiC particle differs from the others reinforced with 100  $\mu\text{m}$  size of SiC particle (Table I). The area percentage of SiC particle of composites reinforced with 50  $\mu\text{m}$  size of SiC particle is larger than that of composites reinforced with 100  $\mu\text{m}$  size of SiC particle. Because the distribution of SiC particle in composite reinforced with 50  $\mu\text{m}$  size of

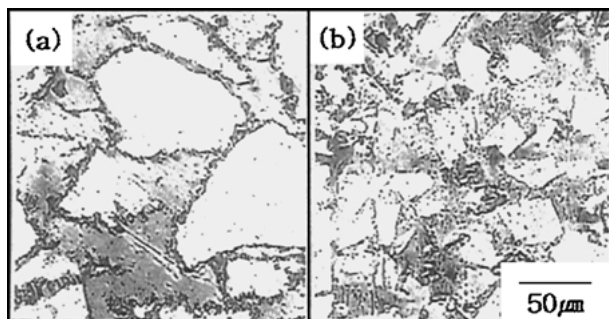


Figure 1 SEM micrographs of Al-5Mg-0.3Si-0.1Cu-0.1Ti/SiCp composites. (a) 100  $\mu\text{m}$  (A3), (b) 50  $\mu\text{m}$  (B3).

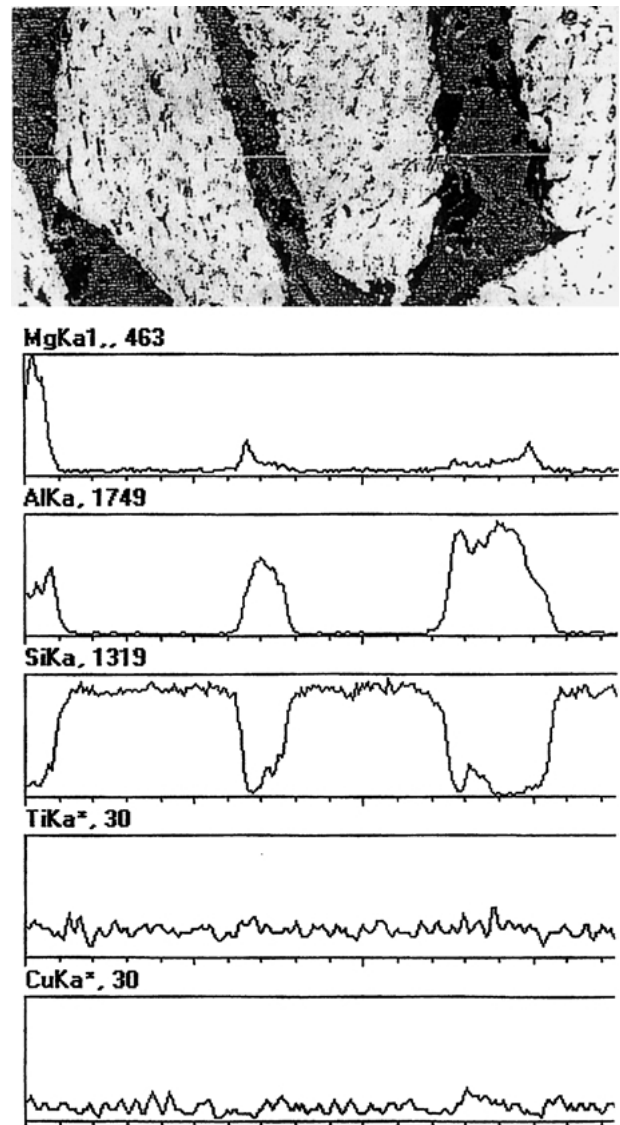


Figure 2 Line analysis by EDX for Al-5Mg-0.3Si-0.1Cu-0.1Ti/SiCp composites.

SiC particle is denser than that of composite reinforced with 100  $\mu\text{m}$  size of SiC particle. The area percentage of SiCp in Al-5Mg/SiCp composite was changed little despite Si, Cu, and Ti addition. The matrix of A2 and A3 specimens appears the interaction zone with grayish color because of the interfacial interactions between SiC particle and aluminum alloy matrix. EDX was carried out in order to analyze interfacial reaction phases. From Fig. 2, Mg was segregated at the interface between SiC particle and matrix in composite A3. This interfacial phase is known as Mg spinel,  $\text{MgAl}_2\text{O}_4$  [7].  $\text{Mg}_3\text{N}_2$  formed by the reaction of Mg with  $\text{N}_2$  gas enhanced the infiltration rate of Al melt into the SiCp bed due to the improvement of the wettability between Al melt and SiCp [4] by reducing the interfacial energy [3].

Fig. 3 shows X-ray maps showing image of Mg, Si, Ti, Cu, and Al element. Mg rich phases appeared at the periphery of SiC particles in composite (A3). These Mg rich phases might be  $\text{Mg}_2\text{Si}$  and  $\text{Al}_4\text{C}_3$  phases formed during infiltration of Al melt into the SiCp bed.  $\text{Al}_4\text{C}_3$  is formed during infiltration of Al melt into SiCp bed, but  $\text{Mg}_2\text{Si}$  is not found in Al-5%Mg/SiCp composite (A1) as shown in Fig. 4.  $\text{Al}_4\text{C}_3$  formed by reaction of

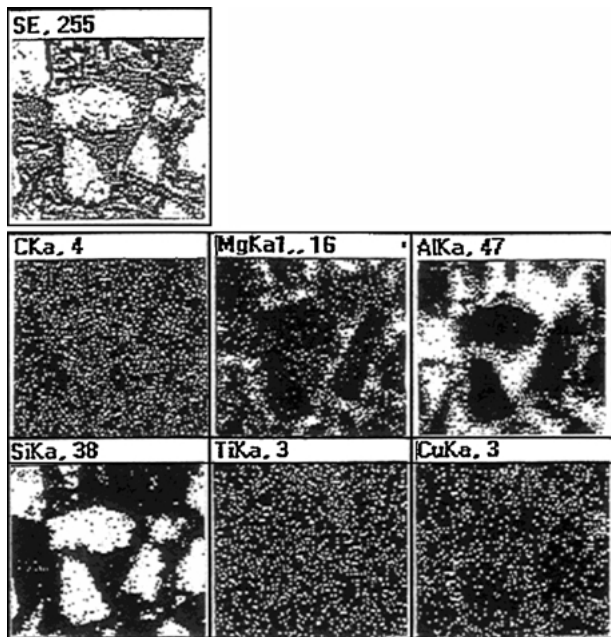


Figure 3 EDX images of Al-5Mg-0.3Si-0.1Cu-0.1Ti/SiCp composites.

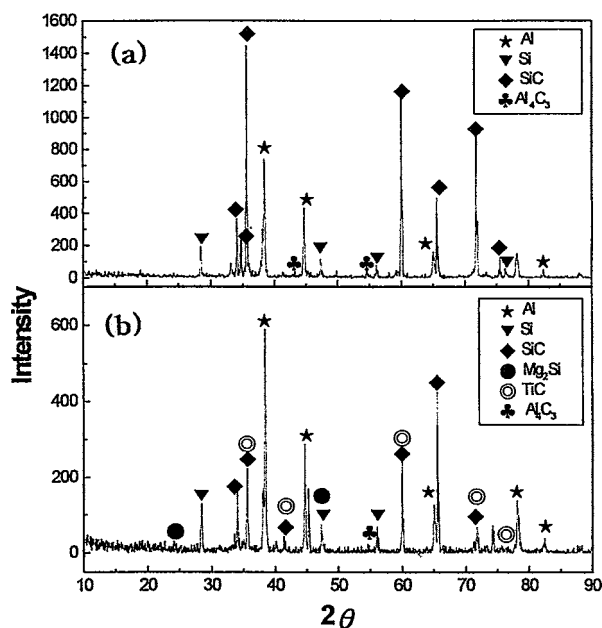


Figure 4 XRD patterns of composites. (a) Al-5Mg/SiCp (A1), (b) Al-5Mg-0.3Si-0.1Cu-0.1Ti/SiCp (A3).

SiCp and Al melt at high temperature deteriorates the corrosion resistance property and mechanical properties such as elongation, strength and toughness of Al-5%Mg/SiCp composite (A1). Therefore, it is necessary to prohibit the formation of  $Al_4C_3$  by adding a lot of Si in Al-Mg alloy [3]. But the composite (A3) containing Si formed  $Mg_2Si$ , while infiltrating Al melt into the SiCp bed.  $Al_4C_3$  is little found in the composite (A3) because of small amount of Si addition in composite. TiC particle formed by the *in situ* reaction; such as  $SiC + Ti \rightarrow TiC + Si$  in Al melt, are shown in the composite containing Ti (A3). But TiC phases were not found by EDX due to lower Ti content.

### 3.2. Age hardening behavior

Table II shows the hardness variation of the as-fabricated Al-5Mg-X(Si,Cu,Ti)/SiCp composites and

TABLE II Hardness of Al-5Mg alloy (R0) and as-fabricated composites

| Alloy No.           | R0 | A0 | A1 | A2 | A3 | B0  | B1  | B2  | B3  |
|---------------------|----|----|----|----|----|-----|-----|-----|-----|
| Hardness ( $H_RF$ ) | 21 | 91 | 91 | 92 | 93 | 101 | 102 | 102 | 103 |

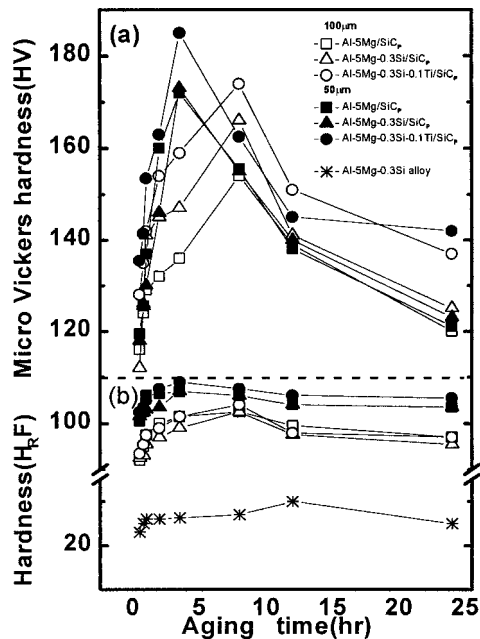


Figure 5 Hardness variation of overall composites (a), matrix of composites and Al-Mg-Si alloy (b).

Al-5Mg (R0) alloy. Hardness of all composites reinforced with  $50 \mu m$  size of SiC particle is higher than that of all composites reinforced with  $100 \mu m$  size of SiC particle. This is because the area fraction and grain size in the composites reinforced with  $50 \mu m$  size of SiC particle are higher and smaller than those in the composites reinforced with  $100 \mu m$  size of SiC particle, respectively. Hardness of Ti added composites A3 and B3 is higher than other composites due to the existence of TiC particles. From Fig. 4, TiC particle having higher hardness than SiC particle was formed by the *in situ* reaction between SiCp and Ti of Al-Mg-Si-Cu-Ti alloy melt.

Fig. 5 shows the hardness variation of matrix and overall region of the composites and un-reinforced Al-Mg-Si alloy aged at  $170^\circ C$  for 8 hrs. Hardness of the composites has significantly increased more than that of un-reinforced Al-5Mg-Si alloy. And response to aging treatment of all the composites was more accelerated than un-reinforced Al-Mg-Si alloy. This is because the grain refinement and dislocation generated in composites had acted as the heterogeneous nucleation site [8]. The grain size of the composites was refined due to the dispersion of SiC particles, and the dislocations were generated at Al and SiCp interface [8]. The hardness of the composites reinforced with  $50 \mu m$  size of SiC particles (B0–B3) is higher than that of the composites reinforced with  $100 \mu m$  size of SiC particle (A0–A3) as shown in Table II. And the age hardening response of composites B0–B3 was higher than that of composites A0–A3 because of the finer grain boundary and high density of dislocation. The hardness curves of all the

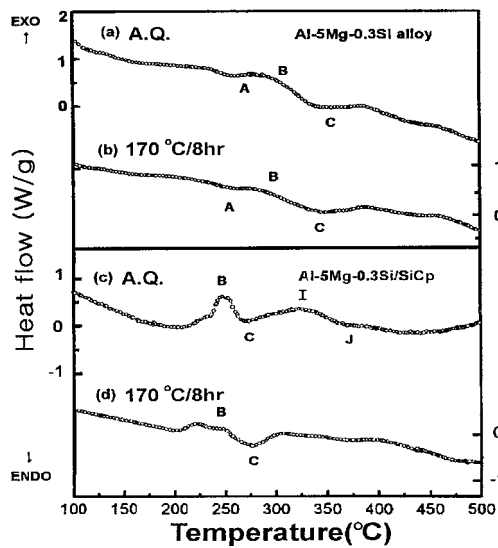


Figure 6 DSC thermograms of Al-5Mg-0.3Si alloy (a), (b) and Al-5Mg-0.3Si/SiCp composites (c), (d) as-quenched and aged at 170°C for 8 hrs.

composites aged at 170°C were almost the same tendency in matrix of composites, but the overall hardness of composites increased more than that of matrix of composites.

Fig. 6 shows the DSC thermograms of as-quenched and aged specimens. From Fig. 6a, the age hardening response of Al-5Mg-0.3Si alloy is not prominent at 170°C. But, Al-5Mg-0.3Si/SiC composites show obviously the aging response in this DSC thermogram of (c). The reaction,  $3\text{SiC} + 4\text{Al} \rightarrow 3\text{Si} + \text{Al}_4\text{C}_3$ , occurs in case of low Si content [4]. Therefore, it is considered that in case of high Si content in matrix, Si and Mg in the matrix make a role to precipitate  $\beta'$  ( $\text{Mg}_2\text{Si}$ ) phase. In Fig. 6c, there are two exothermic peaks (B and I) and one endothermic peak (C), is shown in as-quenched composite (A1). The peaks B and I represent the precipitation of meta-stable  $\beta'$  ( $\text{Mg}_2\text{Si}$ ), and stable  $\beta$  phase respectively [9]. The peak C represents the dissolution of  $\beta'$  phase. In A1 composite, the peak B decreased remarkably after aging at 170°C for 8 hrs. This means that plenty of precipitates of  $\beta'$  in A1 composite were formed after aging treatment at 170°C for 8 hrs. The DSC thermograms of other composites were similar to those of the Al-5Mg-0.3Si/SiCp composite. The Al-5Mg-X(Si,Cu,Ti)/SiCp composites showed the response to aging treatment, but Al-5Mg-Si alloy showed little. The increase in dislocation density of composites is responsible to improvement of the aging response [8].

### 3.3. Wear resistance characterization

Fig. 7 shows the change of wear amount in the as-fabricated and the aged composites. Wear resistance property of the aged composite is superior to that of the as-fabricated composite because of the precipitates of  $\beta'$  phase. At very lower abrading speed (0.5 m/s), the breakage amount of SiC particle is lower, and SiC particle in matrix effectively prevents from contacting matrix and wearing counterpart in all composites [10]. Little or no particle fracture arises in this region; thereby it protects the matrix alloy against the wearing counterpart during sliding contact [11]. But the wearing amount of composite increased as abrading speed increased up

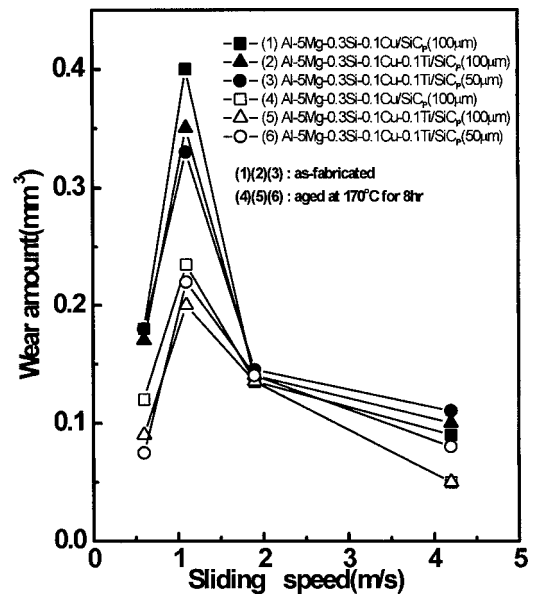


Figure 7 Comparison of wear amount between as-fabricated and aged composites.

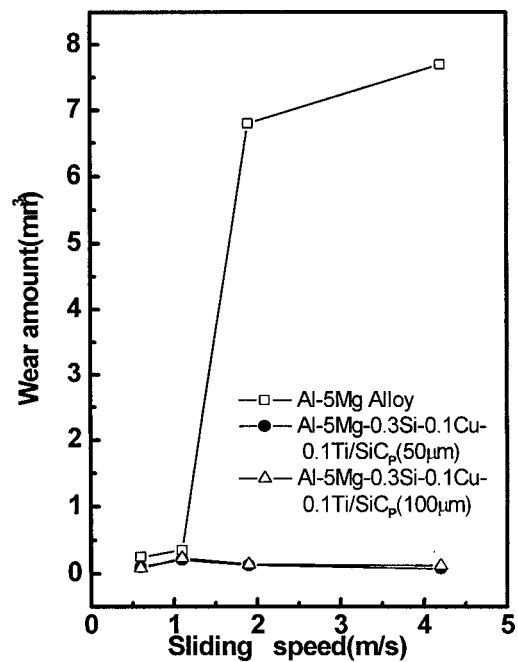


Figure 8 Comparison of wear amount between Al-Mg alloy and aged composites (A3, B3).

to 1.1 m/s. This is because of abrading phenomenon by breakage of SiC particles [12] and delamination of oxidation layer [11]. Fig. 8 shows the wearing amount of aged composites (A3 and B3) and Al-Mg alloy. Wear resistance property of composites is superior to that of the Al-Mg alloy because of the grain refinement, homogeneous dispersion of SiC particles and precipitates. In Al-Mg alloy, the frictional seizure [12] appeared at abrading speed more than 1.1 m/s due to the matrix fusion by the frictional heat. But the frictional seizure was not found in the composites (A3 and B3) aged at 170°C for 8 hrs. The wear amount increased to 1.1 m/s abrading speed in the composites (A3 and B3) as shown in Fig. 8. But at higher abrading speed (more than 2 m/s), the wear amount decreased as abrading speed increased. This is because there are the resistance of SiC

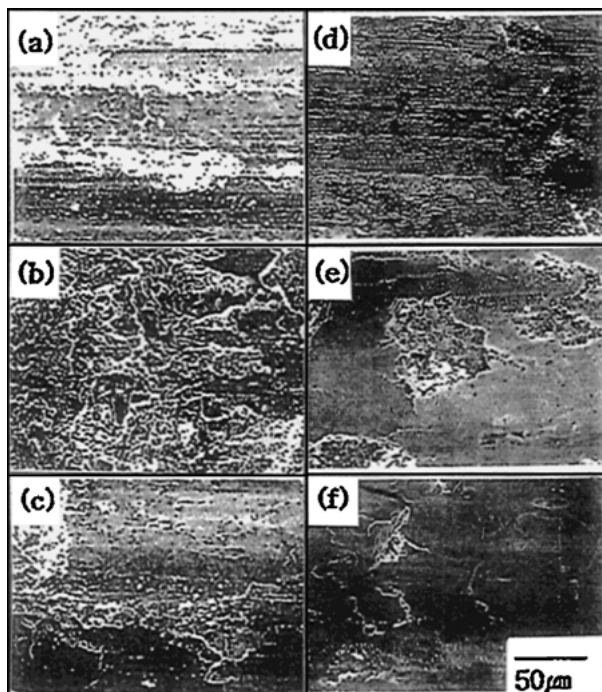


Figure 9 Worn surfaces of composites A3 as-fabricated (a, b, c) and aged (d, e, f). (a), (d) 0.6 m/s; (b), (e) 1.1 m/s; (c), (f) 1.9 m/s.

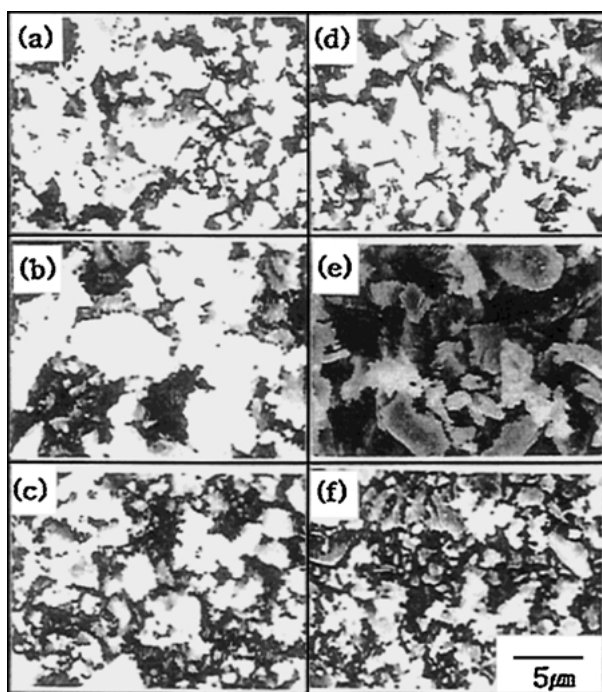


Figure 10 Worn debris of composites A3 as-fabricated (a, b, c), aged (d, e, f). (a), (d) 0.6 m/s; (b), (e) 1.1 m/s; (c), (f) 1.9 m/s.

particles against plastic flow of matrix, and fine debris and dispersion of SiC particles cumulated on abrading surface and obstructed aluminum matrix from frictional contacting with the counterpart. The composite aged at 170°C for 8 hrs is effective enough to prevent the frictional seizure due to hardness increase by the precipitation. Ti added composites (A3, B3) are more effective to prevent the wearing friction than other composites.

The worn surfaces and worn debris of composite (A3) were shown in Fig. 9 and 10 respectively. At abrading speed of 1.1 m/s, worn surface is rough and worn debris

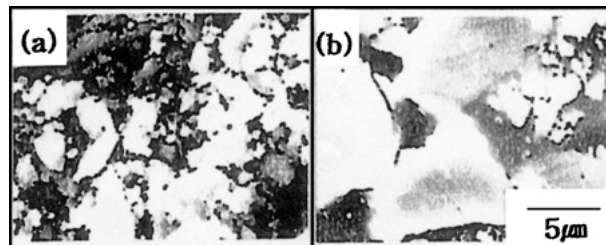


Figure 11 Worn debris of aged composite A3 (a) and Al-5Mg alloy (b) at abrading speed of 1.9 m/s.

is large. But at high abrading speed (1.9 m/s), worn surface is smooth and worn debris is small. Fig. 11 shows the worn debris of Al-5Mg alloy and composite (A3) at abrading speed of 1.9 m/s. The size of worn debris of Al-5Mg alloy is larger than that of composite (A3). These results agree well with the results of wear tests of all the composites (Fig. 7).

#### 4. Conclusions

In both cases of the as-fabricated and aged Al-5Mg-0.3Si-0.1Cu-0.1Ti/SiCp composites had better wear resistance among composites. The wear resistance of all composites was enhanced after the aging treatment at 170°C for 8 hrs due to the precipitation of  $\beta'$ (Mg<sub>2</sub>Si) phase. The wear resistance of the composite fabricated with 50  $\mu$ m of SiC particle is superior to that of the composite fabricated with 100  $\mu$ m of SiC particle. In the Al-5%Mg alloy aged at 170°C for 8 hrs, the frictional seizure appeared more than abrading speed of 1.90 m/s, but in Al-5Mg-(Si,Cu,Ti)/SiCp composites aged at 170°C for 8 hrs, the frictional seizure was not found at abrading speed from 0.5 m/s to 4.3 m/s.

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#### References

1. T. CHOY, M. KOBASHI, H. NAKATA and H. KANEDA, *Materials Sci. Forum* **217-222** (1996) 353.
2. M. K. AGHAJANIAN, M. A. ROCAZELLA, J. T. BURKE and S. D. KEČK, *J. Mater. Sci.* **26** (1991) 447.
3. K. B. LEE and H. KWON, *J. Kor. Inst. Met. & Meter.* **33** (1995) 1101.
4. K. B. LEE, K. W. LEE, S. W. HOE, K. H. HAN and H. KWON, *ibid.* **36** (1998) 242.
5. C. ZWEBEN, *JOM* (1992) 15.
6. I. M. HUTCHINGS, *Mater. Sci. Tech.* **10** (1994) 513.
7. C. G. LEVI, G. J. ABBASCHIAN and R. MEHRABIAN, *Met. Trans.* **9A** (1978) 697.
8. M. VOGELSANG, R. J. ARSENAULT and R. M. FISHER, *Metall. Trans.* **17A** (1986) 379.
9. K. D. WOO, J. S. LEE, M. S. LEE and S. W. KIM, *J. Kor. Foundrymen's Society* **19** (1999) 164.
10. M. TSUJIKAWA, K. ITOH, M. MAEDA and M. KAWAMOTO, *Imono* **61** (1989) 303.
11. S. WILLSON and A. T. ALPAS, *Wear* **212** (1997) 41.
12. M. TSUJIKAWA, T. TOMINAGA and M. KAWAMOTO, *Imono* **65** (1993) 559.

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