

Research Article

Tribological Analysis of Mg₂Si Particulates Reinforced Powder Metallurgy Magnesium Alloy Composites under Oil Lubrication Condition

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For the evaluation of wear behavior of Mg composites under oil lubrication conditions, powder metallurgy Mg₉₇Y₂Zn₁ alloy reinforced with additive Mg₂Si particles were fabricated by the repeated plastic working (RPW) and hot extrusion. The RPW process was effective in refining both Mg₂Si reinforcements and α -Mg grains causing the matrix hardening. When increasing the repetition number of RPW process from 200 to 600 cycles, the particle size of Mg₂Si additives changed from 8 μ m to 1~2 μ m, and α -Mg grain size was 1 μ m or less. With regard to the defensive and offensive properties of Mg alloys reinforced with Mg₂Si dispersoids, the composite had superior adhesive wear resistance compared with the conventional Mg alloys because of its extremely high microhardness of 95~180 Hv by RPW process. The uniform distribution of refined Mg₂Si particles was useful for improving both defensive and offensive properties against AZ31B counter disk specimens. The Mg₂Si prominent dispersoids in the matrix were also effective in forming the oil grooves around them, and caused the low and stable friction coefficient. On the other hand, in the case of the composite containing coarse Mg₂Si particles, severely deep scratches were given on the counter face of the AZ31B disk, and resulted in an unstable and high friction coefficient.

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1. Introduction

Magnesium alloys are applied to engineering components in the automotive industries due to their low density, less than 2 g/cm³, and effective for weight reduction, assisting the improvement of the fuel efficiency [1]. In addition, they also possess a good damping capacity and superior machinability to the other light metals such as aluminum and titanium alloys. Furthermore, Mg matrix composites show outstanding thermomechanical properties because of the hard dispersoids. On the other hand, magnesium silicide (Mg₂Si) intermetallic compounds is one of the useful reinforcements of Mg alloys because of its low density of 1.91 g/cm³, high melting point of 1358 K, 120G Pa Young's modulus, high micro-Vicker's hardness of 600~700 Hv, and low coefficient of thermal expansion of $7.5 \times 10^{-6} \text{ K}^{-1}$ [2, 3]. Mg composites with in situ synthesized Mg₂Si dispersoids were studied by a casting method and solid state reaction process [4~6]. In the casting process, the needle like Mg₂Si

compounds distributed in the matrix caused the reduction of mechanical properties. On the other hand, fine Mg₂Si particles via a solid state reaction between added silicon particles and the Mg matrix were effective for improving the strength of the composites. By employing SiO₂ particles as additives, the microstructural and mechanical responses of Mg alloy composites reinforced with Mg₂Si/MgO hybrid dispersoids were studied [7]. The reduction of SiO₂ particles by Mg was completely conducted by controlling the reaction temperature. It was set as an exothermic temperature of the DTA profile, when using the elemental mixture of Mg-SiO₂ powder. The Mg matrix composites revealed high mechanical properties and good wear resistance due to a good coherence of in situ synthesized Mg₂Si and the matrix [8]. In the previous study, Mg alloy composites reinforced with some additives such as nano-sized alumina, SiC, and feldspar particles were used to evaluate their tribological properties and wear resistance under dry sliding conditions [9~11]. This was because the weight reduction of

Mg alloy composites was effective in reducing the friction loss and saving the consumed energy in operating. The conventional AZ91 alloys and pure Mg were employed as the composite materials, and then the adhesion phenomena mainly occurred on the sliding surface due to the poor heat resistance of the matrix materials. In particular, with regard to AZ91 alloy, the sliding wear map under dry conditions was investigated by changing the applied load and sliding speed [12]. It was concluded that high strength and hardness of the matrix were required to control the adhesive wear phenomena in addition to the intermetallic compounds of the alloy. Yttrium and Zinc additives to Mg alloys were also useful for improving the mechanical properties, in particular high temperature strength due to their long-periodic stacking ordered (LPSO) structures [13, 14]. LPSO phases were formed in not only rapid solidification but also conventional ingot metallurgy process, and then Mg-Y-Zn cast ingots also showed much higher tensile strengths than the conventional AZ series alloys. In this study, the tribological property of powder metallurgy (P/M) $Mg_{97}Y_2Zn_1$ (at%) alloy reinforced with additive Mg_2Si particles was investigated under oil lubrication conditions. In particular, the microstructures of the composites were refined by repeating the severe plastic working on the elemental mixture of Mg alloy and Mg_2Si powders. The effects of the matrix hardness, Mg_2Si particle size, and content of the composite on their wear behavior were experimentally evaluated.

2. Experimental

2.1. Preparation of Mg_2Si Particles. Pure Mg powders, having a mean particle size of $112\ \mu m$ and purity of 99.9%, were used as starting raw materials. Pure silicon powders with $21\ \mu m$ diameter and 99.9% purity were prepared as another raw materials. The elemental mixture of Mg-33.33 at% Si powders was consolidated by a pressure of 15 MPa in vacuum (<4 MPa) by using the spark plasma sintering (SPS) equipment (SPS SYNTEX Inc., SPS-1030). The sintering conditions at 893 K for 600 seconds were sufficient for the solid state reaction between Mg and Si powders to synthesize Mg_2Si intermetallics [15]. XRD analysis on the sintered material indicated the only Mg_2Si peaks, and no peak of Mg and Si of raw powders was detected in the XRD profile. Fine Mg_2Si powder with a mean particle size of $8.2\ \mu m$ was obtained by ball milling (SEIWA GIKEN Co., RM-30) for 7.2 kiloseconds in air.

2.2. Powder Metallurgy Mg Alloy Composite with Mg_2Si via RPW Process. Machined chips of $Mg_{97}Y_2Zn_1$ (at%) cast ingot, containing 30 ppm Fe and 17 ppm Cu as impurities, were used as input materials. The elemental mixture of the above Mg-Y-Zn alloy powders and Mg_2Si additives (Mg_2Si content; 0, 5 and 10 wt%) was prepared by ball-milling process for 7.2 kiloseconds. The repeated plastic working (RPW) process, schematically illustrated in Figure 1 [16], was applied to the elemental mixture to refine Mg_2Si particles and uniformly distribute them over the matrix of the green compact. In RPW process, the alternate operation between

cold compaction and backward extrusion (an extrusion ratio; 5.2) was carried out on the powder mixture filled in the die, which was installed in a 1000 kN screw-driven press machine. The fragmentation of the additive Mg_2Si particles occurred by the above severe plastic deformation during the RPW process. At the same time, the refined particles were embedded in the Mg alloy matrix. A lot of strains, which were effective for the dynamic recrystallization during hot extrusion, were also induced into the matrix of Mg-Y-Zn alloy powder.

The maximum repetitive number was 600 cycles in this study during RPW process. After RPW process with a suitable repetitive number, the columnar green compact was fabricated, and served to hot extrusion process. The green compact via RCP process was heated at 623 K for 300 seconds in nitrogen gas atmosphere, and immediately consolidated by hot extrusion. The extrusion ratio was 30, and temperature of the extrusion container was 673 K. The microstructure coarsening with large grains over $10\ \mu m$ occurred via dynamic recrystallization by heating the green compact at 673 K. Therefore, the heating temperature of the powder compacts was 623 K in this study. Micro-Vicker's hardness measurement, optical microstructure, and scanning electron microscope (SEM, JOEL JSM-6500F) observation were carried out on the extruded Mg-Y-Zn alloy composites reinforced with Mg_2Si particles. α -Mg grain and Mg_2Si particle size distribution of the Mg alloy composite was measured by the image analysis software (Image Pro-plus) on optical microstructure photos.

2.3. Tribological Evaluation by Pin-on-Disk Type Wear Test. Pin-on-disk type wear test equipment (Rhesca Co. Ltd., FPR-2100) was used to evaluate the tribological properties of the above Mg alloy composites under oil lubrication conditions. Pin specimens, having 5 mm diameter and 10 mm length, were machined from the extruded Mg alloy composites. The conventional AZ31B and ADC12 (die-cast Al-12 wt% Si) alloys were used as the reference materials, and also machined to the pin specimen. AZ31B disks were employed as the counterpart materials. The surface roughness of the counter face after polishing was measured by the surface texture measuring instrument (Surfcom 1400D, TOKYO SEIMITSU CO). The R_{max} value was $0.81\sim 0.93\ \mu m$. Wear tests took place in 10W30 motor oil controlled at $308\sim 311\ K$. The applied load on the pin specimen, sliding speed and test time were 100 N, 1 m/s and 10 kiloseconds, respectively. The changes in friction coefficient during wear test were evaluated. The volumetric wear loss of the pin specimens was investigated by measuring the change of the pin length. The sliding surfaces of the pin and disk specimens were observed by optical microscope to investigate wear behavior of each Mg alloy composite.

3. Results and Discussion

3.1. Microstructural and Mechanical Responses of Mg Alloy Composites via RPW Process. Figure 2 shows optical microstructures of hot extruded $Mg_{97}Y_2Zn_1$ alloy

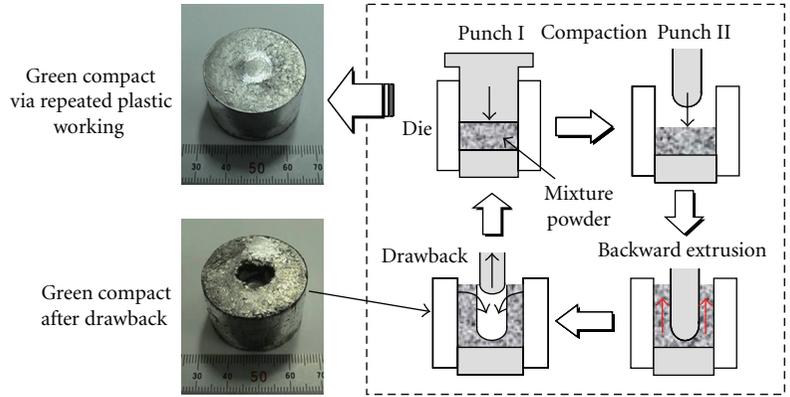


FIGURE 1: Schematic illustration of repeated plastic working (RPW) process on elemental mixture of Mg-Y-Zn alloy powders and Mg₂Si additives to fabricate columnar green compact.

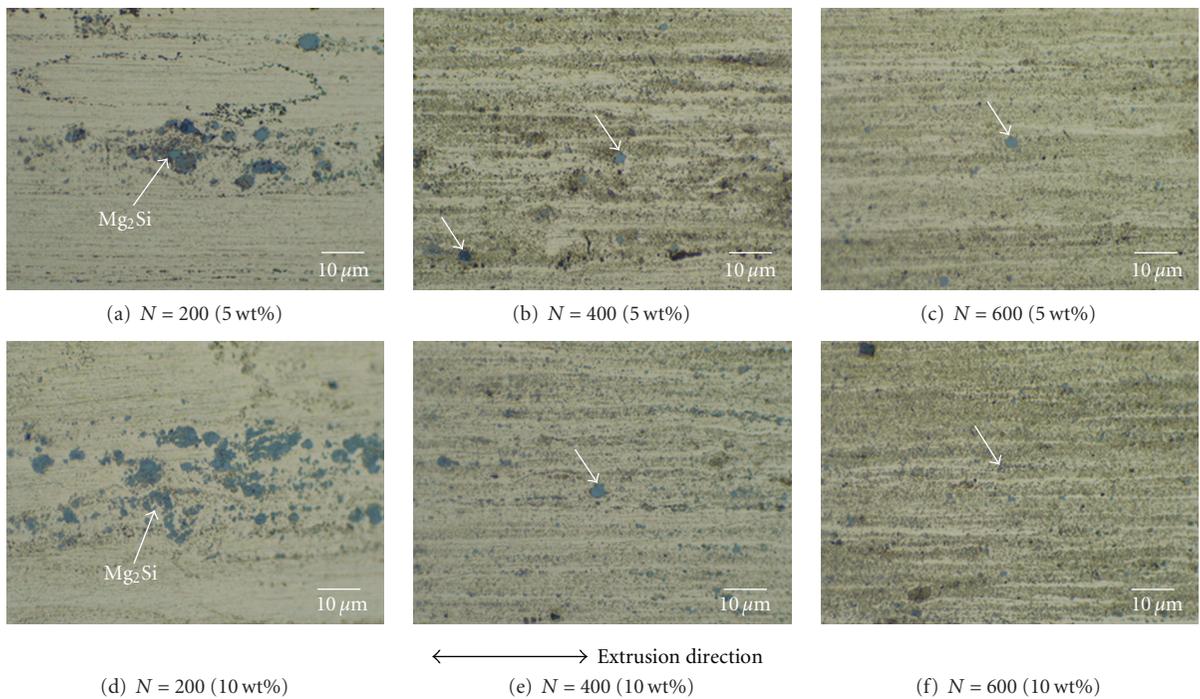


FIGURE 2: Microstructure changes of powder metallurgy extruded Mg-Y-Zn alloy composites reinforced with additive Mg₂Si particles via RPW process with $N = 200, 400,$ and 600 cycles. Mg₂Si content of 5 wt%; (a)~(c) and 10 wt%; (d)~(f).

composites with Mg₂Si particles via the RPW process with $N = 200 \sim 600$ cycles.

In both Mg₂Si contents of 5 wt% and 10 wt%, the refinement and uniform distribution of the additives obviously occurs with increase in the repetition number of the RPW process. For example, the initial mean particle size of Mg₂Si additives is 8.2 μm, and drastically decreases to less than 2 μm after the RPW process with $N = 600$ cycles as shown in Figures 2(c) and 2(f). Macroscopic photos indicate that a large plastic deformation of the matrix is observed, and some primary particle boundaries also exist in the case of 200 cycles (seeing Figures 2(a) and 2(d)). Each material reveals a layer microstructure in the matrix, which is typically caused

by hot extrusion. The distance between layers gradually decreases with increasing the repetition number. Figure 3 shows optical microscope and SEM observation photos on hot extruded Mg₉₇Y₂Zn₁ alloy composites with additive Mg₂Si particles via the RPW process with 400 cycles. Their content is (a) 5 wt% and (b) 10 wt%, respectively. The grain size of both composites is 0.7~1 μm or less via dynamic recrystallization during hot extrusion. The optical microstructures obviously reveal Mg₂Si spherical particles with a diameter of 1~3 μm. In the SEM photos, the white particles correspond to refined Mg₂Si additives. Very fine particles with 300 nm or less, which are impossible to be detected by optical microscope observation, are uniformly

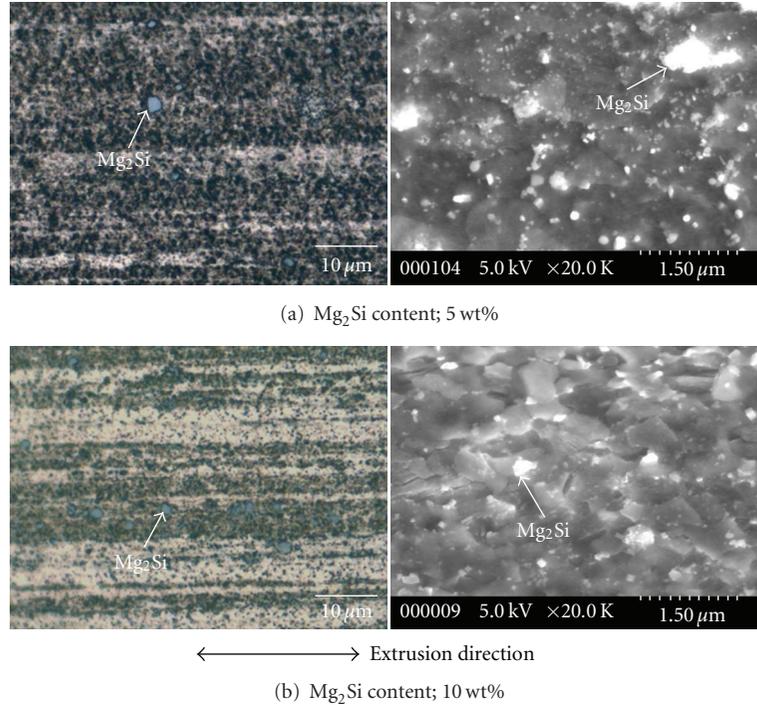


FIGURE 3: Optical microscope and scanning electron microscope observation on hot extruded Mg-Y-Zn alloy composites with Mg_2Si particles of (a) 5 wt% and (b) 10 wt%. Repetition number in RPW process, $N = 400$ cycles.

distributed inside α -Mg grains of the matrix. Figure 4 shows the hardening dependence of hot extruded Mg alloy composites on the repetition number in the RPW process. In each material, the hardness is proportion to the repetitive number because α -Mg grain refinement by dynamic recrystallization occurs with increase in the repetition number. The composite including 10 wt% Mg_2Si is harder than that with no additive when applying RPW process with 600 cycles. In this study, the hardness was measured on the matrix of the composite, except for the Mg_2Si particles which could be detected by optical microscope observation. The reason for this increase of the hardness is considered as below. Very fine Mg_2Si dispersoids with 100~300 nm in diameter, shown in SEM photos of Figure 3, effectively cause the increase of microhardness of the matrix. On the other hand, many strains are induced into α -Mg grains around Mg_2Si hard fine particles prior to the others far from the particles by the repetition of severe plastic deformation during RPW process. They accelerate the formation of fine grains via dynamic recrystallization during hot extrusion. Therefore, the hardness of the Mg alloy via RCP process increases with an increase in the Mg_2Si content of the composite as shown in Figure 4.

3.2. Wear Phenomenon under Wet Sliding. Figure 5 shows the comparison of the total volumetric wear loss of the pin specimens made of $Mg_{97}Y_2Zn_1$ alloy composite with various contents of Mg_2Si additives. The loss was calculated by measuring the changed height of the pin specimen after the wet sliding test. When increasing the additive content

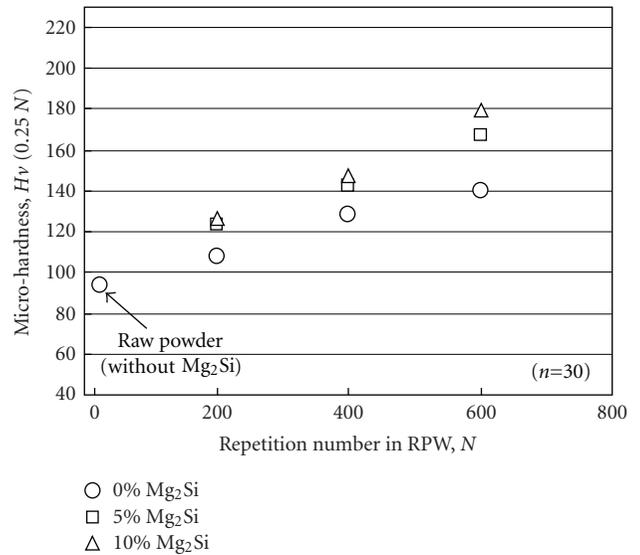


FIGURE 4: Hardness dependence of extruded Mg-Y-Zn alloy composites reinforced with additive Mg_2Si particles on repetition number in RPW process.

of Mg_2Si particles and the repetition number of the RPW process, the wear loss of pin specimens is remarkably reduced. This means that their wear resistance is obviously improved by the hardening of the matrix and the uniform distribution of fine Mg_2Si hard particles. For example, the total wear loss of the composite with 10 wt% Mg_2Si via 600 cycles RPW process is about 35% of that including no

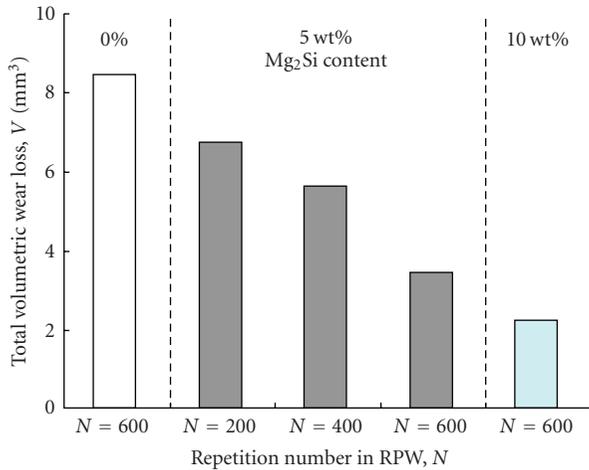
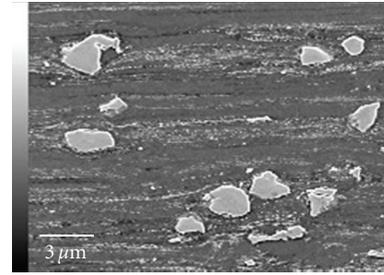


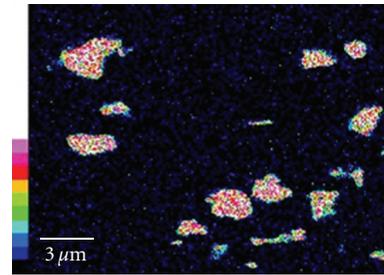
FIGURE 5: Changes in total volumetric wear loss of pin specimens including various contents of Mg₂Si additives.

additives (0%). The discussion on damages of the sliding surface of each specimen is given in below. First of all, Energy Dispersive X-ray Spectroscopy (SEM-EDS, JEOL, JED-2300) analysis was carried out on the sliding surface of the Mg alloy composite reinforced with Mg₂Si additives. As shown in Figure 6, Mg₂Si dispersoids with 1~3 μm diameter reveal round shape, not angular by the severe plastic deformation via RPW process. They are uniformly distributed in the matrix with no segregation. It also indicates no trace and no hollow of Mg₂Si particles detached from the matrix. Figure 7 shows the comparison of sliding surfaces of the pin specimens of Mg-Y-Zn alloy with no Mg₂Si (a) and 10 wt% Mg₂Si additives. The repetition number of RPW process was 600 cycles. In general, the conventional Mg alloy shows the adhesive wear at the sliding surface when it directly contacts the counter face in wear test because of its soft matrix compared to the aluminum alloys and steels [9, 10]. As shown in Figure 7(a), however, the sliding surface of the pin specimen reveals very slight adhesive wear, and a small change of the surface roughness is observed. This is quite different from the previous results on tribological properties of the conventional AZ91D alloy and their matrix composites [17] as mentioned above. This means that high hardness of about 140 Hv of the extruded Mg₉₇Y₂Zn₁ alloy via RPW process is enough to improve the defensive to the adhesion wear due to the plastic deformation in sliding and contacting with the counter disk specimen.

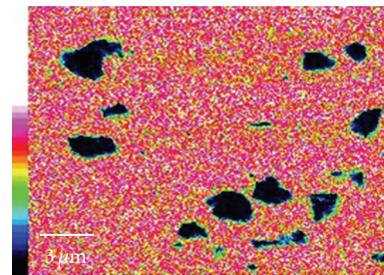
Therefore, the large volumetric wear loss of the Mg-Y-Zn alloy without Mg₂Si additives shown in Figure 5 seems to be mainly caused by the abrasive wear. On the other hand, in the case of the composite reinforced with 10 wt% Mg₂Si particles, very slightly abrasive wear is observed on the sliding surface as shown in Figure 7(b). In particular, the magnified observation indicates that fine Mg₂Si particles are distributed in the matrix, and no particle is detached from the surface after wear test. It is similar to the polished surface observed in the microstructure. This means that mild wear with no adhesion is formed at the sliding surface because in addition



(a)



(b)



(c)

FIGURE 6: SEM-EDS analysis result on sliding surfaces of pin specimens (Mg alloy composite reinforced with 10 wt% Mg₂Si particles via RPW process with 600 cycles). (a) SEM image, (b) element mapping of Si and (c) Mg.

to the matrix hardening as mentioned above, fine Mg₂Si hard dispersoids in the matrix are also effective for the defensive to both abrasive and adhesive phenomena in sliding.

With regard to the effect of Mg₂Si particle size on the wear behavior, Figure 8 shows the sliding surfaces of the pin specimens and AZ31B disks when employing Mg-Y-Zn alloy composite reinforced with 5 wt% Mg₂Si particles via RPW process with (a) 200, (b) 400 and (c) 600 cycles. In the case of N = 200 cycles, coarse Mg₂Si additives with about 20 μm are observed in the matrix, and their particle size gradually decreases with increase in the repetition number of the RPW process. This corresponds well to the microstructure changes shown in Figures 2(a)–2(c). Every pin specimen reveals slight scratches, not adhesive wear on the sliding surface. In the case of N = 200 cycles, however, severely deep scratches are observed at the counter face of AZ31B disk specimen. This means coarse Mg₂Si hard dispersoids attacked the counter disk material in sliding. With increase in the repetition number, the scratches become slight due to the refinement of their hard particles of the pin specimens.

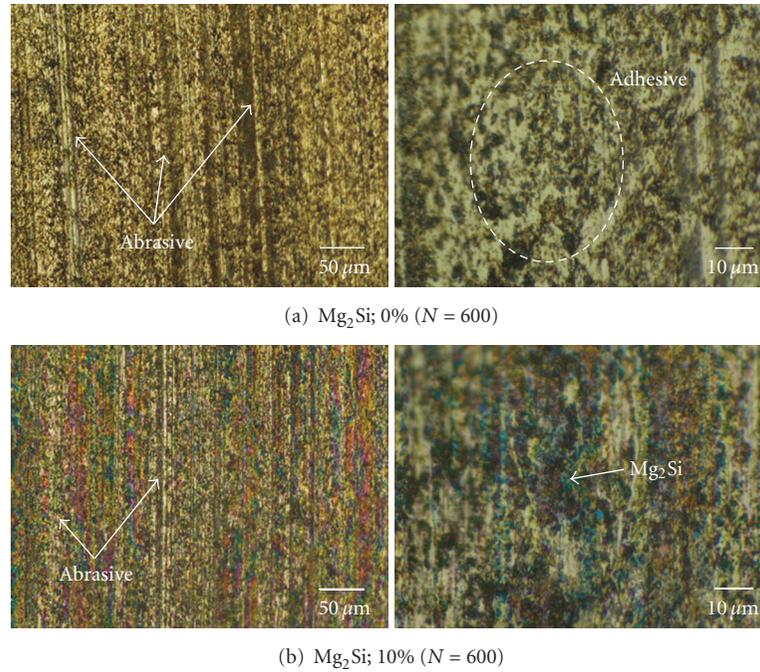


FIGURE 7: Damages on sliding surfaces of pin specimens made of Mg-Y-Zn alloy reinforced with Mg_2Si particles via RPW process with 600 cycles. Mg_2Si content; (a) 0 wt% and (b) 10 wt%.

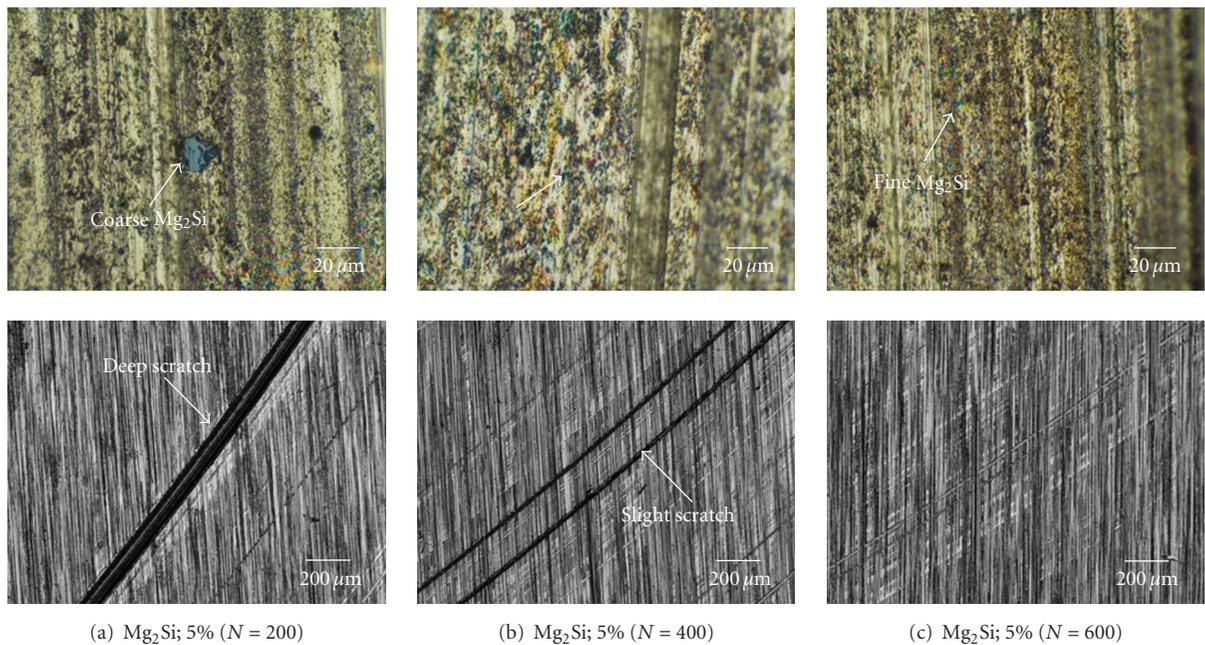


FIGURE 8: Sliding surfaces of pin specimens (upper) and AZ31B disks (lower) when employing Mg-Y-Zn alloy composite reinforced with 5 wt% Mg_2Si particles via RPW process with (a) 200, (b) 400 and (c) 600 cycles.

When employing $N = 600$ cycles, very narrow abrasion are detected on the counter face shown in Figure 8(c). Figure 9 indicates changes in friction coefficient during the wet sliding wear test, which correspond to the damage of the pin and disk specimens as mentioned above. When using $\text{Mg}_{97}\text{Y}_2\text{Zn}_1$ alloy with no Mg_2Si particle ($N = 600$ cycles),

friction coefficient was unstable. In particular, some sudden increment of the coefficient is detected. This is caused by the stick-slip phenomena due to the repetition of adhesive and abrasive wear at the contacting surface between the pin and disk specimen (shown in Figure 7(a)). With increase in the Mg_2Si content (5 wt% and 10 wt%), friction coefficient of

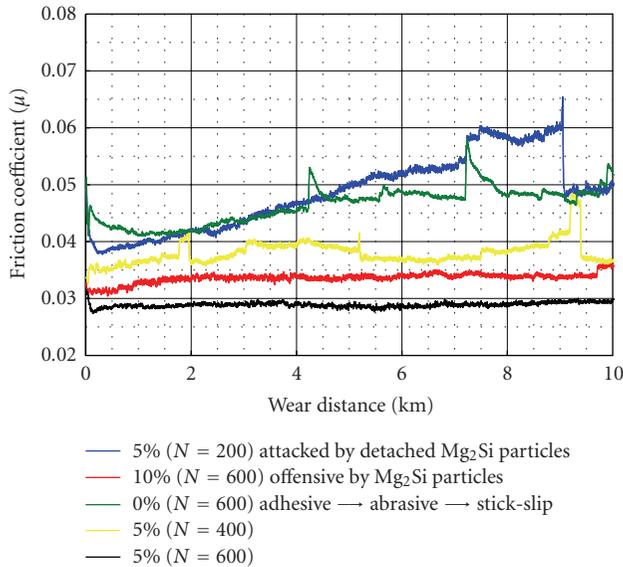


FIGURE 9: Friction coefficient changes in Mg-Y-Zn alloy composites containing various contents of Mg_2Si additives.

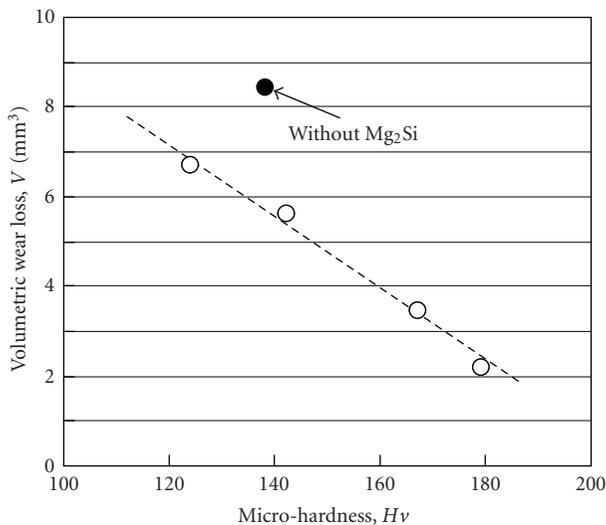


FIGURE 10: Dependence of volumetric wear loss on microhardness of pin specimens made of P/M Mg-Y-Zn alloy composites reinforced with additive Mg_2Si particles.

both materials was constant and stable. As mentioned above, the sliding surfaces of both materials shown in Figures 7(b) and 8(c) revealed very slight damages with extremely narrow abrasion, and seemed as the polished surfaces. In particular, Mg_2Si dispersoids are clearly observed as the prominent dispersoids at the sliding surface because the matrix is softer than Mg_2Si particles and its wear occurs much early. This means that a mixed lubrication condition was stably formed, and the sliding behavior was very mild due to the formation of the oil grooves around Mg_2Si prominent dispersoids. This wear behavior is similar to that in the case of Al-Si eutectic alloys under wet sliding conditions [18]. As a result, friction coefficient became stably lower when Mg_2Si additives

were contained in the Mg alloy. In the case of the Mg_2Si content of 5 wt% via RPW process with 200 cycles, coarse Mg_2Si hard particles have a severely offensive property on the counter disk surface (shown in Figure 8(a)), and the friction coefficient gradually increases due to the abrasive wear of the disk specimen. 10 wt% fine Mg_2Si additives of the Mg composite ($N = 600$ cycles) cause the remarkably offensive effect on the counter material (shown in Figure 7(b)), and result in the stable but slightly high friction coefficient. The distribution of refined Mg_2Si particles and matrix hardening of $Mg_{97}Y_2Zn_1$ alloy composites are suitable to keep the balanced wear resistance. That is, both of the defensive and offensive properties are given to the Mg composites when adding 5 wt% Mg_2Si particles and applying the RPW process with 600 cycles on $Mg_{97}Y_2Zn_1$ alloy powder. The reference materials of AZ31B and ADC12 alloys were used as a pin type specimen to evaluate their tribological properties under the same sliding conditions in this study. In the case of AZ31B alloy, the total volumetric wear loss was about 37 mm^3 , and extremely large compared to the Mg-Y-Zn alloy composite with no Mg_2Si particles shown in Figure 5. The friction coefficient gradually increased from 0.03 to 0.08, and was unstable. The microhardness (Hv) of the matrix of AZ31B was about 46, and quite smaller than that of Mg-Y-Zn alloys via RCP process (microhardness; 97). It was concluded that the poor wear resistance of AZ31B alloys was mainly caused by the extremely low hardness. The large wear loss was also due to no additive of hard particles, such as Mg_2Si and Si, contained in the conventional AZ31B alloy. On the other hand, when ADC12 alloy was used as a pin specimen, the wear loss was about 14 mm^3 , and its friction coefficient was 0.03~0.04, and very stable. This means coarse Si particles are effective to improve wear resistance, and also have an important role to make a stable sliding condition by oil grooves around Si dispersoids as mentioned in the previous reports [18]. Figure 10 shows the dependence of volumetric wear loss on microhardness of pin specimens. This indicates that wear resistance strongly depends on the matrix hardness. That is, the abrasion is a major wear phenomenon in the case of $Mg_{97}Y_2Zn_1$ alloys via RPW process. Furthermore, Mg_2Si fine hard dispersoids are also very effective to improve the wear resistance and prevent the abrasion. The above results are quite different from wear behavior in the case of the conventional Mg alloys resulting in the adhesive wear [10, 19, 20]. This is because $Mg_{97}Y_2Zn_1$ alloys have superior mechanical properties to the conventional ones, and prevent the plastic deformation, causing at the sliding surface. It is concluded that the addition of Mg_2Si particles is also effective for improving the abrasive wear resistance. It is however, important to handle their suitable particle size and content of the composite by microstructure control.

4. Conclusion

The wear behavior of powder metallurgy $Mg_{97}Y_2Zn_1$ alloy composites reinforced with additive Mg_2Si particles via RPW process was investigated under oil lubrication conditions. When increasing the repetition number of RPW process,

the severe plastic deformation by RPW process was effective for the uniform distribution of refined Mg_2Si prominent reinforcements with $1\sim 3\ \mu m$. It also accelerated the matrix hardening (microhardness, $H_v = 95\sim 180$) by α -Mg grain refinement of the composite. The former was useful for improving the wear resistance and offensive property against AZ31B counter disk specimens. It also caused a low and stable friction coefficient less than 0.03 by the oil groove formation around the Mg_2Si particles, making mixed lubrication conditions. The hardening of the matrix was useful to prevent the adhesion wear phenomenon when contacting the AZ31B counter face. When no Mg_2Si hard particle was dispersed in the matrix of the Mg alloys, the adhesive was the main wear mechanism. On the other hand, when coarse Mg_2Si additives were contained in the composite, they severely attacked the counter face with deep scratches of AZ31B disk, and the abrasive was the main wear mechanism.

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