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Wear Behavior of Sintered Magnesium Matrix Composites Reinforced with Mg_2Si Particle under Wet Sliding Conditions[†]

KONDOH Katsuyoshi*, UMEDA Junko**, KAWABATA Kenshi***, SEKI Yoshikazu**** and KAWAMURA Yoshihito*****

Abstract

For evaluation of wear behavior in the sliding conditions, powder metallurgy Mg97Y₂Zn₁ alloy composites reinforced with additive Mg_2Si particles prepared via the repeated plastic working (RPW) process were investigated. The RPW process was effective in refining Mg_2Si particles of 5 to 10 wt% and grains of the matrix via dynamic recrystallization. When increasing the repetition number of RPW process from 200 to 600 cycles, the particle size of Mg_2Si additives changed from 8 μm to 1~2 μm , and the grain size of the magnesium matrix was 1 μm or less. With regard to the defensive and offensive behavior in wet wear test, this composite had superior adhesive wear resistance compared with the conventional magnesium alloys because of its high matrix hardness at elevated temperature. The uniform distribution of refined Mg_2Si particles was useful for improving both defensive and offensive properties against AZ31B counter disk specimens. Deep scratches of the disk surface were caused by coarse Mg_2Si additives, and resulted in an unstable and high friction coefficient.

KEY WORDS: (Magnesium Composite) (Mg_2Si Particle) (Repeated Plastic Working) (Adhesive) (Abrasive) (Friction Coefficient)

1. Introduction

Magnesium alloys are applied to engineering components in the automotive industries due to their low density, less than 2 kg/m³, and they are effective for weight reduction, assisting the improvement of fuel efficiency¹⁾. In addition, they also possess a good damping capacity and superior machinability to the other light metals. Furthermore, magnesium matrix composites show outstanding thermo-mechanical properties because of the hard dispersoids. Magnesium silicide (Mg_2Si) has the possibility to be one effective reinforcement because of its low density of 1.91 kg/m³, high melting point of 1358 K, 120G Pa Young's modulus, high micro Vicker's hardness of 600-700Hv, and low coefficient of thermal expansion of $7.5 \times 10^{-6} K^{-1}$ ^{2,3)}. Magnesium composites with in-situ synthesized Mg_2Si dispersoids were studied by a casting method and solid state reaction process⁴⁻⁶⁾. In the casting process, needle like Mg_2Si distributed in the matrix caused the reduction of mechanical properties. Fine Mg_2Si particles via a solid state reaction between

added silicon particles and the magnesium matrix were effective for improving the strength of the composites. By employing SiO_2 particles as additives, magnesium alloys with Mg_2Si/MgO composite dispersoids were studied⁷⁾. The deoxidization of SiO_2 particles by magnesium was completely conducted by controlling the reaction temperature, which was an exothermic temperature from the DTA profile, where using the elemental mixture of Mg- SiO_2 powder. The magnesium matrix composites revealed high mechanical properties and good wear resistance due to a good coherence of in-situ synthesized Mg_2Si and the matrix⁸⁾. In the previous study, magnesium 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 conditions⁹⁻¹¹⁾. This was because the weight reduction of magnesium alloys was effective in reducing the friction loss and saving the consumed energy in driving. The conventional AZ91 alloy and pure magnesium were employed as the composite materials, and then the

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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 speed¹²⁾. 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 magnesium 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 higher tensile strengths than the conventional AZ series alloys. In this study, the tribological property of powder metallurgy (P/M) $\text{Mg}_{97}\text{Y}_2\text{Zn}_1$ (at%) alloy including the additive Mg_2Si particles, synthesized in the solid state from the elemental mixture of pure Mg powder and Si powders, was investigated under wet sliding 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, and their effect on the mechanical and wear properties were evaluated.

2. Experimental

2.1 Preparation of Mg_2Si particles

Pure Mg powder, having a mean particle size of 112 μm and purity of 99.9%, respectively, and silicon powder of 21 μm and 99.9% purity were prepared as the starting materials. The elemental mixture of Mg-33.33 at% Si powder was consolidated by a pressure of 15 MPa in vacuum ($< 4\text{MPa}$) by using the spark plasma sintering (SPS) equipment (SPS SYNTEX Inc., SPS-1030). The sintering conditions at 893K for 600S were sufficient for the solid state reaction between Mg and Si powders to synthesize Mg_2Si intermetallics¹⁵⁾. XRD analysis on the sintered compact indicated the only Mg_2Si peaks, and no peak of Mg and Si of raw powders. Fine Mg_2Si powder with a mean particle size of 8.2 μm was obtained by ball milling (SEIWA GIKEN Co., RM-30) for 7.2 ks in air.

2.2 P/M Mg-Y-Zn alloy composite with Mg_2Si was obtained via a repeated plastic working process

Machined chips of $\text{Mg}_{97}\text{Y}_2\text{Zn}_1$ (at%) cast ingot, including 30 ppm Fe and 17 ppm Cu as impurities, were used as input materials, and elementally mixed with the above fine Mg_2Si particles. The Mg_2Si content of the mixture was 0, 5% and 10% in this study. The repeated

plastic working (RPW) process, schematically illustrated in Fig.1¹⁶⁾, was applied to this elemental mixture of powders to uniformly mix and to refine Mg_2Si particles.

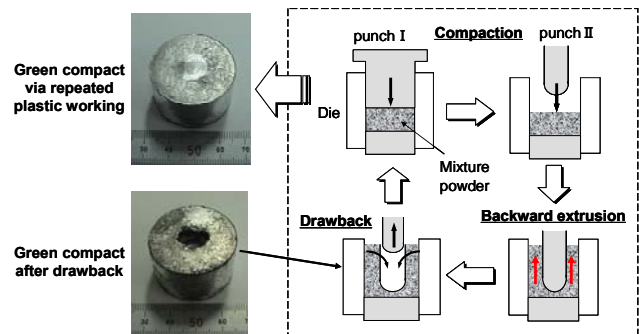


Fig.1 Schematic illustration of repeated plastic working process on magnesium alloy powder and appearance of green compacts.

In this process, the cold compaction and backward extrusion with an extrusion ratio of 5.2 were alternately carried out on the powder mixture filled in the die installed in a 1000 kN screw-driven press machine. The severe plastic deformation was applied to the mixture materials by the repetitive compaction and extrusion at room temperature, and then the fragmentation of the additive Mg_2Si particles occurred during the RPW process. A lot of strain, which was effective for the dynamic recrystallization during hot extrusion, was also induced into the matrix of magnesium alloy powder. The maximum repetitive number was 600 cycles in this study. The green compact via the RCP process was heated at 623 K for 300s in nitrogen gas atmosphere, and immediately consolidated by hot extrusion. The grain coarsening with large grains of 10 μm or more 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. The extrusion ratio was 30, and temperature of the extrusion container was 673 K. Micro Vicker's hardness (Hv) measurement, optical microstructure and field emission scanning electron microscope (FE-SEM, JOEL JSM-6500F) observation were carried out on P/M extruded Mg composites.

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 magnesium composites with Mg_2Si particles under wet sliding conditions. Pin specimens of 5mm diameter were machined from the extruded Mg alloy composites, and AZ31B disks were used as the counterpart materials. Wear tests took place

in 10W30 motor oil controlled at 308~311 K. The applied load on the pin specimen, sliding speed and test time were 100 N, 1 m/s and 10 ks, respectively. The volumetric wear loss of pin specimens after test and changes in friction coefficient during test were evaluated. The damage to the sliding surface of pin and disk specimens was observed by optical microscope.

3. Results and Discussion

3.1 Microstructures analysis and micro-hardness of Mg composites via RPW process

The application of the RPW process to the elemental mixture of $Mg_{97}Y_2Zn_1$ alloy chips and Mg_2Si particles forms fine and homogeneous microstructures of the green compacts. In particular, the refinement of Mg_2Si additives and grains occurs by repeating the severe plastic deformation. The refined Mg_2Si particles are also embedded in the magnesium alloy matrix during this process. **Figure 2** shows optical microstructures of hot extruded $Mg_{97}Y_2Zn_1$ alloy composites with Mg_2Si particles via the RPW process with $N=200\sim600$ cycles.

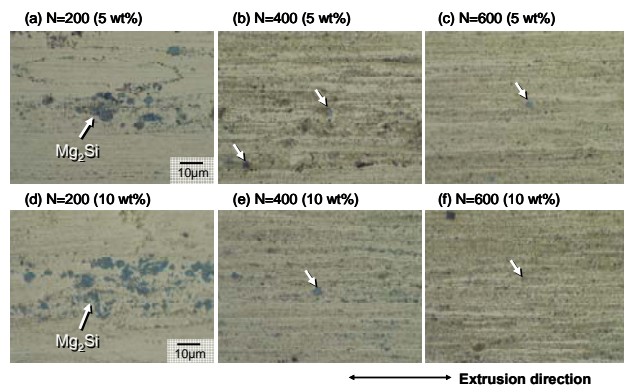


Fig.2 Microstructure changes of extruded magnesium alloy composites with additive Mg_2Si particles via RPW process with $N=200$, 400 and 600 cycles, Mg_2Si content of 5 wt%; (a)~(c) and 10 wt%; (d)~(f).

In both Mg_2Si contents of 5 and 10 wt%, the refinement and uniform distribution of the additives significantly occurs with increase in the repetition number of the RPW process. The initial mean particle size of Mg_2Si additives of 8.2 μm drastically decreases to less than 2 μm after the RPW process with $N=600$ cycles. 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. Each material reveals a layer microstructure in the matrix, which is typically caused by hot extrusion, and its distance gradually decreases with increasing the repetition number. **Figure 3** shows optical microscope and SEM observation photos on hot extruded $Mg_{97}Y_2Zn_1$

alloy composites with additive Mg_2Si particles via the RPW process with 400 cycles. Their content is 5 wt% (a) and 10 wt% (b), respectively. In the SEM photos, the white particles correspond to refined Mg_2Si additives. Very fine particles with 300 nm or less, which are impossible to detect by optical microscope observation, are uniformly distributed inside the grains of the matrix, and indicate a good coherence with them. The grain size of both composites is 0.7~1 μm or less via dynamic recrystallization during hot extrusion.

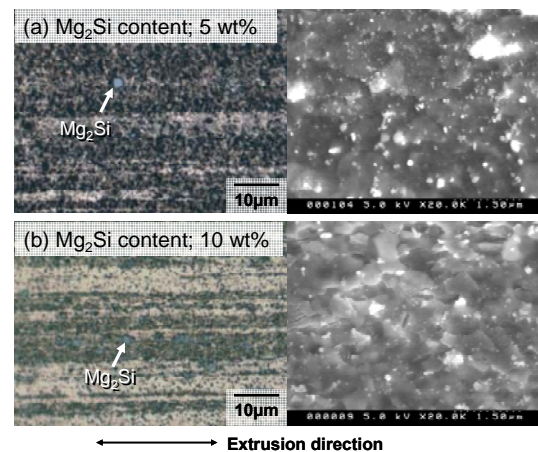


Fig.3 Optical microscope and scanning electron microscope observation on hot extruded magnesium alloy composites with additive Mg_2Si particles of 5 wt% (a) and 10 wt% (b) via RPW process with 400 cycles

Figure 4 shows the hardening dependence of hot extruded magnesium alloy composites on the repetition number in the RPW process. In each material, work hardening caused the increase of its micro-hardness of the matrix with increase in the repetition number.

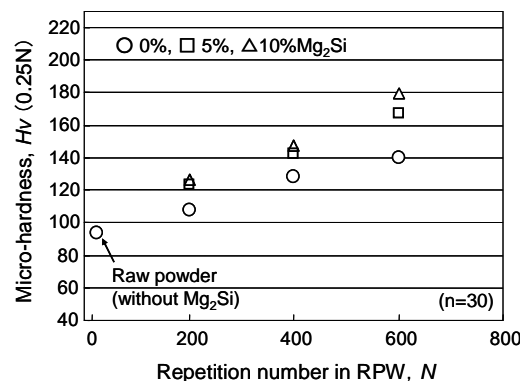


Fig.4 Hardening dependence of hot extruded magnesium alloy composites with additive Mg_2Si particles on repetition number in RPW process.

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, except for the Mg_2Si particles. The reason for

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this increase of the hardness is considered as below. During the repetition of severe plastic deformation on the elemental mixture powder by the RPW process, strains are induced into the grains around the hard fine particles of Mg_2Si additives prior to the others far from the additives, and accelerate the formation of fine grains via dynamic recrystallization. Therefore, the hardness of the magnesium alloy via the RCP process increases with an increase in the Mg_2Si content of the composite as shown in **Fig.4**.

3.2 Wear phenomenon under wet sliding

Figure 5 shows the changes in the total volumetric wear loss of pin specimens made of $\text{Mg}_{97}\text{Y}_2\text{Zn}_1$ 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 of Mg_2Si particles and the repetition number of the RPW process, the wear loss of pin specimens is remarkably reduced, that is, their wear resistance is improved. For example, the total wear loss of the composite with 10 wt% Mg_2Si via 600 cycles RPW is about 35% of that including no additives. The discussion on damages of the sliding surface of each specimen is given in below.

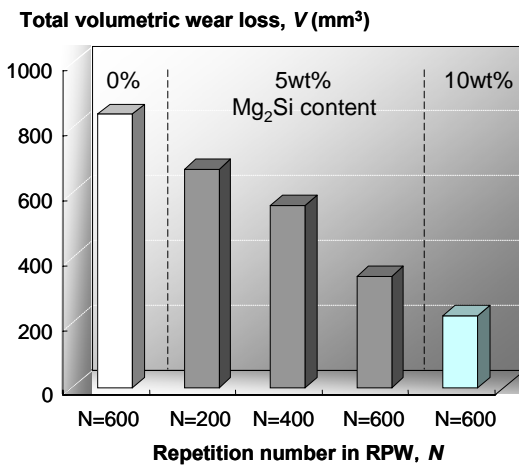


Fig.5 Changes in total volumetric wear loss of pin specimens including various contents of Mg_2Si additives.

Figure 6 shows the comparison of sliding surfaces of the pin specimens without Mg_2Si (a) and including 10 wt% Mg_2Si additives via RPW process after 600 cycles. The former reveals abrasive and adhesive wear phenomena on the surface. In general, the adhesive wear easily occurs at the sliding surface of magnesium alloys due to its soft matrix compared to the conventional aluminum alloys and steels^{9, 10}. In the specimen without hard particles shown in **Fig.6 (a)**, the total damaged area with

the adhesive wear and changes of the surface roughness are very small, and significantly different from the previous results on tribological properties of magnesium matrix composites. This means that matrix hardness of about 140 Hv of the extruded $\text{Mg}_{97}\text{Y}_2\text{Zn}_1$ alloy via the RPW process is enough to improve the defensive to the adhesion phenomenon due to the plastic deformation in sliding and contacting with the counter disk specimen. Therefore, the volumetric wear loss shown in **Fig.5** seems to be mainly caused by the abrasive wear. On the other hand, when using the composite with 10 wt% Mg_2Si particles, very slightly abrasive wear is observed on the sliding surface as shown in **Fig.6 (b)**. In particular, the magnified observation indicates that fine Mg_2Si particles are distributed in the matrix, and no particle is detached from the surface. It is similar to the polished surface observed in the microstructure. That is, mild wear with no adhesion is formed in contacting with the counter material because in addition to the matrix strengthening as mentioned above, the uniformly distributed fine Mg_2Si hard particles in the matrix are also effective for the defensive to both abrasive and adhesive phenomena in sliding.

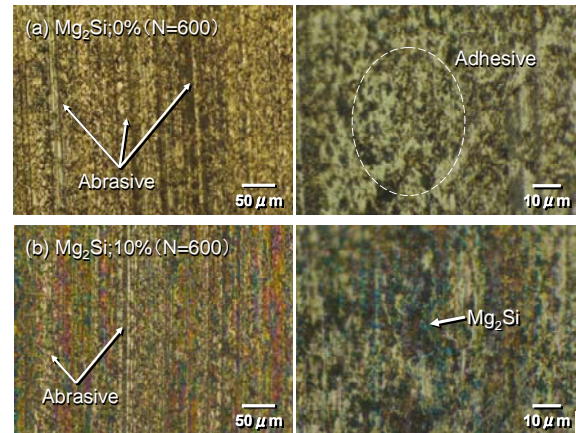


Fig.6 Damages on sliding surfaces of pin specimens with Mg_2Si content of 0 wt% (a) and 10 wt% (b) via RPW process with 600 cycles.

With regard to the Mg_2Si particle size on the wear resistance and offensive property, **Figure 7** shows the sliding surfaces of pin and disk specimens. In the case of N=200 cycles, coarse Mg_2Si additives are observed in the matrix, and they become fine with increase in the repetition number of the RPW process. This corresponds well to the microstructure changes shown in **Fig.2 (a)~(c)**. Every pin specimen reveals slight scratches, not adhesive wear on the sliding surface. However, a deep scratch is detected in the case of N=200 cycles, because coarse Mg_2Si hard particles attacked the counter disk material. With increase in the repetition number, the

scratches become slight due to the refinement of the hard particles of the pin specimens.

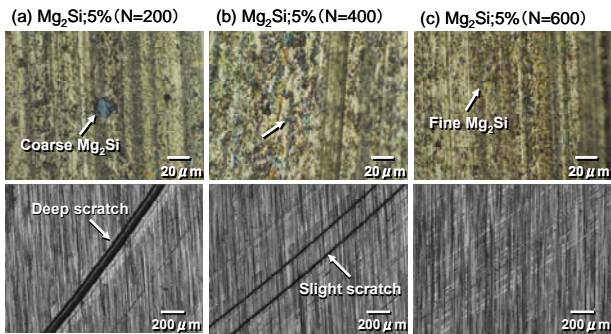


Fig.7 Sliding surfaces of pin and AZ31B disk specimens in employing magnesium alloy composite with 5 wt% Mg_2Si via RPW process with 200 (a), 400 (b) and 600 cycles (c).

Figure 8 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.

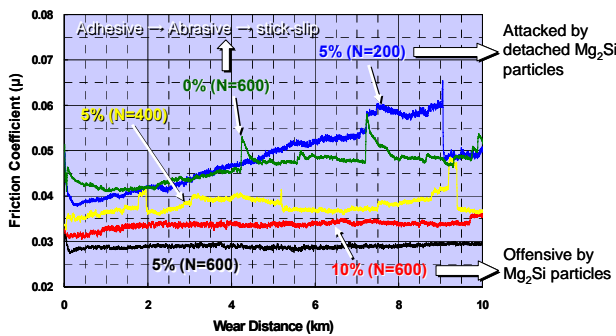


Fig.8 Friction coefficient change of each magnesium composite with Mg_2Si additives when using AZ31B alloy disk specimen as counter materials

When using $Mg_{97}Y_2Zn_1$ alloy with no Mg_2Si particle ($N=600$ cycles), some sudden increases of friction coefficient are detected, and 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 **Fig.6 (a)**). 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 **Fig.7 (a)**), and the friction coefficient gradually increases due to the abrasive wear of the disk specimen. 10 wt% fine Mg_2Si additives of the magnesium composite ($N=600$ cycles) cause the remarkably offensive effect on the counter material (shown in **Fig.6 (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 magnesium composites when adding 5 wt% Mg_2Si particles and applying the RPW process with 600 cycles on $Mg_{97}Y_2Zn_1$ alloy powder. **Figure 9** shows the dependence of volumetric wear loss on micro-hardness of pin specimens.

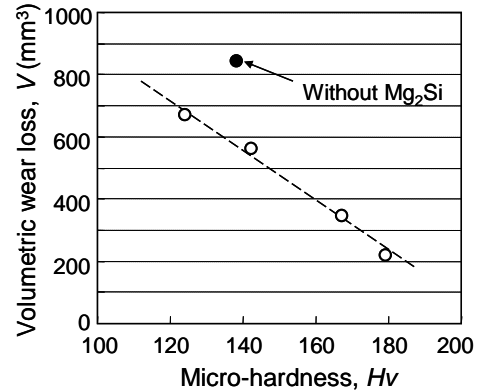


Fig.9 Dependence of volumetric wear loss on micro-hardness of pin specimens made of magnesium alloy composites with additive Mg_2Si particles.

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. It is different from the results in using the conventional magnesium alloys resulting in the adhesive wear^{10, 17, 18}, because $Mg_{97}Y_2Zn_1$ alloys have superior mechanical properties at elevated temperature to the conventional ones and obstruct the plastic deformation at the sliding surface. It is concluded that the addition of Mg_2Si particles is also effective in improving the abrasive wear resistance. It is however, important to handle their suitable particle sizes and content of the composite by microstructure control.

4. Conclusions

The defensive and offensive properties in the sliding wear test of P/M $Mg_{97}Y_2Zn_1$ alloy composites with additive Mg_2Si particles via the RPW process were investigated. When increasing the repetition number of the RPW process, the severe plastic deformation was effective in refining Mg_2Si particles and grains of the matrix of the composite. This produced superior adhesive wear resistance to the conventional magnesium alloys because of its high matrix hardness at elevated temperature. The uniform distribution of refined Mg_2Si particles was useful for improving both the defensive and offensive properties against AZ31B counter disk specimens. Deep scratches of the disk surface were caused by coarse Mg_2Si additives, and resulted in an unstable and high friction coefficient.

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