Silicon Carbide Dispersion Strengthening of Magnesium Using Mechanical Alloying Method*

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To improve the mechanical properties of magnesium alloys, hot pressing was performed to produce a silicon carbide-dispersion-strengthened magnesium (SiCp/Mg), in which SiC particles (SiCp) were milled with pure Mg using an attritor ball mill to reinforce the Mg. In this study, the silicon carbide dispersion strengthening of magnesium using the mechanical alloying (MA) method was investigated. The experimental results are summarized as follows. By increasing the milling energy of the ball mill in which the pAl2O3/Mg MA powder of a previous study was fabricated, the density of the SiCp/Mg was able to be increased to a value higher than that of the pAl2O3/Mg. The SiCp/Mg is about two-thirds as dense as commercial aluminum alloys. The hardness of the SiCp/Mg was about 1.5-fold that of the pAl2O3/Mg and exceeded that of AZ91D, which has the best mechanical properties of all the commercial Mg alloys. The highest value obtained was 95 HV. The bending strength of the SiCp/Mg was better than that of the pAl2O3/Mg. The results of XRD analysis and SEM-EDAX of the SiCp/Mg suggest that SiCp is almost uniformly and finely dispersed in the Mg and that the grain size of the matrix becomes finer with the dispersion.

Keywords: dispersion strengthening, magnesium, silicon-carbide particles, mechanical alloying, powder forming, density, hardness, bending strength

1. Introduction

Recently, automotive lightening has led to an increase in the demand for magnesium (Mg) alloys, which are lightweight substitutes for aluminum (Al) alloys.1) If Mg alloys begin to be used for the pistons and cylinder blocks of engines, the demand for piston rings and cylinder interiors having enhanced wear resistance will increase.

One potential method of improving the wear resistance of Mg alloys is to make a composite of Mg alloys and ceramic particles and/or fibers. As reported in a previous paper,2) hot-pressed discs of alumina particle (pAl2O3)-dispersion-strengthened magnesium powder obtained using the mechanical alloying (MA) method exhibit a hardness value that is equal to or higher than that of commercial Mg alloys.

Heat resistance is required for parts in close contact with thrust chambers, such as the piston crown side or the cylinder head. Silicon carbide (SiC) has a lower thermal expansion coefficient than Al2O3, and also has superior heat conductance and high-temperature strength.

Therefore, in this paper, SiC particles (SiCp) were mixed with pure Mg powder, and, in order to obtain mechanical properties superior to those in the previous paper,3) both the number of balls and the rotation rate of the arm of an attritor ball mill were increased.

Green discs of MA powder were hot-pressed, and the SiCp dispersion strengthening of magnesium using the MA method was experimentally investigated.

2. Experimental Procedures

2.1 Testing powders

Pure Mg powder (Kojundo Chemical Laboratory Co., Ltd.; purity, 99.9%; average particle size, $d = 142.2 \mu m$) and SiCp (Kojundo Chemical Laboratory Co., Ltd.; purity, over 99%; $d = 3.7 \mu m$) were tested. Figure 1 shows the morphologies of the tested powders. The average particle size of the powders and MA powders was measured using a laser-diffraction particle size distribution measuring device (Helo & Rodos, Japan Laser Co., Ltd.) and was expressed in terms of volume average size.

2.2 Composition and forming

Figure 2 shows a trial process for producing SiCp-dispersion-strengthened magnesium. MA powder was obtained with an attritor ball mill3) (attritor, Mitsuji Mining Co., Ltd.). Mixed powders consisting of 50 ml of pure Mg powder and a given amount of SiCp were fed into an Al2O3 container along with stearic acid of 2 mass%2) and $5 mm Al2O3$ balls at an volume of 1 or 3 $\ell$. The Al2O3 agitator arm was rotated in an argon atmosphere at 174 or 296 rpm for 10.6–86.4 ks to mill the mixed powders.

The MA powders were put in a die with a diameter of 20 mm and were cold-compacted into green discs (thickness of 2.00 ± 0.05 mm) using a 100 kN capacity hydraulic press at a given pressure and holding time. The carbon tools,3) coated with BN to prevent adhesion between the tools and with the green discs, were charged with the green discs and set in hot-press equipment (Nems Co., Ltd.). After the stearic acid was removed at 673 K for 3.6 ks, the green discs were hot-compressed at a given temperature, a given pressure, and a given holding time in an argon atmosphere and then furnace cooled into SiCp-dispersion-strengthened magnesium (SiCp/Mg) discs with diameters of 20.37–21.04 mm and thicknesses of 1.02–1.79 mm.

2.3 Density

Both surfaces of the SiCp/Mg discs were polished with abrasive paper (finished with # 1000 emery paper; plate

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thickness tolerance, ±0.05 mm). The size and weight of the discs were measured, and then disc density was calculated.

2.4 Hardness test

The hardness of the SiC<sub>p</sub>/Mg discs was measured using a Vickers hardness tester at 49.03 N for 30 s. The values obtained were the means of the values from 3 discs (at 5 points each).

2.5 Bending test

The polished (finished with # 1000 emery paper) SiC<sub>p</sub>/Mg discs with 1.00 ± 0.05 mm thickness were subjected to a bending test. The bending strength was calculated using the formula used in the three-point bending of beams. The values obtained were the means of the values from 5–6 discs. All the discs failed at the center of the span.

2.6 Microstructure observation and elemental analysis

Microstructural observation and qualitative analysis of both MA powders and the discs were carried out using a scanning electron microscopy (SEM, Jeol Ltd.) system, an energy dispersive X-ray analysis (EDAX, Edax Japan K. K.) system, and X-ray diffraction equipment (Rigaku Co., Ltd.).

3. Results and Discussion

3.1 Lightness of SiC<sub>p</sub>/Mg disc

Figure 3 shows the effects of pAl<sub>2</sub>O<sub>3</sub> and SiC<sub>p</sub> contents on the density of specimens fabricated from MA powder processed for 86.4 ks. The theoretical density of both Mg-SiC and Mg-Al<sub>2</sub>O<sub>3</sub> are indicated by the long and short dotted lines, respectively.

Where, the symbols L and H in parentheses with the specimen names show the cases in which mixture powder consisting of Mg powder and pAl<sub>2</sub>O<sub>3</sub> or SiC<sub>p</sub> is mechanical-alloying (MA)-treated at an arm rotation rate of 174 rpm with a ball volume of 1 ℓ and at 296 rpm with 3 ℓ, respectively. The specimens are expressed as SiC (L), SiC (H), and Al<sub>2</sub>O<sub>3</sub> (L) which are the results obtained in a previous paper.

In addition, the pAl<sub>2</sub>O<sub>3</sub> and SiC<sub>p</sub> contents were expressed as mass percent rather than volume percent, because of the
difficulty in obtaining an accurate powder density. Density increased with Al$_2$O$_3$ and SiC contents, because of an increase in the size of the ceramics, which have a higher density than magnesium. The density of SiC (H) was the highest. As an example, the relative density$^{21}$ of the specimens with a ceramic content of 20% was 96% for both Al$_2$O$_3$ (L) and SiC (L), and was 104% for SiC (H).

Figure 4 shows SEM photographs of 20% SiC (H) and 20% Al$_2$O$_3$ (L) powders MA-treated for 86.4 ks. In the 20% SiC (H) powder, the powder particle shape becomes nearly spherical and almost maintains a constant size (average particle size $d = 47.7 \mu$m). On the other hand, the 20% Al$_2$O$_3$ (L) powder particles become flakelike, do not maintain their size, and show a larger size ($d = 159.6 \mu$m).

This tendency is similar to those of the SiC (H) and Al$_2$O$_3$ (L) powders with other ceramic contents. In addition, the particle size and shape of the SiC (L) powder were similar to those of the Al$_2$O$_3$ (L) powder, and in the 20% SiC (L) powder, the average particle size was 130.2 \mu m.

The contact area between the powders is considered to increase in powder compacting, because the SiC (H) powder was essentially spherical in shape, almost maintained its size, and showed a finer powder particle size than the Al$_2$O$_3$ (L) powder. As a result, sinterability was improved and the density of SiC (H) increased.

The reasons the density of the SiC (H) exceeded the theoretical density are considered to be as follows: (1) an increase in the amount of contamination from the arm, ball, and container; (2) an increase in the composition variation of Mg-SiC due to the prior agglutination to the ball milling parts of Mg powder softer than ceramics particles during the MA treatment; (3) an increase in the amount of Mg oxidation resulting from an increase in the total powder surface area for powdery refinement while the MA powder was taken out from the attritor caused by increasing the milling energy.

The density of the SiC (H) was in the range of 1.8–2.0 g/cm$^3$, which is similar to that of commercial Mg alloys (approximately 1.8 g/cm$^3$). Moreover, it was about two-thirds as dense as commercial Al alloys (2.7–2.8 g/cm$^3$).

3.2 Mechanical properties of SiC$_p$/Mg disc

3.2.1 Hardness

Figure 5 shows the effects of $p$Al$_2$O$_3$ and SiC$_p$ contents on the Vickers hardness of specimens fabricated from MA powder processed for 86.4 ks. For a comparison, Figure 5 shows the results of hardness tests on pure Mg virgin metals (purity, 99.9%), the Mg alloy used for the die-casting, AZ91D (casting), and the wrought Mg alloy, AZ31 (rolling and extrusion). In addition, the hardness of the materials was similar to the nominal hardness.$^{5,6}$
The hardness of the specimens increased with \( p \mathrm{Al}_2 \mathrm{O}_3 \) and \( \mathrm{SiC} \) contents. The hardness of \( \mathrm{Al}_2 \mathrm{O}_3 \) (L) is similar to that of \( \mathrm{SiC} \) (L); however, \( \mathrm{SiC} \) (H) is about 1.5-fold harder than \( \mathrm{Al}_2 \mathrm{O}_3 \) (L) and \( \mathrm{SiC} \) (L). Moreover, the hardness of \( \mathrm{SiC} \) (H) exceeded that of the representative commercial Mg alloy, AZ91D, with \( \mathrm{SiC} \) contents over 5%, and the highest value for 20% \( \mathrm{SiC} \) (H) was 95 HV.

The 0% \( \mathrm{SiC} \) (H) had a hardness of 70HV, and this value was higher than that of the hot-pressed compact fabricated from pure Mg powder (MA-treated for 108ks using a planetary ball mill) by Yamazaki et al.\(^7\).

The main factors causing the hardness of the \( \mathrm{SiC} \) (H) to be higher than that of both \( \mathrm{Al}_2 \mathrm{O}_3 \) (L) in a previous paper\(^2\) and \( \mathrm{SiC} \) (L) are considered to be the density increase caused by MA powder refinement (previously described) and the improvement in the dispersivity of the ceramic particles caused by increasing the milling energy.

Consequently, in order to investigate the dispersion of ceramic particles, the specimen surface was examined by X-ray diffraction measurement, microstructural observation using an SEM, and elemental analysis using SEM-EDAX.

Figure 6 shows the X-ray diffraction patterns of 20% \( \mathrm{SiC} \) (H) and 20% \( \mathrm{Al}_2 \mathrm{O}_3 \)/Mg mechanical-alloying-processed for 86.4ks.

![X-ray diffraction patterns of 20% SiC/Mg (SiC (H)) and 20% pAl2O3/Mg mechanical-alloying-processed for 86.4ks.](image)

The square area of Fig. 7 was magnified and subjected to elemental analysis by EPMA\(^3\) or SEM-EDAX. Figure 8 shows the results for 20% \( \mathrm{SiC} \) (H). From both the X-ray diffraction results shown in Fig. 6 and the elemental analysis results shown in Fig. 8, the gray regions observed in Fig. 7(a) (20% \( \mathrm{SiC} \) (H)) are considered to be Mg, and the white regions are considered to be refined Mg and \( \mathrm{SiC}_p \). Hard \( \mathrm{Mg}_2\mathrm{Si} \), which is newly produced, and comparatively large \( \mathrm{SiC}_p \) particles of several \( \mu \)m in size are uniformly dispersed in the white regions.

On the other hand, from the results of a previous paper\(^2\), the white regions in 20% \( \mathrm{Al}_2 \mathrm{O}_3 \) (L) were considered to be \( p\mathrm{Al}_2 \mathrm{O}_3 \) having a particle size of several \( \mu \)m.

For this reason, 20% \( \mathrm{SiC} \) (H) is considered to have ceramics particles dispersed more finely and more uniformly in Mg than 20% \( \mathrm{Al}_2 \mathrm{O}_3 \) (L).

Both the grain size and the local strain of Mg were determined from the X-ray diffraction patterns for use in the Hall equation:\(^4\)

\[
\beta \cos \theta = \frac{\lambda}{D} + 2\eta \sin \theta
\]

where \( \beta \) is the half-peak width of a specimen, \( \theta \) is the angle of diffraction, \( \lambda \) is the wavelength of the X-ray, \( D \) is the grain size, and \( \eta \) is the local strain.

As a result, the grain size was determined to be approximately 50nm and the local strain was determined to be approximately 0.07 % for 20% \( \mathrm{SiC} \) (H). The values for 20% \( \mathrm{Al}_2 \mathrm{O}_3 \) (L) were approximately 290nm and 0.06%, respectively. The local strain of \( \mathrm{SiC} \) (H) was similar to that of \( \mathrm{Al}_2 \mathrm{O}_3 \) (L), whereas the grain size of \( \mathrm{SiC} \) (H) was about 6-fold smaller than that of \( \mathrm{Al}_2 \mathrm{O}_3 \) (L).

In addition, the grain size and local strain for the 86.4ks MA treated 20% \( \mathrm{SiC} \) (H) powder were approximately 40nm and 0.35%, respectively. Because local strain is released when the MA powder is heated, the strain of the specimen is about 5-fold less than that of the MA powder. However, the grain size after heating the MA powder shows no marked change with respect to the size before heating.

Although the Mg grain recrystallizes and grows after heating the MA powder, grain growth is considered to be inhibited and grain size is kept small by the improvement of \( \mathrm{SiC}_p \) dispersivity.

From these results, the ceramic particles are considered to have improved dispersivity, and a fine grain structure was formed in the Mg matrix by increasing the milling energy.

In view of the fact that an outstanding increase in hardness was obtained by improving of ceramic particle dispersion strength, the effect of MA treatment time on the hardness of the discs was investigated under the MA conditions of an arm rotation rate of 296rpm and a ball volume of 3 l.

From the results, \( \mathrm{SiC} \) (H) obtained a hardness exceeding that of AZ91D with a treatment time over 64.8ks for \( \mathrm{SiC} \) contents of 5% and 10%, and with a treatment time over 21.6ks for a \( \mathrm{SiC} \) content of 20%.

20% \( \mathrm{Al}_2 \mathrm{O}_3 \) (L), which had the maximum value in a
previous paper, was similar to 10.8 ks-MA-treated 20% SiC(H).

3.2.2 Bending strength

Figure 9 shows the effects of \( p\text{Al}_2\text{O}_3 \) and \( \text{SiC}_p \) contents on the bending strength of specimens fabricated from MA powder processed for 86.4 ks. The bending strength decreased as ceramic content increased. The bending strength of SiC(L) is similar to that of \( \text{Al}_2\text{O}_3 \) (L). However, the bending strength of SiC(H) is higher than those of \( \text{Al}_2\text{O}_3 \) (L) and SiC(L). Mg grain refinement caused by increasing the milling energy is considered to be a major factor.

Nobre et al.\(^8\) have reported that bending strength values of

Fig. 7 Surface observation of 20\% SiC\(_p\)/Mg (SiC (H)) (a) and 20\% \( p\text{Al}_2\text{O}_3\)/Mg (b) mechanical-alloying-processed for 86.4 ks.

Fig. 8 The elemental analysis results of Mg (b), Si (c) and C (d) by EDAX of rectangular area in Fig. 7(a). (a) shows backscattered electron image (BEI).
200–220 MPa were obtained for a compressive strain of 2.5% using the 4-point bending test on AZ31 wrought magnesium alloys (length of 160 mm, width of 15 or 20 mm, and thickness of 10 mm).

Although their results cannot be directly compared with the results in this paper, the bending strength of SiC (H) (244–341 MPa) was higher than the reported values for AZ31 alloys regardless of SiC content. 20% Al2O3 (L) was similar to 20% SiC (H) fabricated from MA powder processed for 10.8 ks.

4. Conclusions

Mechanical alloying (MA) powder, consisting of pure Mg powder and SiC particles (SiCp), was hot-pressed, and the SiCp dispersion strengthening of magnesium using the MA method was experimentally investigated. Consequently, the following conclusions were obtained, where the symbols L and H in parentheses with specimen names indicate the cases in which a mixture powder of Mg powder and pAl2O3 or SiCp is MA-treated at an arm rotation rate of 174 rpm with a ball volume of 1 ℓ and at 296 rpm with 3 ℓ, respectively.

(1) The density of SiC (H) hot-pressed discs of SiCp/Mg MA powder obtained by increasing the number of balls and the arm rotation speed of the ball mill compared with those used in a previous report was in the range of 1.8–2.0 g/cm³. The SiC(H) was about two-thirds as dense as commercial aluminum alloys.

(2) By increasing the milling energy, the hardness of SiC (H) was about 1.5-hold those of Al2O3 (L) and SiC (L), and exceeded that of the commercial Mg alloy AZ91D. The highest value was 95 HV.

(3) The bending strength of SiC (H) was better than that of Al2O3 (L) and SiC (L).

(4) SiCp is considered to be finely and almost uniformly dispersed in Mg, and the Mg grain becomes fine in SiC (H).

(5) The main factors of the increase in the hardness are considered to be the density increase caused by MA powder refinement and the improvement in ceramic dispersion strength.

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