Oxide dispersion strengthened magnesium: Effect of sonicated dispersion on the microstructure and mechanical properties

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Abstract

In this study, ultrafine (0.3 µm) aluminum oxide (Al₂O₃) particles were dispersed into commercially pure magnesium powder using the sonication method and processed by press-sinter-extrusion technique. The oxide particles dispersion in the processed magnesium microstructure was reasonably homogeneous and with good interfacial integrity. The dispersed oxide particles refined the grain morphology. They significantly improved the hardness, yield strength, tensile strength, ductility, and work-of-fracture of magnesium when added to a vol.% of up to 2.5. The dispersed oxide particles also changed the fracture mode of magnesium from brittle to ductile nature. However, the study also revealed that the ultrafine oxide particles dispersion was most effective in improving the overall mechanical properties of magnesium when added in an amount of 0.7 vol.%.

Key words: light alloy, oxide dispersion strengthening, sonication, microstructure, mechanical properties

1. Introduction

The growing demand for energy-efficient equipment, conservation of rapidly depleting fossil fuel resources, and the protection of the deteriorating environment has brought attention to the lightweight materials, including magnesium as a structural material. Compared to the density of the existing structural metallic materials [1], the magnesium is thirty percent lighter than aluminum, sixty percent lighter than titanium, and seventy-five percent lighter than steel. Besides the lightweight, the magnesium has excellent castability, good damping capacity, and superior machinability. The magnesium has a long history of structural application [2, 3] and is expected to grow soon significantly. The supply of the magnesium metal is well secured by its abundant natural existence (sixth element in earth crust composition and third element in seawater composition) and the invention of the economically competitive extraction and refining process. Intensive research work is underway globally to improve the intrinsic limitations (i.e., relatively poor strength, ductility, corrosion resistance, and thermal stability) of the magnesium and make it eligible to be a sustainable substitute of the existing structural metallic materials. Alloying with the compatible elements and/or reinforcing with the thermally stable stiffer second phase are considered to be the technically feasible ways to mitigate and/or circumvent these limitations. Between these two choices, the use of the second phase particles as reinforcement has reportedly been able to improve the mechanical properties of magnesium to the extent that it is beyond the limit of the traditional alloying process [4–11]. The shape, size, volume fraction, properties, and chemical compatibility of the second phase reinforcement dictate the extent of their effect on the resultant properties of the processed matrix material. Strengthening of metal by using harder second phase particles is maximized, with a trade-off in ductility, when they are well dispersed in the matrix in very small size and/or in relatively large volume fraction and effectively impede the dislocation motion [11, 12]. However, in the case of magnesium, it has been reported that the presence of extremely fine added second phase particles [6–11] induced metastable precipitation [13], and even the

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grain refinement [14] can strengthen the matrix with simultaneous enhancement of its ductility, an anomalous to the generalized strength-ductility relationship. There is a compositional and/or processing limitation to produce favorable metastable precipitation, which does not exist in the case of the addition of external second phase particles. Added external second phase particles often introduce grain refinement and provide cumulative benefit to the metal matrix. Among all the added particle reinforcements, oxides, and, more specifically, the alumina ($\text{Al}_2\text{O}_3$) in a size range from nanometer to micrometer scale [7, 9, 10], reportedly was most compatible with magnesium to improve the structural properties. The alumina particles were reportedly dispersed in magnesium by using ingot and powder metallurgy processes. One of the earlier studies [10] revealed that the submicron-sized ($0.3 \, \mu m$) ultrafine alumina provided the best combination of strength-ductility in magnesium when compared to the effect of nanometer and micrometer size particles. In this study, the ultrasonic method was used to disperse the different amounts of ultrafine ($0.3 \, \mu m$) alumina particles in magnesium powder and investigated the effect of the added oxide dispersoids on the microstructure and mechanical properties of magnesium. Sonication was expected to disperse the ultrafine oxide particles efficiently and consequently maximize the exploitation of the oxide dispersion strengthening effect in magnesium.

2. Material and procedures

2.1. Material

High purity alpha alumina with an average size of 0.3 $\mu m$ (Baikowski, Japan) in three different vol.%, i.e., 0.7, 1.1, and 2.5 was dispersed in the commercially pure ($\geq 98.5 \%$) magnesium powder (Merck, Germany) with a particle size range of 60–300 $\mu m$.

2.2. Processing

Oxide particles of the required vol.% were added to the magnesium powder in a glass beaker and dispersed using a probe sonicator (VC-50, Sonics) for ten minutes. Acetone was used as suspension media. The oxide dispersed magnesium suspension was kept in the oven at 50 $^\circ C$ to evaporate the acetone and was subsequently compacted to cylindrical preforms (40 mm height with 35 mm diameter) at a pressure of 450 MPa using a uniaxial hydraulic press. The preforms were coated with colloidal graphite, wrapped in aluminum foil, and sintered for 2 h at 500 $^\circ C$ in a tube furnace under inert argon gas atmosphere. Sintered oxide dispersed magnesium preforms were hot extruded at 250 $^\circ C$ to produce 8 mm diameter rods, using an extrusion ratio of 19.1 : 1. Extrusion was conducted in a 150-ton uniaxial hydraulic press, and each of the preforms took one minute to be extruded. Colloidal graphite was used as a lubricant. The preforms were preheated for 90 min in a constant temperature furnace at 300 $^\circ C$ before the extrusion process. The synthesis of the monolithic magnesium without oxide particles was carried out using precisely similar steps.

2.3. Density measurement

Density ($\rho$) of the monolithic and oxide dispersion strengthened (ODS) magnesium was measured on polished extruded samples following Archimedes' principle [9]. Distilled water was used as the immersion fluid. A Mettler Toledo model AG285 Electronic balance, with an accuracy of $\pm 0.0001 \, g$, was used to weigh the samples in water and air media. Theoretical densities of ODS magnesium were predicted using Rule-of-Mixture with an assumption of a fully-dense microstructure free from any dispersoid-matrix interfacial reaction. Vol.% of porosity in the extruded materials was calculated from the difference between the theoretical and experimental density values.

2.4. Microstructural characterization

Microstructural characteristics of the monolithic and ODS magnesium were studied on metallographically prepared extruded samples to investigate the oxide particles dispersion pattern, oxide-magnesium interfacial characteristics, and effect of the oxide particles dispersion on the grain morphology of magnesium. Field Emission Scanning Electron Microscope (FESEM) Tescan Lyra-3 equipped with Energy Dispersive Spectroscopy (EDS) and Meiji MX7100 optical microscope were used for the characterization process. The grain morphology was determined using Matlab based Line-cut image analysis software.

2.5. Mechanical characterization

The bulk and microhardness of the monolithic and the ODS magnesium were determined on polished extruded samples. Bulk hardness measurements were conducted using a Rockwell 15T superficial scale in universal Rockwell hardness testing machine following ASTM E18-17 standard. Vickers microhardness was measured using a load of 50 $gf$ and dwell time of 15 s on a Beuhler MMT-3 automatic digital microhardness tester in accordance with ASTM E384-17 standard.

Extension-to-failure tensile tests on the monolithic and ODS magnesium were carried out on an Instron 3367 machine following the ASTM E8M-16a standard. The tensile tests were conducted on smooth cylindrical specimens, which were machined from the extruded...
Table 1. Results of density, porosity, and grain morphology characterization of ODS magnesium

| Materials          | Al₂O₃ (wt.%) | Density (gm cm⁻³) | Porosity (%) | Grain morphology |
|-------------------|--------------|-------------------|--------------|-----------------|
| Mg/0.0Al₂O₃       | 0.0          | 1.7400            | 1.7310 ± 0.0149 | 0.52            |
| Mg/0.7Al₂O₃       | 1.5          | 1.7548            | 1.7435 ± 0.0140 | 0.64            |
| Mg/1.1Al₂O₃       | 2.5          | 1.7647            | 1.7554 ± 0.0064 | 0.53            |
| Mg/2.5Al₂O₃       | 5.5          | 1.7954            | 1.7845 ± 0.0289 | 0.61            |
| Mg/0.7Al₂O₃ [7]   | 1.5          | –                 | –             | 0.27            |
| Mg/1.1Al₂O₃ [7]   | 2.5          | –                 | –             | 0.09            |

Fig. 1. Representative micrographs showing: 0.3 µm-Al₂O₃ particles distribution pattern and their interfacial integrity in Mg/1.1Al₂O₃ (using SEM & FESEM) in (a) & (b) and grain morphology for Mg and Mg/0.7Al₂O₃ in (c) & (d), respectively.

3. Results

3.1. Synthesis and macrostructural characteristics

Meticulous physical observation of the compacted green and sintered preforms and the extruded rods of monolithic and ODS magnesium did not reveal the presence of any deformation or cracks or any other apparent defects.
Table 2. Results of room temperature mechanical properties of ODS magnesium

| Materials        | Al₂O₃ size (µm) | Hardness | 0.2% YS (MPa) | UTS (MPa) | Ductility (%) |
|------------------|-----------------|----------|---------------|-----------|---------------|
|                  |                 | HR15T    |               |           |               |
| Mg/0.0Al₂O₃     | 43 ± 0          | 37 ± 0   | 111 ± 4       | 188 ± 5   | 7.3 ± 1.1     |
| Mg/0.7Al₂O₃     | 49 ± 1          | 43 ± 0   | 209 ± 2       | 256 ± 5   | 12.4 ± 2.0    |
| Mg/1.1Al₂O₃     | 0.3             | 56 ± 1   | 52 ± 0        | 182 ± 3   | 237 ± 1       |
|                  |                 |          |              |           | 12.1 ± 1.4    |
|                  |                 |          |              |           | (66%)         |
| Mg/2.5Al₂O₃     | 58 ± 0          | 53 ± 1   | 185 ± 2       | 225 ± 3   | 9.9 ± 0.6     |
| Mg/0.7Al₂O₃ [7] | 0.05            | 56 ± 0   | 50 ± 1        | 191 ± 2   | 247 ± 2       |
| Mg/1.1Al₂O₃ [7] |                 | 60 ± 1   | 70 ± 0        | 194 ± 5   | 250 ± 3       |

Density and porosity measurement results for the extruded monolithic and ODS magnesium samples (see Table 1) revealed less than 1% porosity, which indicated the dense material synthesis ability of the parameter used in the processing technique in this study.

3.2. Microstructural characteristics

Microstructural characterization revealed a necklace-type typical homogeneous distribution of the ultrafine oxide particles (see Fig. 1a) with apparent good interfacial integrity (see Fig. 1b). An increasing amount of oxide particle dispersions induced a significantly increasing grain refinement in the magnesium (see Figs. 1c,d and Table 1).

3.3. Mechanical characteristics

Mechanical characterization results showed (see Table 2) that the hardness (both bulk and micro) of magnesium was increased with the dispersion of the increasing amount of oxide particles used in this study.

The results of the room temperature elongation-to-failure tests (see Fig. 2 and Tables 2) revealed that sonication assisted dispersion of ultrafine oxide particles had significantly enhanced the strength, ductility, and limit of energy absorption capacity of the extruded magnesium. It also revealed that the tensile properties of magnesium reached the highest level when dispersed with 0.7 vol.% of oxide particles and gradually decreased with further increment in the vol.% of oxide dispersions. Study of the tensile fracture surfaces revealed that the cleavage-steps representing the relatively brittle mode of fracture of magnesium (see Fig. 3a) transformed into pseudo-dimple (see Fig. 3b) and intergranular crack (see Fig. 3c) due to the presence of oxide particles dispersion, which was indicating the relatively ductile mode of fracture in ODS magnesium.

![Fig. 2. Graph showing tensile stress-strain behavior of monolithic and alumina particles dispersed magnesium.](image)

4. Discussion

4.1. Synthesis of reinforced and monolithic magnesium

Different vol.% of oxide particles were successfully well dispersed into magnesium using sonication [15, 16] and subsequent high extrusion ratio [4]. Finer ceramic particles typically agglomerate into clusters due to the presence of high surface energy induced strong van der Waals attractive forces among themselves. Sonication creates an alternating high and low-pressure waves in the liquid media leading to intense cavitation (i.e., the formation and immediate collapse of small vacuum bubbles) [17], which causes very high-speed impingement of the liquid media on the ceramic particles clusters [18] and deagglomerates them to distribute homogeneously in the space. The oxide ceramic reinforcement particles, along with the mag-
Fig. 3. Representative SEM fractographs showing: (a) brittle cleavage in monolithic magnesium, (b) pseudo-dimple, and (c) intergranular features in the case of 1.1 vol.% alumina particles dispersed magnesium.

Necklace-type distribution of sonication dispersed oxide particles in the ODS magnesium (see Fig. 1a) was observed in this study, and it is typical for externally added ultrafine second phase particles in the powder metallurgy processed metallic material [5, 15]. The tendency of externally added oxide particles agglomeration increased with an increase in their amount, and the apparent reason behind this could be the considerable differences between their initial particles (i.e., $\text{Al}_2\text{O}_3$ was $0.3 \text{ µm}$ and Mg was $60–300 \text{ µm}$). Large size differences between the particles incline the smaller particles (oxide dispersions in this study) to fill the interstitial spaces between the large particles (magnesium powders in this study) during the drying and compaction stages. The high extrusion ratio in the subsequent thermomechanical processing was not effective enough to disperse the clustered interstitial oxide particles of the sintered preform. However, almost zero standard deviation in experimental den-
The significant grain refinement in the processed ODS magnesium (see Figs. 1c,d and Table 1) was considered to be the cumulative effect of the ability of ultrafine oxide particles to nucleate new grains in the magnesium during the recrystallization stage and as well restrict the growth of recrystallized grains by boundary pinning [4, 15, 28] during the grain growth stage. However, the grain refinement ability of externally added oxide particles in magnesium was saturated at a level of 1.1 vol. %, and further increment in their amount became ineffective to enhance the grain refining process proportionally. The limitation was seemingly due to the agglomeration tendency of oxide particles [28]. It should be noted that the grain refinement effect of the submicron size oxide dispersoid used in this study was more effective than that of nanoparticle size oxide dispersoid used earlier [7] (see Table 1).

Minimal porosity in the monolithic and ODS magnesium was corroborated by the experimental density values, as shown in Table 1. It could be attributed to the cumulative effect of enormous compaction pressure, appropriate sintering parameter, and high extrusion ratio used in this study.

4.3. Mechanical characteristics

The hardness of ODS magnesium experienced gradual increment (see Table 2) with the addition of an increasing amount of oxide particles. The increment in the hardness of magnesium can primarily be attributed to the increased resistance to the localized deformation during indentation process due to the presence of an increasing amount of stiffer aluminum oxide particles [4, 5] (elastic modulus is 390 GPa for Al2O3 and 45 GPa for Mg, [1]) and grain boundaries in the microstructure.

The results of the elongation-to-failure tensile test revealed a significant strengthening effect in terms of 0.2% yield strength and ultimate tensile strength of magnesium (see Fig. 2 and Table 2) due to the addition of ultrafine oxide particles. The yield stress of a metallic material is the minimum level of stress required to initiate dislocation motion. It is governed by the dislocation density and magnitude of all the obstacles present in the microstructure to restrict the motion of dislocation under stress.

Synergic effect of the following strengthening mechanisms played a role in the enhancement of yield strength of metal with ultrafine oxide particles dispersoids in the microstructure:

(i) Orowan strengthening mechanism affects the strengthening of metal by dislocation – ‘hard second phase’ interaction and is effective when the dislocation encounters the hard second phase on the slip plane inside the grain. Contribution of this strengthening mechanism in powder metallurgy processed metal is expected to be insignificant [4–10] due to the fact the ‘hard second phase’ (oxide particles dispersion in this study) is mostly located at the grain and/or initial particle boundary.

(ii) Hall-Petch strengthening mechanism affects the strengthening of metal by dislocation – grain boundary interaction and can be predicted by Eq. (1):

\[ \sigma_{HP} = \sigma_0 + K \sqrt{D}, \]

where \( \sigma_0 \) is friction stress, \( K \) is stress direction-dependent constant, and \( D \) is grain size. The Hall-Petch contribution is expected to be significant in the processed ODS magnesium due to the significant grain refinement [11, 12, 14].

(iii) Increased dislocation density strengthening affects the strengthening of metal by increasing the dislocation density, which was created due to the difference in elastic modulus (390 GPa for Al2O3 and 45 GPa for Mg [1]) and coefficient of thermal expansion (7.6 × 10^-6 K^-1 for Al2O3 [1] and 27.045 K^-1 for Mg [29]) between the metal and added second phase (fine aluminum oxide particles in this study). The contribution of increased dislocation density can be predicted by Eq. (2) [30, 31]:

\[ \Delta \sigma = \sqrt{(\Delta \sigma_{EM})^2 + (\Delta \sigma_{CTE})^2}, \]

where \( \Delta \sigma \) represents an increase in the yield strength, \( \Delta \sigma_{EM} \) and \( \Delta \sigma_{CTE} \) are components of strength increment due to elastic modulus (EM) and coefficient of thermal expansion (CTE) mismatch. The components can be equated as:

\[ \Delta \sigma_{EM} = \sqrt{3}\alpha \mu_m b \sqrt{\rho_{EM}} \]

and

\[ \Delta \sigma_{CTE} = \sqrt{3}\beta \mu_m b \sqrt{\rho_{CTE}}, \]

where \( \mu_m \) is metal shear modulus, \( b \) is Burgers vector, and \( \alpha \) and \( \beta \) are two coefficients for dislocation strengthening.

\( \rho_{EM} \) is dislocation density from elastic modulus mismatch and equals to \( 4\gamma/\lambda b^2 \) [32], where \( \gamma \) is plastic shear, \( b \) is Burgers vector, strain and \( \lambda \) is local deformation field length related to the inter-particle dis-
Table 3. Specific strength and work of fracture of ODS magnesium

| Materials          | Al₂O₃ size (µm) | Work of fracture (J m⁻³)* | σ₀.2%YS/ρ (kN/m kg⁻¹) | σUTS/ρ (kN/m kg⁻¹) |
|--------------------|-----------------|---------------------------|-----------------------|--------------------|
| Mg/0.0Al₂O₃       | 12.0 ± 1.1      | 64                        | 108                   |
| Mg/0.7Al₂O₃       | 28.2 ± 4.9      | 119                       | 146                   |
| Mg/1.1Al₂O₃       | 25.0 ± 3.3      | 103                       | 134                   |
| Mg/2.5Al₂O₃       | 19.2 ± 1.1      | 103                       | 126                   |
| Mg/0.7Al₂O₃ [7]   | 19.9 ± 3.9      | 109                       | 141                   |
| Mg/1.1Al₂O₃ [7]   | 15.5 ± 2.6      | 110                       | 142                   |

* Determined from engineering stress-strain diagram using EXCEL software.

The work of fracture for a metal expresses its ability to absorb energy up to fracture under the applied tensile stress, computed using a stress-strain diagram, enhanced significantly (see Table 3) due to the dispersion of oxide particles following the same trend of ductility of the processed ODS magnesium. In essence, the sonicated dispersion of oxide particles made commercially pure magnesium eligible for both strength-based design (higher yield strength when compared to monolithic magnesium) and damage tolerant design (higher work of fracture when compared to monolithic magnesium). It has to be noted that the improvement of ductility and work of fracture of submicron size oxide dispersions containing magnesium in this study was found to be significantly higher than the ductility and work of fracture of an equivalent amount of nanometer size oxide dispersions containing magnesium reported earlier (see Tables 2, 3).

5. Conclusions

1. The sonication process was effective in the dispersion of ultrafine aluminum oxide particles in magnesium metal processed by powder metallurgy technique.

2. Homogeneous distribution of the oxide particles,
significant grain refinement, and presence of minimal porosity in the microstructure indicated the appropriateness of the sonication, compaction, sintering, and extrusion parameters used in this study.

3. Well-dispersed ultrafine oxide particles were effective in the improvement of hardness, yield strength, ultimate tensile strength, ductility, and work of fracture of commercially pure magnesium metal.

4. Ultrafine oxide particles effectively transformed the brittle mode of fracture in monolithic magnesium to ductile mode of fracture in ODS magnesium.

5. 0.7 vol.% of ultrafine aluminum oxide was found to be most effective in the improvement of the overall mechanical properties of magnesium.

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