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Research article

Mechanical properties and wear characterization of Al-Mg composites synthesized at different temperatures

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Abstract: Reactive sintering of Al-Mg powder mixtures containing 5, 10, 15, and 20 wt.% Mg was used to synthesize lightweight composites reinforced with in-situ formed Al_3Mg_2 and $Al_{12}Mg_{17}$ intermetallics. Detailed microstructural investigation and phase analysis were employed to examine the phases in the composites formed at 400 and 450 °C. The creation of particles with the $Al_{12}Mg_{17}$ cores encapsulated by the Al_3Mg_2 phase, which was further covered by a continuous aluminum matrix, was observed in the composites synthesized at 400 °C. If the composites were held at 450 °C, the liquid phase appeared at the Al-Mg interface, and as a result, a two-phase mixture was formed. It was the eutectic composed of the Al_3Mg_2 intermetallic compound and a solid solution of magnesium in aluminum (Al). The introduction of magnesium particles into the aluminum matrix resulted in a decrease in the density of composites, but there was no significant difference in the density of composites sintered at different temperatures. The mechanical behavior of the composites was examined using microhardness and hardness measurements and a room-temperature compression test. The result of using different cooling speeds, with the furnace and quenching in water, was the refining of the grains in the $Al_3Mg_2 + $ (Al) eutectic, resulting in an increase in microhardness. The increase in hardness of the composites was related to the amount of particles introduced. Sintering at 450 °C and the cooling method influenced the hardness and compressive strength of the composites, which were higher by 10% and 13%, respectively, compared to composites sintered at 400 °C. Tribological tests showed that introducing more and more magnesium particles into the aluminum matrix, followed by reactive sintering, increased the wear resistance. On the other hand, the sintering temperature and cooling conditions had little effect on the wear resistance of the Al-Mg composites.

1. Introduction

Aluminum and its alloys have attracted considerable attention, primarily because aluminum is a very common element and one of the lightest structural metals. Low-density aluminum-magnesium alloys and composites with excellent machinability and satisfactory recyclability are evolving as potential replacements for conventional materials in the automotive, aerospace, and electrical industries [1,2]. 5xxx series alloys (containing up to 6.2% Mg by weight) are the most resistant to corrosion (up to 3 times more resistant than the most commonly used 6xxx series alloys) of all aluminum alloys [3]. They are characterized by their very good suitability for mechanical processing and welding. The strength of welded structures made of 5xxx series alloys is slightly lower compared to the strength of structures made of bulk materials. An increase in the Mg content results in an increase in strength properties, but this is associated with a decrease in the deformability of the material and a lower suitability for hot plastic working [4]. Varied particulate-reinforced [5–7], fiber-reinforced [8], and laminated [9,10] aluminum matrix composites have been fabricated. There is no doubt that the properties of the composites depend strongly on the volume fraction, shape, and morphology of the reinforcement. Particulate-reinforced metal matrix composites (MMCs) are attractive due to their easier processing and lower costs compared to other types of composites. Various particulate reinforcements (ceramics, metallic glasses, quasi-crystals, and intermetallics) have been used to produce aluminum matrix composites with improved properties through friction stir forming [11], casting [12], or powder metallurgy [5,7]. The main advantage of powder metallurgy is that it provides very good control over the size, shape, distribution, and volume fractions of reinforcing particles in the matrix. Metallic reinforcements have several advantages over conventional reinforcing ceramic particulates, e.g., metal oxides (Al_2O_3, SiO_2), carbides (SiC, TiC), nitrides (TiN, BN), or borides (TiB_2, ZrB_2), including easy availability and improved recyclability [6]. They also have better compatibility with a metallic matrix, which may create a stronger interface between reinforcement and matrix. The possible reaction between the reinforcement and the matrix, called reactive sintering, can produce new phases (solid solutions or intermetallics) during processing or subsequent heat treatment. The mechanical properties of sintered composites depend on the volumetric changes associated with the transformation, the type of the new phases, and their bonding with the matrix [13]. The degree of interfacial reaction between the reinforcement and the matrix and the type of reaction products can be controlled by appropriate control of the sintering time and temperature, which depend on diffusion. Phase transformations due to the reaction between aluminum matrix and magnesium particulate reinforcements can lead to the formation of two intermetallics, $Al_{12}Mg_{17}$ and Al_3Mg_2 [5]. Unfortunately, these intermetallics belong to the brittle and hard phases and have poor resistance to plastic deformation, which makes them easy to crack under external force [14–16]. During literature studies, a knowledge gap was noticed regarding the influence of the cooling rate of sintered composites from the Mg-Al system on their phase composition, grain size and properties. In this work, an attempt was made to synthesize Al-Mg composites reinforced with $Al_{12}Mg_{17}$ and Al_3Mg_2 intermetallics formed in situ during the reactive sintering of aluminum and magnesium elemental powder mixtures. The main aim was to analyze the effect of the initial weight percent of magnesium, sintering temperature, and

cooling conditions on the microstructural modification and the resulting density, hardness, compressive strength, and wear resistance of the composites.

2. Materials and methods

In this experiment, commercially available aluminum (99.74 at.% Al) and magnesium (99.53 at.% Mg) powders were used. Preliminary tests showed that in order to achieve complete conversion of magnesium particles during sintering at the temperatures and times used in the tests, they had to be smaller than 40 mm in size. Therefore, the average grain size of the used powders was less than 30 mm. The shapes and arrangements of the powder particles are shown in Figures 1 and 2.
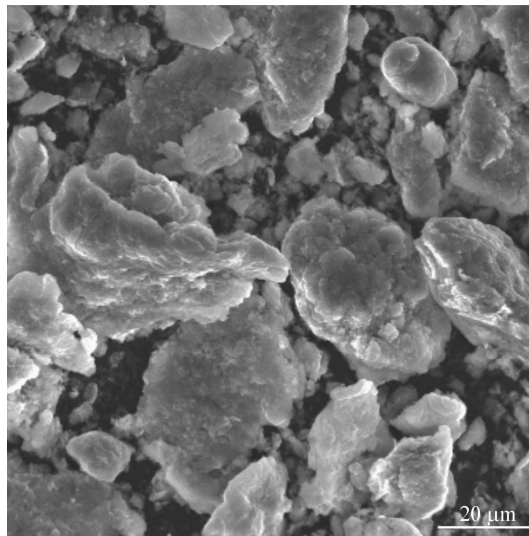

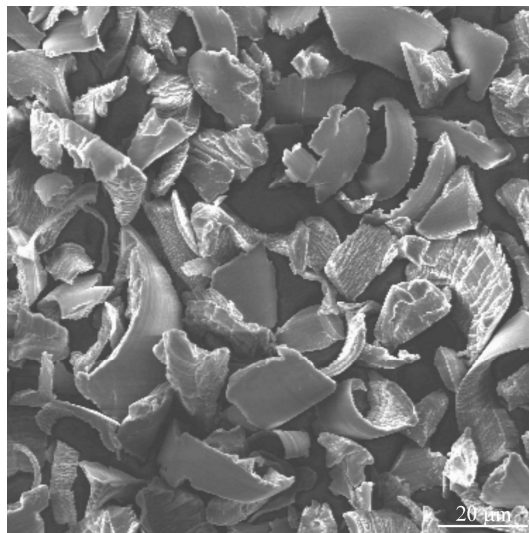**Figure 1.** Scanning electron micrograph of aluminum powder.

Figure 2. Scanning electron micrograph of magnesium powder.

Powder mixtures with different amounts of Mg (5, 10, 15, and 20 wt.%) and pure aluminum powder were blended using a Retsch PM 100 planetary mill with steel balls. The powders were subjected to milling in an argon atmosphere for 0.5 h by using a ball-to-powder ratio (BPR) of 10:1 and 120 rpm as rotation speed. To avoid a possible temperature rise, the blending process was carried out in two steps for 0.25 h, with a rest interval of 0.25 h. The cylindrical specimens, 20 mm in diameter and 10 mm high, made from pure aluminum powder and prepared powder mixtures, were subjected to single-track pressing on a hydraulic press at a compaction pressure of 624 MPa. The green compacts were sintered at two different temperatures, 400 or 450 °C, in a vacuum furnace for 0.5 h by applying uniaxial pressure of 5 MPa, and then the furnace was cooled. In order to investigate the effect of undercooling on the dispersion of solidifying phases formed at 450 °C, some samples were held at 450 °C for 0.5 h and then water quenched. Finally, all samples were consolidated at 300 °C in a vacuum furnace for 0.5 h at a pressure of 10 MPa. Samples intended for microscopic examination were cut transversely using a low-speed diamond saw and then embedded in resin. Then, they were ground with sandpaper with a grit of up to 1200 and polished with a diamond suspension with a grain size of 1 mm. The microstructure analyses of the prepared metallographic specimens were performed using a Nikon Eclipse MA200 optical microscope and a JEOL JMS 5400 scanning electron microscope (SEM). Before the samples were examined with the optical microscope, they had been etched to reveal the grains of aluminum-based compounds using a solution of 0.5 mL HF and 99.5 mL H_2O. The chemical composition of the reaction regions was investigated by energy dispersive X-ray analysis (EDX) using an ISIS 300 (Oxford Instruments). The composition of the phases was determined by comparing the results of the microprobe analysis with the data in the binary Al-Mg phase diagram [17]. In addition, X-ray diffraction using a D/max RAPID2 diffractometer was employed to confirm the presence of the phases. The fabricated materials were subjected to density, hardness, compression, and wear tests. The density of sintered materials was determined by weighing the specimens in air and water using WPA120 hydrostatic scales in line with the EN ISO 2738:2001 standard. The hardness of the materials was measured using the Vickers microhardness method (HV0.1) in accordance with the EN ISO 6507-1:2023 standard and the Brinell method with a sintered carbide ball of 5 mm in diameter at a load of 613 N in accordance with the EN ISO 6506-1:2014 standard. Cylindrical samples of 20 mm in diameter and 10 mm in length were prepared from the sintered materials and then tested at room temperature under quasi-static compression using an Amsler screw machine at a constant crosshead speed of 0.1 mm/min. The tribological tests were performed using a device with a roll-on-block configuration. The roll of Φ35 mm × 9 mm was made of hardened 100Cr6 steel with a hardness of 62 HRC. The blocks, with dimensions of 6 mm × 8 mm × 15 mm, were made of sintered materials. The tests were performed under dry friction conditions with a rotational speed of 130 rpm and a load of 5 N. The loss of weight was measured with an accuracy of 0.0001 g every 10 min during the test, which lasted 1 h. The total path of friction was 864 m. The reported data for all tests are the average values of three tested specimens.

3. Results and discussion

3.1. Microstructural investigations

The microstructural analysis of the sintered materials was performed by an optical microscope and a scanning electron microscope equipped with a system for microprobe analysis. The main aim of

these examinations was the identification of the chemical composition and dispersion of synthesized phases. Figure 3 shows the exemplary microstructures of the composites sintered at 400 and 450 °C.
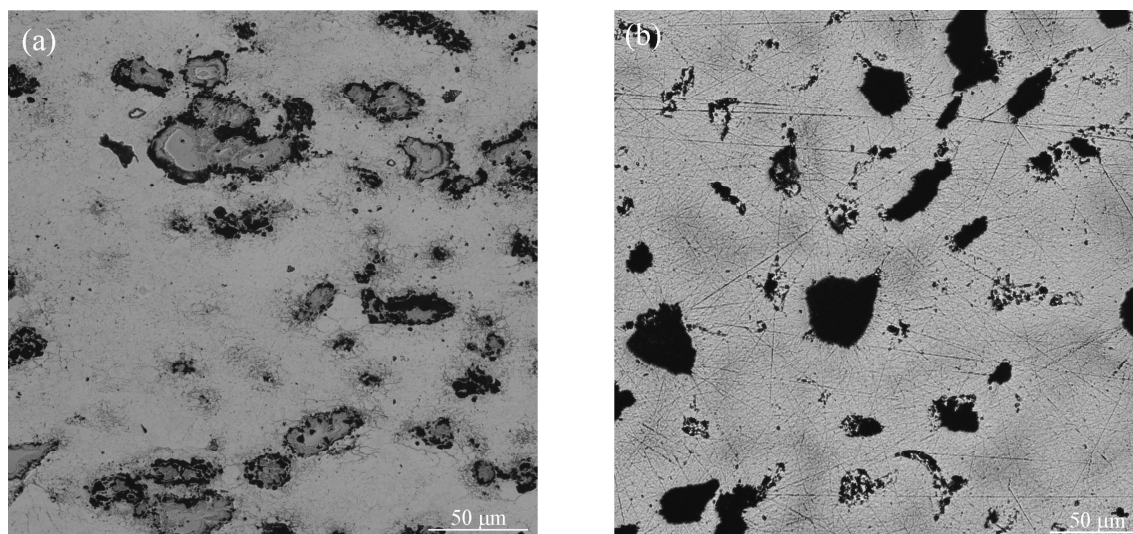

Figure 3. Optical microscope micrographs of the composites containing 10 wt.% Mg sintered at (a) 400 and (b) 450 °C.

The microstructure of the composite (with only one particle and the aluminum matrix) that was annealed at 400 °C for 0.5 h is shown in Figure 4. The following chemical composition of the region marked A, formed close to the aluminum side, was determined using SEM and EDX analysis: 59.24 at.% Al and 40.76 at.% Mg. The result indicates, according to the Al-Mg diagram [17], the development of the Al_3Mg_2 intermetallic compound.

X-ray microprobe analysis of the region marked B in the central part of the particle revealed a chemical composition of 41.11 at.% Al and 58.89 at.% Mg, which suggests that an $Al_{12}Mg_{17}$ compound was formed. The creation of particles with the $Al_{12}Mg_{17}$ cores encapsulated by the Al_3Mg_2 phase, which is further covered by a continuous aluminum matrix, is in compliance with the previously reported results [2,5] obtained during the processing of Al-Mg composites. If the composites were held at 450 °C, the liquid phase appeared at the Al-Mg interface. After 0.5 h of reaction and cooling with the furnace, the dominant part of the particles' microstructure was a two-phase mixture (Figure 5). A narrow layer adjacent to the aluminum (marked C in Figure 5) and the mixture (D + E) can be distinguished. The chemical composition of the layer marked C was as follows: 99.18 at.% Al and 0.82 at.% Mg, which was considered as a layer of magnesium solid solution in aluminum.
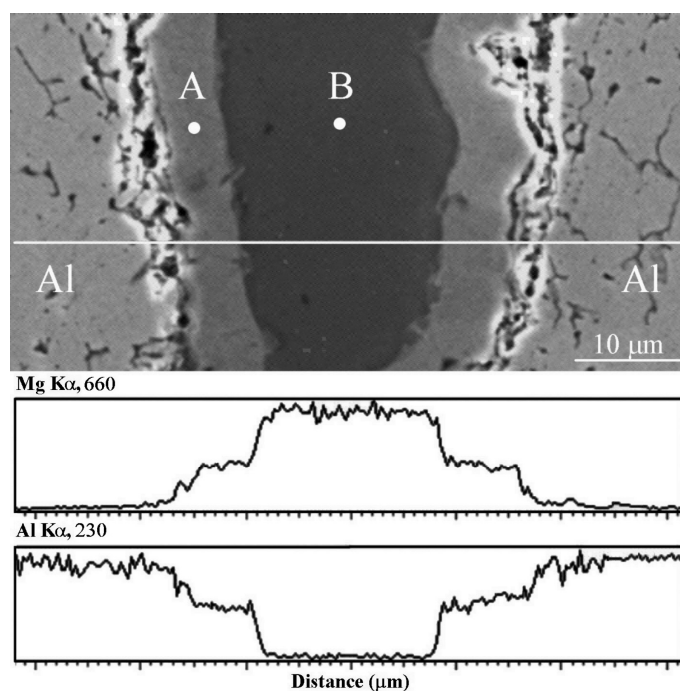
Figure 4. The SEM image and EDX elemental concentration profiles for the particle-matrix region of the composite sintered at 400 °C.
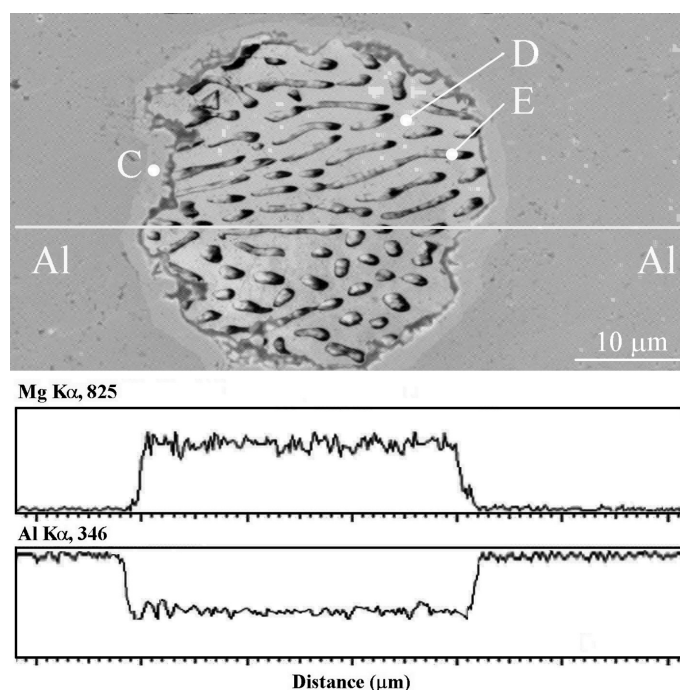

Figure 5. The SEM image and EDX elemental concentration profiles for the particle-matrix region of the composite sintered at 450 °C and then furnace cooled.

The chemical composition of the particle was investigated separately in two phases. The chemical composition of the lighter phase (marked D) was 99.04 at.% Al and 0.96 at.% Mg, which was considered

as a solid solution enriched with magnesium. The composition of the darker phase (marked E) was as follows: 60.11 at.% Al and 39.89 at.% Mg. The received results suggested the presence of the Al_3Mg_2 intermetallic compound. On the basis of the Al-Mg diagram [17], it can be concluded that the two-phase mixture was the eutectic composed of the Al_3Mg_2 intermetallic compound and a solid solution of magnesium in aluminum (Al). The growth activation energies of Al_3Mg_2 and $Al_{12}Mg_{17}$ are 86.18 and 115.33 kJ/mol, respectively. Due to the fact that the growth activation energy of Al_3Mg_2 is smaller than that of $Al_{12}Mg_{17}$, the growth rate of Al_3Mg_2 is faster [18]. As a result of the reaction at 450 °C, this is probably the reason that Al_3Mg_2 is the only intermetallic compound occurring in the eutectic. Similar results were obtained for composites sintered at 450 °C and then quenched in water. The only difference was the grain size refining (Figure 6). What is important is that in the quenched samples, there were not observed cracks produced by possible quenching stresses.
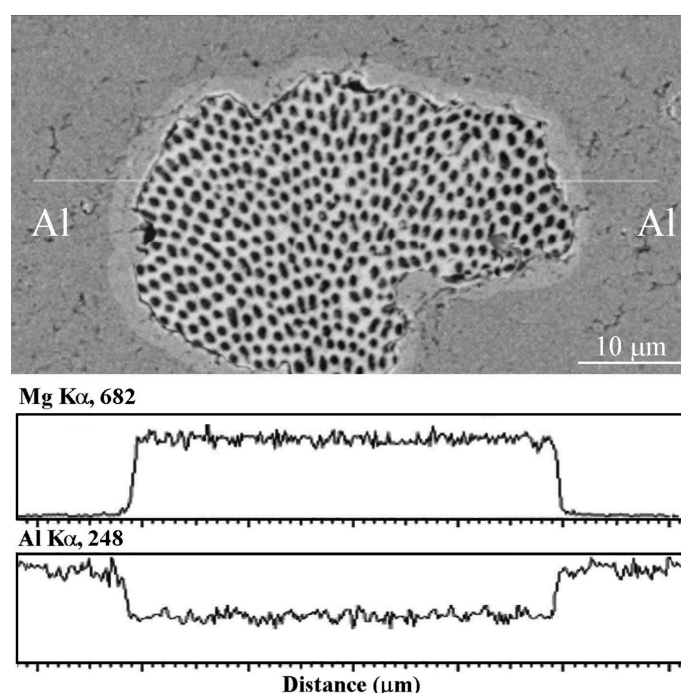

Figure 6. The SEM image and EDX elemental concentration profiles for the particle-matrix region of the composite which was sintered at 450 °C and then quenched in water.

3.2. Density measurements

The results of density measurements for all sintered materials are presented in Table 1. The abbreviations mean the following: FC—furnace-cooled, Q—quenched in water.

The examinations showed that the introduction of magnesium particles into the aluminum matrix resulted in an obvious decrease in the density of composites. It was also found that sintering at a temperature of 450 °C led to a reduction in porosity, especially at the particle-matrix interfaces. This, on the other hand, led to an increase in the density of composites sintered involving the liquid phase compared to composites sintered in the solid state. There was no significant difference in the density of composites sintered at 450 °C and then furnace cooled or quenched in water. The actual densities of all sintered materials ranged from 90% to 94% of the theoretical density.

Table 1. Results of density measurements of sintered materials.

Material	Sintering temperature (°C)	Density (g/cm^3)
Al	400 °C	2.43 ± 0.02
Al + 5 wt.% Mg	400 °C	2.42 ± 0.01
Al + 10 wt.% Mg	400 °C	2.35 ± 0.01
Al + 15 wt.% Mg	400 °C	2.29 ± 0.02
Al + 20 wt.% Mg	400 °C	2.23 ± 0.01
Al	450 °C	2.48 ± 0.01
Al + 5 wt.% Mg (FC)	450 °C	2.46 ± 0.02
Al + 10 wt.% Mg (FC)	450 °C	2.40 ± 0.02
Al + 15 wt.% Mg (FC)	450 °C	2.34 ± 0.01
Al + 20 wt.% Mg (FC)	450 °C	2.27 ± 0.02
Al + 5 wt.% Mg (Q)	450 °C	2.46 ± 0.01
Al + 10 wt.% Mg (Q)	450 °C	2.41 ± 0.01
Al + 15 wt.% Mg (Q)	450 °C	2.33 ± 0.02
Al + 20 wt.% Mg (Q)	450 °C	2.28 ± 0.01

3.3. Mechanical properties

Microhardness measurements were performed for aluminum matrix as well as for intermetallics and eutectic mixtures formed after furnace cooling and quenching in water for all specimens (containing 5, 10, 15, and 20 wt.% Mg). The results of the microhardness measurements were averaged and are given in Table 2.

Table 2. Results of microhardness measurements.

Material	HV0.1	Cooling conditions
Al matrix	52	Furnace cooling
Al_3Mg_2	342	Furnace cooling
$Al_{12}Mg_{17}$	258	Furnace cooling
Eutectic mixture Al_3Mg_2 + (Al)	188	Furnace cooling
Eutectic mixture Al_3Mg_2 + (Al)	265	Water quenching

The increase in solidification rate of the eutectic mixture resulted in refined microstructure (Figures 5 and 6), and therefore a direct relationship between cooling conditions and the microhardness of particles could be observed. Brinell hardness measurements were performed for all investigated materials, and the results are given in Table 3.

Table 3. Results of hardness measurements and compressive strength of sintered materials.

Material	Sintering temperature (°C)	Brinell hardness (HB)	Compressive strength (MPa)
Al	400 °C	19 ± 1.5	184 ± 4
Al + 5 wt.% Mg	400 °C	22 ± 1.7	218 ± 7
Al + 10 wt.% Mg	400 °C	25 ± 1.3	231 ± 5
Al + 15 wt.% Mg	400 °C	31 ± 1.5	260 ± 8
Al + 20 wt.% Mg	400 °C	38 ± 1.8	295 ± 4
Al	450 °C	22 ± 1.4	192 ± 8
Al + 5 wt.% Mg (FC)	450 °C	23 ± 1.6	220 ± 8
Al + 10 wt.% Mg (FC)	450 °C	27 ± 1.5	241 ± 3
Al + 15 wt.% Mg (FC)	450 °C	33 ± 1.2	279 ± 9
Al + 20 wt.% Mg (FC)	450 °C	39 ± 1.6	312 ± 7
Al + 5 wt.% Mg (Q)	450 °C	24 ± 1.3	227 ± 9
Al + 10 wt.% Mg (Q)	450 °C	29 ± 1.5	254 ± 4
Al + 15 wt.% Mg (Q)	450 °C	35 ± 1.6	298 ± 6
Al + 20 wt.% Mg (Q)	450 °C	42 ± 1.7	334 ± 5

The examination showed that the introduction of magnesium particles combined with reactive sintering resulted in an increase in the hardness of the composites compared to sintered aluminum. The variations in the microhardness and hardness of Al-intermetallic composites depended on the sintering temperature and cooling conditions, as shown in Tables 2 and 3. Rapid water quenching of the composites sintered at 450 °C resulted in a 41% microhardness increase of the eutectic mixture with respect to the furnace-cooled one. This is attributed to the dispersion of Al_3Mg_2 and (Al) phases in the formed particles, which enhances the Hall–Petch strengthening effect [6]. When the interaction between the hardness and the microstructure is discussed, many factors should be considered in order to completely understand the strengthening mechanisms [7,14]. These factors include reinforcement ratio, reinforcement type, sintering temperature, and cooling condition. The increase in hardness was related to the amount of particles introduced. An increase in hardness with an increase in the amount of magnesium particles was also reported by Shahid et al. [5] and Tosun et al. [7]. Composites sintered with a liquid phase had slightly higher hardness, especially those that were rapidly cooled. Rapid water quenching of the composites sintered at 450 °C containing 20 wt.% Mg resulted in a 10% increase in hardness in comparison to the composites furnace cooled. Furthermore, such composites were more than twice as hard as sintered aluminum. This effect is attributed to the higher hardness values of $Al_{12}Mg_{17}$ and especially Al_3Mg_2 as compared to the Al matrix. This also corresponds to the decrease in the interparticle distance with an increase in the amount of reinforcement, as represented by the Eq 1:

$$l = d_r \left[\left(\frac{\pi}{6V_f} \right)^{\frac{1}{3}} - 1 \right] \tag{1}$$

where l, d_r, and V_f, are the inter-particle spacing, radius of the particle, and volume fraction of reinforcement, respectively [19]. It was also seen that the hardness of all the composites increases with an increase in the sintering temperature from 400 to 450 °C. This is due to an increase in the relative

density and the formation of particles containing the Al_3Mg_2 intermetallic phase. The changes in microstructure affect the mechanical properties of composites. The hardness of composites increased because the secondary phases were observed in the microstructure. The Al_3Mg_2 intermetallic phase was formed and precipitated at the grain boundaries of (Al) solid solution. When the sintering temperature was increased from 400 to 450 °C, the denser material was obtained due to the decrease in the number of pores, and the hardness increased. It was observed that there was strong interfacial bonding between particles and matrix (Figures 5 and 6), and there were no visible traces of any porosity or inclusions in the interfacial region. Therefore, it is considered that the increase in hardness was the sum of three strengthening mechanisms: solution strengthening (after the diffusion of some magnesium into the aluminum matrix), the effect of Al_3Mg_2 phase precipitations occurring at the grain boundaries, and reinforcement with particles of eutectic mixture. Figure 7 shows the exemplary room-temperature compressive stress-strain curves for the composites containing 20 wt.% Mg sintered at 400 and 450 °C.
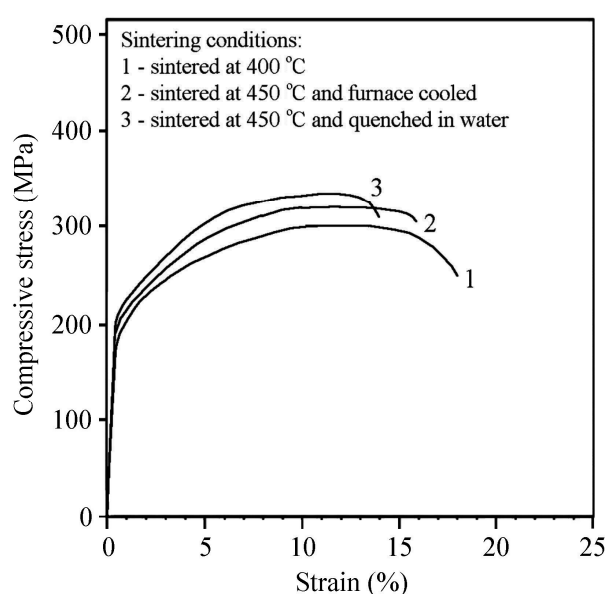

Figure 7. The compressive stress-strain curves for the composites containing 20 wt.% Mg.

The introduction of an increasing amount of magnesium particles combined with reactive sintering resulted in an increase in the yield strength of the sintered composites. Under compressive loading, according to Kim et al. [20], the reinforcement particles move in the loading direction to get closer to each other, and deformation takes place mainly in the matrix. Therefore, the plastic deformability of the sintered composites decreased with an increase in the amount of intermetallic particles. The composites sintered with a liquid phase had generally higher compressive strength than the composites sintered at 400 °C (Table 3). Rapid water quenching of the composites sintered at 450 °C containing 20 wt.% Mg resulted in a 13% increase in compressive strength in comparison to the composites sintered at 400 °C and an 82% increase in comparison to the compressive strength of sintered aluminum. Conversely, the plastic deformability of the composites decreased from 18.5% for composites sintered at 400 °C to 16% for composites sintered at 450 °C and furnace cooled and to 14% for composites quenched in water. The compressive strength of the composites increased with increasing the total amount of intermetallics. On the other hand, a smaller volume fraction of reinforcement leads to a larger distance between reinforcement particles, and deformation takes place

mainly in the matrix [5,7,12]. When the sintering temperature was increased from 400 to 450 °C, the denser material was obtained, and the compressive strength increased. The main reason for this phenomenon was that there was strong interfacial bonding between particles and the matrix. Similarly to the increase in hardness, three strengthening mechanisms were responsible for the increase in compressive strength: solution strengthening, the influence of Al_3Mg_2 phase particles at the grain boundaries, and strengthening with particles of the eutectic mixture. These results are in line with the data reported by Scudino et al. [21] for Al-based composites reinforced with b-Al_3Mg_2 particles. Results indicate that lightweight Al-Mg composites with a wide range of strength and plastic deformation can be successfully synthesized by sintering at different temperatures and with different cooling conditions.

3.4. Wear characterization

The samples for the wear tests were weighed before and after every 10 min during the test, which lasted 60 min. The wear mass loss of the investigated sintered materials gradually increased with increasing wearing time and sliding distance (Figure 8). Tribological tests showed that introducing more and more magnesium particles into the aluminum matrix, followed by reactive sintering, increases the wear resistance. The order of the wear mass loss in the experiment could be ranked as follows: sintered Al < composites sintered at 400 °C < composites sintered at 450 °C quenched in water < composites sintered at 450 °C furnace cooled. The smallest wear mass loss after 60 min was found for the composites containing 20 wt.% Mg sintered at 450 °C and then furnace cooled, and it was found to be about 30% smaller than for sintered aluminum. Nevertheless, the wear mass loss for all composites containing the same amount of magnesium particles was comparable and differed by less than 10%.
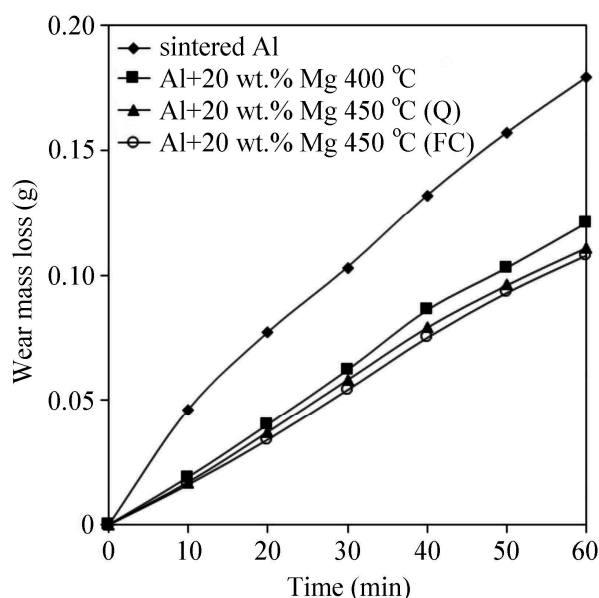

Figure 8. The wear mass loss with testing time for sintered aluminum and the composites containing 20 wt.% Mg sintered at 400 and 450 °C and then furnace cooled or quenched in water.

Figure 9 shows examples of the worn surfaces for the composites containing 20 wt.% Mg sintered at 400 and 450 °C and then furnace cooled or quenched in water after wear testing for 60 min until a sliding distance of 864 m.
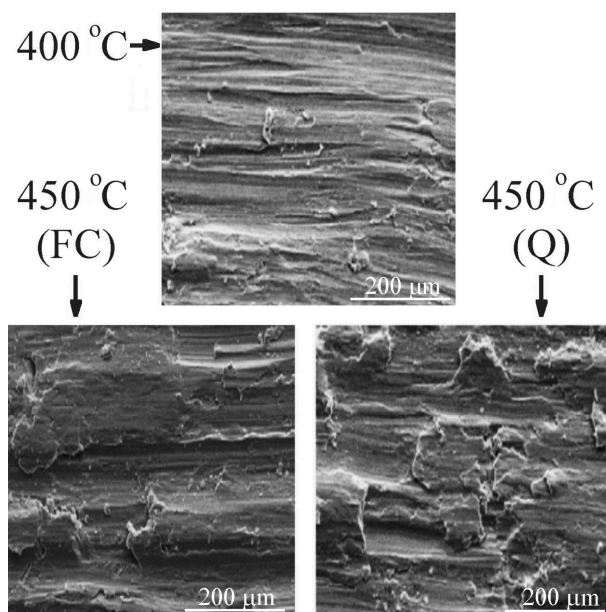

Figure 9. Morphologies of the worn surfaces of tested specimens made of the composites containing 20 wt.% Mg sintered at 400 and 450 °C and then furnace cooled or quenched in water.

The worn surface of the composite sintered at 400 °C was rather smooth, accompanied by slight scratches showing evidence of plastic deformation. The worn surfaces of the composites sintered at 450 °C looked slightly different. They exhibited negligible signs of plastic deformation, and a small amount of debris stuck along the sliding direction. Also, locally, mini-cracked surface layers were present. This was probably related to a better connection of particles with the aluminum matrix, which was created during the formation of composites using the liquid phase. In this case, the particles were torn out together with pieces of the matrix. This type of behavior is typical for aluminum matrix composites and was described in detail by Bodunrin et al. [6].

4. Conclusions

The reactive sintering of Al-Mg powder mixtures was used to synthesize lightweight composites reinforced with in-situ formed Al_3Mg_2 and $Al_{12}Mg_{17}$ intermetallics. Detailed microstructural investigation and phase analysis were employed to examine the phases in the composites formed at 400 and 450 °C. The creation of particles with the $Al_{12}Mg_{17}$ cores encapsulated by the Al_3Mg_2 phase, which was further covered by a continuous aluminum matrix, was observed in the composites synthesized at 400 °C. If the composites were held at 450 °C, the liquid phase appeared at the Al-Mg interface, and as a result, the eutectic composed of the Al_3Mg_2 intermetallic compound and a solid solution of magnesium in aluminum (Al) was formed. The mechanical behavior of the composites was examined using microhardness and hardness measurements and a room-temperature compression test.

The sintering temperature and cooling method influenced the hardness and compressive strength of the composites. Composites sintered with a liquid phase had slightly higher hardness, especially those that were rapidly cooled. Rapid water quenching of the composites sintered at 450 °C containing 20 wt.% Mg resulted in a 10% increase in hardness in comparison to the composites furnace cooled or sintered at 400 °C. Rapid water quenching of the composites sintered at 450 °C containing 20 wt.% Mg resulted in a 13% increase in compressive strength increase in comparison to the composites sintered at 400 °C and an 82% increase in comparison to the compressive strength of sintered aluminum. Tribological tests showed that introducing more and more magnesium particles into the aluminum matrix, followed by reactive sintering, increases the wear resistance. The smallest wear mass loss was found for the composites containing 20 wt.% Mg sintered at 450 °C and then furnace cooled, and it was found to be about 30% smaller than for sintered aluminum. Nevertheless, the sintering temperature and cooling conditions had little effect on the wear resistance of the Al-Mg composites.

Use of AI tools declaration

The author declares that no Artificial Intelligence (AI) tools were used in the creation of this article.

Conflict of interest

The author declares no conflict of interests.

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