Article

Effect of Semi-Solid Processing on the Microstructure and Mechanical Properties of Aluminum Alloy Chips with Eutectic Mg₂Si Intermetallics

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Abstract: This study reports the microstructural changes and mechanical properties of high-strength aluminum alloy chips prepared in the semi-solid state at different temperatures, pressures, and holding times. In semi-solid processes, these processing parameters must be optimized because they affect the microstructure and mechanical properties of the chips. In microstructural analysis, these parameters clearly influenced the spheroidization of the aluminum matrix. The aluminum matrix was uniformly spheroidized after semi-solid processing, and the densities of the final samples increased with the holding time. After 30 min holding time at a given temperature, the density approached the theoretical density, but the compressive strength of the samples seriously deteriorated. Meanwhile, fracture surface investigation revealed a deformed Mg₂Si phase, which is formed through a eutectic reaction. The strength of this phase significantly decreased after increasing the holding time of the semi-solid processing from 10 to 30 min. Therefore, deformation of the Mg₂Si phase caused by diffusion of aluminum into this phase can be a key factor for the decrease in the mechanical properties of samples fabricated with 30 min holding time.

Keywords: high-strength aluminum alloy; semi-solid; microstructure; mechanical properties; fracture surface

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1. Introduction

The semi-solid process (SSP) offers several advantages in industry: short solidification times, low shrinkage, low forming temperatures, and a simple production process [1,2]. Recently, the SSP has been widely researched and applied in lightweight industries and is especially effective for producing lightweight automotive parts with excellent mechanical properties [3].

The main requirement for semi-solid (SS) state formation of alloys is the elimination of the dendritic structure during the solidification process. When the connected grains of a dendritic structure are eliminated, a spheroidal or non-dendritic structure is formed. This process takes place at a temperature between the solidus and the liquids. The microstructure of the alloy must be controlled by considering each variable in the SSP because the time, pressure, and temperature of the SSP influence the spheroidization process (breakage of the dendritic arms to create the spheroidal structure).

SSP-formed alloys with a spheroidal microstructure can produce high-quality casting parts with superior mechanical properties, lower segregation, and fewer other casting defects than in conventional casting [4,5]. In addition, it is known that alloys with a spheroidal microstructure can be easily fabricated by a technology involving thixo forming, rheo forming, and spray forming. Salleh et al. reported that alloys fabricated by thixo forming can reduce macro-segregation, porosity, and forming forces in the shaping process

and can produce a variety of products, especially for the automotive industry [3]. Ivanchev et al. reported that rheo-forming technology is being developed as the demand for quality improvement and weight reduction of parts in the manufacturing industry is increasing, and this technology can minimize casting defects and increase elongation [6]. Malekan et al. found lower segregation and solidification shrinkage in the fine spheroidal structure obtained by the SSP than by conventional casting and solid-forming methods [7]. Judging from these results, parts manufactured by the SSP should possess higher mechanical properties than those manufactured by conventional casting. In fact, many studies have reported the high mechanical properties of products manufactured by the SSP [8–10]. In addition, research on improving the mechanical properties of aluminum (Al) alloys through the SSP is also proceeding steadily, and the mechanical properties of Al alloys have been improved mainly through the Al–Si [11–13], Al–Zn [14,15], and Al–Cu [16,17] alloy series. Unlike conventional SSP studies in which liquid or bulk-type materials are mainly used, Al chips are used in this study in order to investigate the effect of pressure and temperature on SSP structures, and there are few reports on the formation of semi-solid slurries using Al chips [18]. The present study aims to manufacture a high-strength aluminum alloy with improved mechanical properties through the SSP. The Al chips used in this study are made from the Al alloy developed by Kim et al. [19,20]. The alloy contains eutectic Mg₂Si particles, which are considered to be the main reinforcement phase to increase mechanical properties. The SSP was run at different pressures, holding times, and temperatures, and the microstructure and mechanical properties of the resulting alloy were characterized. The compressive strengths, fracture behaviors, and Vickers hardness of the samples fabricated at different holding times were obtained and discussed, along with their microstructures. During the SSP, the eutectic Mg₂Si particles showed deformation and fracture behavior with prolonged holding, and it was confirmed that the behavior of eutectic Mg₂Si particles has a significant effect on the mechanical properties.

2. Materials and Methods

The raw matrix material was Al–8Zn–6Si–4Mg–2Cu wt% alloy with the following composition: commercial-purity Al (99.99%; Nantong Tade Electronic Materials Technical Co., Ltd., Nantong, Jiangsu, China), pure Cu (99.997%; RND Korea Co., Gwangmyeong, Korea), pure Zn (99.99%; Sichuan Lande Industry Company Ltd., Chengdu, China), pure Mg (99.9%; Luoyang Hualing Magnesium Co., Ltd., Luoyang, Henan, China), and commercial crystalline Si (99.9%; Hoshine Silicon Co., Ltd., Pinghu, China). The alloy was processed into chips of length 3 mm and width 1 mm in a crushing apparatus. The chips (8 mg) were placed into a graphite mold to form the alloy. A hot press furnace (HPF; Jungmin Industrial Co. Ltd., Vacuum Hot Press, Bucheon, Korea) capable of uniaxial pressuring was used as the furnace for manufacturing the samples. The K-type thermocouple was used to measure the temperature inside the furnace. The behaviors of the matrix manufactured at various pressures, temperatures, and holding times are described below and summarized in Table 1.

Group A: To optimize the SSP, the holding time and pressure were fixed at 30 min and 80 MPa, respectively, and the final processing temperature was varied as 550, 560, 570, and 580 °C. The best process temperature for the SSP was 570 °C. When the process temperature was reduced to 550 and 560 °C, the Al matrix retained its dendritic structure, whereas the higher temperature (580 °C) exceeded the SS temperature, so the Al matrix was not spheroidized.

Group B: Process temperatures were set at 550, 560 and 570 °C based on the optimum SSP. The process of group B increased the temperature to 450 °C and holding for 10 min. After 10 min holding, the pressure was reduced from 80 MPa to 0 MPa (PI process). The temperature was then increased to the 3 different temperatures, 550, 560 and 570 °C. The Al matrix was spheroidized at all process temperatures, but owing to the low holding time, the samples were porous and their relative densities were below the theoretical density.

Group C: To improve the relative densities of the group B samples, the holding time was increased from 10 to 30 min under the same temperature and pressure conditions. The relative densities were similar to the theoretical density, and the Al matrix was spheroidized at all temperatures.

Table 1. Spheroidization behaviors of the matrix formed at different process temperatures, pressures, and holding times.

Sample Group	Process Temperature (°C)	Pressure (MPa)	Holding Time (Min)	Matrix Behavior
A	550	80	30 (Holding time at final process temperature)	Dendrite arm
	560			Dendrite arm
	570			Spheroidization
	580			Needle shape
B	550	PI process at 450 °C (80 MPa to 0 MPa)	10 (Holding time at 450 °C)	Spheroidization
	560			
	570			
C	550	PI process at 450 °C (80 MPa to 0 MPa)	30 (Holding time at 450 °C)	Spheroidization
	560			
	570			

The heat flow–temperature relationships were analyzed by differential scanning calorimetry (DSC; Rigaku DSC8270, Osaka, Japan). An aluminum chip sample (8 mg) was placed in an alumina pan and heated to 630 °C at 10 °C/min in a nitrogen atmosphere to prevent oxidation, and the sample was cooled to room temperature. The relative densities of the samples prepared in groups A, B, and C were measured by Archimedes' method. The microstructures of the samples were investigated by optical microscopy (OM; Leica DM 2700M RL/TL, Wetzlar, Germany), scanning electron microscopy (SEM; S-4800 Hitachi, Tokyo, Japan), and energy-dispersive X-ray spectroscopy (EDX; Horiba EX-250 X-stream, Tokyo, Japan). The mechanical properties of the fabricated samples were evaluated by a uniaxial compression test (Shimadzu TCE-N300-CE, Kyoto, Japan). Rectangular samples of volume $5 \times 5 \times 10 \text{ mm}^3$ were subjected to compression testing, and the mechanical properties of a single phase in the microstructure were investigated by the Vickers hardness test (Shimadzu HMV-2T, Kyoto, Japan).

3. Results and Discussion

Figure 1 shows the DSC heating curve of the base aluminum alloy used in this study. There are four endothermic peaks in the DSC heating curve, which are referred to as peak 1, peak 2, peak 3, and peak 4, respectively. In peak 1, the $\text{Al}_5\text{Cu}_2\text{Mg}_8\text{Si}_6$ phase first reacted at 476.5 °C. In peak 2, melting of the eutectic Si phase was observed at 527.4 °C. In peak 3, the melting reaction of the Mg_2Si phase occurred at 539.3 °C. Finally, in peak 4, the melting reaction of α -aluminum occurred at 582.5 °C. According to Kim et al., this alloy is reported to have a liquid fraction of 48.6% at 539.7 °C [19]. Based on these results, the SSP temperatures were set in the temperature range where the liquid fraction was close to 50% through the DSC experiments.

Figure 2 compares the relative densities of the samples formed at different SSP pressures and holding times. Groups B and C fabricated by PI exhibited lower densities than group A, although the density improved by increasing the holding time (c.f. group C vs. group B). The theoretical density $D_{\text{high-strength aluminum}}$ of high-strength aluminum was calculated as follows:

$$D_{\text{High-Strength Aluminum}} = (D_{\text{Al}} \times C_{\text{Al}}) + (D_{\text{Zn}} \times C_{\text{Zn}}) + (D_{\text{Si}} \times C_{\text{Si}}) + (D_{\text{Mg}} \times C_{\text{Mg}}) + (D_{\text{Cu}} \times C_{\text{Cu}}) \quad (1)$$

where D_x and C_x denote the true density and volume fraction of element x in the composition, respectively. The true densities of the elements are listed in Table 2.

Figure 1. DSC heating curve of high-strength aluminum alloy.

Figure 2. Relative densities of the samples prepared in groups A, B and C, with the theoretical density shown for comparison.

Table 2. Densities of the elements in the composition alloy.

Element Composition	Al	Zn	Si	Mg	Cu
True density (g/cm ³)	2.7	7.14	2.33	1.73	8.96

The theoretical density of high-strength aluminum calculated by Equation (1) is plotted in Figure 2 for comparison with the measured values. The relative densities of groups A, B, and C were 96.6%, 89.3%, and 95.3% of the theoretical density of high-strength aluminum, respectively. The sharp decrease in the relative density of the group B samples can be explained by the formation of unintended micro-porosity during the PI process of the SSP [21]. The relative density of the group C samples approached the theoretical density because the holding time was increased to 30 min. As shown in Figure 1, the melting reaction of high-strength aluminum alloy began at 470 °C. This result is likely attributable to the reduced pore volume, as the increased holding time at 450 °C enhances local liquefaction on the surface of Al chips [22,23]. In addition, as the holding time

increases, the local liquefaction increases the contact area between the Al chips, which is expected to facilitate the heat diffusion. Therefore, it was inferred that the short holding time caused the low relative density of group B high-strength aluminum produced by the PI process, whereas the longer holding time of group C high-strength aluminum was sufficient to achieve a near-theoretical density.

Figure 3 shows the microstructures of the samples fabricated at various process temperatures, pressures, and holding times. The optimal conditions of Al matrix spheroidization were confirmed in group A. The optimum temperature for spheroidizing the Al matrix was 570 °C (Figure 3c). At process temperatures of 550 and 560 °C, the Al matrix was dendritic (Figure 3a,b, respectively). A dendritic form of the Al matrix indicates that the temperature is insufficient to form a mushy zone. In contrast, the Al matrix formed at the process temperature of 580 °C was needle-like, and dendrites and spheroids were not seen (Figure 3d). In this case, the alloy exceeded the mushy-zone temperature and the liquefaction of the Al matrix was complete rather than local. Under complete liquefaction and pressure effects, the sample was deformed along the stress direction. Panels e, f, and g of Figure 3 are micrographs of the group B samples manufactured through the PI process at 550, 560, and 570 °C, respectively. At the optimal spheroidization temperature (570 °C), uniformly spheroidal forms are seen in the Al matrix. Panels h, i, and j of Figure 3 are micrographs of the group C samples manufactured through the PI process at 550, 560, and 570 °C, respectively. Similar to group B, the Al matrix of the sample prepared in group C also shows a uniform spheroidal shape, depending on the process temperature. These results can explain that the 10 and 30 min holding at 450 °C had a significant influence on the spheroidization of the group B and C samples. The cause of the microstructure difference between group A and groups B and C is thought to be the pressure applied during the process. In the case of group A samples, 80 MPa of pressure was applied at the final processing temperature. However, the pressure was released after holding of 10 and 30 min at 450 °C for group B and C samples. Around 450–470 °C, the $\text{Al}_5\text{Cu}_2\text{Mg}_8\text{Si}_6$ phase becomes liquid and the small volume of liquid plays a critical role in spheroidization of the matrix. Under 80 MPa pressure (group A), the small volume of liquid squeezed out through the large surface of dendrites. However, without pressure (groups B and C), the liquid formed at around 450–470 °C remains between the dendrites and the Ostwald ripening phenomenon takes place with longer holding at temperatures below 560 °C. Figure 4 schematizes the spheroidization mechanism of the Al matrix, which proceeds through rheology and Ostwald ripening during the PI process at the mushy-zone temperature [24,25]. The microstructure of the high-strength aluminum alloy in the mushy-zone state evolves through four stages. First, the dendrite arms are deformed and broken under the applied pressure and temperature. Second, the broken dendritic arms grow into non-dendritic grains through the rheology and Ostwald ripening mechanism in the PI process. Third, the primary α -Al melts along the grain boundaries and grows in association with new particles. Finally, the grains become separated and spheroidization occurs in the mushy-zone temperature range. Increasing the holding time to 30 min (group C) removed the unintended porosity found in group B, thus boosting the relative density to 95.3% of the theoretical density.

Figure 5 shows the EDX line profiles of the chemical elements of the Mg_2Si phase inside the group B and C samples. No pronounced deformation appeared in the Mg_2Si phase of the group B sample (Figure 5a). This result implied that deformation of the Mg_2Si phase could not occur during the short holding time. In contrast, the Mg_2Si phase of group C became coarse in size and showed some evidence of changes in its physical and mechanical properties (Figure 5b). This is interesting because the Mg_2Si phase deformed in group C, although the holding time was 20 min longer in this group than in group B. In general, lengthening the holding time encourages the deformation of the reinforced phase, forming a “liquid pool” or a “liquid island” [7]. Owing to such mechanical deformation, the Mg_2Si phase inside the sample of group C should exhibit a weaker mechanical strength than the Mg_2Si phase inside the sample of group B.

Figure 3. Optical microscopy images of the SSP-fabricated samples: (a–d) group A, (e–g) group B, and (h–j) group C.

Figure 4. Schematic of SS behaviors in the Al matrix.

Figure 5. SEM images and EDX line profiles of the Mg_2Si phase in the (a) group B and (b) C samples formed at 550 °C.

Table 3 lists the Vickers hardness values of the Al matrix, eutectic Si, and Mg_2Si phases in the sample of groups B and C. The Al matrixes and eutectic Si phases of the group B and C samples exhibited similar hardness values, but the hardness of the eutectic Mg_2Si particle was much lower in group C than in group B. The hardness of the Mg_2Si phase is typically 350–750 HV [26], higher than the hardness of the Mg_2Si phase inside the group C sample. The low value in the group C sample was probably attributable to deformation of the Mg_2Si phase during the long holding time.

Table 3. Vickers hardness in each phase of the group B and C samples.

Phase \ Sample Type	Group B (550 °C)	Group B (570 °C)	Group C (550 °C)	Group C (570 °C)
Al	115.3	131.2	114.8	134.6
Eutectic Si	253	255.6	253.8	252.3
Mg_2Si	378.8	382.5	262.3	311.2

Figure 6 shows the compressive stress–strain curves of the samples in groups B and C. As shown in Figure 6, the compressive stress of the samples in both groups B and C increased with the process temperature. At 550 °C, the maximum compressive strength of the sample in both groups was significantly lower than in the samples manufactured at 570 °C. This result implies that increasing the process temperature reduces the pore volume inside the sample, improving the load transfer to the Al matrix and possibly homogenizing the stress distribution under compression. The maximum compressive stress was much higher in the sample of group B than in the sample of group C, probably because the strength of the Mg_2Si phase was superior in group B (recall that the Mg_2Si phase in the sample of group C was deformed; see Figure 5). The higher the strength of the Mg_2Si phase, the higher the resistance to compressive stress when the load transfer in the Al matrix decreases. Additionally, in general, the compressive strength of aluminum is reported to be about 140–250 MPa [27–29]. As shown in Table 4, it was confirmed that the compressive strength of the group B and C samples prepared through the SSP improved compared to the general compressive strength of aluminum.

Figure 6. Compressive yield strengths of the samples formed under different processing conditions: (a) group B and (b) group C.

Table 4. Compressive yield strength of group B and C samples.

Temperature (°C)		550	560	570
Compressive Yield strength (MPa)	Group B	414.9	582.2	723.7
	Group C	350.7	437.6	520.3

Figure 7 shows the fracture behaviors of the Mg_2Si , eutectic Si, and Al matrices in the group B and C samples under compression. Samples in both groups displayed similar fracture behaviors of each phase. The fracture mode of each phase develops in three stages, as shown in Figure 8. First, cracking begins in the elastic region as stress is concentrated in the Mg_2Si phase, and micro-cracks are found only in the Mg_2Si phase. Second, when the maximum compressive stress is reached, the cracks begin propagating along the Mg_2Si phase (Figure 7b,e). Finally, the cracks propagate along the Mg_2Si phase to the eutectic Si and Al matrices (Figure 7c,f). The compression fracture behavior of the Mg_2Si phase confirms a lower maximum compressive strength of the sample in the group C sample than in the group B sample. Meanwhile, panels b and c of Figure 7 confirm larger cracks in the Mg_2Si phase of the group C sample than in the group B sample, which can be explained by the higher compressive stress of the Mg_2Si phase in the group C. The low maximum compressive stress of the sample in group C is probably explained by the reduced strength of the Mg_2Si phase in the group C sample after deformation during the manufacturing process.

Figure 7. SEM images of the samples after compressive strength tests: (a–c) fracture behaviors of group B and (d–f) fracture behaviors of group C (process temperature = 570 °C).

Figure 8. Schematic showing the likely fracture route of high-strength aluminum.

4. Conclusions

The conclusions of this study are as follows:

1. High-strength aluminum alloy with a spheroidized aluminum matrix was successfully fabricated through a pressure-less semi-solid process at 550, 560, and 570 °C. Spheroidized aluminum alloy is characterized by a higher strength than general aluminum. In the case of optimal conditions for spheroidizing the aluminum matrix, the sample could be fabricated at a temperature of 570 °C with a holding time of 30 min and a pressure of 80 MPa. The pressure-less process (pressure released from 80 MPa to 0 MPa at 450 °C) enabled fabricating of high-strength aluminum alloy in which the aluminum matrix was spheroidized with holding times of 10 min and 30 min at temperatures below 570 °C.
2. In the microstructure, uniform spheroidization was observed in the matrix of the high-strength aluminum alloy fabricated by the pressure-less process. The spheroidization of this matrix seems to be caused by the Ostwald ripening mechanism of the dendrite arms destroyed after the pressure-less process.
3. For the high-strength aluminum alloy with a short holding time of 10 min in the pressure-less process, no deformation of the Mg₂Si phase was observed. In contrast, a significantly deformed Mg₂Si phase was observed for the high-strength aluminum alloy with a holding time of 30 min. This result is due to changes in the mechanical properties of the Mg₂Si phase with increasing holding time. In addition, it was found that the strength of the Mg₂Si phase decreased as the holding time increased due to the formation of a liquid pool on the Mg₂Si phase.
4. The high-strength aluminum alloy fabricated by a holding time of 30 min in the pressure-less process had lower compressive strength than the aluminum alloy fabricated by a holding time of 10 min. The Vickers hardness results showed that the main reason for the low strength of the high-strength aluminum alloy is the low strength of the deformed Mg₂Si due to the 30 min holding time.

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