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Improved mechanical properties of near-eutectic Al-Si piston alloy through ultrasonic melt treatment



Jae-Gil Jung^a, Sang-Hwa Lee^a, Jung-Moo Lee^{a,*}, Young-Hee Cho^a, Su-Hyeon Kim^b, Woon-Ha Yoon^a

^a Implementation Research Division, Korea Institute of Materials Science (KIMS), Changwon 51508, Republic of Korea ^b Metal Materials Division, Korea Institute of Materials Science (KIMS), Changwon 51508, Republic of Korea

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1. Introduction

Al-Si alloys have been widely used in high-temperature applications such as in fabricating automobile engine pistons, because of their high strength and excellent resistance to abrasion and fatigue at ambient and elevated temperatures [1]. With increasing demands of high fuel efficiency, lighter pistons that can withstand higher temperatures are required. Thus, many investigators have made extensive efforts to improve the mechanical properties of Al-Si piston alloys.

Transition elements such as Mn [2], Fe [3], Ni [4], Cu [4,5], Cr [6], Co [7] and Zr [8] have been added to improve the mechanical properties of Al-Si piston alloys because these elements form rigid phases that are thermally stable at elevated temperatures [9]. Hence, recent Al-Si piston alloys contain Si (11–23 wt%), Cu (0.5–5.5 wt%), Mg (0.6–1.3 wt%), Ni (0.5–3.0 wt%), Fe (< 1.3 wt%), Mn (< 1.0 wt%) and other elements (Co, Zr, Ti, V, etc.) [10]. The optimization of processing variables such as cooling rate and heat treatment have been also used to improve the mechanical properties of Al-Si piston alloys [11].

Ultrasonic melt treatment (UST) has been applied to improve the mechanical properties of Al alloys because the UST effectively reduces the porosity and refines the microstructures [12–16]. The refining effect of UST has been explained by the hypothesis that the dendritic grains can be broken and distributed by ultrasonic vibrations, acting as heterogeneous nucleation sites for α -Al and/or secondary phases

ABSTRACT

The effects of ultrasonic melt treatment (UST) on the microstructure and mechanical properties of Al-12.2Si-3.3Cu-2.4Ni-0.8Mg-0.1Fe (wt%) piston alloy were systematically investigated. Rigid colonies consisting of primary Si, eutectic Si, Mg₂Si and various aluminides (ϵ -Al₃Ni, δ -Al₃CuNi, π -Al₈FeMg₃Si₆, γ -Al₇Cu₄Ni, Q-Al₅Cu₂Mg₈Si₆ and θ -Al₂Cu) were observed in the as-cast alloys. The sizes of the secondary phases, eutectic cell and grain were significantly decreased by UST because of the enhanced nucleation of each phase under ultrasonic irradiation. The yield strength, tensile strength and elongation at 25 °C were significantly improved by UST mainly because of the refinement of the microstructures. Both tensile strength and elongation at 350 °C were also improved by UST despite the unchanged yield strength. © 2016 Elsevier B.V. All rights reserved.

[12,13]. It has also been suggested that the decreased undercooling required for nucleation during the expansion and collapse of cavitation bubbles that are introduced by ultrasonic vibrations is responsible for the refinement of microstructure [14–16].

Beneficial effects of UST are highly expected in the case of Al-Si piston alloys containing a high volume fraction ($\sim 20\%$) of coarse secondary phases. Sha et al. [17] and Lin et al. [18] reported that at room temperature and elevated temperature (350 °C), UST improved both strength and ductility of hypereutectic Al-Si piston alloys such as Al-20Si-2Cu-1Ni-0.6Mg-0.7Fe-(0–1.1)Co and Al-17Si-2Cu-1Ni-0.4Mg-(0.2–2.0)Fe-(0.4–0.8)Mn; this improvement was attributed to the refinement of pre-eutectic phases such as primary Si and Fe-bearing intermetallic compounds. However, until now, few studies have focused on the effects of UST in near-eutectic Al-Si piston alloys whose microstructure and mechanical properties are dominated by eutectic phases.

Therefore, in this study, UST was applied to near-eutectic Al-12.2Si-3.3Cu-2.4Ni-0.8Mg-0.1Fe (wt%) piston alloy and then microstructural changes (pre-eutectic phases, eutectic phases, grain, etc.) due to UST were quantitatively measured through two-dimensional (2-D) and three-dimensional (3-D) observations. The effects of UST on tensile properties and fracture mechanisms at ambient and elevated temperatures were also investigated.

2. Experimental procedure

The specimens used in the study were provided by Dong Yang

E-mail address: jmoolee@kims.re.kr (J.-M. Lee).

* Corresponding author.

Table 1	
Chemical composition of Al-Si piston	alloys with and without UST (wt%).



Fig. 1. (a) Temperature-solid fraction curve and (b) chemical composition of liquid phase during Scheil-Gulliver solidification of Al-Si piston alloy.

Piston Co. (Ansan, Republic of Korea). One kilogram of near-eutectic Al-Si piston alloy was re-melted at 750 °C in an electric resistance furnace and degassed using Ar gas bubbling filtration (GBF). The degassed melts were poured at 700 °C into a permanent copper mold (245 mm \times 200 mm \times 70 mm³), which was preheated to 200 °C (hereafter referred to as without UST).

The UST was performed for 60s over the temperature range of 750–700 °C using a titanium horn connected with a high speed bipolar amplifier (NF corporation, HSA4052) and a multifunction

synthesizer (NF corporation, WF1945B). The amplitude and frequency of ultrasounds were approximately 20 μ m and 19 kHz, respectively. The titanium horn was preheated to 200 °C to reduce the chill effect of cold horn and then it was immersed to 20 mm below the top melt surface. The ultrasonic-treated melts were poured into the same mold (hereafter referred to as with UST). The chemical compositions of as-cast alloys with and without UST were measured three times per each sample using an optical emission spectroscopy (OES, Thermo Scientific, ARL 3460) and their averaged values were listed in Table 1. To examine the solidification events, the cooling curves of the solidifying alloys were recorded using a K-type thermocouple more than two times to ensure reproducibility.

After the specimens were mechanically polished, the microstructures were observed using an optical microscope (OM, Nikon, MA200) and a scanning electron microscope (SEM, JEOL, JSM-6610LV) equipped with an energy dispersive X-ray spectroscope (EDXS, JEOL, INCA Energy). The grain structure was examined using an electron backscatter diffraction (EBSD) instrument installed in a field emission scanning electron microscope (FE-SEM, TESCAN, CZ/MIRA I LMH). An image analyzer (IMT, i-Solution) was used to quantitatively measure the size, roundness and volume fraction of the secondary phases from ten OM images taken at x1000 magnification. 3-D images of the secondary phases were obtained using an automatic serial sectioning machine (UES Inc., Robo-Met. 3D) and 3-D analysis software (FEI, Avizo Fire 7). X-ray diffraction was performed to characterize the secondary phases using Cu-K α radiation. The density of each alloy was measured using an analytical balance (Mettler Toledo, AG285).

Room-temperature tensile test was performed using an Instron 4206 testing machine with a crosshead speed of 1.5 mm/min. Tensile test was also performed at 350 °C with a crosshead speed of 0.125 mm/min according to ASTM E21 [19] after the samples were isothermally held for 100 h at the test temperature to simulate their operation conditions. Four dogbone-shaped (gage section: $\emptyset 6 \text{ mm} \times 25 \text{ mm}^2$) specimens per each alloy and each testing temperature were used to perform the tensile tests.

3. Results and discussion

3.1. Thermodynamic calculation

Fig. 1(a) shows the temperature vs. solid fraction (f_s) curve of the Al-Si piston alloy without UST during the Scheil-Gulliver solidification, which is calculated using the Thermo-Calc software [20] with the TCAL3 database. A minor element, Ti is excluded in calculation. The primary Si and ε-Al₃Ni phases were formed from liquid at 580 °C (region 2) and 569 °C (region 3), respectively. Then, various eutectic reactions take place at temperatures ranging from 564 to 510 °C. The Cu-free eutectic phase of ε-Al₃Ni was formed up to the medium stage of solidification (regions 4–6 in $f_s < 0.65$), followed by the formation of Cu-containing δ-Al₃CuNi eutectic phase (regions 6–11 in 0.65 < $f_s < 0.85$). The eutectic phases with relatively higher Cu and Mg content such as γ -Al₇Cu₄Ni, Q-Al₅Cu₂Mg₈Si₆, θ-Al₂Cu and M-Mg₂Si form in the final stage of solidification (regions 10–18 in $f_s > 0.85$) after the Cu and Mg are sufficiently enriched in the remaining liquid (Fig. 1(b)).

3.2. Microstructures

Fig. 2(a) and (b) show the OM images of as-cast alloys without and with UST. Several secondary phases were observed and they were categorized into three groups. Dark gray phases were Si (blocky faceted primary Si_p and long platelet eutectic Si_e) and the black phases were Mg_2Si . The other phase of long bright platelets



Fig. 2. Microstructures of as-cast alloys (a, c) without and (b, d) with ultrasonic melt treatment: (a, b) OM, (c, d) EBSD.

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ffect of ultrasonic melt treatment on secondary phases of as-cast Al-Si piston alloy.

Phase	Alloy	Maximum Size (µm)	Average size (µm)	Roundness	Area frac- tion (%)
Si_p and Si_e	w/o UST	73.8	13.6 (± 3.0)	$0.38 \ (\pm 0.11)$	13.6 (+2.0)
	UST	26.8	5.2 (±2.1)	$0.43 \ (\pm 0.13)$	13.1 (±3.2)
Aluminide	w/o UST UST	69.9 29.5	$\begin{array}{c} 14.9 \;(\pm 3.1) \\ 6.3 \;(\pm 0.8) \end{array}$	$\begin{array}{c} 0.18~(\pm0.03)\\ 0.19~(\pm0.02) \end{array}$	$\begin{array}{l} 4.9 \; (\; \pm \; 0.9) \\ 3.7 \; (\; \pm \; 0.4) \end{array}$
Mg ₂ Si	w/o UST UST	50.2 18.4	7.4 (± 1.9) 3.9 (± 1.8)	$\begin{array}{c} 0.39~(\pm0.14)\\ 0.35~(\pm0.07) \end{array}$	$\begin{array}{c} 0.4~(\pm0.3)\\ 0.5~(\pm0.2) \end{array}$

was aluminides. The formation of primary Si at near-eutectic Si concentration (12.2 wt%) is because of the P modification (0.002 wt%) that enhances the nucleation of primary Si during solidification [17,18,21]. Pores were not observed in both alloys

because the melts were degassed prior to the casting by GBF or UST. Similar values of density for alloys without (2.755 g/cm^3) and with UST (2.764 g/cm^3) were also obtained. It was seen that the UST reduced both the sizes of eutectic cell (Fig. 2(a) and (b)) and grain (Fig. 2(c) and (d)).

The size, roundness (R= 4π (area)/(perimeter)² [22]) and area fraction of the secondary phases were quantitatively measured and the results are listed in Table 2. It is obvious that both the maximum and average sizes of Si, Mg₂Si and aluminides were greatly decreased by UST. The similar values of roundness for the secondary phases of both alloys indicate that the morphology was not significantly changed by UST. Even taking into account lower contents of alloying elements for the UST alloy (Table 1), the decrement in area fraction of aluminides in the UST alloy is somewhat large. This might imply larger amounts of solid-solution elements and/or their clustering in the UST alloy [23]. Thus, we are currently investigating to confirm this hypothesis via observation of clusters by means of small-angle neutron scattering and transmission electron microscopy as well as measurements of lattice parameter and electrical resistivity.



Fig. 3. SEM images and EDXS element mappings of as-cast alloys (a) without and (b) with ultrasonic melt treatment.

Table 3

EDXS results of secondary phases observed in as-cast Al-Si piston alloy without ultrasonic melt treatment (Fig. 3(a)).

No.	Element (at%)						Suggested phase	
	Mg	Al	Si	Fe	Mn	Ni	Cu	
1	-	7.9	92.1	-	-	-	-	Si
2	1.0	79.3	2.9	5.5	0.3	8.6	2.3	ε-Al ₃ Ni
3	2.3	67.1	4.6	0.4	-	16.7	8.9	δ-Al ₃ CuNi
4	37.6	33.1	21.1	-	-	0.2	8.0	Q-Al ₅ Cu ₂ Mg ₈ Si ₆
5	4.0	67.6	2.8	-	-	0.3	25.2	θ-Al ₂ Cu
6	11.9	66.7	16.5	2.0	0.3	1.2	1.3	π-Al ₈ FeMg ₃ Si ₆
7	34.5	17.6	47.4	0.1	-	0.1	0.3	M-Mg ₂ Si
8	-	67.0	1.2	-	-	8.6	23.2	γ-Al ₇ Cu₄Ni

Fig. 3(a) shows the SEM image and corresponding EDXS element mappings of the as-cast alloy without UST. Three groups of secondary phases (i.e. Si, aluminides, and Mg₂Si) were also distinguished using the element distribution. Most of them form eutectic conglomerates because of the serial and complex eutectic reactions occurring during solidification as shown in Fig. 1(a). The chemical compositions of the secondary phases (numbers 1–8 in Fig. 3(a)) were measured using SEM-EDXS, and the aluminides were carefully identified by comparing their measured chemical

compositions (Table 3) to those reported in a previous study [9]. The observed aluminides were confirmed as ϵ -Al₃Ni, δ -Al₃CuNi, π -Al₈FeMg₃Si₆, γ -Al₇Cu₄Ni, Q-Al₅Cu₂Mg₈Si₆ and θ -Al₂Cu, agreeing with the solidification simulation (Fig. 1) and X-ray diffraction (Fig. 4). SEM observation also indicates the decreased size and area fraction of aluminides in the UST alloy (Fig. 3(b)), as observed in the OM images (Fig. 2).

3-D microstructures of the secondary phases were observed in order to investigate their morphology, distribution and interconnectivity further. Fig. 5(a) to (d) show the 3-D microstructures of the secondary phases in the as-cast alloy without UST. The coarse blocky primary Si and eutectic Si platelets (roundness =0.38) and thin aluminide platelets (roundness=0.18) were clearly observed in Fig. 5(a) and (b). The Si and aluminides were interconnected, forming huge colonies, although most of each phase was observed as separated in the 2-D image (Fig. 2(a)). As shown in Fig. 5(c), the interconnectivity between the irregularly shaped Mg₂Si phases was insignificant because of their low volume fraction (~0.5%). Although the sizes of Si and aluminides were greatly decreased by UST, their interconnectivities remained, as shown in Fig. 5(e) and (f). Fine Mg₂Si particles with a higher population density were observed after UST (Fig. 5(g)).

The 3-D microstructures of the secondary phases are described together in Fig. 5(d) and (h). High contiguities between Si,



Fig. 4. X-ray diffraction patterns of as-cast alloys (a) without and (b) with ultrasonic melt treatment.

aluminides and Mg₂Si were observed in both alloys without and with UST. Asghar et al. [24] also observed the 3-D network structures of Si and aluminides with high interconnectivity (94– 97%) between them in the as-cast Al-10Si-5Cu-(1–2)Ni (wt%) piston alloys using synchrotron tomography. It is known that these network structures of rigid phases play an important role in the strength of the Al-Si piston alloy, especially at high temperatures, by transferring the external load from the soft Al matrix to the rigid phases [24,25]. Meanwhile, the Mg₂Si phases were observed mainly adjacent to the eutectic platelets of Si or aluminides because of the simultaneous formation of Mg₂Si, eutectic Si and aluminides during eutectic reactions (regions 10–13 in Fig. 1(a)).

It is known that the refinement of microstructure by UST is closely related to the nucleation and growth behaviors during solidification [12–16]. Therefore, the cooling curves of solidifying alloys without and with UST were measured to examine the nucleation and growth behaviors of each phase. As shown in Fig. 6, there are three humps in the cooling curve of the as-cast alloy without UST. The first hump at 576.6 °C and the second one at 569.2 °C are due to the nucleation of primary Si and ε -Al₃Ni, respectively, which are consistent with the equilibrium formation temperatures of primary Si (580.4 °C) and ε -Al₃Ni (569.3 °C). The third hump starting at 555.9 °C results from the formation of eutectic phases of the Al matrix, ε -Al₃Ni and Si, based on thermodynamic calculations (Fig. 1(a)).

As shown in Fig. 6, the nucleation temperatures of primary Si (578.4 °C), ε -Al₃Ni (571.4 °C) and eutectic phases (559.8 °C) increased under ultrasonic irradiation. This indicates that the UST enhances the nucleation of primary Si, ε -Al₃Ni and eutectic phases by decreasing the undercooling required for their nucleation during solidification. Our experimental results agree with those for the refinement of primary Si with higher nucleation temperatures observed in hypereutectic Al-16Si-0.37Fe-0.16Mn (wt%) [14,15] and Al-18.5Si-0.5Mg-3.9Cu-0.24Fe (wt%) [23] alloys. In addition, a

smaller recalescence following the nucleation of both primary Si and eutectic phases was observed in the cooling curve of the UST alloy, indicative of the prolonged nucleation with less inhibition by the release of latent heat. Therefore, it is thought that the microstructure refinement by UST is likely due to the enhanced and prolonged nucleation of secondary phases and the Al matrix.

3.3. Tensile properties

The tensile properties at ambient temperature of the as-cast alloys (Table 4) show that both yield strength and tensile strength were increased by UST, from 147 to 197 MPa and 171-272 MPa. respectively. In addition, the elongation at ambient temperature was also increased by UST, from 0.57 to 1.30%. The improvement of the strengths by UST is mainly attributed to the refinement of microstructures, including grain, eutectic cell and secondary phases (Table 2). The enhanced strengthening of the solid-solution and/or clustering in the UST alloy, which can be inferred from the decreased area fraction of aluminides, is also somehow responsible for the increases in yield and tensile strength. This hypothesis can be supported by the results of Tuan et al. [26] who examined the effect of UST on the age-hardening behavior of Al-1Mg-0.3Sc (wt%) alloy. They reported that the hardness of ultrasonic-treated cast alloy (82 Hv) was much higher than that of nontreated cast alloy (57 Hv) and it was decreased during artificial aging at 300 °C. This might imply that the enhanced strengthening by solid-solution elements and/or their clusters increases the hardness of ultrasonic-treated cast alloy and its strengthening effect is larger than the strengthening by precipitate formed during aging.

Fig. 7(a) and (b) show the tensile-fractured microstructures of the as-cast allov without UST at ambient temperature. A wide area of cleavage patterns with flat facets ('A') was observed. This cleavage pattern is formed by the debonding of coarse primary Si and/ or eutectic Si particles from the Al matrix, which is the main tensile crack reported in the Al-Si piston alloy [27]. Further, Si particles with secondary cracks, indicated by 'B' in Fig. 7(a) and (b), also exist. Meanwhile, agglomeration of broken aluminides without cleavage facets was observed along the dashed lines ('C'), implying that the main crack broke up the aluminides because of their brittle mechanical properties [27]. Fine fractured microstructures with reduced area and size of cleavage facets were observed in the UST sample (Fig. 7(c)). In addition, the cracks in Si and aluminides were not severe in the UST alloy (Fig. 7(d)) compared to the alloy without UST (Fig. 7(b)). This implies that the stress concentrations near smaller Si and aluminides are lower in the UST alloy because of the dispersed pile-up of mobile dislocations during plastic deformation, thereby improving the ductility [28,29].

Table 4 also shows the tensile properties at 350 °C of the as-cast alloys without and with UST. The yield strength at 350 °C did not change, probably because of the complementary effects of UST: the refinement of microstructures and the decreased area fraction of aluminides. Both tensile strength and elongation at 350 °C were improved by UST, from 57 to 60 MPa and from 9.2 to 18.7%, respectively. Fig. 8(a) and (b) show the tensile fractured surfaces of the as-cast alloy without UST at 350 °C. Dimples, which are typical ductile features, were observed at 350 °C unlike the fractured microstructure at 25 °C (Fig. 7). Secondary phases (i.e. Si, aluminides and Mg₂Si) that debonded from the Al matrices were observed inside the dimples. This indicates that the tensile fracture at 350 °C mainly progressed by the debonding of secondary phases from the Al matrices, and then deformation of the ductile Al matrices. Meanwhile, the sizes of both dimples and secondary phases were much smaller in the UST alloy (Fig. 8(c) and (d)), which are responsible for the improved tensile strength and ductility at 350 °C.



Fig. 5. Three-dimensional microstructures of as-cast alloys (a-d) without and (e-h) with ultrasonic melt treatment.

In general, the addition of alloying elements simultaneously increases both the volume fraction and the size of secondary phases. Hence, the concentrations of alloying elements in Al-Si piston alloys have been limited to certain levels to avoid the formation of coarse secondary phases that adversely affect mechanical properties. The present study shows that the UST improves the mechanical properties of Al-Si piston alloy by refining the microstructure. This implies that the mechanical properties may be further improved by



Fig. 6. Cooling curves of solidifying Al-Si piston alloys without and with ultrasonic treatment.

Table 4

expanding the upper limits of the chemical compositions of alloying elements if the sizes of secondary phases are effectively controlled using the UST technique. The effectiveness of UST may be enhanced by the optimization of processing parameters such as the amplitude and the power of ultrasound, treatment time and treatment temperature [30]. Thus, our research group is currently investigating the optimization of the alloy system and processing variables to further improve the mechanical properties of Al-Si piston alloys.

4. Conclusions

The effects of UST on the microstructure and mechanical properties at ambient and elevated temperatures of near-eutectic Al-Si piston alloy were systematically investigated. The main results can be summarized as follows:

(1) As-cast Al-Si piston alloys were composed of primary Si, eutectic Si, Mg₂Si and a variety of aluminides, including ϵ -Al₃Ni,

Tensile properties at ambient and elevated temperatures of as-cast Al-Si piston alloys without and with ultrasonic melt treatment.

Alloy	25 °C			350 °C		
	Yield strength (MPa)	Tensile strength (MPa)	Elongation (%)	Yield strength (MPa)	Tensile strength (MPa)	Elongation (%)
w/o UST w/ UST	$\begin{array}{c} 147\pm2\\ 197\pm8 \end{array}$	$\begin{array}{c} 171\pm5\\ 272\pm24 \end{array}$	$\begin{array}{c} 0.57 \pm 0.05 \\ 1.30 \pm 0.39 \end{array}$	$\begin{array}{c} 38\pm2\\ 36\pm1 \end{array}$	57 ± 0 60 ± 1	9.2 ± 1.9 18.7 ± 5.1



Fig. 7. Tensile fractured microstructures at ambient temperature of as-cast alloys (a, b) without and (c, d) with ultrasonic melt treatment, which were observed (a, c) parallel and (b, d) perpendicular to the tensile direction (TD).



Fig. 8. Tensile fractured microstructures at elevated temperature of as-cast alloys (a, b) without and (c, d) with ultrasonic melt treatment, which were observed (a, c) parallel and (b, d) perpendicular to the tensile direction (TD).

 δ -Al₃CuNi, π -Al₈FeMg₃Si₆, γ -Al₇Cu₄Ni, Q-Al₅Cu₂Mg₈Si₆ and θ -Al₂Cu. Eutectic phases containing higher Cu and Mg content were formed in the latter stages of solidification.

- (2) The UST greatly decreases the sizes of the grain, eutectic cell and the secondary phases (i.e., primary Si, eutectic Si, Mg_2Si and aluminides) because of the enhanced and prolonged nucleation of each phase under ultrasonic irradiation. The interconnected network structures of rigid secondary phases remained after UST.
- (3) The yield strength, tensile strength and elongation at 25 °C were significantly improved by UST, which was mainly attributed to the refinement of microstructures (i.e. grain, eutectic cell and secondary phases). At an elevated temperature of 350 °C, both tensile strength and elongation also increased because of UST.

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