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# Combined effects of ultrasonic melt treatment, Si addition and solution treatment on the microstructure and tensile properties of multicomponent Al—Si alloys



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#### ABSTRACT

The effects of ultrasonic melt treatment (UST) on the microstructure and tensile properties of three multicomponent Al—Si alloys with different Si contents (12, 15, and 18 wt%Si) and solution treatments were systematically investigated at room and elevated (350 °C) temperatures. The microstructures of these alloys consisted of primary Si, eutectic Si, Mg<sub>2</sub>Si, and various types of aluminides, but the average size and area fraction of the primary Si increased with Si content. The application of UST caused a transformation from dendritic to equiaxed cells and a reduction in grain size, with a significant reduction in the size of all secondary phases (i.e., Si, Mg<sub>2</sub>Si and aluminides). Both the strength and ductility at room and elevated temperatures were greatly improved by UST, mainly due to microstructure refinement. Improvements in the high-temperature tensile strength as a result of UST increased with increasing Si contents, due to the enhanced refinement of primary Si by UST with increasing Si contents. The spherodization of rigid phases induced by solution treatment can improve the strength and ductility at room temperature, but can also cause a loss of strength at elevated temperatures due to the relaxation of the interconnected network structure of rigid phases caused by their spherodization.

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#### 1. Introduction

Multicomponent Al—Si alloys have been widely used in high-temperature applications such as automobile engine pistons due to their high strength and fatigue resistance at elevated temperatures and their excellent castability [1]. These alloys generally 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%), and Mn (<1.0 wt%), which are necessary to attain the required mechanical properties by forming thermally stable phases [2,3]. Owing to increasing demand for greater fuel efficiency, many researchers are seeking to develop lighter piston alloys that are capable of withstanding even higher temperatures through optimization of their chemical composition (e.g., Si [4] and transition elements [5–10]) or heat treatment [11–14].

Ultrasonic melt treatment (UST) is one of the more promising means of improving the mechanical properties of Al alloys, as it effectively reduces the porosity while simultaneously refining the microstructure through cavitation-induced dendrite fragmentation and/or cavitation-induced heterogeneous nucleation [15–18]. Indeed, the present authors [19] have shown that both the strength and ductility of near-eutectic Al-12.2Si-3.3Cu-2.4Ni-0.8Mg-0.1Fe (wt.%) alloy are greatly improved at ambient and elevated temperatures due to refinement of its grains, eutectic cells, and secondary phases. Khalifa et al. [20] also reported that the wear resistance of a hypereutectic Al-17Si-4.5Cu-0.6 Mg (wt.%) B390 alloy is improved by UST owing to refinement of the primary Si and Fe-intermetallic compounds.

The refining effect of UST implies that the mechanical properties of a multicomponent Al–Si alloy can be further improved by increasing the amount of Si or transition elements, assuming that the size of the secondary phase is sufficiently reduced by UST. Lin et al. [21] have examined the combined effects of UST and Mn addition on the microstructure and mechanical properties of hypereutectic Al–17Si–2Cu–1Ni-0.4 Mg–2Fe-(0.4,0.8)Mn (wt.%) alloys, and found that high-temperature tensile properties are improved through an increase in the amount of secondary phases and a decrease in their size, as well as the transformation of needle-like  $\beta$ -Al<sub>5</sub>FeSi to a more compact  $\alpha$ -Al<sub>15</sub>(Fe,Mn)<sub>3</sub>Si<sub>2</sub> phase. Sha et al.

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[22] reported similar results with a combination of UST and Co addition, achieving improved high-temperature tensile strength of a hypereutectic Al–20Si–2Cu–1Ni-0.6Mg-0.7Fe (wt.%) alloy.

Although Si also plays an important role in strengthening by forming a large amount (10–20 vol%) of rigid Si particles, there are few reports pertaining to the combined effect of UST and Si addition on the microstructure and mechanical properties of multicomponent Al—Si alloys. This study therefore examines the effects of UST on the microstructure and tensile properties of multicomponent Al—Si alloys containing different Si contents (12, 15, and 18 wt%) in order to understand the effect of Si addition on the beneficial effects of UST (i.e., the refining efficiency of primary Si and strength improvement). The combined effects of UST and solution treatment on the multicomponent Al—Si alloy were also examined to further improve its mechanical properties at room and elevated temperatures.

#### 2. Experimental procedure

Ingots of three multicomponent Al—Si alloys containing different Si contents (12, 15, and 18 wt%) were provided by Dong Yang Piston Co. (Ansan, Republic of Korea). The ingots were remelted at approximately 800 °C in a clay-graphite crucible (Ø120 mm  $\times$  160 mm) located within an electric resistance furnace, and then degassed by Ar gas bubbling filtration (GBF). Once degassed, 1.5 kg melts were poured into a copper book mold  $(245\times70\times200~\text{mm}^3)$  that had been preheated to 200 °C, and were then cooled to room temperature in the mold. Given the formation temperatures of primary Si in 12Si (577 °C), 15Si (617 °C) and 18Si (669 °C) alloys, the melt pouring temperatures used were 700 °C for the 12Si alloy and 750 °C for the 15Si and 18Si alloys. The dimensions of the rectangular ingots were approximately  $180\times30\times90~\text{mm}^3$ .

UST of the melts was performed for 60 s at the temperature ranges of 700–750 °C for the 12Si alloy and 750–800 °C for 15Si and 18Si alloys. A titanium sonotrode ( $\emptyset$ 50 mm) preheated to 200 °C was used to induce ultrasonic waves into the melts with an electric power of 0.4 kW. The amplitude and frequency of ultrasounds were 20  $\mu$ m in air and 19  $\pm$  0.2 kHz in the melt, respectively. The ultrasonic intensity (I) of the ultrasonic device was calculated as 3560 W/cm<sup>2</sup> using Equation (1) [23,24]:

$$I = \frac{1}{2}\rho c(2\pi f A)^2 \tag{1}$$

where  $\rho$  is the density of the aluminum melt (2.6 g/cm³ [25]), c is the velocity of ultrasound (4.8 × 10³ m/s [25]), f is the frequency of ultrasound and A is the amplitude of ultrasound. It is thought that the actual ultrasonic intensity is somewhat lower than the calculated value, considering the loss of amplitude in the melt caused by the friction between the sonotrode and the Al melt. Nevertheless, the ultrasonic intensity is believed to be high enough to activate fully developed cavitation in the Al melt (80 W/cm² [24]). The ultrasonically treated melts were immediately poured into the same copper mold and cooled to room temperature.

The cooling rates of the solidifying melts were measured based on the cooling curves from 650 °C to eutectic arrest temperatures (approximately 560 °C). The cooling rates of 12Si, 15Si and 18Si were 7.5, 7.3 and 7.1 °C/s, respectively, showing a decreasing tendency with increasing Si content which is caused by the increased latent heat released by primary Si formation. The effect of ultrasonic treatment on the cooling rate was insignificant. The chemical compositions of ingots with and without UST were measured using optical emission spectroscopy (OES, Thermo Scientific, ARL 3460), and are listed in Table 1. Note that all ingots contained a small

amount of phosphorus (11–32 ppm) to minimize the size of primary Si particles by enhancing their nucleation using AlP inoculants [26], and that the Ti content is slightly higher in UST alloys due to erosion of the titanium sonotrode during UST [27].

The ingots with and without UST were solution-treated at 490 °C for 2 h and artificially aged at 230 °C for 5 h (hereafter referred to as T7 alloy). Additional 18Si alloy melts with and without UST were cast into the same mold and taken out after solidification was completed at 450 °C. The hot ingots were immediately quenched in water to inhibit precipitation during cooling, and then aged at 230 °C for 5 h (hereafter referred to as T5 alloy).

Samples were taken from each ingot at one half of its length and width, and one-quarter of its height. The density of each sample was measured using an analytical balance (Mettler Toledo, AG285). After mechanical polishing, the microstructure of each sample was observed using an optical microscope (OM, Nikon, MA200) and a scanning electron microscope (SEM, JEOL, JSM-6610LV) with an attached energy diverse X-ray spectroscope (EDXS, JEOL, INCA Energy). Some samples were deep-etched in 18.5% HCl aqueous solution for 25 min to reveal the morphology and interconnectivity of the secondary phases. An image analyzer (IMT, i-Solution) was used to quantitatively measure the size distribution and area fraction of secondary phases from several OM images taken 200 × magnification. The grain structure was observed using an electron backscatter diffraction (EBSD) instrument installed in a field emission scanning electron microscope (FE-SEM, TESCAN, CZ/ MIRA I LMH) using a step size of 10 um. Those grains whose misorientation angle and size were greater than 5° and 100 um were averaged to exclude the influence of primary Si particles.

Room-temperature tensile tests were conducted using an Instron 4206 universal testing machine with a crosshead speed of 1.5 mm min $^{-1}$ , as per ASTM E8/E8M-13a [28]. After the alloys were isothermally held for 100 h at the testing temperature to simulate their service conditions, tensile tests were performed at 350 °C with a crosshead speed of 0.125 mm min $^{-1}$  in accordance with ASTM E21-09 [29]. Dogbone-shaped (gage section: Ø6 mm  $\times$  25 mm) specimens were used for all tensile tests.

#### 3. Results and discussion

#### 3.1. Calculation of Scheil-Gulliver solidification

Fig. 1(a) shows the temperature vs. solid fraction ( $f_s$ ) curve of 12Si alloy without UST during Scheil-Gulliver solidification, which was calculated using Thermo-Calc software [30] and the TCAL 3 database. The alloying elements Si, Cu, Ni, Mg, Fe, and Mn were included in the calculation along with all of their formable phases. Note that in the pre-eutectic region, primary Si, ε-Al<sub>3</sub>Ni and β-Al<sub>9</sub>Fe<sub>2</sub>Si<sub>2</sub> phases are formed at 576.5, 570.3 and 568.6 °C, respectively, with the same pre-eutectic phases being formed in 15Si (Fig. 1(b)) and 18Si (Fig. 1(c)) alloys. Furthermore, both the formation temperature ( $T_i$ ) and solid fraction ( $f_s$ ) of the primary Si significantly increase with increasing Si contents, as shown in Table 2. Meanwhile, similar values of  $T_i$  were observed for ε-Al<sub>3</sub>Ni (570–578 °C) and β-Al<sub>9</sub>Fe<sub>2</sub>Si<sub>2</sub> (566–570 °C) in the three alloys.

The eutectic reactions (i.e., eutectic Si and IMCs) of all three alloys took place within a temperature range of 560–510 °C. Among the eutectic IMCs, Cu-free eutectic  $\epsilon\text{-Al}_3\text{Ni}$  first forms ( $f_s<0.6$ ), followed by Cu-containing eutectic  $\delta\text{-Al}_3\text{CuNi}$  (0.6 <  $f_s<0.8$ ). Eutectic IMCs with higher Cu and Mg contents, such as  $\gamma\text{-Al}_7\text{Cu}_4\text{Ni}$ ,  $\theta\text{-Al}_2\text{Cu}$ , and M-Mg2Si, form in the final stage of solidification ( $f_s>0.8$ ) once the Cu and Mg solutes are sufficiently enriched in the remaining liquid (Fig. 1(d)).

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