Effects of La intermetallics on the structure and tensile properties of thin section gravity die-cast A357 Al alloy

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Abstract

In current research different amounts of La (0.01, 0.03, 0.05, 0.07, 0.1, 0.3, 0.5 and 1 wt%) were added to A357 aluminum alloy to investigate the structure and tensile properties of thin section parts prepared by gravity die-casting. The cast specimens were examined by using optical microscopy (OM), scanning electron microscopy (SEM), tensile testing and determination of quality index values to evaluate the modification efficiency of the alloy by La addition. Obtained results revealed that La reduces the grain size of A357 alloy from 620 μm to minimum 300 μm and improves the morphology of eutectic silicon in as-cast condition. Al_4La and AlSiLa intermetallic phases were detected through the microstructural studies. The optimum amount of La was found to be 0.1 wt% by enhancing ultimate tensile strength (UTS) of the alloy from 226 MPa to 267 MPa. Further improvement in tensile strength was achieved after T6 heat treatment for 0.1 wt% La added specimens, in which UTS showed the highest value (420 MPa). The addition of 0.1 wt% La also increased El.% values from 3.8 % to 6.8 % in non-heat treated and from 6.2% to 9% in T6 heat treated alloys.

Keywords: Al alloy; Tensile properties; Lanthanum; Heat treatment

1. Introduction

Al-Si-Mg alloys, a group of heat treatable cast Al-Si alloys are progressively being used in critical structural applications in automotive, aerospace and general engineering industries on account of its several advantages involving excellent casting characteristics, good corrosion resistance, welding, superb mechanical properties and particularly, high strength-to-weight ratio in heat-treated condition [1,2].

The mechanical properties of Al-Si cast alloy are controlled by two principal parameters: (a) those are governed by the casting process and (b) those are influenced by the physical metallurgy such as chemical composition, heat treatment and microstructural features [3,4]. Tensile properties and fracture behavior of Al-Si alloy, particularly the ductility, are accepted to be mainly controlled by the microstructural features normally the morphology and size of α -Al primary phase, silicon particles, intermetallics and other precipitate phases which may be presented in the microstructure [5,6].

In general, under normal cooling condition in hypoeutectic Al-Si alloys, α -Al primary phase grows in dendritic form and eutectic silicon particles normally present as coarse, acicular needles and nonuniform structure introducing stress concentration sites for crack initiation. Consequently, the mechanical properties of such alloys are accepted to be weak in as cast condition [7].

There are many different methods to modify the morphology and size of α -Al primary phase and eutectic silicon particles, such as chemical modification (addition of certain elements), quench modification (rapid cooling rate), thermal modification (heat treatment) and etc [8-11]. Several chemical elements are known to be used for chemical modification such as Sb [9], Na [10] and Sr [12]. The addition of rare-earth metals to Al-Si alloys has also been reported to act

as proper modifiers [13-20]. For example, Yi et al. [13] have reported that the addition of La into A390 alloy has little modifying influence on Si particles. Tsai et al. [14] have also investigated the effect of La on A356 alloys. They found that eutectic silicon in A356 alloy is not well refined until the amount of La increases up to 1 wt%. They have also reported that La addition has no influence on UTS results. Recently, Mousavi et al. [15] investigated the effect of mishmetal (combination of rare earth metals, mainly La and Ce) and heat treatment on the microstructure and tensile properties of A357 Al-Si casting alloy, suggesting that the addition of trace amount of mishmetal improves tensile strength by modifying eutectic Si crystals. In 2012, Jiang et al. [16] showed that La and Ce not only refine eutectic silicon particles but also reduce the size of α -Al primary phase effectively.

Further investigation by the use of an appropriate solution treatment can modify the morphology of eutectic silicon particles. T6 heat treatment, including solid solution strengthening and then precipitation of the fine coherent particles inside the microstructure during the aging process, has been used to improve the mechanical properties of the alloy. T6 treatment comprises solutionizing at a relatively high temperatures (530-550 °C), then quenching in water and finally artificial ageing (155-210 °C) [21-24].

In this study, different amounts of La were added into A357 Al-Si alloy to investigate its effect on the microstructural characteristics, tensile properties and fracture behavior of A357 thin section castings before and after T6 heat treatment. Effort was made to introduce the main mechanisms of refinement, modification and fracture after adding La to A357 alloy.

2. Experimental procedure

Pure Al (99.8%), Si (99.5%) and Mg (99.9%) were used to produce primary A357 ingots, in the beginning of this work. After melting Al in a 10 Kg graphite crucible via an electrical resistance furnace and heating up to 800 °C, Si and Mg were added into the molten Al to prepare A357 alloy. The chemical composition of A357 primary ingots prepared in this study is shown in Table 1.

Table 1

Then, remelting of chopped primary ingots was carried out in a small SiC crucible (1 Kg capacity). When the temperature reached 780 °C, Al-20 wt% La master alloy was added into the melt to obtain different amounts of La (0.01, 0.03, 0.05, 0.07, 0.1, 0.3, 0.5 and 1 wt%) in the alloy. In each trial, Al-20 wt% La master alloy was added into the remelted alloy, held for about 10 min in the molten metal. Before stirring and cleaning off the dross and pouring, the melt was stirred manually with a graphite rod for about 1 min to ensure complete mixing. Alloys with different compositions were poured into a preheated cast iron mould (280 °C) to prepare thin section castings. This mould was made according to ASTM B108/B108M-14 standard (Fig. 1a). Some specimens were made and tested in the as-cast condition and the rest were subjected to T6 treatment. For this purpose, they were solution treated in an accurate electrical furnace at 540 °C for 8 h, then quenched in water to room temperature, and finally aged at 170 °C for 7 h prior to air cooling.

Fig. 1

For structural studies, the specimens were selected from the gauge length portion of the test bars (with 6 mm diameter), as seen in Fig. 1b. Metallographic specimens were ground, polished according to the standard metallographic procedure and etched by HF (5%) to reveal

the microstructure. In order to develop grain structure of the as-cast alloys, polished samples were macro-etched using 45% HCl, 15% HNO₃, 15% HF and 25% water solution for 30 s prior to rinsing with warm water. Some samples were also deep etched in 20% NaOH solution for 45 min to dissolve aluminum matrix for scanning electron microscope (SEM) studies. The average grain size of the cast specimens was calculated according to ASTM E112 standard. Microstructural parameters were determined using an optical microscope equipped with an image analysis system (Clemex Vision Pro. Ver. 3.5.025). The microstructural characteristics of the specimens were also examined by SEM performed in a Vega©Tescan SEM, equipped with energy-dispersive spectroscopy (EDS) system. Phase identification was also performed by x-ray diffraction (XRD), using PHILIPS binary diffractometer and applying Cu-k α radiation.

Tensile test specimens were prepared according to ASTM B557M-10 standard, as seen in Fig. 1b. Tensile testing was carried out by a computerized testing machine (SANTAM STM-20) at the constant cross-head speed of 1 mm/min at room temperature.

Fracture surface characterization studies were carried out on the tensile fractured samples in order to provide valuable insight into the various fracture mechanisms operative during tensile loading. Fracture surface characterization studies were primarily accomplished using similar SEM.

3. Results and discussion

3.1. Structural characterization

Fig. 2 depicts a typical microstructure of A357 alloy in as-cast condition.

Fig. 2

From Fig. 2 and previous investigations, it can be seen that the microstructure of the unmodified A357 cast alloy consists of primary α -Al dendrites and Si particles in eutectic area that are evenly dispersed around the Al dendrites to form a cell pattern [14-17].

The optical micrographs of A357 alloys with different La contents in both as-cast and T6-heat treated conditions are shown in Fig. 3.

Fig. 3

From Fig. 3, it is clear that the microstructure of A357 alloy is notably changed by adding different levels of La. Large micrographs shown on top of Fig. 3a-d, related to the as-cast specimens, exhibit that La addition not only changes Si morphology, but also introduces new phase/phases in the alloy microstructure.

Secondary electron images of La added A357 specimens, shown in Fig. 4, clearly reveal that silicon particle size is changed from large particles to fine fibers.

Fig. 4

As seen in Fig. 4a, eutectic silicon particles are more irregular or acicular in La free A357 alloy but they change to clusters of fine fibers by adding 0.05 wt% La (Fig. 4b) or fine individual particles or broken fibrous when the amount of added La reaches to 0.1% (Fig. 4c). The modifying effect of Sr on Si crystal is well known in literature. For example, El Sebaie et al. [21] have shown the strong modification effect of Sr on eutectic silicon particles. Some researchers have reported similar mechanism for La on modifying the eutectic silicon phase due to their diagonal relationship in the periodic table of the elements, according to an impurity induced twinning. Besides, the growths of eutectic silicon particles are limited by concentrating of La on the surface of eutectic silicon particles, because La atomic radius is larger than silicon. Subsequently, the morphology of eutectic silicon phases alters to fine

fibrous structure [16,25]. According to an impurity induced twinning modified Si fibers include more twins than the unmodified ones and have a rough microfaceted surface and each surface imperfection is a potential site for branching. Therefore, the silicon fibers are tend to bend, curve and split in the chemically modified eutectic to create a fine microstructure. Lu and Hellawell [25] believe that a growth twin is generated at the interface when the atomic radius of the element has the accurate size relative to the radius of Si ($r_{\text{element}}:r_{\text{Si}} = 1.65$). Tsai et al. have also mentioned that La element is found to be in the atomic radius range proposed to cause chemical modification [14].

Table 2 shows quantitative results of the microstructural studies for A357 alloy specimens with various La concentrations in as-cast and T6-heat treated conditions.

Table 2

As can be seen in Table 2, significant reduction is seen in size, area and the aspect ratio of eutectic silicon particles in A357 alloy with different La contents. By adding 0.1 wt% La, eutectic silicon particles are seen to be fully modified and their size decreases from 13.8 μm to 7.3 μm . In the present case, the maximum reductions in particle length, area and the aspect ratio of the as-cast A357-0.1% La alloy are 47%, 54% and 57%, respectively. Variation in Si particle size by La addition before applying heat treatment can be similar to the Sr modified Al-Si-Mg alloy systems [16,25]. It is important to note that the size of silicon particles does not change considerably when using more La (1 wt%), as shown in Table 2 and Fig 3d. This can be due to the presence of new intermetallics in La added specimens.

From Tables 2 and Fig 3e-h, it can be seen that T6 heat treatment has a profound effect on the spheroidization of Si crystals, especially in La added specimens. The maximum reduction values in particle length, area and the aspect ratio of the heat treated samples by the addition of

La are 37%, 58% and 16%, respectively. During solution treatment at 540 °C for 8 h, it is expected that the size of Si particles reduces clearly in both La free and La modified alloys. But such modification effect on the size and morphology of eutectic silicon particles after T6 treatment is not observed by the addition of 1 wt% La. It has been also reported that the addition of La reduces the coarsening of the eutectic silicon particles during solution heat treatment [15,16]

Fig. 5 demonstrates the macrostructures of A357 samples with different La contents.

Fig. 5

From Fig.5, it can be seen that the increase in La content lead to a perceptible reduction in the grain size of the cast alloy. Fig. 6 also depicts the variation in average grain size of La modified specimens, obtained from macrostructural measurements.

Fig. 6

From Fig. 5 and Fig. 6, it is clear that in comparison with La free alloy, the addition of 0.1 wt% La reduces the grain size of the cast specimens about 50%. Therefore, the optimum amount of La for obtaining a finer structure is 0.1 wt%.

The mechanisms of grain refinement in A357 alloy by adding rare earth elements have been discussed by several researchers [16,26,27]. The general ideas developed to understand such refinement can be applied to this system too. Essentially, La can thermodynamically react with other exiting elements in the melt to form intermetallic particles capable of introducing proper nucleating sites for α -Al phase during primary stage of solidification. On the other hand, the solubility of La in Al is exceedingly low and during solidification, La element can also be rejected ahead of the solid-liquid interface throughout the time of α -Al grain growth. Thus, a strong constitutional undercooling will take place, which limits the growth and makes further

nucleation of α -Al grains possible [27,28]. The formation of La intermetallics phase (as seen in Fig 3b-d, top right) may lead to reduction in the grain size of La added alloy.

Incorporating small amount of La may form in the melt, even less than 0.1 wt%, can cause extreme reduction in grain size, as seen in Fig. 5 and Fig. 6. When trace amount of La is added to the alloy, eutectic reaction may occur at 637-642 °C, due to the following reaction [16]:

Large numbers of nucleation sites are supplied by the eutectic reaction for the α -Al primary phase. Owing to the rather high melting point of Al-La containing intermetallic compounds (above 1200 °C) and also similarity between lattice constant of Al-La intermetallics and α -Al primary phase, it is highly expected that such intermetallics act as heterogeneous nucleating sites for α -Al primary phases [16]. Based on the EDS analysis, Fig. 7 confirms the presence of Al and La containing products.

Fig. 7

Consequently, by adding very small amount of La (< 0.1 wt%) to the alloy, high melting point intermetallic phases (such as Al_4La) can be formed in the molten alloy which may act as a heterogeneous nucleating site for α -Al primary phase with the globular morphology [16].

In agreement with previous studies [14-16], the EDS analysis and XRD patterns of A357-3 wt% La alloy show new intermetallics (as shown in Fig. 8 and Fig. 9). Similarly, some reports have shown that $Al_{11}La_3$ and Al_4La intermetallics are these new phases [16,29].

Fig. 8

Fig. 9

Previous researchers have shown that at higher La concentrations (> 0.1 wt%) new intermetallic phase (AlSiLa) is identified as well [30,31]. The formation of AlSiLa intermetallic compound begins below the solidus temperature of Al-Si alloy and its contribution to the heterogeneous nucleation could hardly be expected [26]. For this reason such intermetallic is formed by rejecting La through dendrites, retarding the growth rate of α -Al primary and eventually further reduction in grain size of the modified alloy occurs. This could also be explained by growth restricting factor (GRF) of the alloy which increase with adding La to the alloy. As depicted in Fig. 10, SEM micrograph of 1 wt% La added A357 alloy shows similar result, while the series of x-ray images corresponding to specific element are exhibited in Fig. 10b-f.

Fig. 10

From Fig 3b-d, it is also clear that when the amount of La is less than 0.1 wt%, La intermetallics are more globular, but by increasing La content (> 0.5 wt%) they become more acicular or flake shape. Furthermore, in A357 alloy with optimum La level, not only the grain size of the alloy is decreased, but also the size and morphology of eutectic silicon particles are obviously improved. All these structural modifications have the potential to improve mechanical properties of the alloy.

3.2. Tensile properties

Fig. 11 shows typical engineering stress-strain curves of A357 alloy with different La contents, before and after T6 heat treatment. The variation of UTS and elongation values after adding La to the alloy is seen in Fig. 12 in as-cast and T6 conditions.

Fig. 11

Fig. 12

In comparison with La free alloy, Fig. 11 and Fig. 12 depict that the addition of La improves tensile properties of the cast specimens in as-cast and heat treated conditions. Also, applying T6 heat treatment to the specimens leads to an enhancement of both UTS and elongation values. From Fig. 12a, it is evident that the addition of 0.1 wt %La improves UTS value of the alloy from 226 MPa to 267 MPa. Major improvement is achieved by heat treating of the alloy containing 0.1 wt% La in which UTS reaches to the highest value of 420 MPa. Fig. 12b shows that the addition of 0.1 wt% La to the alloy increases El.% from 3.8% to 6.8% in as-cast specimens and from 6.2% to 9% in T6 heat treated alloys.

This enhancement in tensile results is mainly related to the structural changes in the cast alloy. As mentioned before, La addition to A357 alloy causes a structure with reduced grain size, fine silicon particles and modified α -Al primary phases and the presence of fine La containing intermetallics. But, a reducing trend is seen in tensile strength and elongation values of the alloy with higher La contents (> 0.1 wt%). According to the obtained UTS and elongation results, it is evident that the addition of 0.1 wt% La is most effective in improving the tensile properties of the alloy in the both as-cast and T6 heat treatment conditions, as shown in Fig.11 and Fig. 12. The presence of fine La containing intermetallic phase with globular morphology in the microstructure at lower La contents (≤ 0.1 wt%), shown in Fig. 3b and Fig. 7, provides less stress concentration sites and crack initiators with less negative effect on tensile properties. It can be noted that the presence of these intermetallics are barriers for dislocation movement, consequently, every dislocation gliding over the slip plane adds one loop around the particle [32]. These loops exert a back stress on dislocation source which must be overcome for additional slip to take place. This leads to an increase in strength in both as-cast and T6 heat-

treated [33,34], but adding La at higher concentrations (more than 0.1 wt%) causes a significant reduction of UTS values due to the formation of new intermetallic phase (AlSiLa) with flake and needle shape morphology (as shown in Fig. 3d and Fig. 8), may lead to high levels of stress concentration which can provide appropriate sites for crack nucleation and therefore the reduction of tensile properties. These hard La-rich intermetallics are so detrimental to the tensile properties of the alloy and its toughness. Previous works have shown that the rupture mechanism of aluminum alloy was changed by adding trace amount of rare earth elements, and the transgranular/intergranular mixed mode of fracture was reported [35,36].

To quantify the overall tensile properties of an alloy the quality index (Q.I.) which is defined as $Q.I. = UTS + \alpha * \text{Log} (El. \%)$ was utilized, where α is 150 for Al-Si-Mg alloy [14,15,37-39]. This concept arose from the combination of UTS and elongation values are a reliable parameter in quantifying tensile properties for engineering applications. It is much more descriptive of the true tensile properties of castings than either the tensile strength or the elongation alone. The quality index Q.I. evaluated for each alloy is also shown in Fig. 13.

Fig. 13 clearly shows that the addition of 0.1 wt% La leads to an enhancement of the quality index value from 309 MPa to 392 MPa. Besides, after T6 treatment the quality index increases from 467 MPa to 561 MPa. The maximum values of Q.I. for 356 or 357 alloys in as-cast and T6 heat treatment condition were found to be 380 MPa and 520 MPa, respectively [15]. From these results, it is evident that T6 heat treatment is a critical function in enhancing tensile results of La added A357 alloy in optimum condition.

Fig. 13

3.3. Fracture characteristic

Fig. 14 and Fig. 15 demonstrate the fracture surfaces of A357 alloy castings without La and with 0.1 wt% and 1 wt% La before and after T6 heat treatment, respectively.

Fig. 14

Fig. 15

It is evident from Fig. 14a that the fracture surface of La free A357 alloy in as-cast condition exhibits quasi-cleavage feature and a brittle fracture nature. After T6 heat treatment few dimples are seen in the fracture surface of the La free alloy and a mixed quasi-cleavage and dimple morphology is observed.

As shown in Fig. 14b, the fracture face of the specimen containing 0.1% La demonstrates fully dimple-like appearance. So, more ductile behavior is expected in La modified alloy compared to the La free 357 alloy. As shown in Fig. 14b, the dimples are deep and dispersed uniformly, which results in elongation value improvement. Similar results have been reported by some investigators. They have reported that La addition to A357 alloy converts the fracture behavior from brittle, with quasi-cleavage facets to ductile, with fine dimples [15,16].

By comparing the fracture surfaces of specimens in as-cast and T6 heat treatment conditions, it can be noted that T6 heat treatment encourages ductile fracture mode.

Fig. 14c exhibits the fracture surface of the alloy at higher La contents (1 wt%). The presence of La-rich intermetallic compound with flake or needle like morphology plays an important role in changing the fracture behavior of the alloy from ductile to brittle [6,15,16,40]. Aggregation of these brittle intermetallic compounds can make potential sites for stress concentration resulting in a significant deterioration of elongation.

In A357 and A356 alloys, the cracking of silicon crystals in eutectic region and other intermetallic particles initiate damage, because of the development of internal stresses in the particles by plastic deformation [6]. As mentioned before, at higher concentration of La, the increased particle size permits population of defect to be introduced in the particle, which lead to have a lower fracture stress [6,15,16,40]. Alexopoulos et al. have shown the mechanism of void growth and coalescence at Si particles [40]. They also noted that when silicon particles become spherical and small the intergranular fracture and fractured Si particles are happened. Jiang et al. [16] are reported that the fracture mode of unmodified A357 alloy is transgranular but with addition of La fracture mode change from transgranular to intergranular, leading to superior ductility. As a consequence, when Si crystals are changed to fine and spherical by adding 0.1 wt% La to A357 alloy the fracture happens by dimple rupture and also, it can be concluded the intergranular fracture mode may occur that causes superior ductility

4. Conclusions

The influences of La on the structural characteristics and tensile properties of A357 (Al-7% Si-0.5% Mg) alloy in both as-cast and T6 heat treatment condition were investigated. The following conclusions can be drawn:

- (1) The addition of La to A357 alloy modified the eutectic silicon particles significantly. Al_4La and $AlSiLa$ intermetallic compounds were found in the microstructure of the La modified alloy. Increasing the amount of La led to change the morphology of intermetallics from globular to flake shape.
- (2) La addition was found to be powerful in reducing the grain size of A357 alloy under as-cast condition from 620 μm to minimum 300 μm . The reduction in the grain size of the modified

alloy was attributed to the effects of the Al_4La intermetallic as a heterogeneous nucleation sites ($< 0.1 \text{ wt\%}$) and retarding the growth rate of $\alpha\text{-Al}$ primary phase by AlSiLa intermetallic at higher La levels.

(3) It was found that the addition of La improved the tensile properties of A357 alloy compared to the unmodified A357 alloys. T6 heat treatment also enhanced tensile properties of the modified and non-modified alloy significantly.

(4) Fracture surface examination of as-cast alloy showed a brittle mode of fracture. After modifying the structure by La, dimples and cracked particles were found in the fractured surface, indicating ductile mode of fracture. The use of higher La contents ($\geq 1 \text{ wt\%}$) revealed that fracture behavior changed from ductile to more brittle. Also, fractured surfaces of T6-heat treated alloy showed fine dimples which are the characteristic of the ductile mode of fracture.

Acknowledgements

The authors gratefully acknowledge University of Tehran for lab facilities and Iran National Science Foundation for financial support of this work.

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Figures captions

Fig. 1. (a) cast iron mould and (b) tensile testing sample.

Fig. 2. Optical micrograph of A357 alloy casting.

Fig. 3. The microstructures of A357 alloys with different La content: (a,e) 0%, (b,f) 0.1 % La, (c,g) 0.5% La and (d,h) 1% La alloy in (a–d) as-cast condition and (e–h) after heat treatment.

Fig. 4. SEM micrographs of deep-etched samples with : (a) no La; (b) 0.05 wt.% La and (c) 0.1 wt.% La, showing variation in morphological features of Si particles.

Fig. 5. Macrostructures of A357 alloy with: (a) 0% La , (b) 0.05 % La (c) 0.1 % La and (d) 0.5 % La.

Fig. 6. The variation of average grain-size of A357 alloy castings with different La contents.

Fig. 7. (a) SEM image of as-cast A357-0.1wt.% La alloy with relative EDS point analysis of zone A (b) with the line scan analysis.

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TABLES

Table 1. Chemical composition of A357 casting alloy (wt%)

Alloy	Si	Mg	Fe	Cu	Mn	Zn	Al
A357	7.20	0.49	0.09	< 0.01	< 0.01	< 0.01	Base

Table 2. Si particle characteristics in various A357 alloy samples

Sample	No	La (wt%)	particle length (μm)	particle area (μm^2)	Aspect ratio (%)
As-cast	L0	0	13.8 $\pm$ 1.1	16.2 $\pm$ 2.2	4.6 $\pm$ 0.4
	L0.05	0.05	8.4 $\pm$ 0.4	8 $\pm$ 1.1	2.5 $\pm$ 0.2
	L0.1	0.1	7.3 $\pm$ 0.4	7.3 $\pm$ 1.2	2 $\pm$ 0.2
	L0.5	0.5	9.3 $\pm$ 0.6	10.2 $\pm$ 1.9	2.7 $\pm$ 0.3
	L1	1	12.7 $\pm$ 0.9	16.1 $\pm$ 2.0	3.6 $\pm$ 0.4
Heat treated	LH0	0	9.3 $\pm$ 0.4	11.5 $\pm$ 3.2	2 $\pm$ 0.2
	LH0.05	0.05	7.6 $\pm$ 0.3	8.9 $\pm$ 2.9	1.9 $\pm$ 0.2
	LH0.1	0.1	5.9 $\pm$ 0.3	4.8 $\pm$ 0.5	1.7 $\pm$ 0.1
	LH0.5	0.5	8.4 $\pm$ 0.4	9.8 $\pm$ 3.3	1.7 $\pm$ 0.3
	LH1	1	15.7 $\pm$ 0.7	21.6 $\pm$ 4.1	1.8 $\pm$ 0.4