

# Microstructural Evolution of Cast Hyper-Eutectic Al-18% Si Alloy during Cyclic Semi-Solid Heat Treatment

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## Abstract

The cyclic semi-solid heat treatment represents a promising technique for improving microstructure and mechanical properties of a wide range of metallic alloys. In the current research the influence of cyclic semi-solid heat treatment on microstructure of Al-18% Si alloy containing 0.8% Fe has been studied. All specimens were heated in an electrically heated resistance furnace with heating rate of  $10^{\circ}\text{C}\cdot\text{min}^{-1}$  to  $585^{\circ}\text{C}$ . For a complete one cycle heat treatment (5 min heating time), samples after 5 min holding at  $585^{\circ}\text{C}$  were cooled to a temperature of  $550^{\circ}\text{C}$  in still air cooling and the samples were taken out immediately for water quenching. It was found that heat treatment cycles should be limited to 3 cycles or less in order to maintain fine grain size and globular structure without agglomeration and coalescence. Cyclic semi-solid heat treatment changes morphology of iron-rich intermetallics phases to be plate-like and fine plate iron-rich intermetallics phases, in stead of needle-like iron-rich intermetallics phases that are observed in as-cast samples. Cyclic heating shows a relatively higher hardness for all heating cycles compared with as-cast one due to its finer and globular structure. Cyclic semi-solid heat treatment technique results in lower coarsening rate constant compared with isothermal heat treatment one due to coarsening discontinuous effect.

## Keywords

Microstructural, Cyclic, Semi-Solid Heat Treatment, Al-18% Si, Microstructure, Hyper-Eutectic, Cast

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## 1. Introduction

Generally speaking aluminum alloys have good mechanical properties, high corrosion strength and low density. Nowadays the fields of application of this kind of alloys are mainly the ones in which weight reduction is a critical factor, such as in aerospace applications, and in the field of transport in general. Outstanding castability, good wear resistance and low thermal expansion coefficient, together with reduced density, make hyper-eutectic Al-Si alloys such as A390 extensively used in the automotive industry. Based on the Al-Si system, Cu and Mg are commonly added as alloying elements to make these alloys heat treatable, and provide a way to enhance their mechanical properties by precipitation hardening. Normally, their mechanical properties are determined by the phase composition, morphology, distribution and size of precipitate formed during decomposition of the metastable supersaturated solid solution obtained by solution treatment and quenching [1]-[3].

Iron improves hot tear resistance and decreases the tendency for die sticking or soldering in die casting. Increasing iron content is, however, accompanied by substantially decreased ductility. Iron reacts to form a myriad of insoluble phases in aluminum alloy melts, the most common of which are  $\text{FeAl}_3$ ,  $\text{FeMnAl}_6$ , and  $\alpha\text{AlFeSi}$ . These essentially insoluble phases are responsible for improvements in strength, especially at elevated temperature. As the fraction of insoluble phase increases with increased iron content, casting considerations such as flowability and feeding characteristics are adversely affected. Iron participates in the formation of sludging phases with manganese, chromium, and other elements [4].

The metallurgy of aluminum and its alloys fortunately offers a wide range of opportunities for employing thermal treatment practices to obtain desirable combinations of mechanical and physical properties. Through alloying and temper selection, it is possible to achieve an impressive array of features that are largely responsible for the current use of aluminum alloy castings in virtually every field of application. Although the term heat treatment is often used to describe the procedures required to achieve maximum strength in any suitable composition through the sequence of solution heat treatment, quenching, and precipitation hardening, in its broadest meaning, heat treatment comprises all thermal practices intended to modify the metallurgical structure of products in such a way that physical and mechanical characteristics are controllably altered to meet specific engineering criteria [4].

The influence of semi-solid isothermal heat treatment on microstructure of hyper-eutectic Al-18% Si alloy has been studied in previous work [5]. It was found that the optimum semi-solid heating treatment condition was achieved at the temperature of  $565^\circ\text{C}$  for the range of 15 to 25 min. Otherwise, increasing the heating time above 25 min increased the chance for grain coarsening [5]. Regarding those points mentioned above, this work is carried out to study microstructural (grain size and grain sphericity) evolution of Al-18% Si alloy containing 0.8% Fe (iron-rich intermetallics) during cyclic semi-solid heat treatment for short heating time range of 5 to 20 min to maintain the optimum of both grain size, grain sphericity and iron-rich intermetallics morphology.

## 2. Materials and Methods

The Al-18% Si alloy studied in the present study was melted in heat resistance furnace using rods stock of 50 mm diameter. The chemical composition of alloy samples is shown in **Table 1**. After flux treated, the melt was held at  $715^\circ\text{C}$  for 5 min and then poured into a permanent mould dimensioning  $20\text{ mm} \times 20\text{ mm} \times 180\text{ mm}$ . The differential scanning calorimetric analysis (DSC) for the studied Al-Si alloy was conducted showing that eutectic temperature ( $T_E$ ) of  $570.5^\circ\text{C}$  and Liquidus temperature ( $T_L$ ) of  $665^\circ\text{C}$ .

**Table 1.** Chemical analysis of Al-Si samples, wt%.

Element	wt%	Element	wt%	Element
Si	17.20	Mn	0.02	Si
Cu	4.10	Sn	0.06	Cu
Mg	0.50	Ti	0.05	Mg
Fe	0.80	V	0.03	Fe
S	0.01	$T_L$ ( $^\circ\text{C}$ ) <sup>a</sup>	665	S
Ni	0.31	$T_E$ ( $^\circ\text{C}$ ) <sup>b</sup>	570.5	Ni

Specimens of approximate dimensions  $20 \times 20 \times 20$  mm were cut for cyclic heat treatment as well as microstructure examination and hardness measurements. All specimens were heated in an electrically heated resistance furnace with heating rate of  $10^\circ\text{C}\cdot\text{min}^{-1}$  to  $585^\circ\text{C}$ . For a complete one cycle heat treatment samples after 5 min holding at  $585^\circ\text{C}$  were cooled to a temperature of  $550^\circ\text{C}$  in still air cooling and were taken out immediately for water quenching. For two cyclic heat treatment samples after 5 min holding at  $585^\circ\text{C}$  were cooled to a temperature of  $550^\circ\text{C}$  in still air cooling and were heated again to  $550^\circ\text{C}$  for the next 5 min heating time were cooled to a temperature of  $550^\circ\text{C}$  in still air cooling and finally the samples were taken out immediately for water quenching (see **Figure 1**).

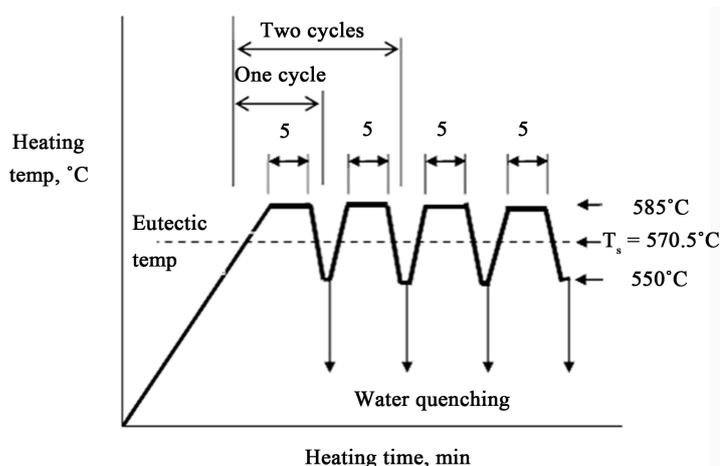
Specimens in either as cast or heat treated condition were grinded, polished, etched with a solution consist of 0.5% HF and 99.5%  $\text{H}_2\text{O}$  and examined metallographically using an optical microscope and photomicrographs were taken. Primary silicon particle sphericity ( $S = 4\pi \times (\text{area of the grain}/\text{grain circumference}^2)$ ) and primary silicon particle size, were measured and analyzed with Scentis image analyzer software (with errors 5%) and JOEL JSM-5410 Scanning Electron Microscope (SEM) with EDS for microanalysis. Rockwell hardness test were also performed using 1/16 inch diameter ball and 100  $\text{kg}_f$  load.

### 3. Results and Discussion

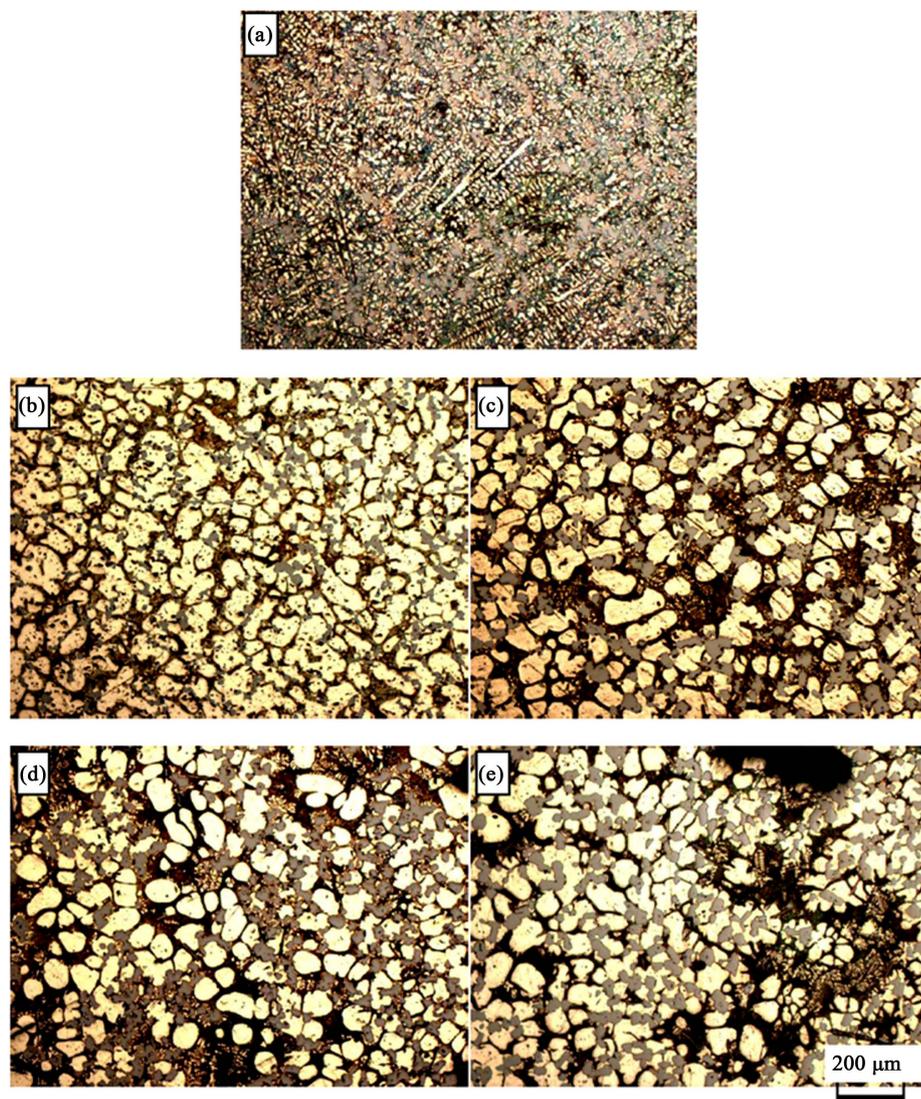
The as-cast and cyclic semi-solid heat treated (from 1 to 4 cycles) microstructures of cast Al-18% Si alloy containing 0.8% Fe shown in **Figure 2**. It can be seen from **Figure 2(a)** grains of as-cast Al-18% Si alloy clearly exhibits the Primary Si and eutectic Al-Si (eutectic  $\alpha$  phase + eutectic Si). It can also be see some denderitic  $\alpha$ -Al ferrite phase and non-uniform distribution of primary Si phase in as-cast samples microstructure. Previous Study [4] reported that presentence of  $\alpha$  (Al) dendrites could be due to the higher cooling rate in the as-cast condition. On the other hand, a globular  $\alpha$ -Al grain and relatively uniform distribution of primary Si phase can be observed for cyclic semi-solid heat treated one up to 3 cycles (see **Figures 2(b)-(d)**). For samples that heat treated for 4 cycles, both of  $\alpha$ -Al grain and primary Si phase showing agglomeration condition (see **Figure 2(e)**).

**Figure 3** and **Figure 4** show effect of cyclic semi-solid heat treated on grain size and grain sphericity of primary Si particle and for  $\alpha$ -Al grain. For all measurements and up to 3 cycles, a significant effect of cyclic heat treatment on improving the grain microstructure is observed, whereas, cyclic semi-solid heat treated samples has a relatively fine and more globular grain compared with as-cast one. By increasing the number of cycles above 3 cycles, grain size increases and sphericity decreases for both primary Si particle and  $\alpha$ -Al grain.

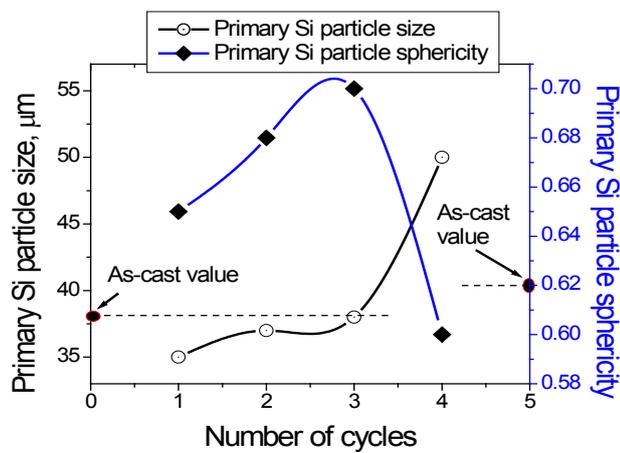
**Figure 5** shows microstructure of cyclic semi-solid heat treated samples for 3 cycles as shown in **Figure 5(a)** and 4 cycles as shown in **Figure 5(b)**. Agglomeration and coalescence of both primary Si particle and  $\alpha$ -Al grain during 3 and 4 cycles are observed. The microstructure evolves; extensive coarsening by increasing the number of cycles (heating time) that will have a deleterious effect on the final mechanical properties of the heat treated samples. In addition, if the grain size is too large, it will limit production of thin section products with considerable mechanical and physical properties.



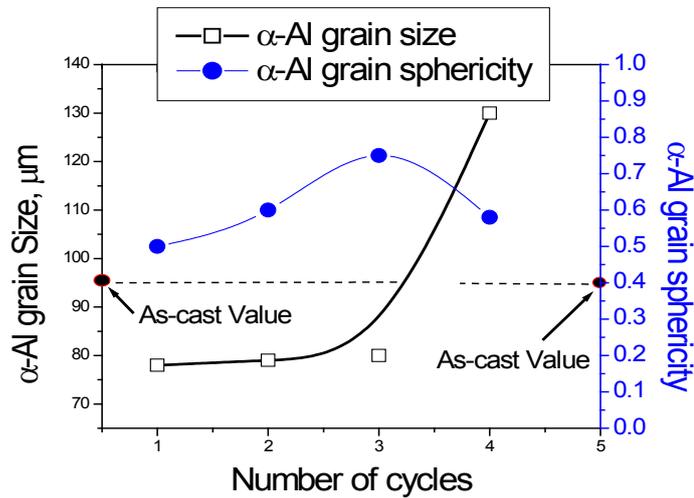
**Figure 1.** Schematic illustration of the cyclic heat treatment process.



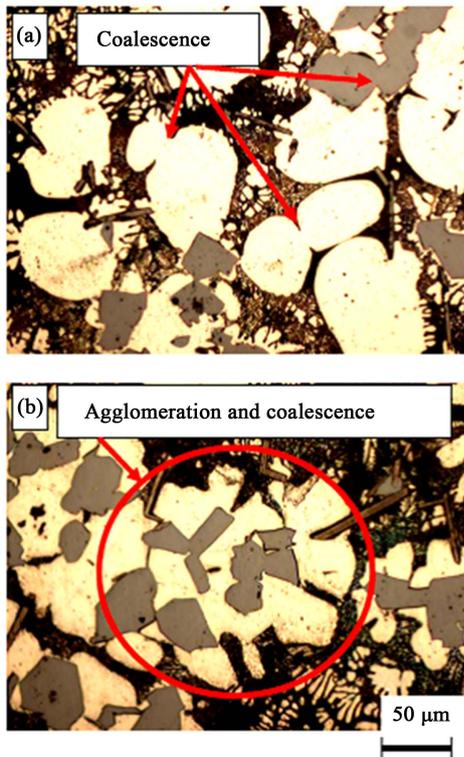
**Figure 2.** Microstructure of semi-solid cyclic heat treatment of Al-18% Si at 585°C for (a) As-cast; (b) One cycle; (c) Two cycles; (d) Three cycles; (e) Four cycles.



**Figure 3.** Effect of number of semi-solid heat treatment cycles on primary Si particle size and its sphericity.



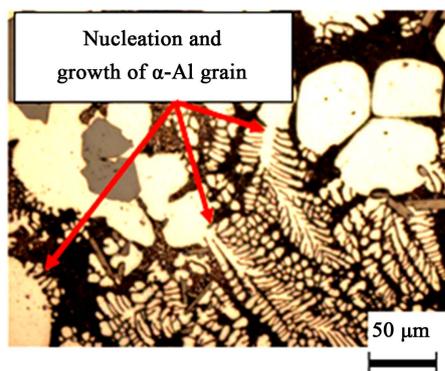
**Figure 4.** Effect of number of cycles for semi-solid heat treatment on  $\alpha$ -Al grain size and its sphericity.



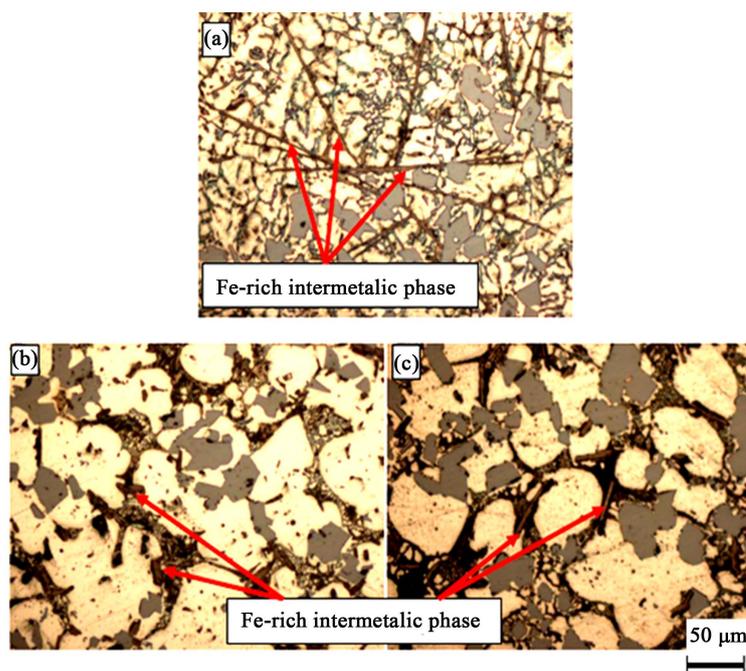
**Figure 5.** Agglomeration and coalescence of  $\alpha$ -Al and primary Si Grain for (a) Three cycles and (b) Four cycles heating times.

**Figure 6** shows a nucleation of new dendritic  $\alpha$ -Al grains start at 3 cyclic semi-solid heat treatment. It is clear that this new dendritic  $\alpha$ -Al grain starts its heterogamous nucleation on pre-exist globular  $\alpha$ -Al grain.

**Figure 7** shows the effect of cyclic semi-solid heat treatment on the morphology of Fe-rich intermetallics. The microstructure of Al-18% Si alloy for as cast and semi-solid cyclic heated samples at 585°C for two cyclic and three cyclic heating times is characterized by needle-like iron-rich intermetallics phases for as-cast samples, otherwise, plate-like and fine plate-like iron-rich intermetallics phases was observed for cyclic semi-solid heated samples. Fe-rich intermetallic phase shown in **Figure 7** is confirmed experimentally by backscattered images



**Figure 6.** Nucleation and growth of  $\alpha$ -Al grain from liquid at 585°C for three cyclic heating times.



**Figure 7.** Change of Fe-rich intermetallic phase for (a) As-cast; (b) Two cyclic and (c) Three cyclic heating times.

and EDS analysis shown in **Figure 8** significantly influenced by cyclic semi-solid heat treatment. Previous studies [6]-[9] stated that a reduction in wear resistance of 1.2 Fe alloy compared to the base alloy (LM28 alloy) can be explained based on the microstructural features of the alloys. Addition of iron to the LM28 alloy led to the precipitation needle-like  $\beta$ -phase intermetallic in the matrix.  $\beta$ -Al<sub>15</sub>FeSi needle-like intermetallics are hard and brittle phases. They exist as discrete particles with a highly faceted nature in the alloy matrix. Accordingly, it has relatively low bond strength with the matrix and the interfacial regions between this phase and the matrix become quite prone to microcracking. Moreover, sharp edges of the  $\beta$ -needles introduce severe stress concentrations in the matrix of the alloy [10].

**Figure 9** shows a quantitative measurement for a relation between Fe-rich intermetallic length and number of cycles. It is clear that cyclic semi-solid heat treatment has a great effect on the Fe-rich intermetallics length, since a value of about 50 - 60  $\mu$ m Fe-rich intermetallics length was obtained from about 160  $\mu$ m Fe-rich intermetallics length for the as-cast condition.

**Figure 10** shows hardness measurements of both as-cast and cyclic semi-solid heat treated Al-18% Si alloy samples. It is clear that hardness values for cyclic semi-solid heating increase with increasing numbers of cycles.

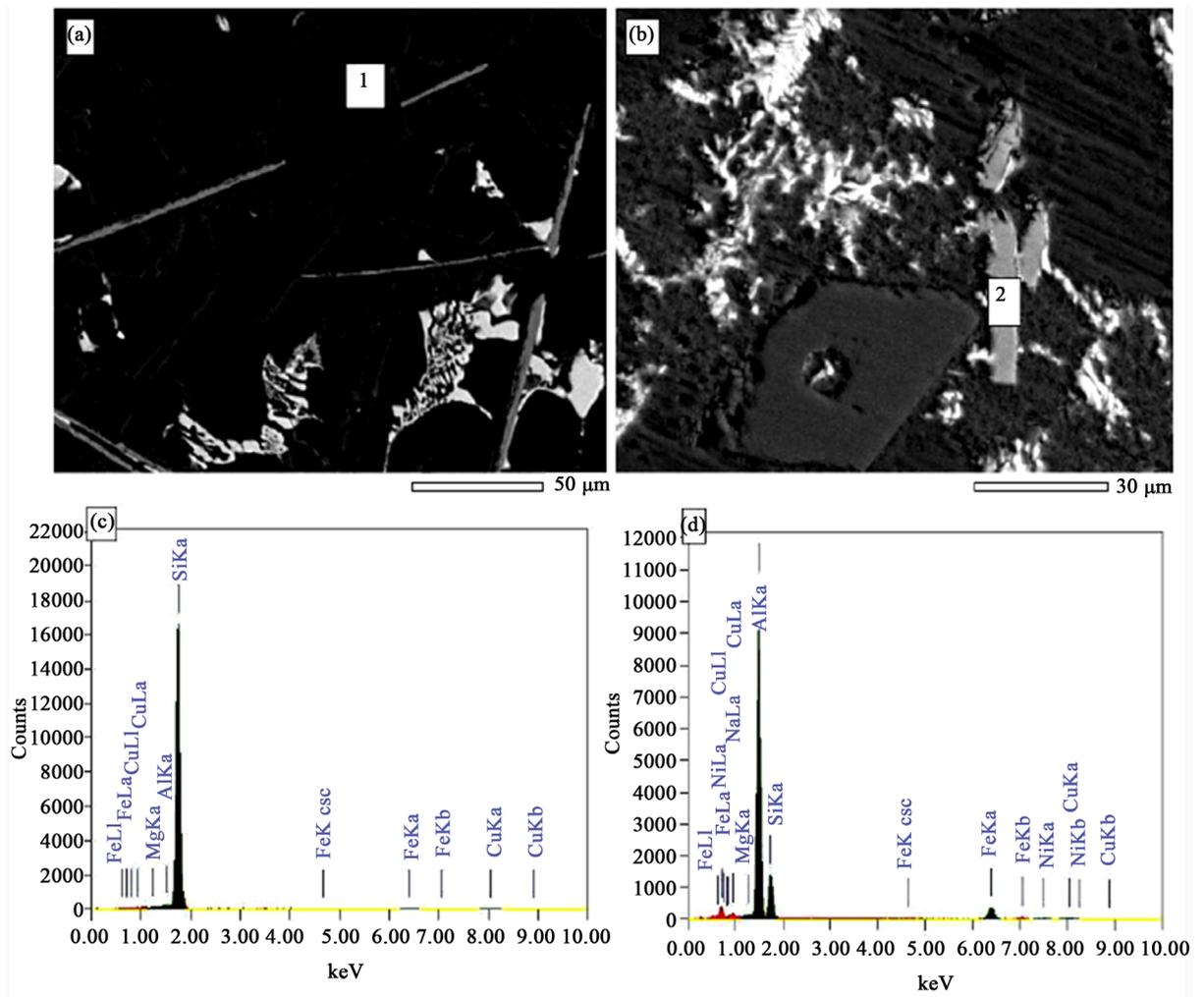


Figure 8. Backscattered images Showing and EDS analysis of locations marked point 1 (primary Si phase) and point 2 (Fe-rich intermetallic Phase) for (a) & (c) as-cast sample and (b) & (d) semi-solid cyclic heat treated for 2cycles sample.

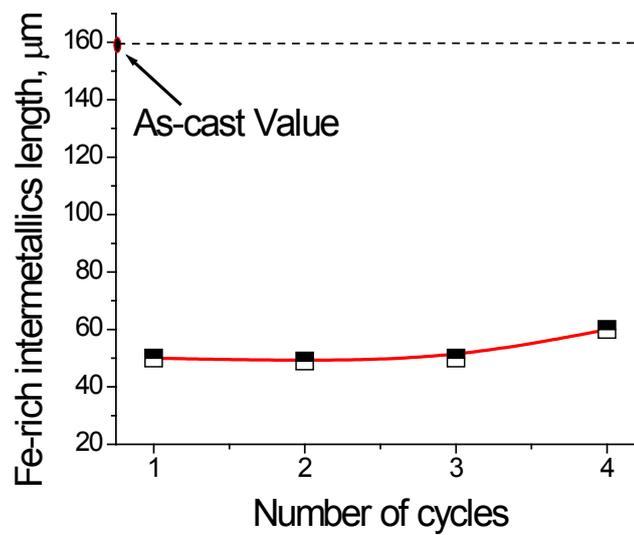


Figure 9. Fe-rich intermetallics length as a function of number of cycles.

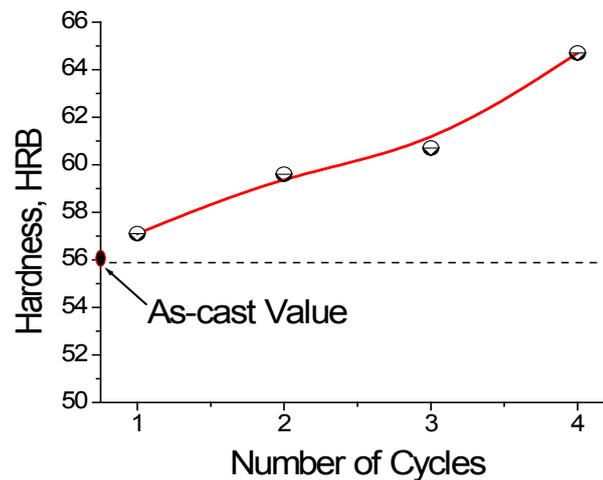


Figure 10. Hardness as a function of number of cycle.

Hardness values for cyclic semi-solid heat treated samples are relatively higher than the hardness value of as-cast one for all heat treating cyclic conditions in this study.

Previous research [11] indicated that during partial re-melting, the alloy was heated up to a temperature at which the solid and liquid phases coexist in equilibrium. Its features lie in obtaining the desirable nominal liquid fraction through the control of temperature, and realizing long-time holding to ensure complete transition from dendritic or rosette to spherical. However, a long-time holding often results in the coarsening of grains, which is detrimental to the mechanical properties of produced parts. During partial melting and then holding in the semi-solid state, coarsening first proceeds through the coalescence of dendrite arms. After coalescence, phase coarsening will take place through the dissolution of the small globules and the grain numbers will then decrease [12].

The coarsening behavior of different semisolid alloys by using the Lifshitz, Slyozov and Wagner (LSW) theory has studied [13]-[15] which gives a simple form as follows.

$$d^3 - d_0^3 = Kt$$

where  $d$  is the average particle diameter at time  $t$ ,  $d_0$  at time  $t = 0$  and  $K$  is the coarsening rate.

Comparing to the previous study [5] dealing with microstructural evolution of hyper-eutectic Al-18% Si alloy during semi-solid isothermal heat treatment, the coarsening rate for isothermal and current study cyclic heating of Al-18% Si alloy was calculated by using Equation (1). It was found that cyclic heat treated Al-18% Si alloy is showing lower coarsening rate constant than that of the isothermal heat treated one ( $K_{\text{Cyclic}} \sim 43\% K_{\text{Isothermal}}$ ) because of coarsening discontinuous actions of cyclic heat treatment.

During the isothermal treatment in semi-solid state, increasing heating time resulted in higher liquid fraction in the parts. As the liquid fraction is increased most of the precipitated phases, which are strengthening phases in the structures, are continuously dissolved in the liquid phase resulting in higher coarsening rate [16]. Otherwise, during the cyclic heat treatment, the short time heating followed by cooling will relatively decreases the rate of precipitates migration to liquid. The relatively high precipitates remain in solid phase could be the reason of relatively decreasing in the coarsening rate of cyclic heat treatment.

#### 4. Conclusions

Experimental studies were carried out to determine the influence of cyclic semi-solid heat treatment on the microstructure of Al-18% Si alloy containing 0.8% Fe. The following conclusions are made from the findings:

- 1) Cyclic semi-solid heat treatment improves the microstructure to be a relatively fine and more globular compared with as-cast one.
- 2) Heat treatment cycles should be limited to 3 cycles or less in order to maintain fine grain size and globular structure without agglomeration and coalescence.
- 3) Needle-like iron-rich intermetallics phases were observed in as cast samples, while plate-like and fine plate

like iron-rich intermetallics phases were observed for all cyclic semi-solid heat treatment samples.

4) Cyclic heating shows a relatively higher hardness for all heating times compared with as-cast one due to its finer and globular structure.

5) Cyclic semi-solid heat treatment shows lower coarsening rate constant compared with isothermal heat treated one due to the coarsening discontinuous effect.

6) The obtained data help in improving the processing conditions of optimized preheating condition for thixo-forming of Al-18% Si alloy.

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