MICROSTRUCTURAL STUDIES OF Ti-REFINED Al2014 ALLOY PREPARED BY SIMA PROCESS

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Abstract: *In this study, Al2014 alloy refined with Al-5%Ti-1%B master alloy was prepared by strain-induced melt activated (SIMA) process. The main variables of the SIMA process were cold working, holding time and temperature in semi-solid state. Cold working was applied on specimens by upsetting technique to achieve 10%, 20% and 30% height reduction. Cold worked specimens were heat treated in semi-solid state at 585 °C, 595 °C, 605 °C, 615 °C, 625 °C and 635 °C and were kept in these temperatures for different times (20 and 30 min). Observations through optical and scanning electron microscopy were used to study the microstructural evaluation. The results revealed that fine and globular microstructures are obtained by applying 30 % height reduction percentage and heat treating in 625 °C for 30 min. Comparison between refined and unrefined Al2014 alloy after applying SIMA process showed that Al-5%Ti-1%B master alloy has no significant effect on average globule size but makes the final structure more globular.*

Keywords: *Aluminum alloy; Grain refinement; SIMA process; Microstructure*

1. INTRODUCTION

The most common aluminium alloys which have been used in important industrial applications such as aerospace and automotive are 2xxx and 7xxx series. In comparison with other aluminum alloys, even steel and titanium alloys, they have high strength per weight ratio [1]. Producing parts with aluminum alloys via usual manufacturing routs is accompanied by certain drawbacks including the formation of porosity, hot tears and segregation which may act as potential crack source during service [2]. Therefore, there have been considerable efforts to minimize these problems which resulted in introducing more advanced semi-solid forming (SSF) routes such as rheoforming and thixoforming [3-5]. Thixocasting is one of the SSF techniques. In this technique, solid specimens are heated between solidus and liquidus temperatures and then formed into desired shapes. In this technique, the microstructure of first specimen directly affects the final mechanical properties [6]. The ideal microstructure is globular, because in this case tension is distributed uniformly while forming and tension concentration in the corners and sharp edges of the products is reduced significantly[7]. In order to produce globular microstructure, one of the most efficient routes is electromagnetic stirring but this method needs complex equipments and imposes high costs and in addition the final structure of EMS produced billets are not completely globular [8]. Recently, the new modified strain-induced melt activated (SIMA) process introduces the desired structures by deformation and a following heat treatment in the temperature range of the mushy zone. During these stages residual strain is stored and causes evolving a globular structure while heat treating in semi-solid state. Parameters such as heating time, temperature and the degree of cold working are critical factors in controlling the microstructure in SIMA process [9].

Several research works related to the SIMA process have been already carried out on aluminum alloys. Applying SIMA process on 7xxx series aluminum alloys refined with Al-8B master alloy have been carried out by some researchers showed that with the increase of strain, average grain size decreases and the sphericity and roundness of the particle increases in less reheating times [9]. The research work on Al2024 alloy by Choi et al. have shown that with increasing effective strain and simultaneously with securing uniform distribution of effective strain during cold working, the degree of sphericity increases [10]. Sirong et al. also showed that for cold-rolled bars of Al2024 alloy, the grain size decreases from 72 µm to 52 µm, after heating in an optimum temperature between solidus and liquidus for an appropriate holding time [11]. Emamy et al. showed that Ti refined 6070 aluminum alloy reveals a finer and more globular structure after applying SIMA process in comparison with unrefined one [12].

The aim of current research is investigating the effect of grain refinement by Al-5%Ti-1%B and main variables of SIMA process on final microstructure of Al2014 alloy.

2. EXPERIMENTAL PROCEDURE

Al2014 alloy ingots were produced by melting pure Al, Cu,Mg, Si and Mn-containing master alloy in an electrical resistance furnace and pouring in a cast iron mould. The chemical composition of this alloy is shown in Table 1. The refinement of the alloy was made by adding different amounts of Al-5%Ti-1%B grain refiner to achieve 0.01%, 0.03%, 0.05%, 0.1% 0.3% and 0.5% Ti in final composition of the Al2014 alloy. For this purpose, the chopped Al2014 ingots were remelted in a graphitic crucible in 760 ------- and different amounts of Ti containing master alloy were added to the molten alloy. Degassing was carried out by submerging dry C2Cl6 tablet into the molten alloy for about 2 min. After cleaning off the dross from the melt, it was poured into an iron mould, as shown in Fig. 1.

The microstructural examination was carried out to obtain the optimum amount of titanium through average grain size variations. The cast specimens with and without optimum amount of titanium were machined to cylindrical shapes (30 mm in diameter and 40 mm in height) before applying SIMA process. Schematic of SIMA process is shown in Fig. 2.

Upsetting was applied on machined specimens to achieve different amounts of height reduction (10%, 20% and 30%). Height reduction percentage (HR%) was calculated according to Eq. (1).

$$HR\%=100(h_o-h)/h_o \tag{1}$$

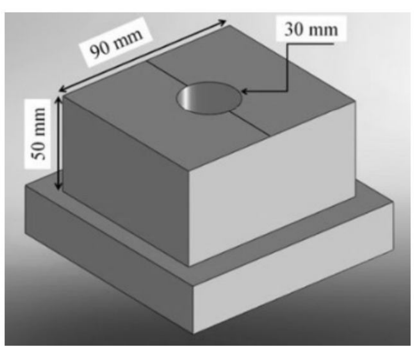

Fig. 1. Schematic drawing of casting mould.

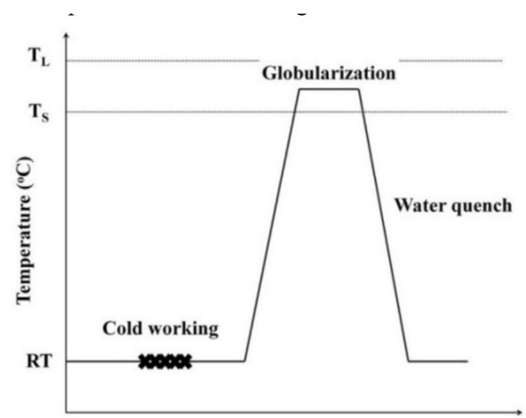

Fig. 1. Schematic drawing of casting mould.

Table 1. Chemical composition of Al2014 alloy.

Element	Cu	Mg	Mn	Si	Al
Wt. %	4.62	0.48	0.71	0.84	Rem.

In Eq. (1), ho and h are the measured heights before and after upsetting, respectively.

In the next step, cold-worked unrefined and Ti-refined Al2014 alloys containing optimum amount of Ti were machined into specimens with 10 mm diameter and 15 mm height for applying heat treatment in semi-solid state. The specimens were heat treated in the temperature range of 585-635 °C by a calibrated induction furnace (calibration deviation ±1°C). Specimens were entered in temperature reached furnace for 20 and 30 min and consequently they were quenched in cold water. It has been reported that for Al-Cu alloys (2xxx series), by increasing temperature from 585 °C to 635 °C, solid fraction decreases from 90% to 30% approximately [11]. This temperature range is an appropriate region for applying SIMA process. To reveal the microstructures of heat treated samples, the cut sections were polished and then etched by Keller'sreagent (2 ml HF, 3ml HCl, 5ml HNO_3 and 190 ml H_2O).

The average grain sizes were measured by using image analysis software (Clemex Vision Pro. Ver.3.5.025). Calculations of average grain sizes were made according to the ASTM E112-10 standard.

In order to determine the progress of globularization, Eq. (2) was used to determine sphericity factor.

$$F = 4\pi S/P^2 \tag{2}$$

where F is sphericity factor which varies between 0 and 1, S is average grains area and P is average grain circumference [13].

Further microstructural characteristics of the specimens were examined by scanning electron microscopy (SEM) performed in a Cam Scan MV2300 SEM, equipped with an energy dispersive X-ray analysis (EDX) accessory.

3. RESULTS AND DISCUSSIONS

3. 1. Effect of Al-5%Ti-1%B on Al2014 alloy in as-cast condition

Fig. 3 illustrates typical microstructures of Al2014 alloy refined with different Ti contents (0-0.5 wt.%). From Fig. 3, it is seen that Ti addition results in a finer structure in comparison with un-refined Al2014 alloy. The variation of Ti addition with average grain size is shown in Fig. 4.

From Fig. 4, it is clear that 0.03 wt.% Ti addition reduces the grain size of the alloy significantly (from 1000 μm to 120 μm) and

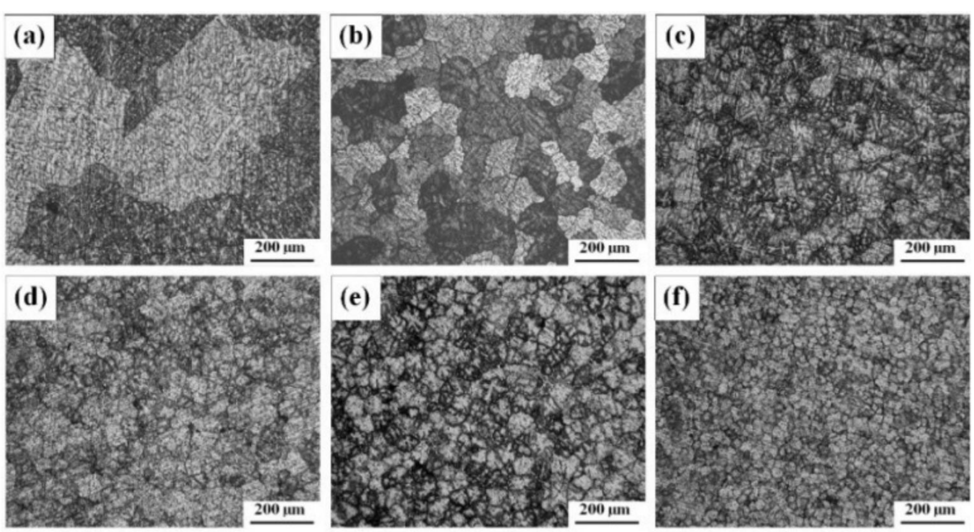

Fig. 3. Structures of Al2014 alloy, before (a) and after adding (b) 0.01% Ti, (c) 0.03%Ti, (d) 0.05% Ti, (e) 0.1%Ti and (f) 0.5% Ti.

adding more Ti (> 0.03 wt.%) has marginal effect on the average grain size. According to the results, the addition of 0.6 wt.% A-5%Ti-1%B (0.03 wt.% Ti) has been selected as the optimum amount of the grain refiner.

Fig. 3 reveals that in Ti-refined Al2014 alloy, (α) Al primary phase has a rosette-like morphology which is surrounded by inter-dendritic secondary phases (Fig. 3b-g). In order to achieve fine microstructures of aluminum alloys, several works confirm the strong effect of Al-5%Ti-1%B master alloy in altering microstructure of Al alloys from coarse dendritic to a fine equi-axed structure by adding trace amounts of Ti (<0.1% Ti) [14-17]. Several mechanisms have been proposed for grain refining process of Al alloys after adding Ti containing master alloy [14-16]. The most probable mechanism is introducing Al3Ti particles which provide appropriate nucleation sites for Al alloys during solidification [17].

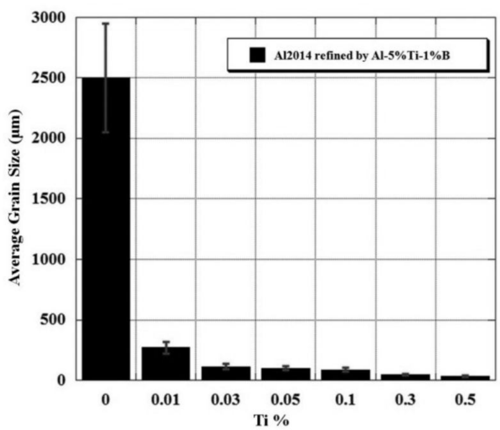

Fig. 4. The effect of Ti addition (wt.%) on average grain size of Al2014 alloy.

3. 2. Effect of SIMA process on the microstructures of Ti-refined Al2014 alloy

Fig. 5 illustrates the microstructure of Ti refined Al2014 alloy after applying SIMA process with 10% height reduction and keeping specimens in the temperature range of 585-635 °C for 30 min.

From figures 3 and 5, it can be seen that SIMA process altered dendritic and relatively coarse microstructure to a fine and relatively globular one. There are some presented mechanisms for producing a fine and globular microstructure

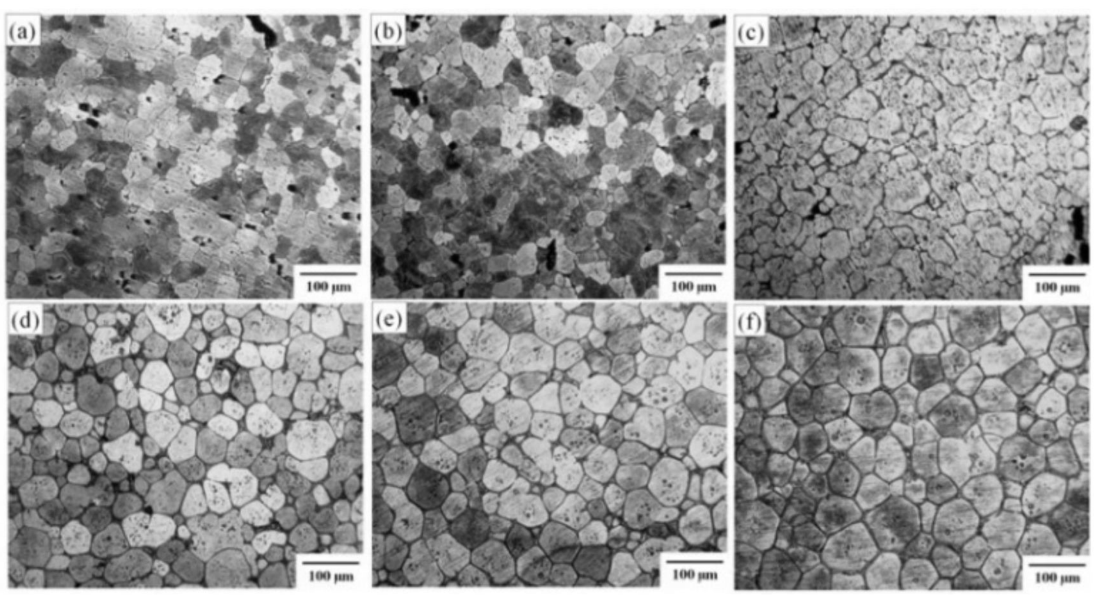

Fig. 5. Microstructures of Al2014 alloy containing 0.03 wt.% Ti, prepared by SIMA process at (a) 585 °C (b) 595 °C (c) 605 °C (d) 615 °C (e) 625 °C (f) 635 °C, for 30 min. with 10% HR.

after applying SIMA process but the most probable one for this phenomenon has been suggested by Kirkwood [18].

According to this mechanism, recrystallization of previously deformed specimens in semi-solid state is the main reason of such modification. Furthermore, when the energy of the newly formed grain boundaries (σ_b) is twice greater than of the energy of the solid/liquid interface (σ_{sl}), liquid phase affects those recrystallized grain boundaries and a fine and globular microstructure can be introduced. Sirong et al. have also developed a model for SIMA process [11]. According to this model, while heat treating in semi-solid state, remelting of grain boundaries starts first, because of the presence of higher level of free energy in this area. This may cause the microstructural changes from dendritic to globular. The relationship between curvature of grain surface and reduction in equilibrium melting point, which is presented in Eq. (3), can clear this fact.

$$\Delta T_r=(-2\sigma T_M V_S K)/\Delta H \qquad (3)$$

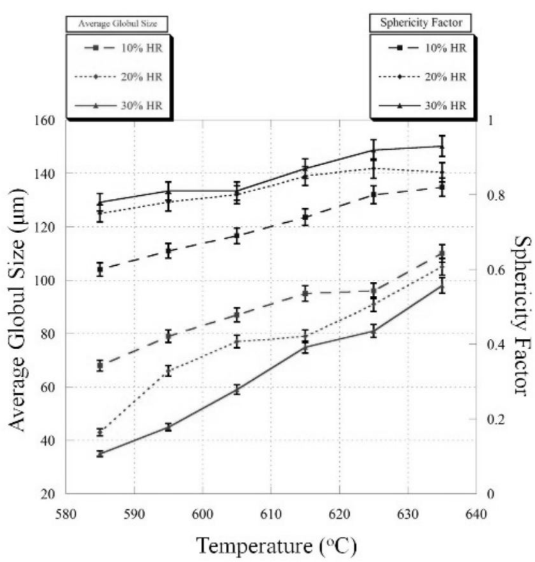

Fig. 6. Variations of average grain size and sphericity factor in Ti-refined Al2014 alloy with 10, 20 and 30% HR, versus holding temperature in semi solid state for 20 min.

In Eq. (3), $T_r = T_M - T$ is the decrease in equilibrium melting point, T_M is the equilibrium transformation temperature, K is the mean surface curvature of the solid, V_S is the solid volume, σ is the surface tension and $\Delta H = H_S - H_L$ (which is negative) is the molar change in enthalpy of the solid and liquid. When the surface curvature is positive, the equilibrium melting point is reduced at the dendrite tip, and Tr in the above equations is positive. The more the surface curvature, the more the equilibrium melting point is reduced. Thus, the sharp tip of the grain will melt and the grain will change to a globular one without sharp corners. Furthermore, producing a fine microstructure after SIMA process can be contributed to increase in the density of dislocations and formation of dislocation walls during cold working which make subgrains. In second step of SIMA process, while heat treating in semi-solid state, this subgrains turn to independent grains and make the microstructure finer [11, 12].

Figures 6 and 7 illustrate variations of average grain size and sphericity factor in Ti refined Al2014 alloy with different amounts of pre-deformation (10, 20 and 30% HR) versus holding

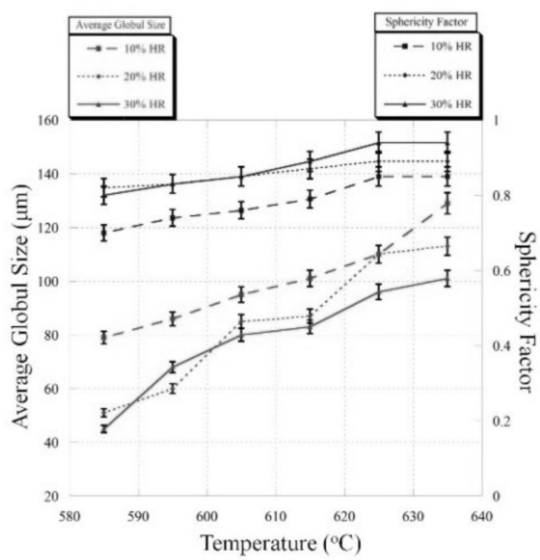

Fig. 7. Variations of average grain size and sphericity factor in Ti-refined Al2014 alloy with 10, 20 and 30% HR, versus holding temperature in semi solid state for 30 min.

temperature in semi solid state for 20 and 30 min, respectively. From figures 6 and 7 it is clear that, specimens with higher amount of pre-deformation (30% HR) have a finer final structure.

Results show that pre-deformation is the main parameter of SIMA process in comparison with temperature and holding time. It is clear that a finer microstructure is produced when higher amount of pre deformation is used. It is concluded that when Al2014 alloy is kept in semi solid state for 30 min, 10% HR is not able to make the structure fine enough because 10% HR is not sufficient to active mechanisms of grain dividing [19].

With the increasing pre deformation, more distortion energy is stored in the forms of vacancies, lattice defects and dislocation multiplications, which would provide the driving force for recrystallization during heat treating in semi solid state. With the increasing driving force, the recrystallized grains become finer. In addition, the higher the pre deformation ratio is, the greater the overall grain boundary and sub-grain boundary area will be. This leads to greater potentiality for the development of recrystallized nuclei [20]. As mentioned before, figures 6 and 7 illustrate that byincreasing the temperature of heat treating in semi solidstate causes to achieve a more globular but coarse structure.

Coarsening is a common phenomenon while increasing the temperature during heat treatment in semi solid state. Coalesence of adjoinig grains and ostwald ripening are two main mechanisms have suggested for this phenomenon [21, 22]. Previous works showed that presence of high liquid fraction in SIMA process decreases the adjoinment between grains and possibility of grain coalesence and makes ostwald ripening as the dominated mechanism of grain coarsening [23].

It is seen that by increasing the temperature, sphericity factor increases too. It is clear that in 625 °C, structure is nearly globular and sphericity factor is more than 0.9. The extensive dependence of globularization to the temperature can be due to the proposed globularization Mechanism. According to previous explanations, the most probable mechanism of globularization is melting the grains with sharp tips and altering irregular shapes to globular structure. According to the proposed mechanism, by increasing the

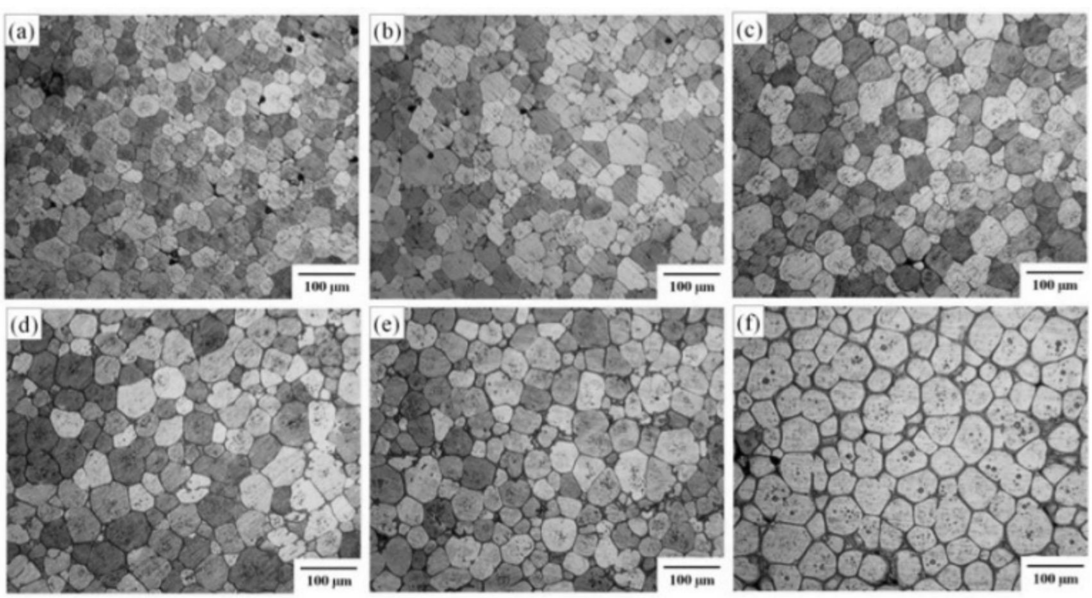

Fig. 8. Microstructures of Al2014 alloy containing 0.03 wt.% Ti, prepared by SIMA process at (a) 585 °C (b) 595 °C (c) 605 °C (d) 615 °C (e) 625 °C (f) 635 °C, for 30 min. with 30% HR.

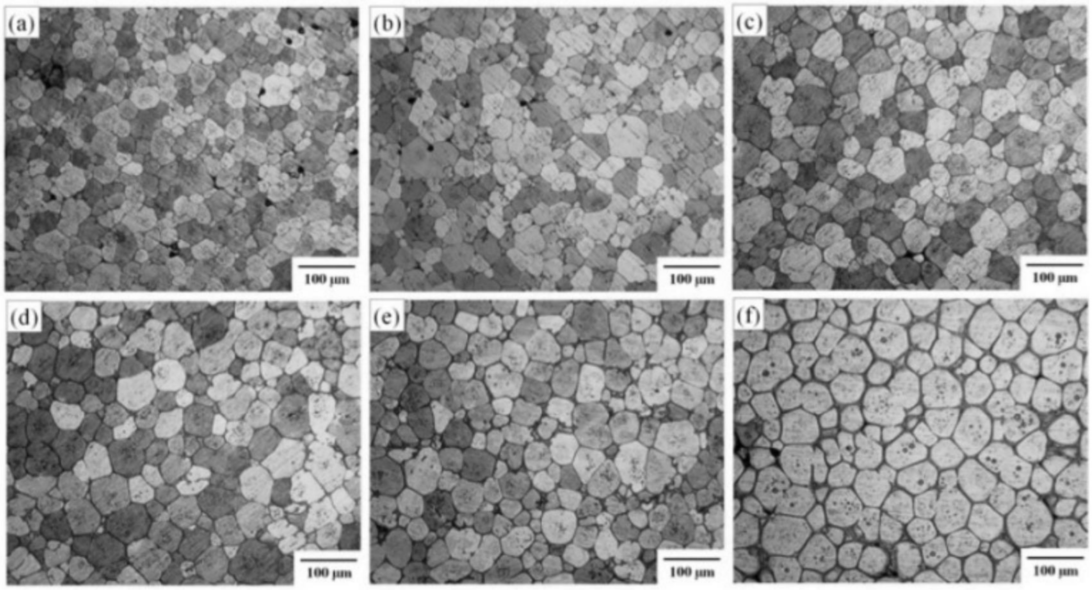

Fig. 9. Microstructures of un-refined Al2014 alloy prepared by SIMA process at (a) 585 °C (b) 595 °C (c) 605 °C (d) 615 °C (e) 625 °C (f) 635 °C, for 30 min. with 30% HR.

temperature, the grain with lower curvature can be easily melted and a globular microstructure is obtained [11].

As expected, after melting all tips and corners in the microstructure, using higher temperatures cannot be effective. For this reason increasing the temperature (more than 625 °C) does not enhance the sphericity factor results and diagram reaches a saturation area. As mentioned before, an appropriate structure for SSF routes, is a fine and globular one. Results revealed that by increasing the holding temperature in semi solid state, both average grain size and sphericity factor increase too.

According to figures 6 and 7, no more grain growth will occurred when holding time increased from 20 to 30 min, but result in a more globular structure.

Holding Ti-refined Al2014 alloy with 30% HR in 625 °C for 30 min, has caused to achieve a globular structure that has not grown extremely. Sphericity factor and average globule size in these conditions are 0.94 and 96 µm, respectively.

Figures 8 and 9 illustrates the microstructure of Ti-refined and un-refined Al2014 alloy containing 30% HR and heat treated in semi solid state (585-635 °C) for 30 min, respectively. Fig. 10 also shows the variation of average grain size and sphericity factor of the un-refined and Ti-refined alloy specimens containing 30% HR as a function of holding temperature for 30 min. results showed that there is no significant difference between globules size in un-refined and Ti-refined Al2014 alloy after applying SIMA process but the final structure in case of Ti-refinement, is significantly more globular. It can be concluded that grain refining effect of SIMA process is too strong that there is no significant difference between average globules size in primary grain refined and un-refined specimens. Achieving a nearly fully globular microstructure after applying SIMA process on Ti-refined Al2014 alloy can be attributed to successfulness of Al-5Ti-1B master alloy in altering the primary structure from dendritic to fine and rosette-like one.

Fig. 11 demonstrates the SEM micrograph and EDX analysis of the alloy in the selected point of the eutectic region. According to previous investigation, the microstructure of such alloy contains similar Cu-intermetallic in the eutectic network [24]. According to theoretical points of view, there is a direct relationship between mechanical properties and microstructural features of alloys. Cáceres et al. have shown that in alloys with a soft matrix and hard particles like intermetallics, mechanical properties depend on hard particle characteristics such as equivalent particle diameter, aspect ratio and average distance [25].

Fig. 12 illustrates the microstructures of Ti-refined Al2014 alloy before and after applying SIMA process. It is important to note that all specimens were heat treated at 625 °C. From Fig. 12a, it is seen that in as-cast Al2014 alloy intermetallic phases such as Al2Cu have been located continuously in interdendritic regions, while the microstructure of specimens prepared by SIMA process clearly shows fragmented particles in eutectic network with a lamellar structure (Fig. 12b). As expected, cold working during SIMA process may break hard intermetallics to finer particles and heat treatment in semi-solid state results in preparing eutectic network to soaks intermetallics particles that are broken during pre-deformation stage. Interesting results of applying SIMA process are not only due to the modification of dendritic structure but also altering continuous coarse intermetallic to lather lamellar morphology in eutectic areas or grain boundaries. The main reason of this modification of intermetallics comes back to pre-deformation applying on specimens during SIMA process. Because intermetallics are seen as one of the main sources of crack initiators and fast intergranular crack propagation in the microstructure, fine distribution of these phases may result in altering the failure mechanism. It has been reported that initiation toughness in lamellar structures is improved by crack branching and microcracking ahead of the crack tip [26].

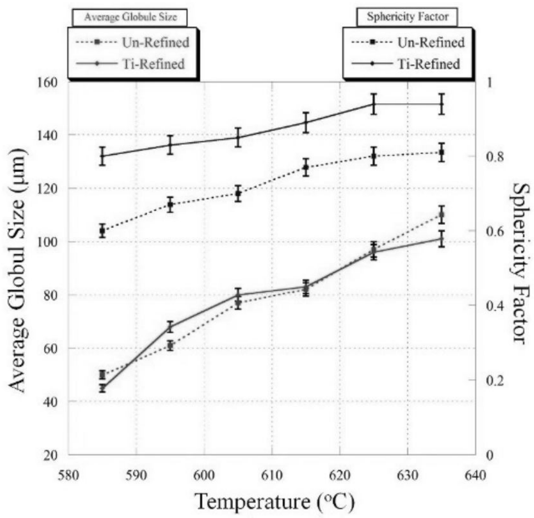

Fig. 10. Variations of average grain size and sphericity factor of the un-refined and Ti-refined Al2014 alloy containing 30% HR versus holding temperature for 30 min.

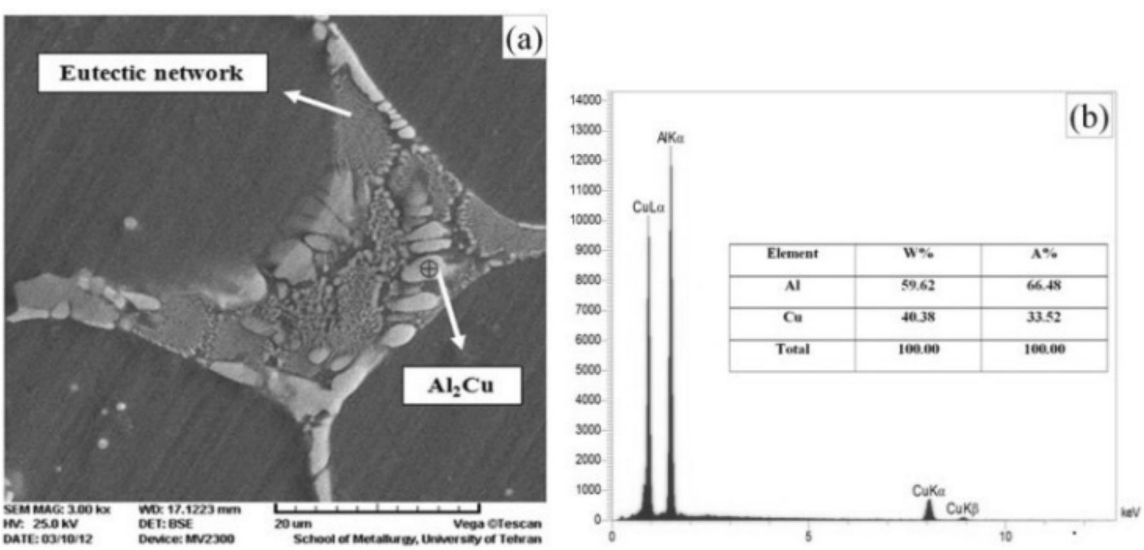

Fig. 11. (a) SEM micrograph and (b) EDX analysis of Al2Cu particles.

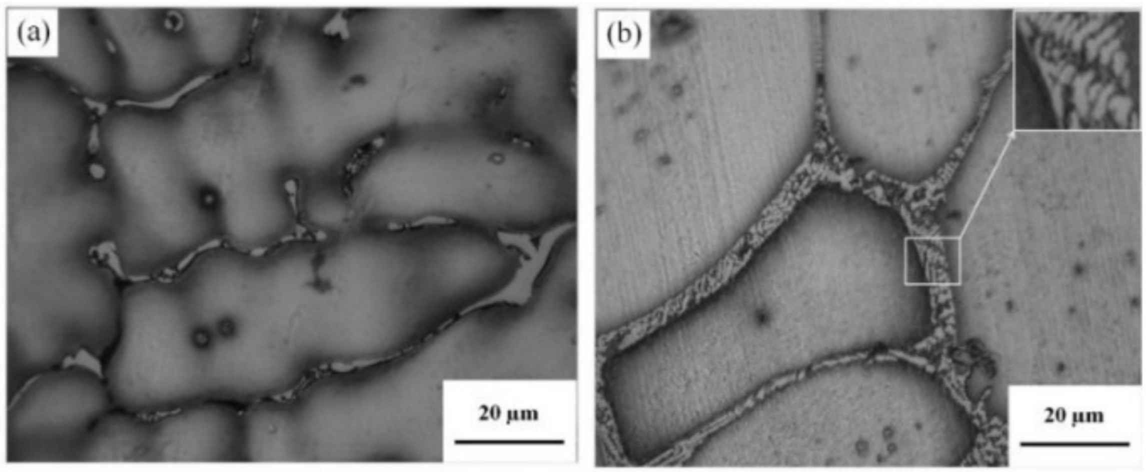

Fig. 12. The microstructures of Al2014-0.03% Ti (a) in as-cast condition and (b) prepared by SIMA process with 30% HR.

CONCLUSIONS

1. Addition of 0.03 wt.% Ti to Al2014 alloy reduced its average grain size from more than 1000 μm to about 120 μm in as-cast condition.
2. In comparison with as-cast condition, a finer and more globular microstructure obtained by applying SIMA process. The most important factors in globularization were found to be high reduction percentage and temperature. The results showed that the optimum condition in SIMA process of Al2014-0.03%Ti is 30% height reduction and heat treating in 625 °C for 30 min which reaches to a structure with average globule size of 96 μm and sphericity factor of 0.94.
3. SIMA process also showed a clear influence on the morphology and particle size of intermetallics in eutectic areas. It was found that in as-cast Al2014 alloy, intermetallics are formed continuously between the grains or dendritic arms whereas, in specimen prepared by SIMA process, a lamellar structure surrounds the grains.
4. Ti-refinement had a great influence on sphericity factor of final structure after applying SIMA process but did not change the globules size, significantly.

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