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Microstructure Evolution of Semi-Solid 7075 Aluminum Alloy During Reheating Process

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Microstructural evolution of semi-solid 7075 Al alloy manufactured by strain-induced melt activation (SIMA) process was investigated. The effects of different processing parameters, such as isothermal temperature and holding time on the semi-solid microstructures (the liquid volume fraction, average grain size, and degree of spheroidization of the solid particles) during partial remelting have been investigated on 7075 Al alloy that was extruded by an extrusion ratio of 20 before remelting. Experiments of remelting were carried out in the range of 560-610 °C for 10, 20, and 30 min holding time and then the specimens were quenched in cold water. Microstructure of quenched samples were observed under optical microscope and then analyzed via image analysis. The results showed that high semi-solid isothermal temperature would increase the liquid volume fraction and accelerate the spherical processing of the solid particles. Furthermore at long holding time, the globular grains coarsened slightly and the average grains size are increased. The experimental results showed that the optimum process parameters, should be chosen at isothermal temperature of 580 °C with the holding time, <30 min.

Keywords 7075 aluminum alloy, isothermal holding time, isothermal temperature, partial remelting, semi-solid microstructure

1. Introduction

Semi-solid forming (SSF) technology, developed at MIT during the 1970s by Spencer and co-workers (Ref 1), has received considerable interest in recent years. Study interest of SSF has been generated in recent years due to its combination of both casting and forging and significant advantages over the conventional process (Ref 1). In this technique, the alloy is heated to temperatures at which solid and liquid phase coexist in equilibrium and is then subjected to a forming process. The SSF technique offers some advantages, such as reduced flow stress during shearing, over the traditional metal processing methods, viz., casting, forging, and powder metallurgy (Ref 2). The key requirement of SSF is the thixotropic slurry with non-dendritic morphologies of the solid phase (Ref 3). The SSF is a very potential near-net shape technology suitable for producing all kinds of metals and alloys in the mushy state (Ref 4).

There are several methods for obtaining globular microstructure, such as mechanical or electromagnetic stirring, the addition of grain refining elements, spray casting, and rapid cooling, have been reported to obtain near equiaxed grain structures (Ref 1). Alternatively, the strain-induced melt activation (SIMA) process produces the desired structures by deformation and a following heat treatment in the mushy zone. In this route, the material is deformed by extrusion or other processes and then reheated to semi-solid state in which recrystallization occurs and liquid metal penetrates in the recrystallized grain boundaries thus resulting in solid globular particles surrounded by liquid (Ref 5, 6). Parameters such as heating time, temperature, and the degree of cold working, are critical factors in controlling the semi-solid microstructures in the SIMA process (Ref 3). The most common alloys used for the SSF technique have been 7xxx or 2xxx series wrought Al alloys or certain cast alloys (Ref 7). This study is focused on high strength 7075 wrought Al alloy. It is typically used for aerospace applications and is heat treatable to obtain a yield strength of 505 MPa and 11% elongation (Ref 3-5).

In partially liquid metals, the most important parameters are the volume fractions of the solid and liquid phases, because they crucially influence the viscosity. The phase fractions for a certain temperature between the solidus and liquidus temperatures under equilibrium conditions can be read from the appropriate phase diagram. If equilibrium conditions are assumed, the solid-phase fraction can be derived thermodynamically from the lever rule. For non-equilibrium conditions, the calculation is possible with different methods. A famous method is the Scheil-Gulliver model, for which it is assumed that no diffusion in the solid phase occurs. This assumption is only valid, however, for short holding times or high cooling rates (Ref 2). For the determination of the liquid volume fraction, various processes are used which define the solid-phase fraction directly or by means of its effect on special physical properties such as thermodynamic data, thermal processes [differential thermal analysis (DTA), differential scanning calorimeter (DSC)], and quantitative metallography by means of quenched samples from the semi-solid interval (Ref 2). In practice, these three methods are mostly used.

The two main mechanisms of grain coarsening play an important role during partial remelting. One of the coarsening mechanisms is the coalescence of grains. Grain growth by coalescence by grain boundary migration is dominant at short

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times after liquid is formed, at low volume fractions of liquid. Liquid fraction increases with increasing of the isothermal temperature and holding time. Because of the liquid phase soakage, it is difficult for the adjoining grains to coalescence continuously. Under these conditions, Ostwald ripening is the dominating mechanism of grain coarsening in the stage of high liquid fraction, in which grains continuously coarsen and the small grains gradually melt (Ref 8).

Some studies on the microstructure evolution of semi-solid slurry of AA5013 aluminum alloy (Ref 7), 2024 aluminum alloy (Ref 9, 10), and AZ91D magnesium alloy (Ref 6, 11) have been carried out. However, little was reported on the microstructure evolution of 7075 Al alloy semi-solid slurry prepared by SIMA. In the present work, the evolution of microstructure of 7075 Al alloy produced by the SIMA method and the mechanism of the globular particles formation during the isothermal treatment were investigated.

2. Experimental Procedures

In this study, 7075 wrought Al alloys were used. Their chemical composition is shown in Table 1. At the first step for removing dendritic microstructure of the as cast 7075 Al alloy, it had been homogenized in 470-480 °C temperature range for 12 h and then was cooled in air. The alloy was hot extruded at 420 °C from round initial ingot with 200 mm diameter to rectangular exit section bar with an extrusion ratio of 20. Cubic specimens of $10 \times 10 \times 10$ mm have been used in this experiment. The dependency of liquid fraction on temperature for the semi-solid 7075 Al alloy with an extrusion ratio of 20 were conducted on a LINSEIS L624HDSC integrated thermal analyzer with a constant heating rate of 10 °C/min. The liquid volume fraction vs. temperature curve is shown in Fig. 1.

From the Fig. 1, semi-solid temperature of 7075 Al alloys was found to be 540-650 °C. In order to study the effect of isothermal process parameters (time and temperature) on the semi-solid microstructures, the pre-deformed samples were heated from room temperature to various temperatures: 560, 570, 580, 590, 600, and 610 °C with an average heating rate 1.9 °C/s in electric resistance furnace. When the heating temperature, respectively, reached to predetermined values, samples was isothermally held for 10, 20, and 30 min and immediately taken out for water quenching to keep microstructure morphology of semi-solid slurry in room temperature.

The liquid fraction, average grain size, roundness, and shape factor (SF) of the solid phase measured using an image analyzing system (Clemex Software). SF of solid grains are calculated in each case by applying the Eq 1 (Ref 12).

$$SF = \frac{1}{\left(\sum_1^N P^2/4\pi A\right)/N} \qquad \text{(Eq 1)}$$

In this equation, SF, A, N, and P are shape factor, area, the number, and perimeter of solid particles, respectively. In accordance with Eq 1, SF of globular particles is close to one. In order to examine the evolution of microstructure in the semi-solid state (grain growth and spheroidization) and volume fraction of liquid as a function of the holding time and isothermal temperature, the alloys were subjected to isothermal exposure at different temperatures and times. Grain size was measured in SIMA alloy using the lineal intercept method.

Table 1 The chemical composition of 7075 wrought aluminum alloy used in this experiment (wt.%)

	Zn	Mg	Cu	Fe	Si	Cr	Mn	Ti	Al
Al 7075	5.60	2.40	1.40	0.42	0.40	0.26	0.13	0.01	Rem

Samples for microstructure characterization were prepared by the standard metallurgical technique, followed by etching in a Keller's reagent (mixed acid solution of HF, HCl, and HNO_3). The microstructure of the samples was observed under metallographic microscope. Three samples for any case and three regions in each sample are used to obtain these data for Fig. 9-17.

3. Experimental Results and Discussion

3.1 Microstructure

The initial as-received microstructure of the extruded billet is shown in Fig. 2. Microstructure is very similar to hot-worked metals with elongated grains and intermetallic particles aligned in the extrusion direction. The microstructure evolution during the SIMA process can be divided into four stages including: (1) deformation, (2) recovery and recrystallization, (3) partial remelting, and (4) spheroidizing and grains coarsening. The deformation ratio, produce a great influence on both the second and third stages. During the plastic deformation of the samples, internal strain energy is stored in the forms of dislocation multiplication, elasticity stress, and vacancies. The energy increases with the plastic deformation ratio, which promote the morphological transition from dentritic to globular structure, in other words globularization occurs faster and globularity of solid grains will be higher.

During heating the extruded 7075 billet to the semi-solid range, in order to decrease the free energy, the vacancies will combine, and dislocations will climb and cross slip, which provides the driving force for recovery and recrystallization. These processes produce nearly equiaxed polygonal grains with

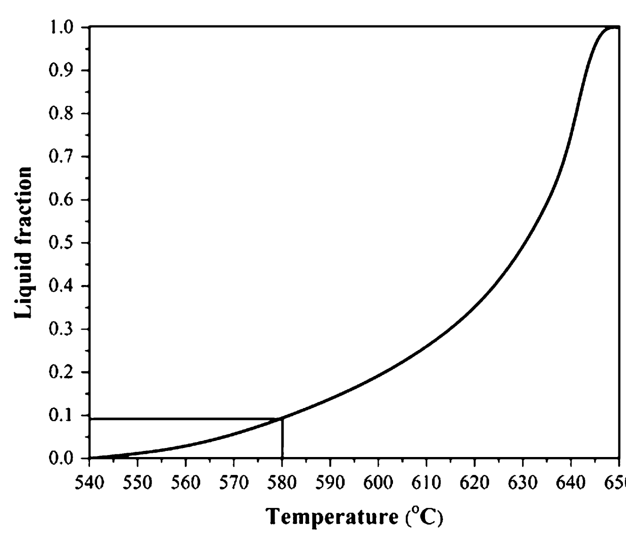

Fig. 1 Liquid fraction vs. temperature in solidification range obtained in constant heating rate of 10 °C/min

high-angle boundaries. At the same time because the holding temperature is over the eutectic line, partial melting starts and the liquid penetrates the recrystallized boundaries. When the material is reheated above the solidus, the first reaction to occur is the formation of liquid by the reaction of $MgZn_2$. The E-phase particles ($Al_{12}Mg_2Cr$ and $Al_{18}Mg_3Cr_2$), which pin grain boundaries migration, start to dissolve in the liquid and this initiates the recrystallization as the grain boundaries start to become unpinned. It appears that the formation of liquid is stimulating the recrystallization (Ref 13).

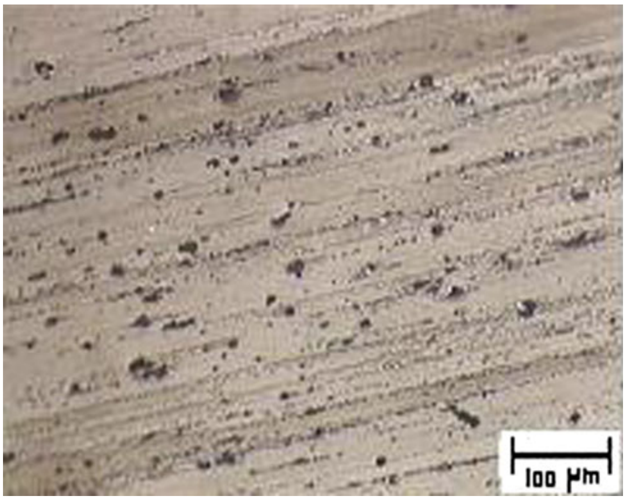

Fig. 2 As extruded microstructure of the wrought 7075 before reheating process

The eutectic phase at the grain boundary remelted first because of its higher solute concentration and formed small amount of liquid phase. The condition for grain boundary wetting is satisfied when: $2\gamma_{S/L} \le \gamma_{S/S}$, where $\gamma_{S/L}$ is the solid/liquid interfacial energy and $\gamma_{S/S}$ is the energy of the solid grain boundary. As soon as liquid is present at these boundaries, the grains begin to grow so resulting in spheroids surrounded by liquid. Then the periphery of solid grains remelted partially through the solute diffusion at the solid/liquid interface. As a result, the liquid fraction increase continuously (Ref 13, 14). The microscopic observation reveals that the recrystallization has completed and the partial melting of grain boundaries occurs for a very short holding time (Fig. 6, isothermal holding at 580 °C for shorter time than 30 min). In this process, the material melts locally at the grain boundaries due to the marginally enriched alloying elements and the associated lower solidus temperature. Contents of Cu at grain boundary increased, this means that the low melting structure at grain boundary was much influenced by Cu (Ref 5).

Microstructures of semi-solid 7075 Al alloy with an extrusion ratio of 20 during partial remelting at the isothermal temperatures of 560, 570, 580, 590, 600, and 610 °C, respectively, with the holding time of 10, 20, and 30 min are shown in Fig. 3-8.

The microstructures consist of α-Al solid particles, liquid phase, and the intragranular liquid droplets inside the solid particles. When the alloy has been heated just above the solidus, grain boundaries were gradually penetrated by liquid due to the dissolution of the last solidified phase of low melting

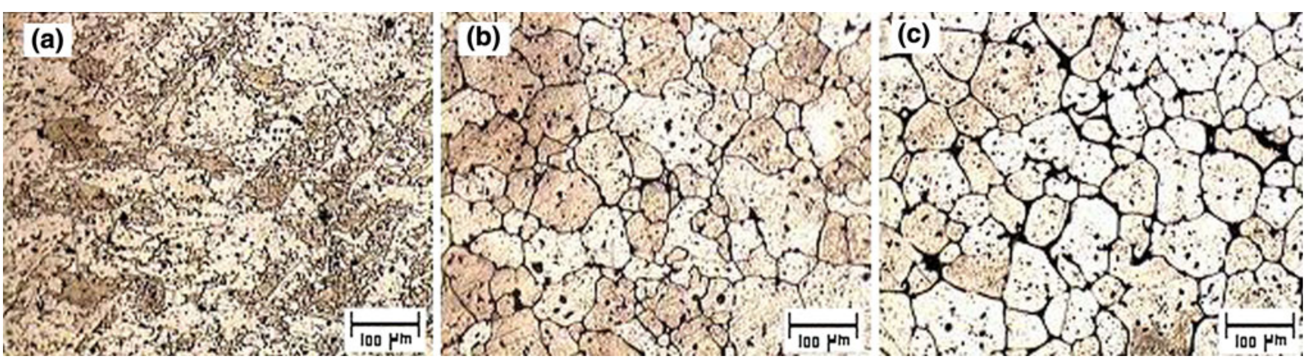

Fig. 3 Microstructures of 7075 alloy reheated at 560 °C for (a) 10, (b) 20, and (c) 30 min

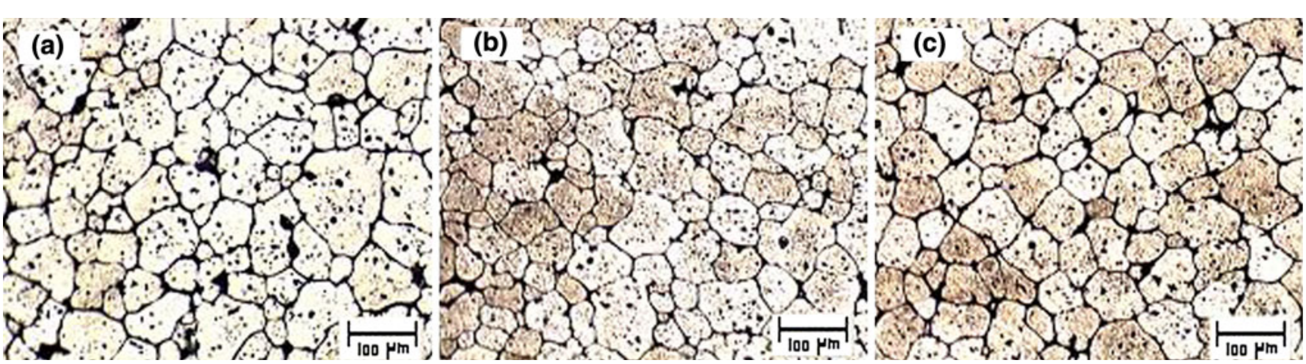

Fig. 4 Microstructures of 7075 alloy reheated at 570 °C for (a) 10, (b) 20, and (c) 30 min

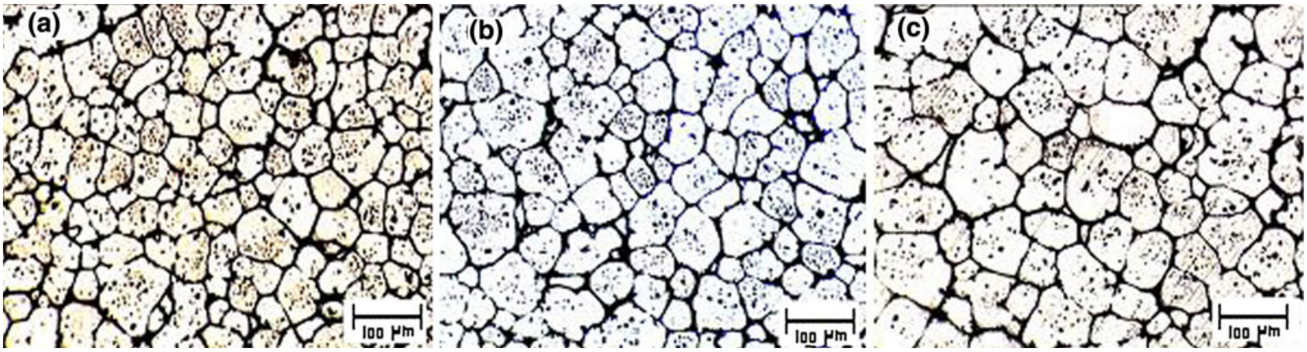

Fig. 5 Microstructures of 7075 alloy reheated at 580 °C for (a) 10, (b) 20, and (c) 30 min

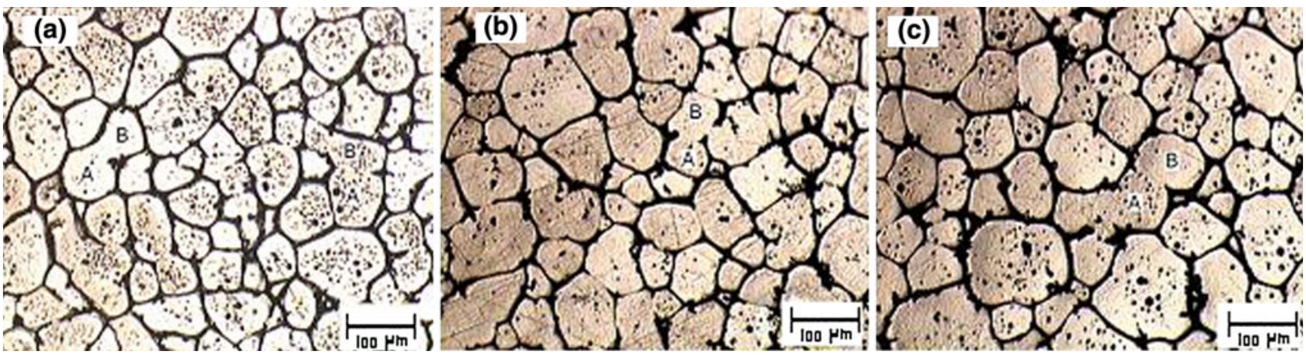

Fig. 6 Microstructures of 7075 alloy reheated at 590 °C for (a) 10, (b) 20, and (c) 30 min

temperature. As soon as liquid phase has formed at grain boundaries, grain spheroidization, and coarsening were activated simultaneously. From the Fig. 3-8, it could be observed that with increasing holding time and isothermal temperatures the liquid phases increase, the amount of the semi-solid particles reduce, the grains size grow larger and the shape of the grains become more globular.

The microstructures of the samples held at low temperatures 560 and 570 °C for 10, 20, and 30 min are shown in Fig. 3 and 4. It is observed that at these temperatures liquid contents will be low and liquid was mostly located along with the grain boundaries and at triple points between grains. Due to lower isothermal temperature, the liquid phase is not so much to soak into the grain boundaries. Hence, the grain boundaries are discontinuous. As shown in Fig. 3 and 4 the grain sizes will be less uniform, which will be make thixoforming more difficult. The microstructures of the samples held at 580 °C for 10, 20, and 30 min are shown in Fig. 5. It could be observed that coarse grains disappeared and globular grains were produced and grains are fine with average grain size < 70 μm and SF of about 0.7, indicating the successful preparation of semi-solid slurry. As may known the required conditions for thixoforming would be the grain sizes < 100 μm and with SF more than 0.6 (Ref 15). Overall, when the samples were reheated at 580 °C for < 30 min, the net-globular and primary grains are turned into fine, globular ones, which are desirable for thixoforming.

The microstructures of the samples held at 590 °C for 10, 20, and 30 min are shown in Fig. 6. Ostwald ripening and grain coalescence operate simultaneously and independently as soon as liquid is formed. It has been shown that the coalescence frequency is proportional to the number of adjacent grains. Figure 6 shows that obvious grain coarsening had occurred in the semi-solid. Disappearance of grain boundaries resulting in coalescence is seen in Fig. 6, where the grain boundaries between grains A and B have disappeared, leading to the formation of a larger grain with irregular shapes. The semi-solid slurry with coarse grains has an adverse effect on microstructure and mechanical properties of final thixoformed part.

The microstructures of the samples held at 600 and 610 °C for 10, 20, and 30 min are shown in Fig. 7 and 8. It is observed that the microstructure consisted of near-globular solid grains with a liquid film around them. Furthermore, the grain boundary liquid film was thicker with increasing isothermal holding temperature. The further observation indicated that the amount of solid grains had a tendency to reduce with elevating of temperature. The coalescence and coarsening occur between the adjoining grains at the high temperatures and globular structures formed at short holding time (< 20 min). When the isothermal temperature is 600 and 610 °C, grains are separated from each other because of liquid phase soakage between the grain boundaries as shown in Fig. 7 and 8. However, the grain coarsening is very evident and the size of some grains reached to 100 μm.

3.2 Effect of Holding Time

It could be observed that with the increase of isothermal time the liquid phases increase and the size of the grains grow larger. The mainly mechanism for structural coarsening is coalescence. When the holding time is long enough to make the solid volume fraction lower down, the Ostwald ripening

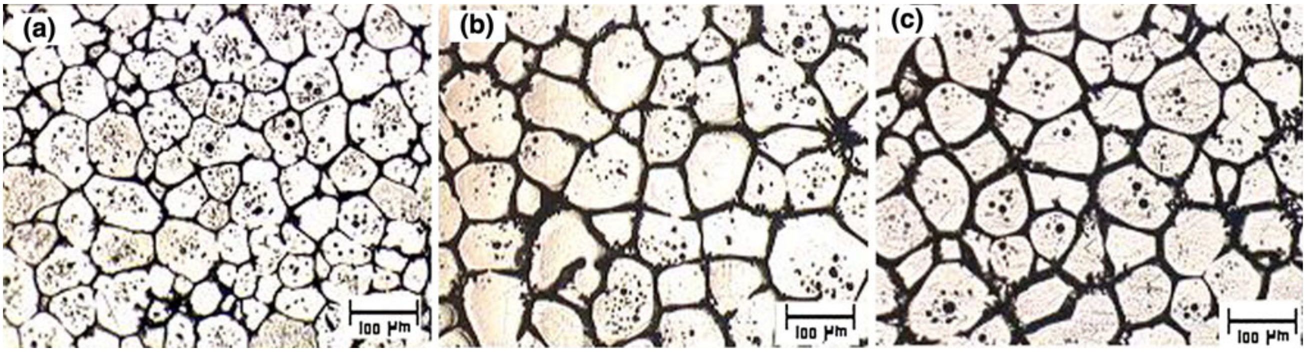

Fig. 7 Microstructures of 7075 alloy reheated at 600 °C for (a) 10, (b) 20, and (c) 30 min

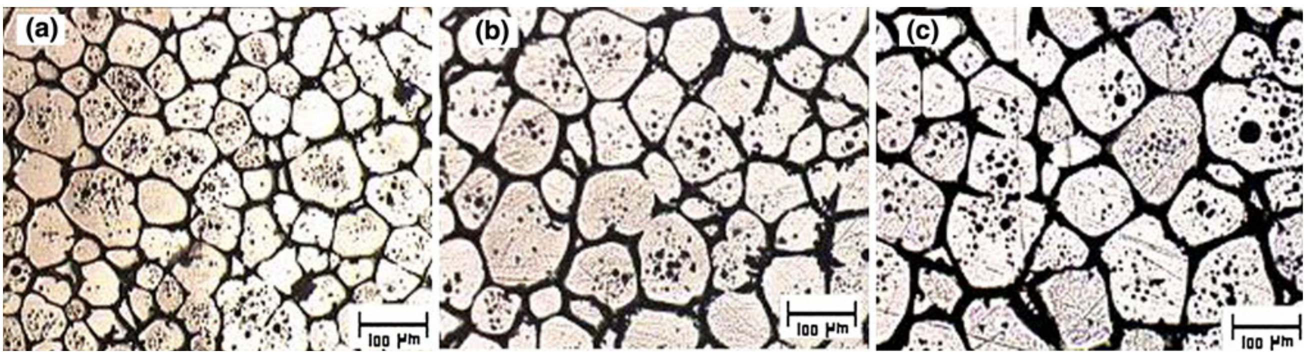

Fig. 8 Microstructures of 7075 alloy reheated at 610 °C for (a) 10, (b) 20, and (c) 30 min

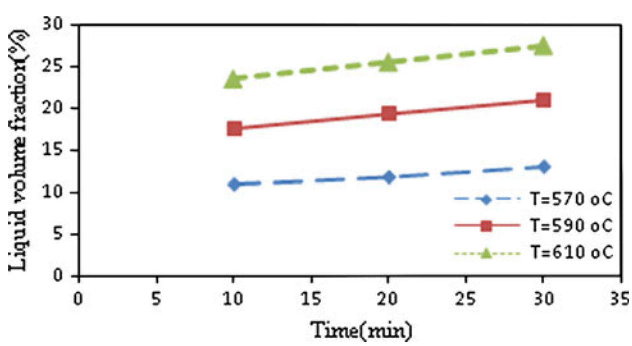

Fig. 9 The effects of holding time on the liquid volume fraction of 7075 alloy

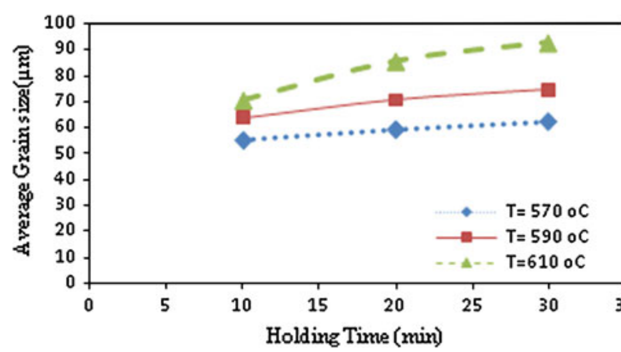

Fig. 10 Effects of holding time on average grain size of 7075 alloy

mechanism also begin to have an effect on structural coarsening.

The liquid volume fraction and variations of average globular grain size in the semi-solid microstructure as a function of different holding time at 570, 590, and 610 °C were illustrated in Fig. 9 and 10, which is done using the digital image analysis. As indicated in Fig. 9, liquid volume fraction increases with the increasing holding time. As shown in Fig. 9, when 7075 Al alloy was held for 10, 20, and 30 min at 590 °C, the liquid volume fraction of semi-solid slurry were 17.5, 19.3, and 21%, respectively.

Figure 10 shows the effects of holding time on average grain size of 7075 Al alloy semi-solid slurry prepared by SIMA. As indicated in Fig. 10, primary solid grains coarsened with the increasing holding time. When 7075 Al alloy was held for 10, 20, and 30 min at 590 °C, the average grain sizes of semi-solid slurry were 55.14, 65.54, and 70.75 μm, respectively. After reheating for 20 min at 590 °C, it can be seen from Fig. 8 that the shape of most grains is near to spherical. However, the grain coarsening is very evident and the size of some grains reached to 100 μm.

3.3 Effect of Isothermal Temperature

Figure 11 shows the effects of isothermal temperature on the liquid volume fraction. It was found that with the temperature varying from 560 to 610 °C for the partial remelting, the volume fraction of liquid phase gradually increased in the semi-solid microstructure with elevating of temperature. Figure 12 shows the effects of isothermal temperature on the average solid grains size. The average grains size increases with rising

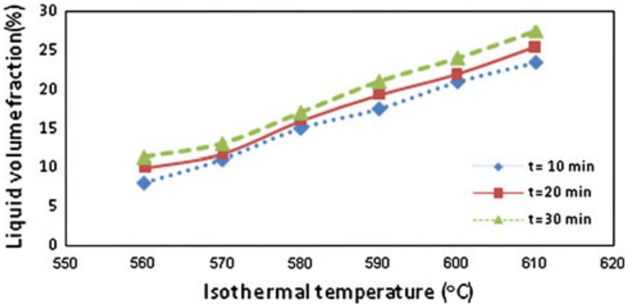

Fig. 11 The effects of temperature on the liquid volume fraction of 7075 alloy

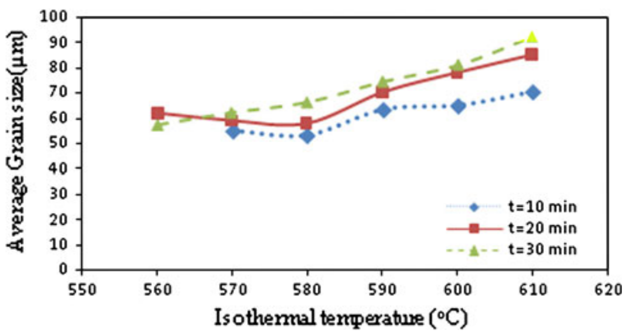

Fig. 12 Effects of isothermal temperature on average grain size of 7075 alloy

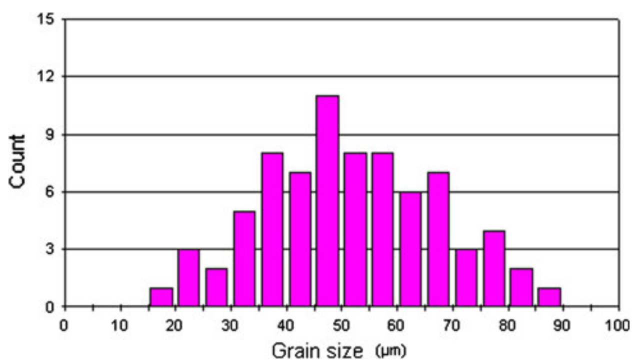

Fig. 13 Grain size distribution of 7075 alloy after 10 min holding at 580 °C

of temperature. With the increasing of isothermal holding temperature, ripening mechanism has an effect on the average solid particles size. Also because of the effects of interface curvature, the solid grains easily become globular.

For a given slurry system with a fixed solid fraction, particle distribution in the liquid matrix has an important influence on the slurry rheology and strong effect on the quality of the semi-solid processed components. Figure 13 shows that the distribution of grain size of 7075 Al alloy after 10 min holding at 580 °C. The grain size has a relatively Gaussian distribution for all samples. As a result, the expected uniform distribution of grain size can be observed with short holding time in high temperatures (580-610 °C). Figure 14 and 15 show the change of roundness and SF of the grains as a function of the temperature. Roundness of grains based on the definition in

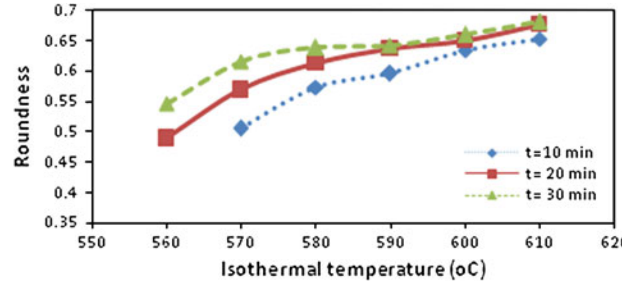

Fig. 14 Dependence of roundness of grains in the 7075 alloy on the isothermal temperature

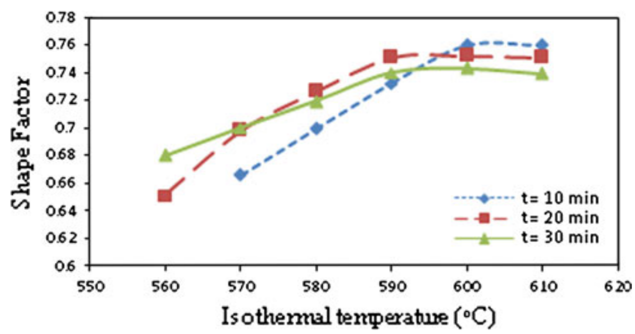

Fig. 15 Effects of isothermal temperature on shape factor of 7075 alloy slurry prepared by SIMA

CLEMEX Software are calculated in each case by applying the Eq 2.

$$\text{Roundness} = 4 \times \text{Area}/\pi \times \text{length}^2, \qquad \text{(Eq 2)}$$

where length is longest feret and feret means distance between two parallel tangents on each side of an object. Roundness give a value in the range (0 1), 1 for a perfect circle and close to 0 for a very narrow elongated grain.

It could be seen that with the increasing of temperature, the roundness of the solid particles increases and the shape of the solid particles becomes more globular. The SF, is an important parameter for thixoforming because it strongly influences the flowability and the viscosity of the material. In general, longer holding times and higher temperatures in the partial liquid state result in grains with more roundness. With increasing temperature and time, liquid soak aging leads to increasing roundness and globularity. In all time (10, 20, and 30 min), SF reach 0.75, indicating the best globularity of semi-solid slurry. For ideal round and globular grains, the SF takes the value 1. When isothermal temperature increased from 560 to 610 °C, SF of primary solid phase increased from 0.65 to 0.75, indicating the better globularity of semi-solid slurry.

Where samples are heating at low temperatures (560-570 °C), with increasing time, the SF of the solid particles is increased and their shapes become more globular. In high temperatures (580-610 °C) where holding time is longer, grain coarsening is occurred by Ostwald ripening and coalescence of grains mechanisms. These lead to the formation of a larger grain with irregular shapes and the globularity of primary solid grains reduced. Hence, the SF decreases with increasing time (it is more obvious for holding times more than 20 min at high temperatures).

3.4 Measurement of Liquid Volume Fraction and Grain Growth

The experimental determination of the volume fractions is mostly done with DSC (or DTA) and by means of quenching experiments from the partial liquid state. Figure 16 shows the curves of fraction liquid vs. temperature estimated by MTDATA/Scheil (Ref 16) compared with the curves estimated by DTA analysis at a constant heating rate of 10 °C/min and metallographic data for 7075 wrought Al alloy with an extrusion ratio of 20 that has been investigated in present work (for 20 min). However, it is clear that the chemical composition of 7075 Al alloy which is provided in this study reveals a little difference in weight percents of alloying elements, but this curve) MTDATA/Scheil (has been used as a comparative diagram.

In summary, it can be said that all three methods provide a rough prediction of the phase fraction. The use of thermodynamic data provides information on the maximum width of the semi-solid interval. However, consideration of the prior thermal history and its microstructure is currently not possible. For the metallographic determination of the average phase contents by means of quenching experiments, the random and uniform distribution of the liquid and solid phases in the sample volume has to be ensured. Furthermore, the phase concentrations should not change significantly during quenching.

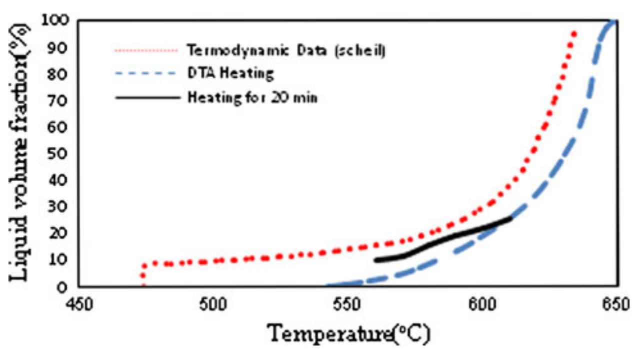

Fig. 16 Liquid fraction vs. temperature given by MTDATA non-equilibrium (Scheil) (Ref 16) and DTA (10 °C/min) analysis compared with metallographically determined liquid-phase contents for 7075 alloy

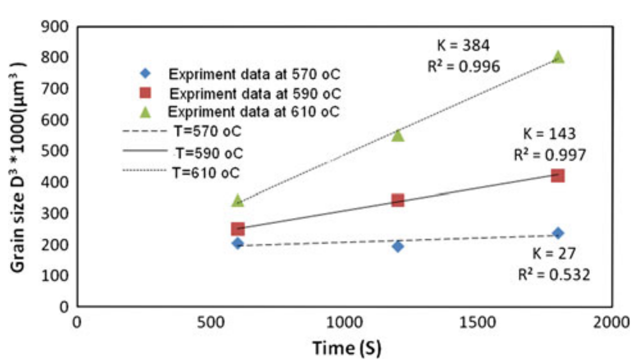

Fig. 17 Relationship between third power of particle diameter and holding time for the 7075 alloy. The solid lines represent the curves fitted to the classic LSW equation at $n = 3$

In order to describe the morphology of a semi-solid alloy, coarsening effects of the microstructure, such as Ostwald Ripening, coalescence as well as agglomeration and particle shape have been considered. In general, to study the kinetics of grain growth and the coarsening behavior is studied using the LSW (Lifshitz-Slyozov and Wanger) relationship (Ref 17, 18):

$$D_t^3 = Kt + D_0^3 \quad \text{(Eq 3)}$$

In this equation, D_t is the average grains diameter after soaking time t, D_0 is the initial average grains diameter, and K is the growth rate constant.

Figure 17 shows the experimental points and plot for the third power of grains diameter as a function of holding time at isothermal temperature of 570, 590, and 610 °C during partial remelting of 7075 Al alloy. The LSW equation in 590 °C is $D^3 = 166400 + 143t$.

The coarsening rate K can be obtained from the slope of the straight lines correlating the cubic D (diameter) and time (t). The coarsening rate K of 143 μm^3/s was reckoned up according to data in Fig. 17. Both experimental points and regression lines are plotted in this figure, it can be found that both experimental points and regression lines show good agreement and the coarsening kinetics follows the LSW theory.

4. Conclusions

When the temperature increases above the solidus point, semi-solid microstructure of 7075 Al alloy, consists of globular grains with grain boundary liquid films. During partial remelting, liquid volume fraction, the grains size and morphology are influenced by the isothermal temperature and holding time. High semi-solid isothermal temperature increases the liquid volume fraction and accelerates the spherical evolution of the solid grains. Furthermore at long holding time, the globular grains coarsened slightly and the average grains size are increased. For the 7075 Al alloy with an extrusion ratio of 20, the optimal process parameters during partial remelting should be chosen at isothermal temperature of 580 °C with the holding time <30 min. That is because this condition produces a fine and equiaxed microstructure which is desirable and suitable for subsequent semi-solid processing. Coalescence and coarsening of grains at low liquid phase fraction occur between the adjoining grains. Because of the liquid phase soakage, the Ostwald ripening appears to be the main mechanism of grain coarsening during partial remelting of 7075 Al alloy at high liquid phase fraction. The microstructure evolution during the SIMA process can be divided into four stages including: (1) deformation, (2) recovery and recrystallization, (3) partial remelting, and (4) spheroidizing and particle coarsening. In order to obtain globular grains, cold or hot deformation before isothermal heat treatment is necessary. In all times, SF increase to a near constant value of 0.75, indicating the successful preparation of semi-solid slurry with satisfied globularity of grains. The SIMA process is an effective method for producing 7075 Al alloy with the spherical grains required for SSF treatment. It was demonstrated that LSW theory could be used to describe the coarsening process of fine and spherical solid particles in 7075 Al alloy semi-solid slurry prepared by SIMA.

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