Journal of Alloys and Compounds 509 (2011) 402–408

Contents lists available at ScienceDirect

Journal of Alloys and Compounds

journal homepage: www.elsevier.com/locate/jallcom

Study on the effects of the compression ratio and mushy zone heating on the thixotropic microstructure of AA 7075 aluminum alloy via SIMA process

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ARTICLE INFO

Article history:
Received 7 June 2010
Received in revised form 6 September 2010
Accepted 8 September 2010
Available online 17 September 2010

Keywords:
Thermomechanical processing
7075 aluminum alloys
Semi-solid
SIMA

ABSTRACT

The effects of compression ratios on the microstructure evolution of semisolid Al7075 alloy produced by the strain induced melt activation (SIMA) process were investigated. The samples were cold deformed by compression into the different heights up to 40% reduction. The isothermal holding treatments within mushy zone of the alloy were carried out at 610, 620 and 625 °C for predetermined time intervals.

The results revealed that the average grain size gradually reduced with the increase in the compressive ratio. While the compressive ratio surpassed 30%, the descending trend above was not as evident as that of below 30% reduction. The optimum condition presenting the minimum grain size and the maximum shape factor with the highest uniformity is discussed. During the subsequent mushy zone heating, the recrystallization was induced in the deformed samples by the increasingly accumulated strain energy.

1. Introduction

Semisolid metal (SSM) processing has been recognized as a technique offering several potential advantages over casting or solid state forming such as reducing flow stress during shearing, producing components with a higher performance and reduced porosity compared with conventional casting. Several reviews are available [1–4]. The key feature that permits the shaping of alloys in the semisolid state is the absence of dendritic characteristics from the morphology of the solid phase and introduces the unique thixotropic behavior [5].

There are various types of semisolid metal processing [3,4].

'Rheocasting' refers to the process in which the alloy is cooled into the semisolid state and injected into a die without an intermediate solidification step; 'rheomoulding' is allied to polymer injection molding, and uses either a single screw or a twin screw. Thixo- route usually refers to the processes in which an intermediate solidification step does occur (there are exceptions to this e.g. 'thixomolding' which is mostly used to produce magnesium alloy components). 'Thixocasting' tends to take place with relatively high liquid fraction, in contrast to 'thixoforging', which take place with lower liquid fraction. An intermediate process called 'thixoforming' covers both thixocasting and thixoforging.

The way to obtain semisolid slurries is the key to semisolid metal processing. Except the mechanical and magneto-hydrodynamic stirring process [6–8], the strain induced melt activation (SIMA) process [9,10] is the most promising technology for the preparation of semisolid slurries, because of its simplicity and low equipment costs. Also more homogeneous and finer equiaxed microstructures can be obtained by the SIMA process [11]. The SIMA process consists of several discrete steps: (a) cold deformation or quenching of a hot extruded or rolled bar to induce sufficient strain, (b) reheating the prepared billet to the semisolid temperature range and holding isothermally for a short time. In this step, following partial remelting, an extremely fine, uniform and non-dendritic spherical microstructure is generated; and (c) the semisolid slurry with equiaxed grains is thixoformed. In the whole process, only one part of the alloy is melted. Therefore, the atmosphere protection adopted in the normal casting process can be omitted.

There is a strong drive to near net shape thixoforming process of 7075 aluminum alloys [12–16], which are often shaped by extensive costly machining of wrought material with much wastage. Thermo-mechanical treatment is the most important aspect in controlling the semisolid microstructures in the SIMA process. Also understanding of the development of the spheroidal microstructure in the semisolid state will enable optimization for the practical application. Nevertheless, no comprehensive and accurate researches has been performed on Al 7075 alloys by investigators to determine and suggest optimum condition for the production of such refined and globular microstructures as feed stock by means of SIMA. In the present investigation, the effects

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doi:10.1016/j.jallcom.2010.09.042

Table 1
The composition of starting material.

Zn	Mg	Cu	Cr	Mn	Fe	Balance
5.1–6.1	2.1–2.9	1.2–2	0.18–0.28	0.3	0.5	Al

of compression ratio, as well as holding time and temperature at the semisolid state on the microstructural characteristics of 7075 aluminum alloys specimens were investigated.

2. Experimental procedure

The commercially pure starting material, within AA 7075 aluminum alloy specification (Table 1), was used for the present investigation. The liquid fraction–temperature relationship was determined using differential scanning calorimetry (DSC). Samples of about 5 mm in diameter and 20 mg weight were cut, weighed and put into carbon pans with carbon lids in an argon atmosphere. The DSC tests were carried out using a Dupont 910 differential scanning calorimeter. The samples were heated to 680 °C at 10 K/min and cooled to room temperature at the same rate. The heat flow and temperature were monitored by thermocouples to obtain heating and cooling curves. The liquid fraction versus temperature curve was obtained by integrating under the curves.

Cylindrical samples, Ø = 31 mm in diameter and L = 34 mm in height, were machined from the starting material and were heated to 470 °C for 60 min to relieve the previous forming stresses and finally cooled in the air. Cylindrical samples were compressed into different reduction heights at room temperature, which are 10%, 15%, 20%, 30% and 40% in this study. After cold compression, all samples were machined again to Ø = 20 mm in diameter (and the same in height for all reductions) to have uniform strain in the core section. A hole of 1.6 mm was drilled into the centre of each sample in order to insert a thermocouple for monitoring the temperature of the samples. Then the samples were heated in a resistance heating furnace, for predetermined time and temperature (within the mushy zone). Heating cycles were interrupted at various stages. The samples were quenched and their microstructure was examined. Samples were prepared by standard metallography procedures down to 0.25 μm diamond paste. They were then etched using Keller solution.

Optical micrographs of the samples were taken using a UNION VERSAMET 3 equipped with OLYMPUS E300 digital camera, and the liquid fraction estimated for each heat treatment. This was performed using image analysis (quantitative measurements), by measuring the volume fraction of the primary α-Al phase particles, within the quenched liquid matrix. All quantitative measurements were carried out for a minimum of 500 grains at a sample by means of CLEMEX-PROFESSIONAL EDITION image analyzer software.

3. Results and discussion

A curve for liquid fraction versus temperature derived from the DSC result is shown in Fig. 1. Note that the liquid content apparent in the micrographs is not indicative of the quantity present at temperature because the quench is not sufficiently rapid for the liquid content to be entirely ‘frozen in’. During the quenching experiments, some liquid phase deposited onto the existing solid surfaces, appearing to be ‘solid’ in the quenched microstructures [17].

An optical micrograph of the as-received Al7075 aluminum alloy is shown in Fig. 2. The microstructure consists of unrecrystallized grain structure with various small dispersed particles, which are characteristics of such alloys [11]. Fig. 3 shows the microstructures of as-deformed alloys with different compression ratios (10%, 15%, 20%, 30% and 40%) after isothermal holding at 620 and 625 °C for 300 s. In general, as shown in Fig. 3, grains appear more refined and uniform as the compression ratio increases. During compression of the alloys, dislocation density increases due to internal crystal lattice distortion. The increase in compression ratio leads to increase in lattice distortion and internal strain energy, which is stored in the forms of vacancies, lattice defect and dislocation multiplication [18].

The present results provide evidence that supports the deformation-recrystallization mechanism as the main governing mechanism responsible for the formation of the spheroidal microstructure via the SIMA process. Distortion energy will provide the driving force for recovery and recrystallization during heating process. During recovery, sufficient thermal energy is supplied to allow the dislocations to rearrange themselves into lower energy configurations which occur near melting point. After that, the residual distortion energy stored in the alloys provides the driving force for recrystallization. This process consists of the replacement of grains containing high concentrations of dislocations with new sub-grains containing much lower dislocation densities. It has been shown that when the misorientation of such sub-grains is larger than a critical angle i.e. 20 in aluminum alloys the solid-liquid interface energy γ_{SL} becomes less than half grain boundary surface energy γ_{gb}. When this condition is satisfied ($\gamma_{gb} > 2\gamma_{SL}$) liquid penetrates the grain boundary minimizing the local surface energy. Solid particles, whose grain boundaries are not wetted, become interconnected by coalescence and form agglomerates [19].

As shown in Fig. 3(a and f), when the compression ratio is 10%, the microstructure consists of unrefined solid particles which can mean that the microstructure breaking up mechanisms are not active or effectively active [18]. Microstructural comparison and calculated results of samples with 10%, 15% and 20% compression ratios for both 620 and 625 °C temperatures may identify the critical compression ratio which the microstructure has the potential to change to the relatively homogenous and refined grains. The microstructure of the sample with 15% compression ratio (Fig. 3(b and g)) mainly consists of unequiaxed and unrefined grains, in other words it seems that the sample with 15% compression ratio

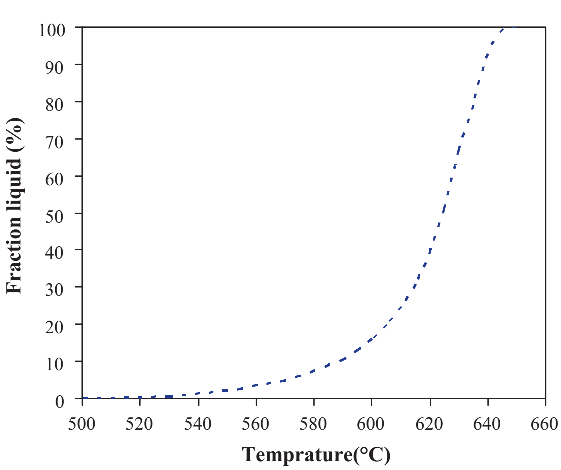

Fig. 1. Fraction liquid vs. temperature estimated from DSC heating curves.

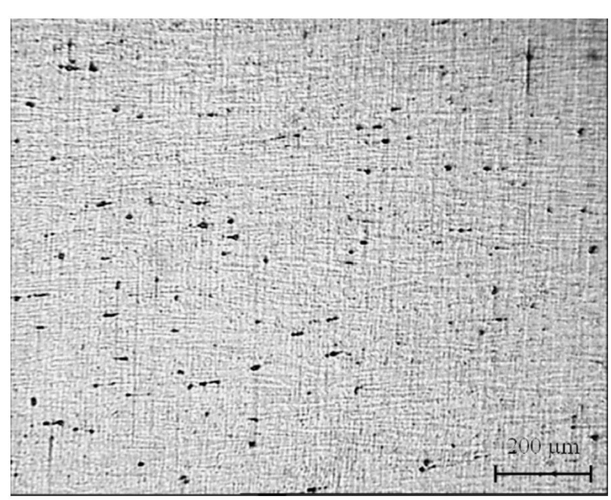

Fig. 2. An optical micrograph for the as-received Al7075 aluminum alloy.

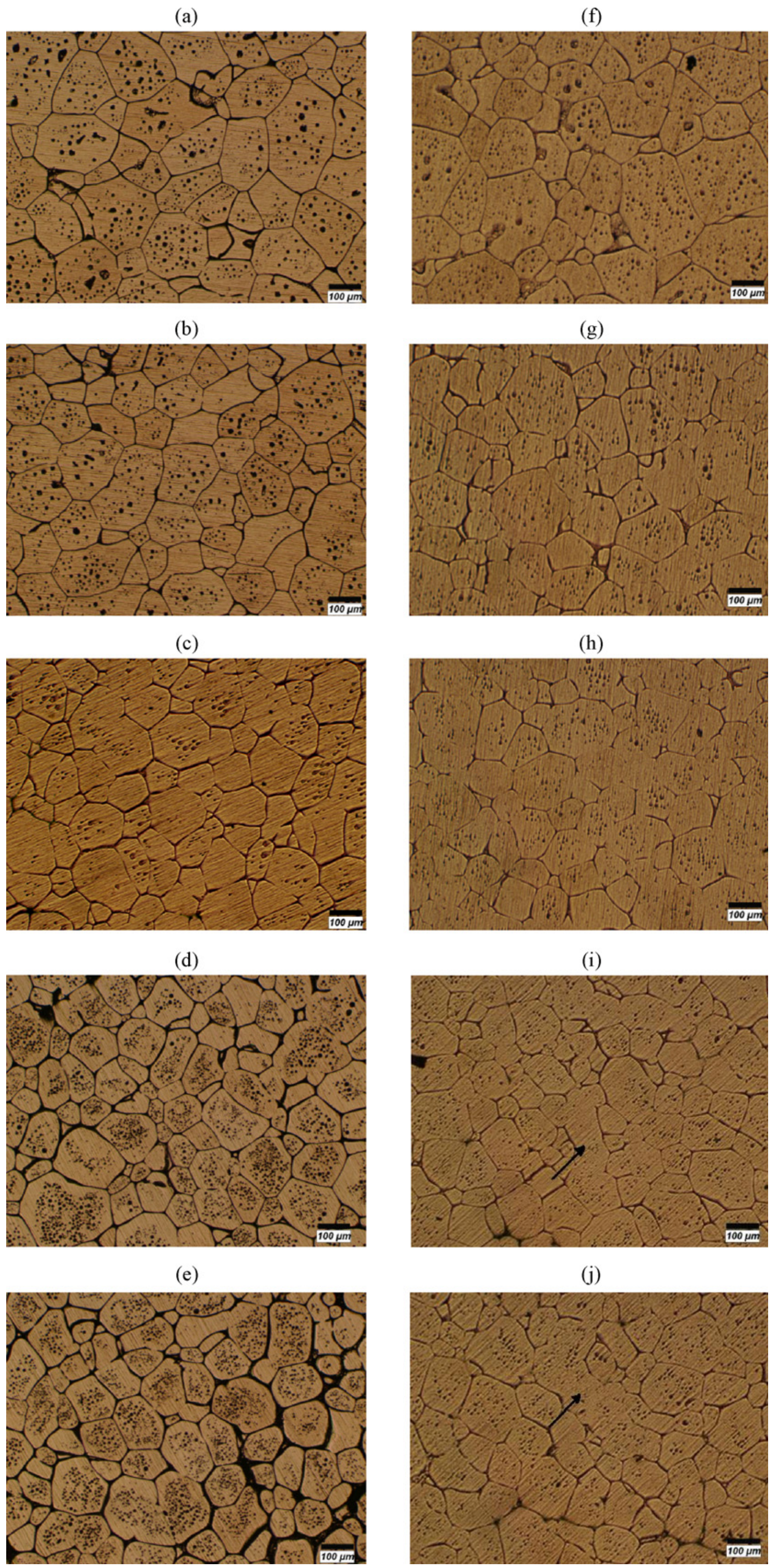

Fig. 3. The microstructures of transverse section of as-deformed alloy at room temperature for different compression ratios: (a) 10%, (b) 15%, (c) 20% (d) 30% and (e) 40% after isothermal holding at 625 °C for 300 min and (f) 10%, (g) 15%, (h) 20% (i) 30% and (j) 40% after isothermal holding at 620 °C for 300 s.

has no potential to change to a suitable globular microstructure for thixoforming process at the mentioned temperatures. In the sample with 20% compression ratio (Fig. 3(c and h)) grains are refined with highly uniform structure. The present results reveal that the critical compression ratio is between 15% and 20%. It means that for SIMA and RAP processing of Al 7075 alloys; at least 20% ± 2 strain is needed for preparing feedstock for thixoforming processes. Lee et al. [20] stated that in the case where the cold working is below

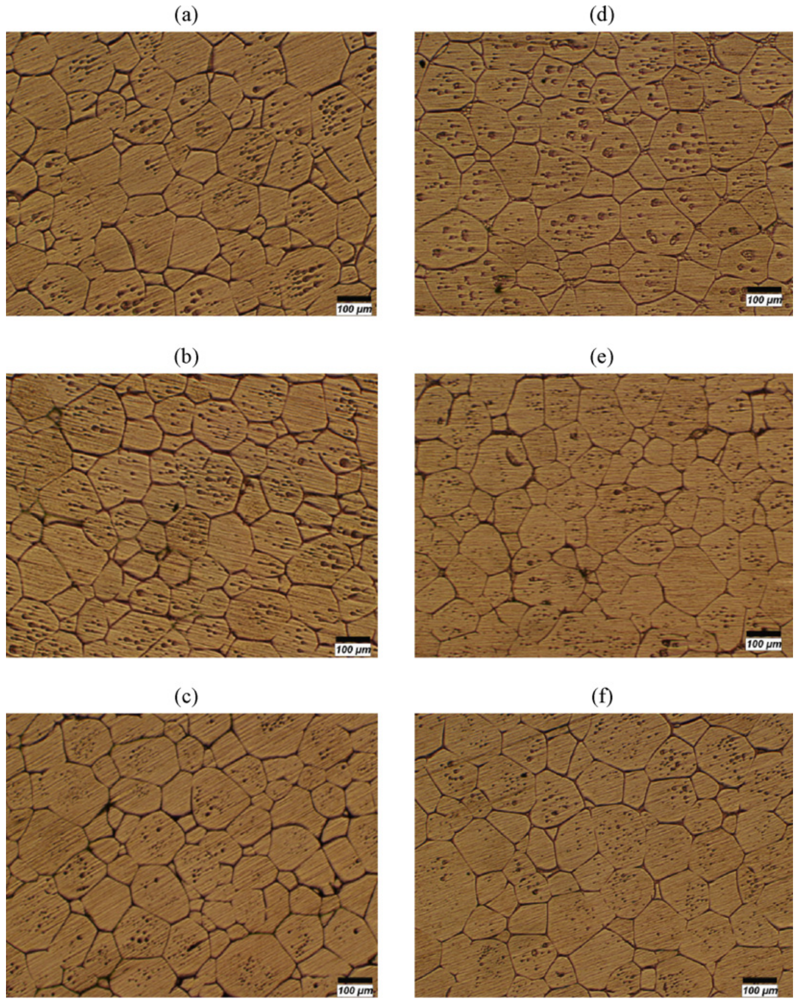

Fig. 4. The microstructures of transverse section of as-deformed alloy at room temperature for different compression ratios: (a) 20%, (b) 30% and (c) 40% after isothermal holding at 625 °C for 600 s and (d) 20%, (e) 30% and (f) 40% (i) after isothermal holding at 620 °C for 600 s.

27%, dendritic structures resulted and grain size became less uniform. Our results also confirm the mentioned critical compression ratio for Al 7075 alloy.

As shown in Fig. 3(d and e), through the inducement of enough strain, the microstructure of samples after isothermal holding consists of homogeneous, globular solid particles surrounded by liquid film. The shape factor improved to 0.73 and 0.79 for isothermal holding at 620 and 625 °C, respectively, which is suitable for semisolid processing [2]. The results also revealed that, for 30% and 40% compression ratios at 620 °C (Fig. 3(i and j)) unusual elongated grains were observed which will be dealt with later on.

Fig. 4 shows the microstructures of as-deformed alloys with different compression ratios (20%, 30% and 40%) after isothermal holding at 620 and 625 °C for 600 s. A comparison of Fig. 3 with Fig. 4 shows that increasing holding time at a constant temperature leads to the elimination of elongated grains, and microstructure becomes more uniform and homogenized which is a critical problem in the semisolid processes for the production of feedstock ingots [21,22]. In addition, there was no significant change in the average grain size by increasing holding time (Fig. 5). A qualitative study and microstructural observations indicate that samples with 30% compression ratio may exhibit a more uniform microstructure in comparison with 20% and 40% compression ratios. And also the shape factor at 625 °C increased to 0.82, 0.79 and 0.76 for 40%, 30% and 20% compression ratios, respectively and at 620 °C improved to 0.76, 0.77 and 0.71 respectively for 40%, 30% and 20% compression ratios (Fig. 7).

Both isothermal holding temperatures of 620 and 625 °C are considered as temperatures with high liquid fraction. Feedstock with lower liquid fraction has also special interests due to unique advantages like being less sensitive to temperature, avoids metal splash at high ram speed, allows laminar flow at high ram speed and gives smooth surface without stick-lip phenomenon [23]. The 610 °C holding temperature, in comparison with 620 and 625 °C will results in low liquid fraction (see Fig. 1). Fig. 5 shows the microstructural evolution of as-deformed alloys with different compression ratios (10%, 15%, 20%, 30% and 40%) after isothermal holding at 610 °C for 600 s.

The microstructures in Fig. 5 have the potential to alter to a refined, equiaxed and uniform microstructure, after meeting the critical value. Also calculated results (Figs. 6 and 7) indicate clearly that the optimum condition can be reached in 30% and 40% compression ratios at 610 °C: the average grain size was less than 100 μm(Fig. 6) and shape factor was almost above 0.7(Fig. 7).

A drawback of high solid fraction in the semisolid forming process is the high viscosity of material: as the geometry gets more complex; higher speed flow is needed to decrease the viscosity of the thixotropic material. The lack of liquid with such high solid fraction promotes macro-porosity and nonsheared material. This results in inhomogenetity of the part and freezing flow. This freezing flow, promoted by the cold tool and the decreasing speed of

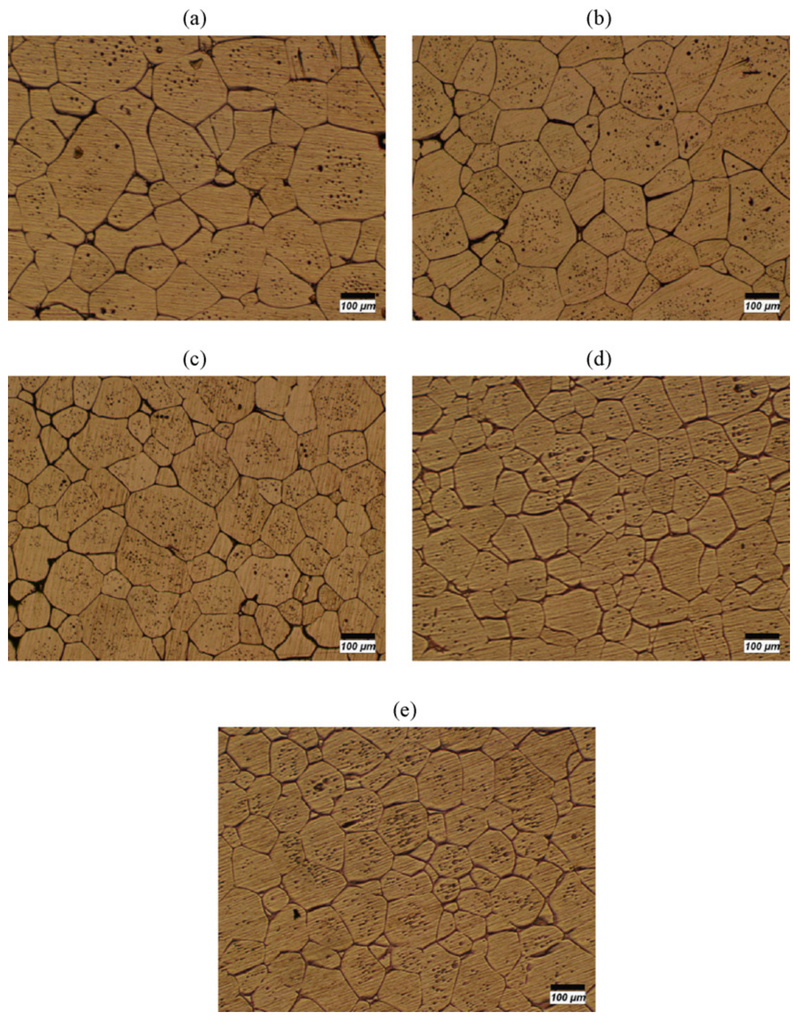

Fig. 5. The microstructures of transverse section of as-deformed alloy at room temperature for different compression ratios: (a) 10%, (b) 15%, (c) 20%, (d) 30% and (e) 40% after isothermal holding at 610 °C for 600 s.

arm, promotes the solid/liquid segregation resulting in defects like hot cracking [23].

The general tendency, where a reduction in the average globular grain size with an increase in compressive ratio of the alloys, for 625, 620 and 610 °C is illustrated in Fig. 6. Results show a precipitous decreasing character in the average grain size for the Al7075 alloy with the compressive ratio below 20%, which is depicted on the left region of the curve in Fig. 6. An increase in the

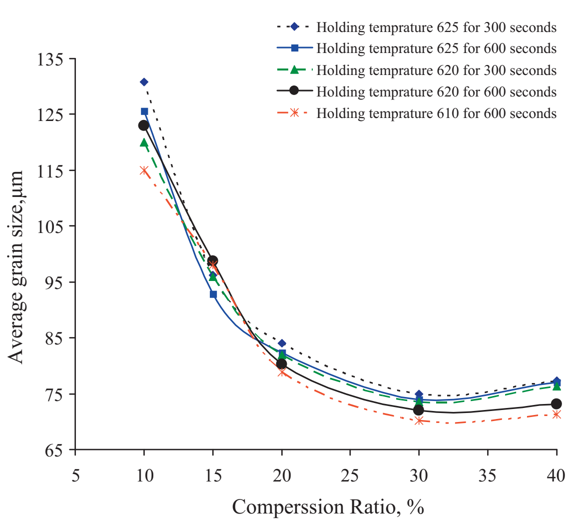

Fig. 6. Variation in the average grain size by applying different compression ratios and soaking times in 625, 620, and 610 °C holding temperature.

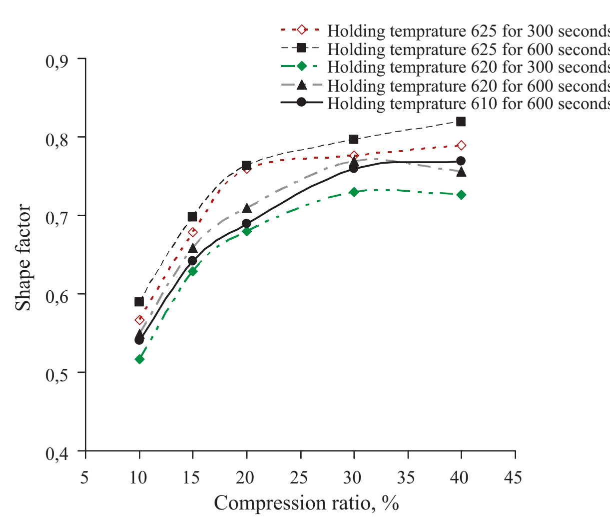

Fig. 7. Variation in the shape factor of by applying different compression ratios and soaking times in 625, 620, and 610 °C holding temperature.

compressive ratio from 20% to 30% leads to a gentle reduction of the average grain size in the semisolid Al7075 alloys, as illustrated by the middle region in Fig. 6. For the right region of the curves in Fig. 6, although the compressive ratios of Al7075 alloy increased from 30% to 40%, no significant reduction was observed in the average grain size of grains in the semisolid Al7075 alloys. The same tendency was reported by Lee et al. [20] for Al 7075 alloys and they stated that for cold working above constant region grains became larger and non-uniform.

The main reason for precipitous decrease in the average grain size might be that the deformation causes the strain energy to rapidly accumulate and is abundantly stored in the compressed alloy to reach a critical value. When a critical value is reached (compression ratios between 20% and 30%), during forming process, the accumulating rate of the strain energy descends by degrees in the cold work which possibly weakens the effect of recrystallization in the evolution of globular grains in the semisolid microstructure [18].

According to the present results no significant reduction was observed on the average grain size of the alloy in the semisolid state by the increase in the compression ratio from 30% to 40%. There may be two different explanations for such phenomena:

By increasing the compression ratio, the breaking up mechanisms become active and the grain size rapidly decrease as the increasing of compression ratio. The critical size of steady recrystallization nucleus reduces with increase of compression ratio and the number of nucleation rises at the same time [24]. We may expect that during holding time in sample with 40% compression ratio very fine equiaxed grain are formed and then grain growth and agglomeration lead to grain coarsening and cause the same average grain size in comparison with sample with 30% compression ratio.

Considering the situation between 20% and 30% ratios, it seems that the effect of compression (deformation) in the evaluation of the microstructure is weakened, strain energy stored in alloy by deformation was the driving force of grain nucleation and growth during recrystallization reaches a peak value and may not exceed in the microstructure easily more than the peak value, so the increase in the strain energy in Al7075 alloys is relatively limited.

To clarify the more probable of the two explanations we have to further examine Fig. 3(i and j) which shows the microstructures of as-deformed alloys with 30% and 40% compression ratios after isothermal holding at 620 °C for 300 s. Elongated grains in samples are observed and are shown by an arrow in the figures. It was suggested before that the main mechanism in the breaking up of the microstructure is recrystallization mechanism, these results show that during soaking time, recrystallization has not occurred completely and the liquid has not fully penetrated into sub-grains. So it seems that the explanation (A) is not valid according to the our observations at 620 °C for both 30% and 40% compression ratios which may have the same effects on the microstructure: there are same elongated grains and almost with the same average grain size. It seems probable that the explanation (B) is correct: by increasing compression ratio vacancies, lattice defect and dislocation (which form strain energy) may be neutralized; for example two dislocations with opposite burger vector neutralize each other. Also there is clear and deep discussion by Lin et al. [18] for the effect of deformation on the reduction of globular grain size for AZ91D alloys which is in accordance with our discussion.

Fig. 7 displays morphological variations of solid grains versus compression ratio at different mushy zone heating conditions. The general tendency is a sharp improving of shape factor by increasing compression ratio by 20% and then follows by a gentle increase. Also prolonged holding times and high temperature within mushy zone have significant effects on the improvement of shape factor. Samples with different solid grain size and shape factor have different semisolid behavior during semisolid forming processes. Shape factor of solid particles has a larger effect than grain size in semisolid behavior of material [22].

Based on the experimental results obtained by microstructure observations shown in Figs. 3–5 and the calculated results obtained by image analyzer software shown in Figs. 6 and 7, it is obvious that there is an optimum condition for holding time and compression ratio in which the best refined and globular microstructures are obtainable. Optimum condition can be determined through statistical points of view [25]. But it is not the whole matter; also the economic points of view should be taken into consideration in accordance with the quality of structure which is the driving force for development of different processes. For example, it could be concluded that as far as shape factor and globularity is concerned, higher induced strain plus shorter heating time can be equivalent to lower induced strain plus longer heating time. The equivalent quantitative value of these variables can be determined by modeling and more experiments. In general we may propose that, 30% compression ratio would be an ideal ratio because it shows the optimum average grain size and more uniform microstructure in all cases and an increase in compression ratio will not yield significant change in the average grain size, although the shape factor and globularity can be improved by increasing compression ratio to 40%. Also it is completely clear that at least 600 s is needed for the material to get the uniform and globular microstructure at a constant temperature.

4. Conclusions

Quenching experiments have revealed the microstructural evolution and the morphology of Al7075 alloy in the semisolid state with a globular microstructure. During the heating process, with increasing the compression ratio, the average solid grain size decreased and the degree of spheroidization tended to improve. The descending trend in the average grain size of Al 7075 alloys in the semisolid state as a function of compression ratio experienced three different stages, which respectively exhibited precipitous, gentle and constant characters. The deformation refined the grains in the semisolid microstructure due to the recrystallization mechanism; the chosen 30% was proved as an appropriate compressive range for the as-received alloys to obtain the ideal semisolid microstructure in the semisolid process. The processing parameters have great influences on the microstructure of semisolid Al7075 alloy in the SIMA process. To obtain the fine, uniform, and spheroidal microstructure the processing parameters must be selected properly.

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