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SOĞUK DEFORMASYON ORANI VE YENİDEN ISITMA SICAKLIKLARININ SIMA İŞLEMİ İLE ÜRETİLEN AA7075 ALAŞIMININ MİKROYAPISI ÜZERİNE ETKİSİ

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Özet

Gerinimin neden olduğu ergiyik/sıvı aktivasyonu (SIMA) işlemi ile üretilen AA7075 alaşımının mikroyapısı üzerine soğuk deformasyon oranı ve yeniden ısıtma sıcaklıklarının etkisi araştırılmıştır. Bu amaçla, farklı oranlarda (%10, %20 ve %30) soğuk deforme edilen kütükler 600 °C, 606 °C ve 611 °C sıcaklıktan hızla suda soğutularak mikroyapı değişimleri incelendi. Artan soğuk deformasyon oranı ile tane büyüklüğünün azaldığı ve şekil faktörünün arttığı, artan yeniden ısıtma sıcaklıklarında da tane büyüklüğü ve şekil faktörünün arttığı gözlenmiştir. Homojen küresel yapıya sahip bir tiksotropik yapı oluşturmak için minimum %20 soğuk deformasyonun gerekli olduğu belirlenmiştir. Artan yeniden ısıtma sıcaklığıyla şekil faktörünün değişiminin anlamlı olmadığı belirlenmiştir.

Anahtar Kelimeler: AA7075 alaşımı, SIMA işlemi, Tiksotropik yapı, Yeniden ısıtma sıcaklığı, Soğuk deformasyon oranı, Yarı katı işlem.

EFFECT OF COLD DEFORMATION RATIO AND REHEATING TEMPERATURES ON MICROSTRUCTURE OF AA7075 ALLOY PRODUCED BY SIMA PROCESS

Abstract

The effect of cold deformation ratio and reheating temperatures was investigated on the microstructure of AA7075 alloy produced by the strain-induced melt activation (SIMA) process. For this aim, cold deformed billets with different ratios (10%, 20%, and 30%) were quenched in water from 600 °C, 606 °C, and 611 °C, and microstructural evolution was studied. It was observed that grain size reduced, shape factor increased with the increasing cold deformation ratio, and grain size and shape factor increased with the rising reheating temperatures. It was determined that a minimum of 20% cold deformation is required to produce a thixotropic structure with a homogeneous globular microstructure. It was also observed that the change in shape factor was insignificant with increasing temperature.

Keywords: AA7075 alloy, SIMA process, Thixotropic structure, Reheating temperature, Cold deformation ratio, Semi-solid Processing.

1. INTRODUCTION

Semi-solid processing (SSP), which is a near-net shape metal forming process, is performed by thixotropic properties of metal alloys (mainly Al and Mg alloys)(Helen V Atkinson, 2005; Fan, 2002; Flemings, 1991; Kirkwood, 1994). The SSP methods required that the alloys with nondendritic, fine, equiaxial, and globular grain structures for forming in the mushy zone (Helen V Atkinson, 2005; de Figueredo, 2001; Fan, 2002; Flemings, 1991; Flemings & Martinez, 2006; Kenney et al., 1988; Kirkwood, 1994; Martinez & Flemings, 2005; Nafisi & Ghomashchi, 2005; Xia & Tausig, 1998).

There are many methods, such as mechanical or electromagnetic stirring, the addition of grain refiners, and the cooling slope used to obtain globular structures (Helen V Atkinson, 2005; Fan, 2002; Flemings, 1991; Kirkwood, 1994). Among these production methods, SIMA is an ideal candidate with significant commercial advantages of simplicity and low equipment costs to produce fine and globular structures (Dışpınar & Türkeli, 2006; Saklakoğlu, Çolakoğlu, & Gençalp, 2009; Türkeli & Akbaş, 1996).

In this route, the alloy is deformed by extrusion or other processes and then reheated to a semi-solid state where recrystallization occurs. Liquid metal penetrates the recrystallized grain boundaries, thus resulting in solid globular particles surrounded by liquid (de Figueredo, 2001; Fan, 2002; Flemings & Martinez, 2006; Martinez & Flemings, 2005). 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 (Dışpınar & Türkeli, 2006; Saklakoğlu et al., 2009; Türkeli, 1993; Türkeli & Akbaş, 1996; Young, Curtis, & James, 1983).

SIMA method, the most common feedstock preparation method in thixoforming of especially wrought Al alloys, includes cold deformation and reheating stages under recrystallization temperature following melting and casting process after hot working processes such as extrusion, rolling, etc. (Young et al., 1983). Dislocation density increases depending on the cold deformation rate applied to material produced by extrusion or cold forming method. With the increase of dislocation density, potential nucleation sites increase for recrystallized grains, and as a result, grain size reduces (Dışpınar & Türkeli, 2006).

The SIMA method is more economical than other feedstock methods (such as magnetic stirring) to produce globular microstructure, enabling better thixotropic properties. SIMA method has been used commercially in feedstock production for semi-solid processing (Kirkwood, 1994; Nafisi & Ghomashchi, 2005; Türkeli, 1993; Türkeli & Akbaş, 1996).

In the SIMA method, obtaining globular grains depends on recrystallization temperature. In addition to this, the cold deformation ratio influenced recrystallization temperature. As the cold deformation ratio increase, globular grains can be obtained at lower temperatures (Akar, 2011; Akar & Mutlu, 2010; Erzi, Gursoy, Yüksel, Kirtay, & Dispinar, 2018; Guner, Dispinar, & Tan, 2019; Saklakoglu, Saklakoglu, Tanoglu, Oztas, & Cubukcuoglu, 2004; Saklakoğlu et al., 2009; Taneroglu, Akar, & Kilicli, 2013).

The present study investigates the influence of cold deformation ratio and reheating temperatures on the microstructural evolution of AA7075 alloy, which was produced by the SIMA process.

2. EXPERIMENTAL STUDIES

2.1. Material

The AA7075 alloy used in experimental studies was obtained in 40 mm diameter and 3000 mm length, and its chemical composition is given in Table 1. ARL 3460 model optical emission spectrometry was used for the determination of the chemical composition of the alloy.

Table 1. Chemical composition of AA7075 alloy used in experimental studies (weight-%)

Zn	Mg	Cu	Mn	Si	Cr	Ti	Fe
5.599	2.033	1.329	0.183	0.383	0.157	0.007	0.741
Ni	Pb	Sn	Zr	V	Ga	Se	Al
0.008	0.043	0.012	0.001	0.006	0.014	0.001	Remain

2.2. SIMA method

SIMA method includes the process of cold deformation of the cast and hot-extruded alloys and reheating to semi-solid temperature and rapid cooling (Figure 1). In this study, for feedstock production with the SIMA method, cold deformation was applied at a different ratio on AA7075 alloy samples in 40 mm diameter and hot extruded. Then, they were cooled rapidly in water at different reheating temperatures. The deformation ratio was calculated with Equation 1.

$$\%\,\varepsilon = \frac{l_o - l_s}{l_o} x100 \qquad \text{(Equation 1)}$$

Here, % ε is the total deformation ratio, and l_o and l_s are the first and last length of material, respectively.

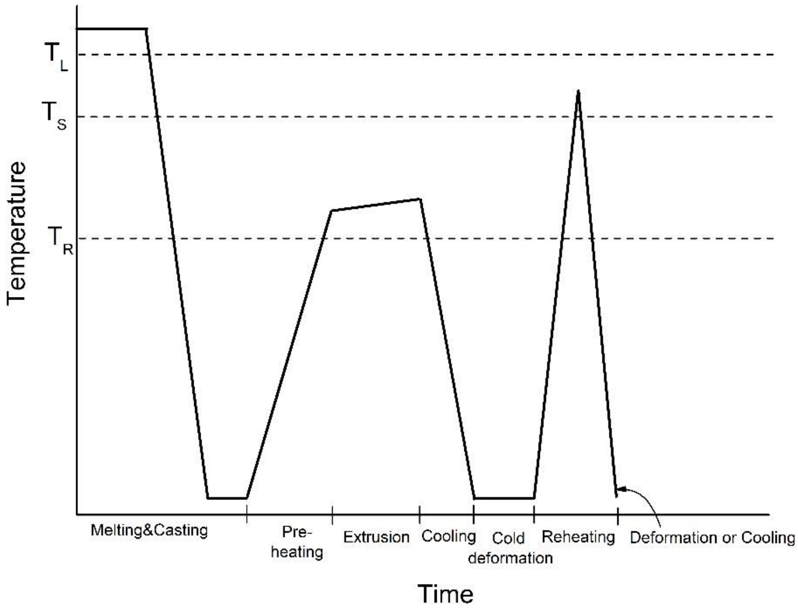

Figure 1. Schematically representation of the SIMA process in the time-temperature diagram (Young et al., 1983)

In order to apply deformation at different rates (10%, 20%, 30%, and 40%), samples cut according to height calculated in Eq. 1 were compressed vertically in a 250-ton hydraulic press at room temperature in 2 mm/min speed until the last height is 75 mm. 40% of deformed samples cracked in the middle by forming buckling (Figure 2). In the studies, 30% deformation could be conducted at most at samples in 40 mm without buckling.

The diameter of samples which were 40 mm before deformation, increased between 45-48 mm after deformation, depending on the deformation ratio. Before samples were heated to a semi-solid temperature with the SIMA method, they were turned and brought into 40 mm and 55 mm.

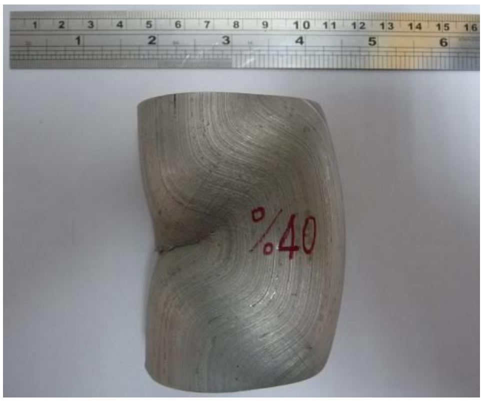

Figure 2. 40% cold deformed sample

2.3. Determination of Semi-solid Temperature Interval

Semi-solid processing is generally carried out at temperatures corresponding to a 30%-50% liquid ratio [1, 16]. In determining the liquid ratio, which changes according to the temperature of the AA7075 alloy, the DTA curve, and "partial area" method was used [17]. Liquid ratios according to a temperature calculated with this method are given in Figure 3. Temperatures that provide 30%, 40%, and 50% liquid ratios were determined as 600°C, 606°C, and 611°C, respectively.

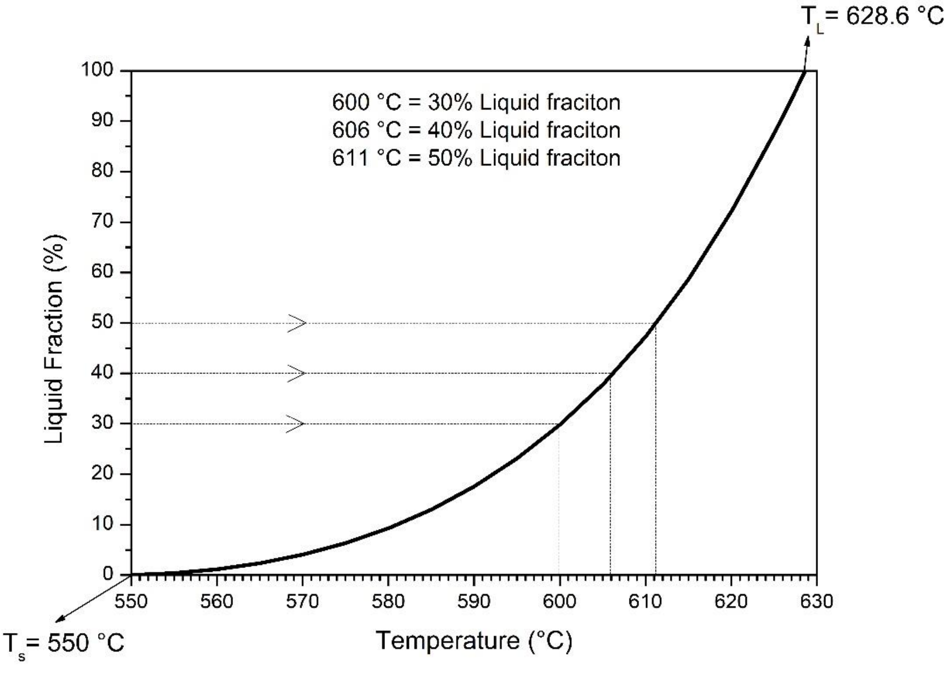

Figure 3. The changes in the liquid fraction depend on the temperature.

2.4. Reheating Experiments

A schematic view of the experiment set used in reheating and cooling the water process is given in Figure 4. In the semi-solid heating process, the sample placed in the coil is dropped in a container full of water with the help of a pneumatic system very rapidly when it reaches the semi-solid structure. The time for semi-solid temperature included within the coil falling into the container is around 2 sec.

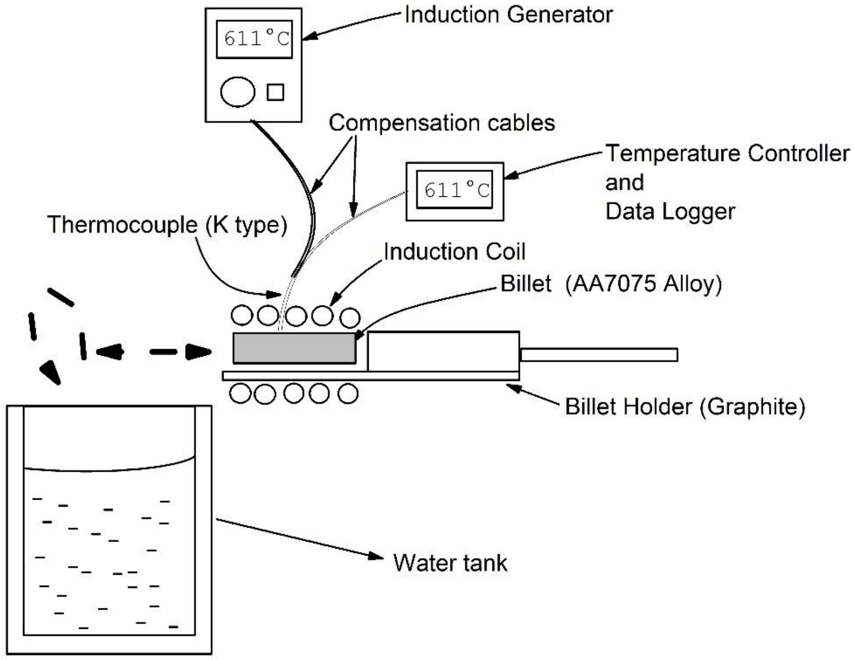

Figure 4. Schematically representation of the set-up used in the SIMA process to heat semi-solid temperature and quenching of the sample

2.5. Metallographic Studies

Microstructural characterization studies were carried out through an optical microscope. Samples that were cold deformed at different ratios and rapidly cooled from a reheating (semi-solid) temperature to produce pre-material with globular microstructure were cut vertically, as seen in Figure 5.b. Later on, three microstructure samples were excluded from the edge, middle, and center of the middle part of the samples (Figure 5.c).

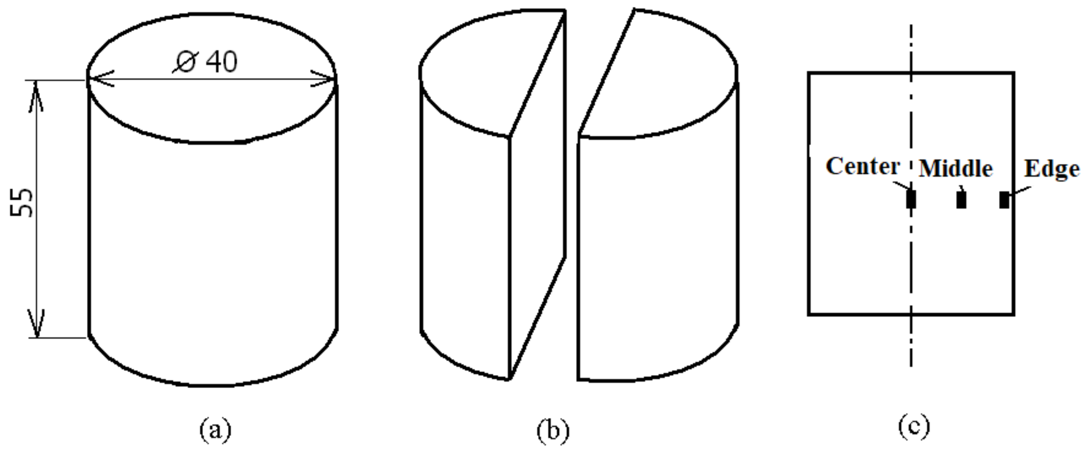

Figure 5. Metallographic examination of the sample produced by the SIMA process; a) sample, b) longitudinally cut a sample, and c) examined regions

Following sanding with Sic sanding material no 220, 400, 800, and 1200 respectively, metallographic samples were cut with the abrasive cut device; they were polished with 6 µm and 3 µm diamond paste and 5 µm silica. The solution was used as an etchant for the Keller (190 ml H2O + 5 ml HNO3 + 3 ml HCl + 2 ml HF).

Leica DFC 320 model digital camera connected Lecia DM 4000 M Model optic microscope was used to observe microstructures. In grain size measurement, the linear intersection method was used with the help of Leica Q550 MW image analysis software.

The globularity index of the feedstocks was determined with the help of the shape actor formula (Witulski, Morjan, Niedick, & Hirt, 1998) given in Equation 2.

$$\text{Shape factor} = \frac{4\pi A}{P^2} \qquad \text{(Equation 2)}$$

Here, A is the area of grain that is measured, and P is the perimeter of the grain. Measurements were taken from regions corresponding to 9 mm^2 (3x3 mm) of edge, middle, and center regions shown in Figure 5. Five hundred measurements were made from each sample to determine grain size, and the shape factor and average values were determined.

3. RESULTS AND DISCUSSIONS

3.1. The Effect of Cold Deformation Ratio and Temperature on Microstructure

In Figure 6 to Figure 8, microstructure changes in different parts of samples applied to different deformation ratios and cooled from different reheating (semi-solid) temperatures are observed. With increasing reheating (semi-solid) temperature and cold deformation amount, globular structure formation has become more distinctive.

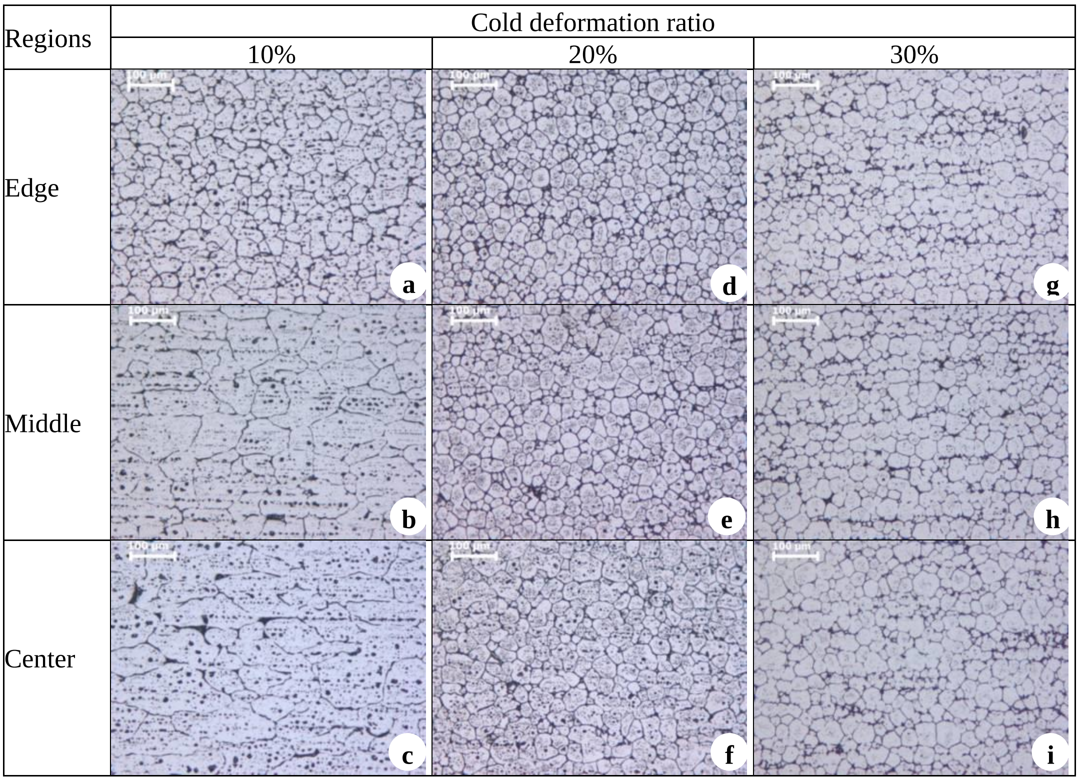

Figure 6. Microstructures of the AA7075 alloy water quenched from 600°C after deformed at various cold deformation ratios

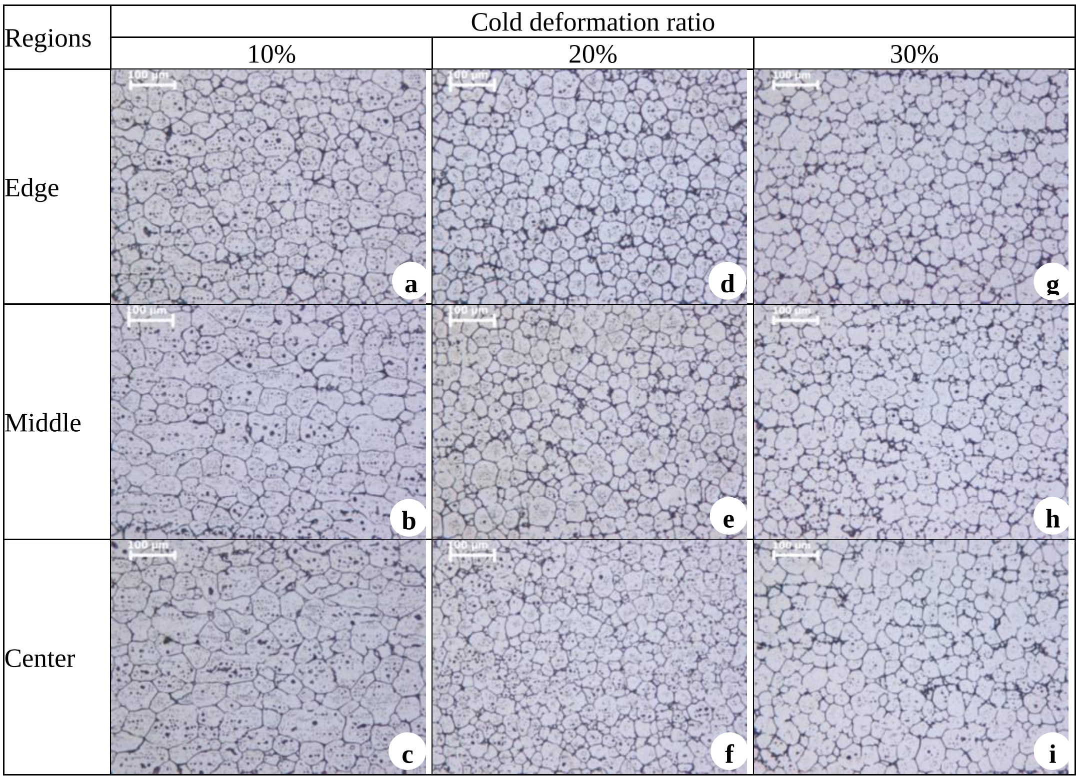

Figure 7. Microstructures of the AA7075 alloy water quenched from 606°C after deformed at various cold deformation ratios

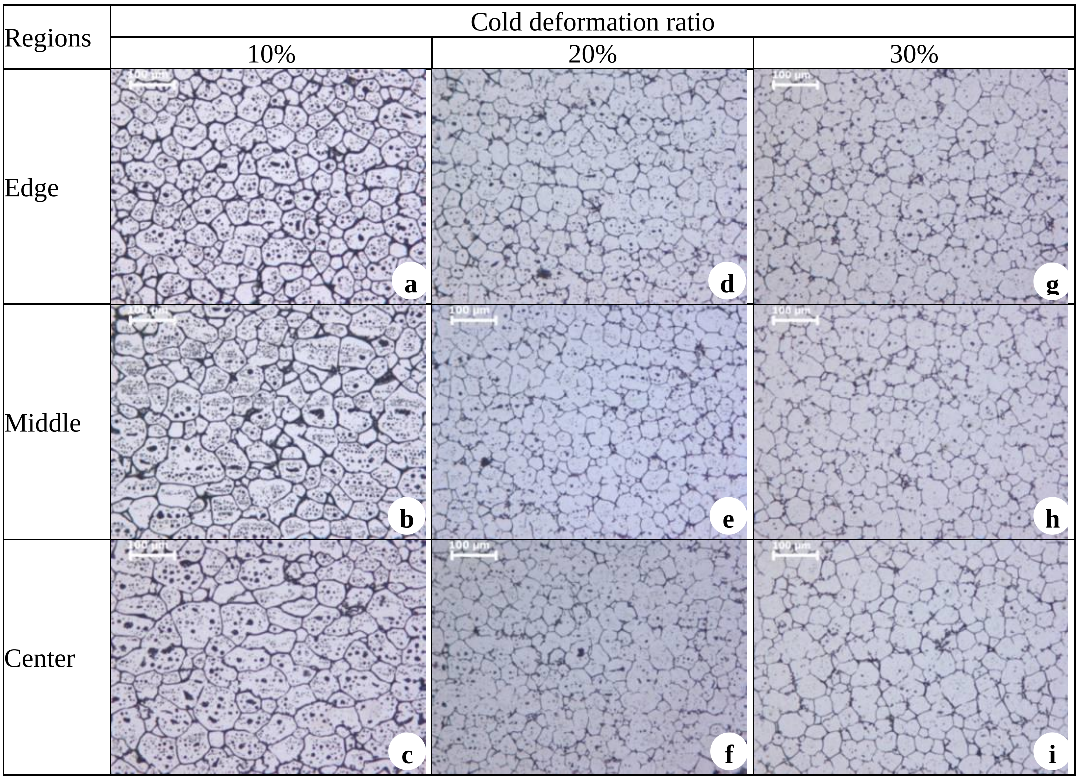

Figure 8. Microstructures of the AA7075 alloy water quenched from 611°C after deformed at various cold deformation ratios

Cold deformation and reheating produced different grain sizes nearly the same from the edge, middle and central area for each cold deformation ratio of samples (Figure 6.g-6.i, Figure 7.g-7.i, and Figure 8.g-8.i). These results show that cold deformation spreads throughout the sample, and the induction coil and heating are homogeneous.

While there is a grain structure with coarse and low shape factor in 10% cold deformed sample (Figure 6.a-6.c, Figure 7.a-7.c and Figure 8.a-8.c), fine and more globular grain structure is obtained in 20% and 30% cold deformation ratio (Figure 6.d-i, Figure 6.d-i and Figure 8.d-i).

Recrystallization was partly attained at 10% cold deformed sample due to insufficient internal strain. Recrystallization occurred in 20% and 30% cold deformation applied samples during heat treatment at the semi-solid state, and globular grains were formed. Akar and Mutlu (Akar & Mutlu, 2010) stated that the SIMA method's 10% cold deformation ratio does not produce a globular structure for AA2024 alloy.

In Figure 6.a-6.c, Figure 7.a-7.c, and Figure 8.a-8.c, a microstructure shows a low shape factor after 10% cold deformation. This is considered insufficient internal energy due to low deformation and few nucleations for recrystallization.

3.2. The Effect of Cold Deformation Ratio and Temperature on Grain Size

The change in the mean grain size due to the cold deformation ratio and reheating to the reheating (semi-solid) temperature is given in Figure 9 and Figure 10. In contrast, average grain size decreased with the increasing cold deformation ratio (Figure 9), and average grain size increased with the increasing reheating temperatures (Figure 10). It is stated through different studies that these two parameters are effective on grain size (Helen Victoria Atkinson, Burke, & Vaneetveld, 2008; Sang-Yong, Jung-Hwan, & Young-Seon, 2001; Türkeli, 1993).

Similar to this study, Sang-Yong et al. (Sang-Yong et al., 2001) reported that in 7075 alloy, grain size decreased with increasing cold deformation, and grain size increased with increasing reheating (semi-solid) temperature and holding at a reheating (semi-solid) temperature in feedstock production with the SIMA process.

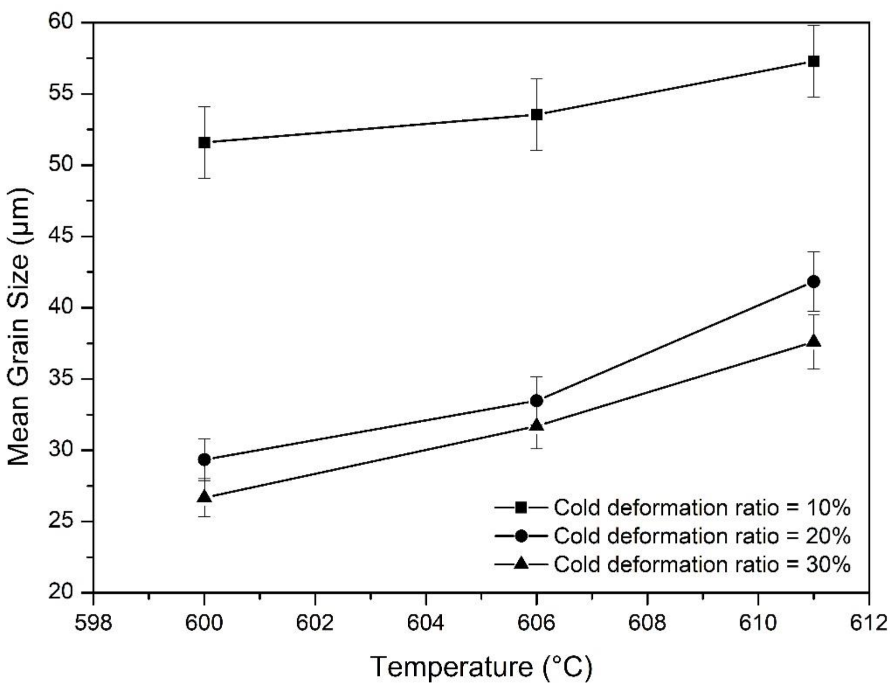

Figure 9. The relationship between reheating temperature and grain size

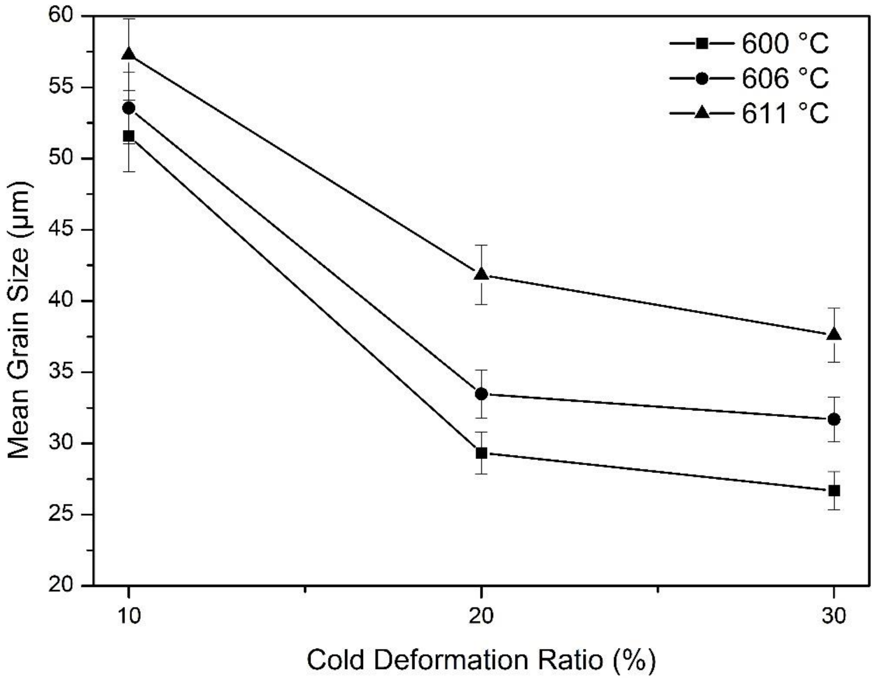

Figure 10. The relationship between cold deformation ratio and grain size

The increase in grain size with increasing temperature is explained through the "coarsening" mechanism in which surface energy is the driving force (Verhoeven, 1975). When the holding duration at reheating (semi-solid) temperature is as long as to allow solid diffusion, the "Ostwald ripening" mechanism begins to be effective in grain growth (Porter & Easterling, 2009; Verhoeven, 1975). However, the Ostwald ripening mechanism was ineffective since there is no process of holding at a reheating (semi-solid) temperature.

Another mechanism can explain grain coarsening with increasing temperature. With increasing temperature, grains minimize their potential interface thermodynamically and make them more stable; grains grow when their surface free energy increases when the total grain boundary (interface) is decreased, and total surface free energy decreases (Porter & Easterling, 2009; Verhoeven, 1975).

The decrease in average grain size with increasing cold deformation ratio is attributed to the high nucleation amount caused by the high-strain ratio in recrystallization (Porter & Easterling, 2009; Verhoeven, 1975), as a result of internal strain increased by cold deformation in recrystallization, while the growth rate of grains slows down nucleation speed increases. Finer structures can be obtained in higher nucleation amounts (at high cold deformation ratios) (Porter & Easterling, 2009).

Average grain size at reheating (semi-solid) temperature should be below 100 µm; homogeneous material flow should be enabled, and fine sections and mold gaps should be filled entirely for thixoforming (H. Atkinson, Kapranos, Liu, Chayong, & Kirkwood, 2002; Helen V Atkinson, 2005; Birol, 2007; Chayong, Atkinson, & Kapranos, 2004; Garat, 1998; Jung & Kang, 2000; Kim & Kang, 2000; Witulski et al., 1998). Since the average grain size of samples produced with the SIMA process using 20% and 30% cold deformed billets are between 25–45 µm, it was determined that they are suitable for semi-solid metal forming.

3.3. The Effect of Cold Deformation Ratio and Temperature on Shape Factor

The change in shape factor due to the cold deformation ratio and the reheating (semi-solid) temperature is given in Figure 11 and Figure 12. The shape factor increases due to increasing reheating (semi-solid) temperature (Figure 11) and the cold-deformation ratio (Figure 12).

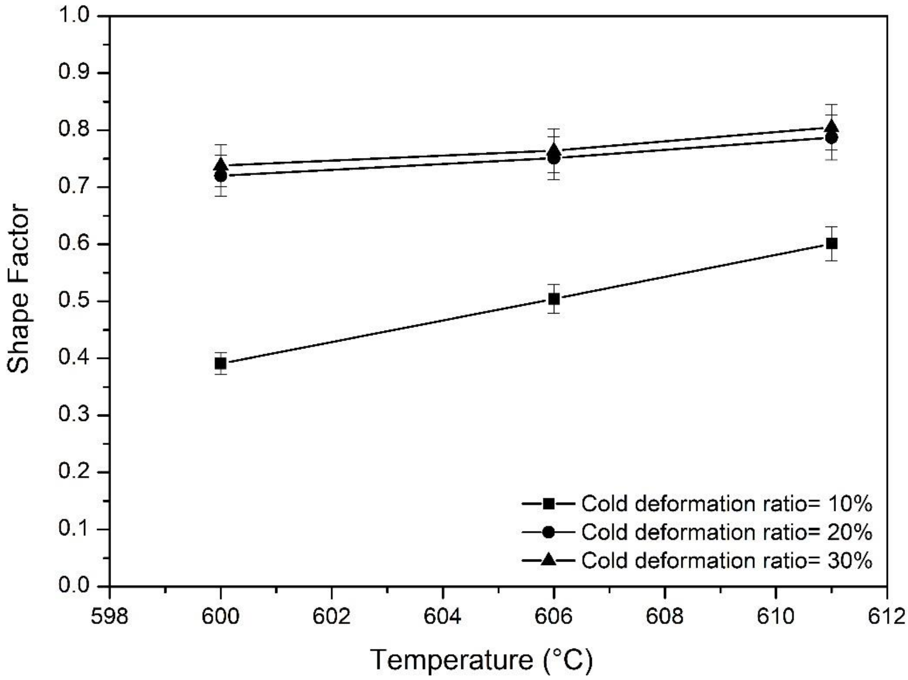

Figure 11. The relationship between reheating (semi-solid) temperature and shape factor

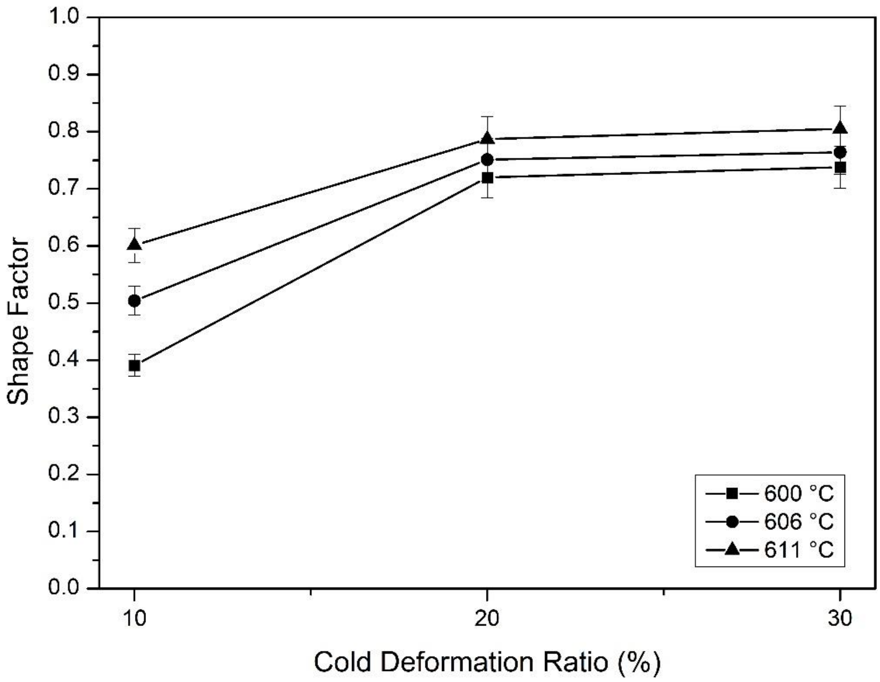

Figure 12. The relationship between cold deformation ratio and shape factor

The increase of shape factor due to increasing reheating (semi-solid) temperature can be explained through increasing liquid phase ratio. Since the heating temperature at the semi-solid area is above eutectic temperature, partial melting begins. Melting occurs at grain boundaries with high-energy deformation and phase areas with a low melting point. In order to reduce energy at the grain boundary, recrystallized equaxial grains melt and depart from each other at the end where they contact. At the same time, a eutectic phase that has a low melting temperature above the solidification temperature also melts. The melted eutectic liquid phase penetrates grain boundaries with energy and enhances equaxial grains separation. Equaxial grains are surrounded by liquid-convex areas on the solid-phase surface, surrounded by liquid melt (Guner et al., 2019). The existence of high amounts of liquid phase causes grains to become more globular. Similarly, Dong et al. (Dong, Cui, Le, & Lu, 2003) state that increasing reheating (semi-solid) temperature increases the globularity index degree in the same alloy.

The low shape factor ratio (0.4-0.6) at 10% cold deformed sample can be attributed to recrystallization due to the insufficient internal energy of this sample. Polygonal coarse grains were obtained from this sample due to insufficient internal energy (Figure 6.a-6.c, Figure 7.a-7.c, and Figure 8.a-8.c). As is seen in Figure 12, the shape factor increases with the increasing cold deformation ratio (0.7-0.8). Recrystallization occurs at 20%, and 30% cold deformed samples result from fine, globular structure (Figure 6.d-6.i, Figure 7.d-7.i and Figure 8.d-8.i). for a successful thixoforming at industrial practices, samples (feedstocks) are desired to have shape factor above 0,6 (Friedrich, Arnold, Sauermann, & Noll, 2009; Guner et al., 2019; Kapranos, Liu, Atkinson, & Kirkwood, 2001).

The increase of cold deformation from 20% to 30% significantly increases the shape factor. Liu et al. (Liu, Atkinson, Kapranos, Jirattiticharoean, & Jones, 2003) stated in a thixoforming study of high-resistance wrought Al alloys that 0-5 min. Holding duration at reheating (semi-solid) temperature increases shape factor, but longer holding durations (5-30 min.) cause a decrease in shape factor. According to these results, it is understood that holding duration at reheating (semi-solid) temperature is more effective on the shape factor. However, Önsel (Önsel, 2005) emphasizes that in feedstock production with the SIMA method, the reheating (semi-solid) temperature is more effective than holding duration in thixotropic structure formation with a globular structure (Önsel, 2005). This study determined that increase in shape factor is more distinctive with increasing reheating (semi-solid) temperature (Figure 11). Liu et al. (Liu et al., 2003) state that long holding duration at reheating (semi-solid) temperature cause macro-segregation at the center and edge of the sample.

4. CONCLUSIONS

The influence of cold deformation ratio and reheating temperatures were investigated on the microstructure of AA7075 alloy, which was produced by the strain-induced melt activation (SIMA) process. The following conclusions can be drawn from the presented study;

1. The grain size decreased while the shape factor increased with an increasing cold deformation ratio in feedstock production.
2. While grain size increases with increasing reheating temperature, it was determined that the change in shape factor is not significant at a 20% cold deformation ratio.
3. It was determined that at least 20% cold deformation is required for obtaining homogeneous thixotropic structure production by the SIMA method in the AA7075 alloy.
4. There is no need to soak at a reheating (semi-solid) temperature to obtain globular structure production in the AA7075 alloy, which is applied with 20% and above cold deformation

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