Recrystallization Behavior of 7175 Al Alloy during Modified Strain-Induced Melt-Activated (SIMA) Process

Young Buem Song¹*, Kyung-Tae Park² and Chun Pyo Hong¹

¹Department of Metallurgical Engineering, Yonsei University, Seoul 120-749, Korea
²Division of Advanced Materials Science and Engineering, Hanbat National University, Taejon 305-719, Korea

A modified strain-induced, melt-activated (SIMA) process for semi-solid processing of alloys was proposed. In order to examine the applicability and effectiveness of the modified SIMA process, the recrystallized microstructures of a high strength 7175 Al alloy prepared by the modified SIMA processes were macroscopically compared to that of conventional process. The modified SIMA process employed casting, two stage homogenization, warm multi-forging, and recrystallization and partial melting (RAP) instead of the conventional process consisted of casting, hot working, cold working and RAP. For the alloy processed by the conventional SIMA process, the recrystallized grain size decreased with increasing the cold rolling reduction ratio up to 20% and then almost unchanged. RAP treatments of the 20% cold rolled alloy at above 600°C and for longer than 30 min resulted in significant grain growth. In case of the modified SIMA process, the alloy multi-forged with the accumulated strain of about 7 and RAP at 575°C for 10 min exhibited the uniform equiaxed recrystallized grain structure similar to that of the conventional SIMA process under the optimum conditions. The improved processing efficiency of the modified SIMA process over the conventional one, i.e. removal of hot working and saving of RAP time, is attributed to the enhanced recrystallization kinetics due to a high density of Mg(Zn,Al,Cu) precipitates which are formed by two stage homogenization, acting as the preferential recrystallization sites and due to the driving force imposed by relatively large amount of uniform deformation by multi-forging.

Keywords: semi-solid processing, strain induced melt activated (SIMA) process, homogenization, multi-forging, recrystallization, aluminum alloy

1. Introduction

Semi-solid processing (SSP)¹ in which metal forming is carried out in a mushy zone provides very effective assess for near net shaping of metals and alloys.² The success of SSP strongly depends on the rheological characteristics of materials affected by their microstructures, such as phase fraction, grain size and shape, phase network state, etc., at given processing conditions.³,⁴ In general, it is known that the skeleton structure of fine solid spheroids without dendrites is the most suitable for SSP.⁵ In order to produce such sound initial microstructure for SSP, several techniques such as magneto-hydrodynamic (MHD) casting and spray casting have been developed and commercialized.⁶–⁸ Since liquid is cooled to a mushy zone in MHD casting and spray casting, these processes are difficult to apply to the alloys which are sensitive to hot cracking and inadequate to economic fabrication of the small-size semi-solid billets. In order to overcome such deficiencies, an alternative process called the stress induced, melt activated (SIMA) process was developed.⁹ The SIMA process involves the four processing stages: casting, hot working, cold working, and recrystallization and partial melting (RAP).⁹,¹⁰ In the SIMA process, an alloy having the sufficient stored energy by cold working is heated to a mushy zone. During heating, recovery and recrystallization occur before liquid formation with the aid of the stored energy. Reaching the mushy zone, liquid is formed by preferential melting at grain boundaries with high energy state, and penetrates into high angle boundaries of recrystallized grains. Accordingly, the amount and distribution of the stored energy by cold working is the most critical factor in the SIMA process since they control the recovery and recrystallization kinetics and the uniformity of the resultant microstructure.

The present investigation was motivated by two main considerations. First, the distribution of the stored energy by cold rolling is usually inhomogeneous across the work piece section,¹¹ causing non-uniform microstructure at the RAP stage. Therefore, the alternative working techniques which can distribute the large amount of the stored energy more homogeneously throughout the work pieces should be employed to ensure the microstructure uniformity. Second, in case of the difficult-to-work alloys, the amount of cold rolling is limited in order to avoid cracking; for instance, about 20% for the high strength wrought Al alloys. In such a case, the selection of optimum RAP conditions becomes important to obtain the desired skeleton structure with fine equiaxed grains for successful SSP. Accordingly, it is essential to figure out the effects of RAP conditions on the microstructures of the alloys having the cold working limit in the conventional SIMA process.

The purpose of this study is two-fold; (a) introduction of a modified SIMA process and its application to a high strength 7175 Al alloy, and (b) examination of the separate effect of each stage of the conventional SIMA process on recrystallization of the same alloy in a mushy zone. A modified SIMA process includes homogenization and warm multi-forging instead of hot working and cold working in the conventional SIMA process (Fig. 1). Multi-forging is an effective process for microstructure refinement.¹²–¹⁶ In order to examine the effectiveness of the present new process, the RAP microstructures developed by both conventional and modified processes were compared.

2. Experimental Procedures

For the present investigation, a high strength 7175 Al alloy
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3. Results and Discussion

3.1 Conventional SIMA process

3.1.1 Effect of cold working

In order to examine the effect of cold working amount on RAP microstructure, the hot rolled plates were cold rolled with the reduction ratios of 5–32% and then recrystallized at 575°C for 30 min. The representative OM micrographs of RAP microstructure are shown in Fig. 3. Without cold rolling (0%), RAP microstructure consisted of equiaxed grains with the presence of liquid phase at some grain boundaries. However, the grain size distribution was bimodal, small grains of ~20µm and large grains of ~100µm. The grain size was steadily decreased up to 15% cold rolling. By further cold rolling, the grain size remained nearly unchanged in association with dynamic recovery, and its distribution became more uniform.

The effect of cold rolling on the RAP microstructure can be explained in terms of the grain deformation-recrystallization model.17) As the cold rolled alloy is still in the solid state during heating to the mushy zone, recovery and recrystallization occur at the expense of the stored energy. By holding at the mushy zone, liquid formation occurs preferentially at high angle grain boundaries (HAGBs) of recrystallized grains since these HAGBs are in the higher energy state than the strain free recrystallized grain interiors. Concurrently, liquid penetrates into HAGBs by a capillary effect, causing the fragmentation and/or enclosure of grains. The increment of cold rolling amount accelerates recovery and recrystallization kinetics and provides the larger number of recrystallization sites. Therefore, the more severe cold rolling is imposed, the finer grain size is obtained.

3.1.2 Effect of RAP temperature

The 20% cold rolled alloy was recrystallized in the temperature range of 400–625°C for 30 min and the resultant microstructures are shown in Fig. 4. As discussed in the preceding section, the RAP experiments were conducted on the 20% cold rolled samples since cold rolling over 20% may be harmful due to cracking for the present alloy without further grain refinement. The cold rolled microstructure remained up to 500°C, i.e. no recrystallization. The partially recrystallized microstructure appeared at 525°C. Fully recrystallized equiaxed grains of 50–60μm were obtained at 550 and 575°C. At 600°C, significant grain growth
occurred and most grains were surrounded by liquid layer. The microstructure at 625°C consisted of coarse grains with localized liquid pools.

The semi-solid with 50–70% solid can bear its own weight and so exhibits the solid-like behavior. When it is subject to shear deformation, it behaves as high viscosity liquid.18) In

Fig. 3  Variation of the recrystallized grain size with the cold rolling reduction ratio (RAP at 575°C for 30 min) in conventional SIMA processed 7175 Al alloy.

Fig. 4  Optical micrographs of the RAP microstructures at various RAP temperatures (20% cold rolling and RAP for 30 min) in conventional SIMA processed 7175 Al alloy.
Fig. 5, the solid fraction of the alloy is predicted as a function of the RAP temperature by the Scheil’s equation\(^\text{19}\) of

\[
\begin{align}
\frac{f_S}{C_0} &= \frac{1}{k} \left( \frac{T_M}{C_0} \right)^{\frac{1}{1-k}} \\
V_f &= \frac{f_S}{1 - (T_M - T_L)}
\end{align}
\]

where \(f_S\) and \(V_f\) are the weight and volume fraction of solid respectively, \(T_M, T_L\), and \(T\) are the melting, liquidus, and RAP temperatures, respectively, \(\rho_S\) and \(\rho_L\) are the solid and liquid density, respectively, and \(k\) is the partitioning coefficient. As shown in Fig. 5, the solid fraction at 575 and 600\(^\circ\)C are \(0.80\) and \(0.60\), respectively. Therefore, RAP at 600\(^\circ\)C seems to be desirable for SSP but it should be reminded that a coarse grain structure at 600\(^\circ\)C as revealed in Fig. 4 deteriorates the rheological properties by lowering the flow resistance of semi-solid during shear deformation of SSP.\(^\text{20}\)

### 3.1.3 Effect of RAP time

The microstructural change of the 20% cold rolled alloy with RAP time at 575\(^\circ\)C is shown in Fig. 6.

Recrystallized grains appeared at 5 min. Beyond 10 min, microstructure was mostly covered with recrystallized equiaxed grains and grain growth occurred continuously with increasing time by the Ostwald ripening mechanism. During Ostwald ripening, small grains with large boundary curvature remelt into liquid and large grains grow by receiving solute atoms from liquid with higher solute content.\(^\text{21-26}\) When grain growth is controlled by the volume diffusion of solute atoms in the solid state, the grain growth behavior generally obeys the third power non-ideal grain growth law\(^\text{27,28}\) of

\[
d^3 - d_0^3 = Kt
\]

where \(d\) is the grain size at time \(t\), \(d_0\) is the initial grain size, and \(K\) is the grain growth rate coefficient. In order to check the applicability of eq. (2) to the present case, \(\Delta d^3\) \((= d^3 - d_0^3)\) is plotted against RAP time in Fig. 7. As seen in Fig. 7, the data exhibit the excellent linearity, indicating that grain growth of the alloy in the semi-solid state was rate-controlled by volume diffusion.

### 3.2 Modified SIMA process

#### 3.2.1 Microstructure developed by rehomogenization and multi-forging

A representative SEM micrograph of the alloy after rehomogenization at 482\(^\circ\)C for 3 h followed by furnace cooling is shown in Fig. 8. After rehomogenization, two types of phases were observed; one coarse boundary phases (‘A’ in Fig. 8) and the other rod-shaped precipitates at grain interior (the area ‘B’ in Fig. 8). By EDS and XRD analysis, the former was identified as eutectic Al\(_7\)Cu\(_2\)Fe or Al\(_2\)CuMg intermetallic compounds with low melting point.\(^\text{29}\) These are probably formed at interdendrites by solute rejection during partial melting. The latter was identified as Mg(Zn,Al,Cu)\(_2\) precipitated during rehomogenization. These rod-shaped Mg(Zn,Al,Cu)\(_2\) precipitates with the length of \(\sim 5\mu\text{m}\) are enough to act as the preferential sites for stored energy accumulation during subsequent multi-forging, and, in turn, for recrystallization during RAP.\(^\text{30}\) The microstructures developed by warm multi-forging at 250\(^\circ\)C are shown in

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**Fig. 6** Optical micrographs of the RAP microstructures at various RAP times (20% cold rolling and RAP at 575\(^\circ\)C) in conventional SIMA processed 7175 Al alloy.
Fig. 7 A plot of $\Delta d^3$ vs RAP time showing the validity of volume diffusion controlled grain growth.

Fig. 9 Optical micrographs of microstructures developed by multi-forging in new modified SIMA processed 7175 Al alloy. (a) no multi-forging (b) $\Sigma e = 7.0$ (c) $\Sigma e = 9.5$.

3.2.2 RAP microstructure

After multi-forging with $\Sigma e = 7.0$ and 9.5, RAP was conducted at 575 and 600°C for only 10 min and the resultant RAP microstructures are shown in Fig. 10. Without multi-forging, the grain size was $\sim 130 \mu m$ regardless of RAP temperature. In addition, the shape of most grains was out of spheroid and the liquid pools locally existed at some grain boundaries. This liquid localization was mainly attributed to the first melting of coarse boundary eutectic phases with low melting point since no strain-induced melting was expected. By imposing multi-forging, the grain size was considerably refined. The grain size at 575°C was slightly smaller than that at 600°C. It was quite noticeable that the grain size developed by only 10 min RAP of the modified SIMA process ($\sim 50 \mu m$) was comparable to that produced by 30 min RAP of the conventional SIMA process ($\sim 55 \mu m$). The RAP time saving of the modified SIMA process resulted from the enhanced recovery and recrystallization kinetics due to imposition of relatively large deformation by multi-forging and uniform distribution of a large number of recrystallization sites, i.e. Mg(Zn,Al,Cu)$_2$ precipitates, formed by rehomogenization.

3.2.3 Advantages of the modified SIMA process

The present modified SIMA process exhibits several advantages over the conventional one. First, the hot working stage in the conventional SIMA process is removed and so the modified process can be applied to the alloys sensitive to hot working. Second, more uniform microstructure can be

Fig. 9. The number and size of coarse boundary phases (the black patches in Fig. 9) were considerably reduced and their distribution became homogeneous by repetitive multi-forging, resulting in relatively uniform microstructure. In addition, the coarsening of Mg(Zn,Al,Cu)$_2$ precipitates concurrently occurred due to warm multi-forging temperature of 250°C.
achieved by homogeneous distribution of fine precipitates and uniform deformation by multi-forging. Third, the RAP time is remarkably saved by enhanced recrystallization kinetics associated with a high density of fine precipitates acting as the preferential recrystallization sites and relatively large amount of deformation by multi-forging. Finally, the semi-solid billets can be fabricated without the dimensional limit, while the conventional SIMA process employing cold rolling cannot produce the heavy sectioned plates or bars due to cold cracking. Even if extrusion is employed instead of rolling, cold extrusion is extremely difficult especially for large size billets.11)

4. Conclusions

(1) In the present investigation, the separate effects of each stage (cold working, and RAP temperature and time) of the strain-induced, melt-activated (SIMA) process on recrystallization of a high strength 7175 Al alloy for its semi-solid processing were investigated. In addition, to overcome the deficiencies of the conventional SIMA process, a modified SIMA process in which homogenization and warm multi-forging were employed instead of hot working and cold working in the conventional one was proposed.

(2) For the alloy processed by the conventional SIMA process, the recrystallized grain size decreased with increasing the cold rolling reduction ratio up to 20% and then remained nearly unchanged with further cold rolling. RAP treatments of the 20% cold rolled alloy above 600°C and longer than 30 min resulted in significant grain growth. The grain growth behavior of the alloy well obeyed the third power grain growth law, indicating the volume diffusion controlled growth.

(3) The alloy which was prepared by the modified SIMA process of multi-forging with the accumulated strain of ~7 and RAP at 575°C for 10 min exhibited the uniform equiaxed grain structure similar to that of the alloy processed by conventional SIMA process with 20% cold rolling and RAP at 575°C for 30 min.

(4) The improved processing efficiency of the modified SIMA process over the conventional one, i.e. removal of hot working and RAP time saving, results from the accelerated recrystallization kinetics associated with a high density of Mg(Zn,Al,Cu)₂ precipitates formed by rehomogenization acting as the preferential recrystallization sites and with the driving force for recrystallization by a large amount of uniform deformation by multi-forging.

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