Backward extrusion of 7075 Al alloy in the semisolid state

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In this study, 7075 Al alloy which has low extrudability has been thixoformed by backward extrusion process. The stress induced and melts activation (SIMA) route was used to obtain the required semi-solid feedstocks for thixoforming. Microstructure evolutions during partial remelting were studied at different temperatures. Results showed that a fine and globular microstructure can be obtained by the SIMA route. Back-extrusion of 7075 Al alloy was performed in the semi-solid state at 580°C for 10 min holding time. Microstructure and mechanical properties of thixoformed components before and after the T6 heat treatment were examined at room temperature. The weak tensile properties and low hardness values in the as-thixoformed parts were improved by subsequent heat treatment.

Key words: Semi-solid processing, 7075 Al alloy, microstructure, mechanical property.

INTRODUCTION

Semi-Solid Forming (SSF) is a novel technology in forming near net shape components in the 21st century, offering several potential advantages over conventional casting and forging technologies such as high product quality and low forming efforts (Haitao and Miaouquan, 2005; Fan, 2002; Atkinson, 2005). The key to SSF is fabricating the semi-solid materials with a globular microstructure (Fan, 2002). Many methods such as mechanical or electromagnetic stirring, the addition of grain refining elements, and cooling slope are used to obtain globular microstructures (Fan, 2002; Atkinson, 2005). Among the production methods, SIMA is an ideal candidate with significant commercial advantages of simplicity and low equipment costs. In this route, the material is deformed by extrusion or other processes and then reheated to semisolid range, in which recrystallization occurs and liquid metal penetrates in the recrystallized grain boundaries thus resulting in solid globular grains surrounded by liquid (Chayong et al., 2005; Dong et al., 2003; Sang-Yong et al., 2001; Zhang et al., 2009).

Thixoforming and rheoforming are two forming processes lately developed (Tahamtan et al., 2008). It enables the forging of parts with complex shapes and gives high mechanical properties. Both thixoforming and rheoforming are based on the semi-solid state; the first includes the melting of the alloy, while the second one applies the solidification of the melted alloy (Becker et al., 2008). In thixoforming, the alloy is only partially liquid and the shrinkage is much less than that of a full molten alloy.

The semisolid processing is widely used to produce feedstock and final products of wrought and casting Al alloys. In this research, we focused on high strength 7075 wrought aluminium alloy, which is typically used in aircraft structural parts and other highly stressed applications where very high strength and good resistance to corrosion are required. For reduction of problems, which occur in hot extrusion of this alloy (such as low extrudability and high applied pressure on the die), the back-thixoextrusion was examined in this research.

Various advantages mentioned help researches to focus on the semi-solid forming of aluminium alloys (Rovira et al., 1999; Zhao et al., 2010; Birol, 2008; Hong-min et al., 2008). Some of these advantages are the extrusion behavior of Al-Cu alloys in the semisolid state (Rovira et al., 1999), the mechanical behavior and microstructure during thixoforming of semi-solid ZK60-Y magnesium alloys at a high solid content (Zhao et al., 2010) and the microstructural evolution of semi-solid
Table 1. Composition of aluminum alloy used in this study (wt %).

<table>
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<tr>
<th></th>
<th>Zn</th>
<th>Mg</th>
<th>Cu</th>
<th>Fe</th>
<th>Si</th>
<th>Cr</th>
<th>Mn</th>
<th>Ti</th>
<th>Al</th>
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<td></td>
<td>5.6</td>
<td>2.4</td>
<td>1.4</td>
<td>0.42</td>
<td>0.4</td>
<td>0.26</td>
<td>0.13</td>
<td>0.01</td>
<td>Bal</td>
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EXPERIMENTAL PROCEDURE

In this study, 7075 Al alloy rod with an extrusion ratio of 20:1 was used. The chemical composition of this alloy is given in Table 1.

Cubic specimens of 10 mm sides were used to investigate the semisolid microstructures after the reheating process. In order to determine the appropriate temperature for semisolid forming, the predeformed samples were reheated from room temperature to various temperatures: 560, 570, 580, 590, 600 and 610°C in an electric resistance furnace. When the heating temperature reached the predetermined values, samples were isothermally held for 10 min and then immediately taken out for quenching into water at room temperature. Samples for microstructure characterization were prepared by the standard metallurgical technique, followed by etching in a Keller’s reagent.

The billet and backward extrusion toolings (H13 hot-work tool steel) were used in the experimental and are shown in Figure 1. The cylindrical billets of 40 mm diameter and 30 mm length were machined from as-received billet. K-type thermocouple was used for monitoring the reheating temperature.

To perform the thixoforming, the back-extrusion toolings and billet were reheated isothermally at 580°C for 10 min in a resistance furnace and then the billets were back-extruded with the hydraulic extrusion equipment, with the punch speed of 0.5 mm/s. Then the thixoformed sample and toolings were air quenched to stop the microstructural changes. The thixoformed samples were vertically sectioned for microstructural study by optical microscope at the marked regions as shown in Figure 2a.

The tensile samples were machined from back-extruded parts with and without T6 (solution treated at 465°C for 16 h, followed by quenching and then aged at 125°C for 24 h) (Chayong et al., 2005) as shown in Figure 2a, and round tensile samples were prepared according to Figure 2b (ASTM E8. Standard Test Methods for Tension Testing of Metallic Materials. ASTM international). Tensile tests were performed at room temperature using a Zwick universal testing machine. Tensile properties values represent the average value of at least three test results. The macro-hardness examination was performed on an Optical Brinell–Rockwell–Vickers hardness tester, by imposing a load of 10 kg for 10 s. The average of hardness of the three parts was taken as the hardness of a product.

RESULTS AND DISCUSSION

Microstructure of reheated samples

Forming in the semisolid state requires that an alloy have a globular and fine grain microstructure. Therefore, to examine and select the optimal process conditions for semisolid forming, microstructure evaluation was performed in the samples, which were reheated to different temperatures in the semisolid range. The semisolid microstructures are shown in Figure 3.

The microstructures at each temperature were analyzed separately and the optimal temperature was selected. The microstructure of reheated samples consists of solid globules, which is surrounded by liquid. A part of the liquid is entrapped inside the solid globules.
Figure 2. (a) Location of tensile specimen, and (b) schematic for tensile specimens [ASTM-E8].

Figure 3. Microstructure of the semisolid 7075 Al alloy during remelting with the holding time of 10 min at the isothermal temperatures of (a) 560, (b) 570, (c) 580, (d) 590, (e) 600, and (f) 610°C.

and consequently does not participate in the deformation. From a mechanical point of view, this entrapped liquid contributes to increase the solid fraction (Favier et al., 2009). Figure 4 displays a typical microstructure of a semi-solid 7075 alloy.

During the plastic deformation of the samples, internal strain energy is stored in forms of dislocation multiplication, elasticity stress and vacancies. During the reheating, as the temperature increases, recrystallization occurred in the solid state and new grains nucleate and grow with different (low and high) angle grain boundaries (Zesheng et al., 2008). When the temperature reaches to above solidus, the high-energy grain boundaries of these new grains are penetrated by liquid, leading to the fragmentation of original grains to small equiaxed grains. The presence of liquid causes grain growth and spheroidization of the newly grains. The isothermal time and temperature in semi-solid state are two important factors to control the grain growth and degree of globularity (Mohamamdi et al., 2011).

Coarsening occurs with two mechanisms: namely coalescence and Ostwald ripening (Mohamamdi et al., 2011). Driving force for coarsening decreases surface energy. In coalescence mechanism, coarsening occurs with coalescence of neighboring grains controlled by solid - solid contacts and grains contacts. From Figure 3, it was concluded that the SIMA route is an effective method for producing 7075 Al alloy with the fine and spherical
grains required for semisolid forming treatment. As the reheating temperature increases, equiaxed grains were obtained, the amount of liquid in the grain boundaries increased and the α-Al grains became separated from each other. On the other hand, the grains tend to grow with increasing temperature. The semisolid alloy with coarse grains has an adverse effect on microstructure and mechanical properties of final thixoformed part. It was observed that from the microstructures of the samples held at 580°C for 10 min (Figure 3c), globular grains were produced, and the grains were fine with an average grain size of less than 70 μm and shape factor of around 0.7, indicating the successful preparation of semisolid alloy based on the results of the previous work of Mohamamdi et al. (2011).

**Microstructures of back-thixoextruded part**

For reaching the desired mechanical properties after backward-thixoextrusion, some parameters are important such as shape, size and distribution of grains and they are related to temperature of process, duration on this temperature, extrusion ratio in SIMA step and post heat treatments of specimens. The experimental results showed that when the billet was held at 580°C with the 10 min holding time, the microstructure of the billet was composed of fine and spherical grains and the remnant was composed of liquids. The reheated billet at these conditions is more suitable for the semisolid forming at high solid fraction; therefore the semisolid forming process was performed at these conditions.

The billets machined from the as-received 7075 Al alloy bar were successfully thixoformed and isothermally reheated at 580°C for 10 min, *in situ*, in a laboratory hydraulic press. In the above experiment, high quality part of 7075 Al alloy with a smooth surface finish could be obtained using the semi-solid backward extruding process.

The microstructures overall of the cross-section of thixoformed part are shown in Figure 5. Through
microstructural examination of thixoformed parts, fine and globular microstructure, which are characteristic microstructure of thixoformed parts, were observed in most of the cross-sectional area of a part (region 4), which was directly compressed; most of the liquid was extruded and it gradually moved up to the upper region of the part (region 1) and showed the greatest compression of the solid particles. This implies that the solid globules cannot flow easily like liquid phase, and they remain at the bottom of the extruded sample and the liquid phase expels out. Therefore, there is liquid fraction near the top edge of the part (region 1) more than that found elsewhere, and liquid segregation was observed as shown in Figure 5a. When the billet was back-thixoextruded with the high solid fraction, the globular grains in the semisolid metal were surrounded by the liquid film, and they flow as a viscoplastic fluid under the extruding force. The forming process is mainly composed of sliding between solid grains and the plastic deformation of solid grains.

Quantitative measurements of liquid volume fraction and average grain size in the thixoformed part were carried out on three different images of each region. The average amounts of liquid fraction and average grain size were plotted against the positions in Figure 6. However, very high cooling rates were necessary in order to reveal accurately the extent of the liquid phase in the semisolid state. Attempts to quench the thixoformed part into cold water failed to accurately identify the microstructure properties of the thixoformed part. So the thixoformed samples were quenched to room temperature in air. In the semi-solid process, microstructure distribution was different at various locations, due to the semi-solid nature. Liquid segregation occurred due to the separation phenomena of the solid grain and the liquid phase. Liquid segregation which occurred during the forming process can be reduced by the control of punch velocity and solid fraction. As shown in Figures 5 and 6b, region 4 was directly compressed, and the grain size decreased. The material at this region underwent severe plastic deformation, and the fine grains indicate that recrystallization has occurred. Reheating of the billet before forming must be accurate and homogeneous and should be quick to avoid excessive grain growth. Induction heating was preferred because of its high heat penetration and good controllability.

**Mechanical properties**

The tensile properties for backward-extruded specimens in the semisolid state with and without T6 heat treatment are shown in Table 2. The ultimate tensile strength (UTS) and elongation of the as-thixoformed samples without heat treatment is approximately 254 MPa and 5.8%, respectively. The UTS and elongation of specimens without T6 temper have a low value, which is due to the fact that eutectic liquid phases are concentrated in the inter-globular regions and as “pools” within the globules result in formation of shrinkage porosities after solidification, thereby reducing elongation of specimens. This problem is resolved to some extent with T6 heat treatment. It is realized that the
T6 heat treatment should be performed at 465°C for as-thixoformed part to dissolve non-equilibrium and low melting point phases completely and homogenise this inhomogeneous structure. During the T6 heat treatment, segregation of liquid reached its lowest value. And the solute elements in the matrix are in the supersaturated condition and tend to precipitate out during aging. These intermetallic precipitates enhance the mechanical properties by precipitation hardening. Therefore, after heat treatment, the average UTS and elongation increased to 482 MPa and 10.8%, respectively. It was revealed that the tensile properties of the as-thixoformed parts were improved and close to the as-extruded materials’ properties.

Figure 7a shows the secondary electron image for the fracture surface of the thixoformed specimen without T6 temper. With attention to the presence of shrinkage porosities in grain boundaries, the fracture occurred completely intergranular, and the grain deformation rarely happened which resulted to brittle fracture. Figure 7b shows a secondary electron image for the fracture surface of one specimen related to backward-thixoextrusion product under T6 tempering treatment. High deformation of grains and ductile fracture of tensile specimens were observed. The fracture surfaces of this specimen represent a mixed mode of failure involving both dimple and cleavage fracture. Dimple type of fracture exhibits numerous cuplike depressions that are the direct result of the micro void coalescence. The micro voids nucleate at regions of localized strain discontinuity, such as that associated with second-phase particles, inclusions, and grain boundaries. As the strain in the material increases, the micro voids grow in the coalescence and eventually form a continuous fracture surface. Cleavage fractures are transgranular, whereas low-energy fractures are mainly derived from the separation of atomic bonds.

Figure 8 shows the results of hardness measurement at four positions from the center of thixoformed part to the top edge. As shown in Figure 6b, the solid grains' size at
position (4) was relatively smaller than that of other regions. As shown in Figure 8, the center region (region 4), which was directly compressed, have the maximum hardness, and the hardness decreased gradually from the center toward the edge position of the thixoformed part. The hardness for region (1) was HV = 100 ± 6; however, in region (4), the hardness was HV = 140 ± 5. Another phenomenon that influenced the hardness value was liquid segregation. Liquid segregation is the major practical problem in semi-solid processes, which leads to heterogeneous mechanical properties in a final product. Low hardness values in the as-thixoformed parts were improved by T6 heat treatment. Post-forming heat treatment is one of the key parameters for improving the mechanical properties of thixoformed parts.

Conclusions

When the temperature increases above the solidus point, semi-solid microstructure of 7075 Al alloy consists of globular grains with liquid grain boundary films. For the 7075 Al alloy with an extrusion ratio of 20:1, the optimal process parameters, during partial remelting, were chosen at the isothermal temperature of 580°C with 10 min holding time. Through microstructural examination of thixoformed parts, fine and globular microstructures, which are characteristic microstructures of thixoformed parts, were observed in most of the cross-sectional area of the as-thixoformed part. The weak tensile properties and low hardness values in the as-thixoformed parts were improved by subsequent T6 heat treatment. As a result of fractography of the tensile specimens, it was realized that the fracture of the as-thixoformed parts has a brittle appearance, while the fracture surface of the thixoformed part with post heat treatment specimens has a mixture shape.

REFERENCES