Investigation on the microstructural evolution and mechanical properties of semi-solid Al-5Zn-3Mg-2Cu alloy based on recrystallization and partial remelting

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Abstract. The present research envisages the investigation of the optimized semi-solid process parameters of the Al-5Zn-3Mg-2Cu alloy. The effects of remelting temperature and holding time on the microstructure and mechanical properties of modified Al-5Zn-3Mg-2Cu alloy were studied by the recrystallization and partial remelting (RAP) process. The results reveal that with an increase in the semi-solid holding temperature, there was an increase in the average grain size of the Al-5Zn-3Mg-2Cu alloy, but when the temperature was increased to 635ºC, the grain roundness decreased owing to the grain coalescence and growth. After holding at 635ºC for 32 min, the grains were partially transformed into dendrites. Further, with the prolongation of the holding time, the grain size of alloy gradually grew and the roundness increased, and after holding 32 min at 625ºC, the grain roundness began to decrease. The optimum semi-solid process parameters were: 615ºC × 32 min and 625ºC × 16 min. The Al-5Zn-3Mg-2Cu alloy parts were prepared by thixoforming process. Under the conditions of 615ºC × 32 min and 625ºC × 16 min, the parts were formed completely and the surface of the parts was smooth and clean. Under the condition of holding 32 min at 615ºC, the tensile strength and elongation at the break of the parts correspondingly reached 576 MPa and 9.5% respectively, while they reached 589 MPa and 11%, respectively under the condition of holding 16 min at 625ºC.

1. Introduction

Thixoforming is an important semi-solid processing technology that combines the thixotropic characteristics of the semi-solid materials and the suitable plastic forming ability of the deformed aluminum alloy, thereby providing a new idea in the development and application of high-performance aluminum alloy products [1-4]. Mohammadi et al [5] studied the microstructural evolution of the semi-solid 7075 Al alloy manufactured by the strain-induced melt activation (SIMA) process. They indicate that the optimum process parameters should be chosen at the isothermal temperature of 580ºC with a holding time of <30 min. Wang et al [6] studied the microstructural coarsening of the 6061 aluminum alloy semi-solid billets prepared via the RAP process, revealing that the coarsening rate gradually increased with an increase in the isothermal holding temperature. Mohammadi et al [7] studied the microstructural evolution and mechanical properties of the back-extruded Al 7075 alloy in the semi-solid state. They found that the post-forming heat treatment is one of the key parameters for improving the mechanical properties of the thixoformed parts. Currently, the most preferred alloy in different industries is the Al-5Zn-3Mg-2Cu alloy, which is a lightweight high-strength deformed aluminum alloy and has a wide application in the aerospace parts, automobile bodies and electronic product casings [8-10]. However, there is no research done on the semi-solid
forming process of the Al-5Zn-3Mg-2Cu alloy. Therefore, in order to obtain the best semi-solid process parameters, and thereby promote its engineering application, the microstructure and mechanical properties of Al-5Zn-3Mg-2Cu alloy were studied by RAP process in the present study.

2. Experimental procedure

The chemical composition of the tested Al-5Zn-3Mg-2Cu alloy is presented in table 1. The differential scanning calorimetry (DSC) curve was measured by SDT- Q600 differential scanning calorimeter. The DSC curve of the Al-5Zn-3Mg-2Cu alloy is showed in figure 1. The low melting point compound begins to melt at 585ºC, which is also the solid phase line temperature of the alloy, and then reaches the peak at 635ºC, ending at 641ºC, which is the liquid phase line temperature of the alloy. Therefore, for Al-5Zn-3Mg-2Cu alloy, the suitable semi-solid processing temperature range is 585ºC ~ 641ºC. In this study, the remelting temperatures selected for Al-5Zn-3Mg-2Cu alloy were 585, 595, 605, 615, 625, and 635ºC respectively, and the holding time were 4, 8, 16, and 32 min, respectively.

Table 1. Chemical composition of the test alloy (%).

| Element | Zn | Mg | Cu | Cr | Mn | Si | Fe | Al |
|---------|----|----|----|----|----|----|----|----|
| Content | 5.17 | 2.53 | 1.57 | 0.25 | 0.082 | 0.07 | 0.02 | Bal. |

The sample was placed into a graphite tray protected by argon and was heated to 700ºC at the rate of 10ºC/min, and then cooled to room temperature at the same rate. The initial billet of the Al-5Zn-3Mg-2Cu alloy was first homogenized, then extruded and deformed. The deformed billet was then heated to the semi-solid temperature range. The target semi-solid structure was obtained by an isothermal treatment for a certain period of time. The ϕ 8 × 12 mm sample was intercepted from the deformed sample by the wire cutting method. The axial direction of the sample was parallel to the extrusion direction of the initial billet. The sample was heated using a resistance furnace with a heating rate of 150 ºC/min, and the sample was held for 10~15 s. When the sample reached the preset temperature, the temperature inside the sample became uniform and consistent, and the holding time of the sample in the furnace was included in the isothermal holding time. When the sample reached the holding time, the cooling of the sample was completed within 3 s, so as to maintain the instantaneous morphology of the sample to the greatest extent. Water abrasive paper was used to polish the sample without obvious scratches, and polishing machine, velvet polishing cloth, and 0.5 pm diamond polishing paste were used to polish the cross section of the sample to the mirror. The reagent used to corrode the cross section included 2 ml HF + 3 ml HCl + 5 ml HNO₃ + 190 ml H₂O, and the corrosion time was 8~10 s. The microstructure of the samples was observed by metallographic microscope. The grain size and shape factor were analyzed by the image analysis software, in which the average grain size and shape factor were defined by equations (1) and (2) [11]:

\[
\text{Average grain size} (d) = \frac{1}{n} \sum_{i=1}^{n} d_i
\]

\[
\text{Shape factor} (S) = \frac{A}{P^2} \times 100
\]
where $SF$ is the shape factor, $d$ is the grain size, $A$ is the area, $N$ and $P$ are the number and perimeter of solid grains, respectively. Al-5Zn-3Mg-2Cu alloy parts were prepared by the thixoforming process. The mechanical properties were tested at room temperature using the WDW-200D universal material testing machine. The tensile rate of the samples was 0.1 mm/s. Under each condition, three tensile samples were tested and their average values were taken.

3. Results and discussion

3.1. Microstructure of extruded Al-5Zn-3Mg-2Cu alloy

Figure 2 shows the metallographic microstructure of the extruded cross section and the longitudinal section of the Al-5Zn-3Mg-2Cu alloy. It can be observed from figure 2 that a large number of un-recrystallized solid particles are distributed in the microstructure, while some second phase particles are dispersed at the grain boundary. In figure 2(b), it can be seen that along the extrusion direction, the grain is noticeably elongated to form a banded structure.

3.2. Microstructure evolution with increasing holding temperature.

Figure 3 depicts the metallographic microstructure of the Al-5Zn-3Mg-2Cu alloy after holding for 16 min at 585–635°C, and figure 4 shows the average grain size and shape factor, under these conditions. After holding the temperature of the Al-5Zn-3Mg-2Cu alloy at 585°C for 16 min, most of the solid particle boundaries were not infiltrated by the liquid phase, the grains were connected to each other, and the undissolved mesophase was distributed at the grain boundary. When the holding temperature was raised to 595°C, the liquid phase was obviously increased, and some of the grains were infiltrated by the liquid phase. However, the shape of the solid phase particles was irregular, and the number of the second phase particles was greatly reduced due to the dissolution into the liquid phase. Simultaneously, because of the incomplete spheroidizing process, the grain size distribution was uneven. When the holding temperature was increased to 625°C, almost all the solid particles were separated by the liquid phase. However, when the holding temperature was increased to 635°C, the size of solid phase particles continued to increase, though the shape factor decreased when compared with the temperature of 625°C. This is attributable to the coarsening of the grains, leading to the
decrease in the grain roundness.

![Figure 3](image3.png)

Figure 3. Metallographic microstructure after holding for 16 min at different temperatures: (a) 585°C; (b) 595°C; (c) 605°C; (d) 615°C; (e) 625°C; (f) 635°C.

![Figure 4](image4.png)

Figure 4. Average grain size and shape factor after holding for 16 min at different temperatures.

Figure 5 shows the metallographic microstructure of the Al-5Zn-3Mg-2Cu alloy after holding for 32 min at 635°C ~ 645°C. As can be seen from figure 5(a), a serrated structure appeared at the boundary of some solid phase particles at 635°C for 32 min, which was due to the unsteady growth of the solid particles during water quenching. In the cooling process, the solid phase particles grew in the direction of the liquid phase between the solid phase particles. Due to the enrichment of the solute elements, the composition aggravated at the front end of the solid-liquid interface during cooling. This caused the growth mode of the solid phase particles to change from the plane to cellular shape. Finally, it transformed in the form of a dendrite growth [12]. With the increase of semi-solid holding temperature, the solute enrichment degree at the solidification front of solid-liquid interface increased, resulting in the aggravation of the unstable growth state and leading to a more obvious dendrite shape, as depicted in figure 5(b). Therefore, from the point of view of solid phase particle roundness, the holding time should be minimized when the semi-solid temperature exceeds 635°C.
Figure 5. Metallographic microstructure after holding for 32 min at different temperatures: (a) 635ºC; (b) 645ºC.

3.3. Microstructure evolution with increasing holding time

Figure 6. Metallographic microstructure at 615ºC for different holding time: (a) 4 min; (b) 8 min; (c) 16 min; (d) 32 min.

Figures 6 and 7 represent the metallographic microstructures of the Al-5Zn-3Mg-2Cu alloy after holding for 4-32 min at 615ºC and 625ºC, respectively. Figure 8 shows the average grain size and shape factor at different holding times. As shown in figures 6 and 8(a), the liquid phase film began to form at the grain boundary when the holding time reached to 4 min at 615ºC. However, owing to the low content of liquid phase, the liquid phase film was not continuous. When the holding time reached to 16 min, the liquid phase discernibly increased, more solid phase particles were infiltrated, and a small number of droplets appeared in the solid phase particles. When the holding time reached 32 min, the liquid phase increased further, most of the solid phase particle boundaries were infiltrated, and the roundness of grains was clearly improved. With the prolongation of the holding time, the mesophase in the microstructure gradually dissolved, and the dissolution of the low melting point phase directly resulted into the increase of the liquid phase. The formation of the liquid phase dissolved the dispersed...
phase in the microstructure, and the pinning effect of these relative grain boundaries disappeared gradually. The mobility of grain boundaries was greatly improved, thereby enhancing the recrystallization ability and spheroidizing rate of the microstructure.

It can be determined from figures 7 and 8(b) that the change of grain size at 625°C for different holding times was similar to that at 615°C; the average grain size increased with the prolongation of holding temperature time; and at 625°C, when the holding time was 16 min, the grain shape factor reached the maximum value. When the holding time was increased to 32 min, the shape factor decreased and the grain roundness decreased.

![Figure 7](image1.png)

**Figure 7.** Metallographic microstructure after different holding times at 625°C: (a) 4 min; (b) 8 min; (c) 16 min; (d) 32 min.

![Figure 8](image2.png)

**Figure 8.** Average grain size and shape factor at different holding times: (a) 615°C; (b) 625°C.

3.4. *Mechanical properties of Al-5Zn-3Mg-2Cu alloy under different partial remelting temperatures*

Figure 9 shows the room temperature mechanical properties of the Al-5Zn-3Mg-2Cu alloy holding time from 4-32 min at 615 and at 625°C. It can be observed from figure 9 that the tensile strength of the Al-5Zn-3Mg-2Cu alloy reached the maximum at the holding time of 32 min and holding
temperature at 615ºC, and the tensile strength reached the maximum at the holding time of 16 min and holding temperature at 625ºC. The elongation after breaking initially increased and then decreased with the prolongation of holding time. It reached the maximum when the holding time was 16 min, and continued to increase till the holding time was 32 min. After breaking, the elongation decreased. The average grain size and shape factor of the solid phase particles had an important influence on the tensile properties of the sample. Fine equiaxed grains can achieve good strength and ductility at the same time [13]. At 615ºC, as the holding time increased, the shape factor increased, and the strength of Al-5Zn-3Mg-2Cu alloy increased. However, due to the long-term heat preservation, the grain coarsening occurred, and the elongation decreased after the holding time at 32 min. When held at 625ºC for 32 min, the grain merging increased and the shape factor decreased, thereby leading to a decrease in the strength and the elongation. From the above analysis, the optimum semi-solid processing temperature and the holding time of the Al-5Zn-3Mg-2Cu alloy were determined to be 32 min at 615ºC and 16 min at 625ºC.

Figure 9. Mechanical properties at different temperatures and holding times.

The optimized semi-solid processing method prepared the Al-5Zn-3Mg-2Cu parts. The forming process of the parts is shown in figure 10. After extrusion and deformation of the original billet, the original billet was cut into the required size and heated to the semi-solid temperature by induction heating. It was then held at that temperature for a certain period of time, forming parts with the forging equipment.

Figure 10. Forming process of parts.

The parts prepared by the thixoforming technology are exhibited in figure 11. The bodies of the parts were completely formed and the surface was smooth and clean. The mechanical properties under different semi-solid process conditions were tested at room temperature. Under the conditions of holding time of 32 min at 615ºC, the tensile strength and the elongation at break were 576 MPa and 9.5%, respectively, and the tensile strength and the elongation at break were 589 MPa and 11%,
respectively, under the condition of holding time of 16 min at 625°C.

Figure 11. Parts prepared by using the thixoforming technology.

4. Conclusions
The average grain size of the Al-5Zn-3Mg-2Cu alloy increased with the increase in the semi-solid holding temperature, but when the temperature increased to 635°C, the grain roundness decreased because of grain coalescence and growth. When the alloy was held at the temperature of 635°C for 32 min, the local grain was transformed into a dendrite form.

The grain size of the Al-5Zn-3Mg-2Cu alloy gradually grew up and its roundness increased after holding at 615°C for different periods of time. After holding at 625°C for 32 min, the grain roundness began to decrease. The optimum semi-solid process parameters were: 615°C × 32 min and 625°C × 16 min.

The Al-5Zn-3Mg-2Cu parts prepared by the thixoforming process had a complete forming and a smooth and clean surface under the conditions of 615°C × 32 min and 625°C × 16 min. Under the condition of holding for 32 min at 615°C, the tensile strength and the elongation at break of the parts bodies were 576 MPa and 9.5% respectively. Furthermore, under the condition of holding 16 min at 625°C, the tensile strength and the elongation at break reached 589 MPa and 11.0%, respectively.

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