Microstructural evolution of Al–Zn–Mg–Cu–(Sc) alloy during hot extrusion and heat treatments

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Abstract

The microstructural evolution during hot extrusion and post heat treatment was investigated for two kind of Al–Zn–Mg–Cu–(Sc) alloy and AA7075. The microstructure of as-extruded bar is mainly comprised of recovered structure for all alloys, however, different restoration processes are observed during post heat treatment. For AA7075 and S1, which contains 0.1% Sc with relatively higher Zn and Cu content than S2, recovery still proceeds during the heat treatment, while the recrystallization becomes main restoration process during the heat treatment for S2.

The differences in abnormal grain growth and hardening behavior between S1, AA7075 and S2 during the heat treatment are discussed in connection with the restoration processes and resultant microstructures.

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Keywords: Scandium; Microstructure; Recovery; Recrystallization

1. Introduction

Small quantity of Sc addition on Al alloys has attracted scientific and industrial interests because a significant improvement in weldability as well as an increase of the strength and thermal stability can be achieved [1,2]. It is reported that fine Al3Sc precipitates play an important role in the increase of strength and retardation of recrystallization during annealing [3,4]. The detailed precipitation kinetics and strengthening have been studied systematically for some series of Al–Sc binary alloys [5,6]. Recently, the precipitation behavior of Al3Sc in deformed Al matrix and its interaction with recrystallization is also investigated [7]. Most of previous works for Al alloys containing Sc mainly focused on the precipitation and recrystallization for annealed states after cold rolling.

For Al alloys containing Sc, the precipitation behaviors and its interaction with restoration process during hot working such as extrusion and post heat treatment are also important in an industrial point of view. Little works, however, has been carried out in this field and many aspects involved with hot working of Al–Sc alloys remain unclear. In present work, for introductory study, the microstructural evolution of Al–Zn–Mg–Cu alloys containing 0.1% of Sc is investigated for hot extrusion process and post heat treatment. The hardness changes during the processes are discussed in connection with the restoration processes. The possible mechanism of abnormal grain growth on the surface of extruded bar during post heat treatment is also discussed.

2. Experimental procedures

The chemical compositions of Al alloy used in present study are listed in Table 1. Two kinds of Al–(Sc) alloy and AA7075 were prepared. The basic compositions of S1 and S2 are similar to that of 7XXX series Al alloy except for 0.1% addition of Sc, but the content of Zn and Cu for S1 is slightly higher than those for S2. Billets of which diameters was 5in. were continuously cast by hot-top process using mother alloy containing 2% of Sc. The billets were hot extruded to make T-shape bar at 380 °C after preheating of 1h. The shape and dimension of extruded bar was shown in Fig. 1. The extrusion ratio was 38:1 and extrusion speed was 0.017 mm/s.
Table 1
Chemical compositions of tested Al alloys

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<tr>
<th></th>
<th>Zn</th>
<th>Mg</th>
<th>Cu</th>
<th>Mn</th>
<th>Cr</th>
<th>Si</th>
<th>Fe</th>
<th>Zr</th>
<th>Sc</th>
<th>Al</th>
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<tbody>
<tr>
<td>S1</td>
<td>7.9</td>
<td>2.0</td>
<td>1.9</td>
<td>–</td>
<td>0.03</td>
<td>–</td>
<td>–</td>
<td>0.11</td>
<td>0.1</td>
<td>Bal.</td>
</tr>
<tr>
<td>S2</td>
<td>5.2</td>
<td>2.0</td>
<td>0.3</td>
<td>0.3</td>
<td>0.03</td>
<td>–</td>
<td>0.11</td>
<td>0.1</td>
<td>0.1</td>
<td>Bal.</td>
</tr>
<tr>
<td>AA7075</td>
<td>5.6</td>
<td>2.5</td>
<td>1.6</td>
<td>–</td>
<td>0.23</td>
<td>0.4</td>
<td>0.5</td>
<td>–</td>
<td>–</td>
<td>Bal.</td>
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Table 2
Heat treatment condition

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<th>Solution treatment</th>
<th>Aging</th>
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<tr>
<td>S1</td>
<td>480 °C/2h</td>
<td>120 °C/24h</td>
</tr>
<tr>
<td>S2</td>
<td>465 °C/2h</td>
<td>120 °C/24h</td>
</tr>
<tr>
<td>AA7075</td>
<td>465 °C/2h</td>
<td>120 °C/24h</td>
</tr>
</tbody>
</table>

Then the extruded bar was heat-treated under the condition shown in Table 2. Solution treat temperature was 480 °C for S1 and S2, and 465 °C for AA7075, respectively. Aging temperature was 120 °C for whole specimens.

The microstructural evolutions during the hot extrusion and heat treatment was analyzed with the optical microscope except for the microstructural characterization of as-extruded bar. The analysis with optical microscope did not clearly show the microstructural details of as-extruded bar, therefore, in present study, electron back-scattered diffraction (EBSD) mapping was used for microstructural characterization. The EBSD mapping was carried out with INCA CRYSTAL from Oxford Instrument Ltd., which is installed in JEOL 6500F SEM. The hardness change according to the process condition was measured with the conventional method.

3. Results

3.1. Microstructural evolution

Microstructures of the billets are shown in Fig. 2. The billets are heated to 380 °C and held for 1 h followed by water quenching to investigate the initial microstructure before hot extrusion. The grain size is about 100 μm and fine precipitates are dispersed in grain interior for all specimens.

Microstructural evolutions after hot extrusion are shown in Fig. 3. An EBSD mapping was used for the analysis of the central region of extruded bar. The solid line in Fig. 3 indicates the boundary of which misorientation angle exceeds 5° and it can be thought that this solid line represents the configuration of grain boundary in extruded bar. As shown in Fig. 3, severely elongated grains are observed in all extruded bar which suggest that dynamic or static recovery be the main restoration process during or after the hot extrusion. It is thought, therefore, the recrystallization does not proceed at this central region.

Even though the initial grain size is almost same for all billets, Fig. 3 shows the average widths of elongated grains in extruded bar have somewhat different value for each of

![Fig. 1. The shape and dimension of extruded bar.](image1)

![Fig. 2. Optical micrographs of billet before hot extrusion.](image2)
the specimen. The average width measured from Fig. 3 is 2.0 μm for S1, 3.3 μm for S2 and 2.6 μm for AA7075, respectively. But it is unclear whether the metal flow characteristics during the extrusion can affect the difference in average width of elongated grains because un-uniform distribution of grain size is often observed in the initial billet, so it can affect uneven microstructure of extruded bar.

Microstructural evolutions after heat treatment of the extruded bar are shown in Figs. 4 and 5. One can notice from the overall microstructure in Fig. 4 that a characteristic microstructure is evolved around specimen surface for S1 and AA7075 during the heat treatment. Detailed microstructural observation (Fig. 7) indicates that the characteristic microstructure is evolved due to the abnormal grain growth. The thickness of abnormal grain growth region is about 150 μm for S1 and 70 μm for AA7075. On the other hand, the abnormal grain growth is rarely observed for S2.

Another significant difference in microstructural evolution during the heat treatment is also found between S1, AA7075 and S2. Fig. 5 shows the optical microstructure of extruded bar after solution treatment and aging. It can be seen that restoration process of S1, AA7075 and S2 during heat treatment proceeds to different way. The elongated grains observed in S1 and AA7075 indicate that only recovery still proceeds during the heat treatment, while fine equiaxed grains evolved in S2 suggest that recrystallization occur during the heat treatment.

From the trend of microstructural evolution during the heat treatment, which is summarized in Table 3, it seems that the abnormal grain growth and the restoration process have some interaction with each other. A possible mechanism will be discussed later in more detail.

Table 3

<table>
<thead>
<tr>
<th></th>
<th>Abnormal grain growth</th>
<th>Restoration process</th>
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<tbody>
<tr>
<td>S1</td>
<td>Observed</td>
<td>Recovery</td>
</tr>
<tr>
<td>S2</td>
<td>Not observed</td>
<td>Recrystallization</td>
</tr>
<tr>
<td>AA7075</td>
<td>Observed</td>
<td>Recovery</td>
</tr>
</tbody>
</table>
3.2. Hardness changes during hot extrusion and heat treatments

Fig. 6 shows the hardness changes according to the process. Two kinds of hardening behaviors can be observed during the heat treatment. For S1 and AA7075, the increase of hardness is more remarkable during the heat treatment than that during the hot extrusion. On the other hand, the hardness increase of S2 is rather slow during the heat treatment compared to that of S1 and AA7075, and so S2 has lower hardness than S1 and AA7075 after the heat treatment.

Slight increase of the hardness of AA7075 during the hot extrusion indicates that rapid recovery has occurred to release the stored energy of deformation. This sufficient recovery during hot extrusion can hinder the recrystallization during the heat treatment and that is why the main restoration process of AA7075 is still recovery during the heat treatment as shown in Fig. 5(c). For S2, the increase of hardness suggests that deformation energy is stored to some extent during the hot extrusion, which can lead to recrystallization during the heat treatment as shown in Fig. 5(b). It is not clear why recrystallization does not proceed during the heat treatment of S1, although similar level of hardness increase is observed during the hot extrusion. The increase of precipitates fraction due to higher content of Zn and Cu for S1, may be responsible because it can inhibit the recrystallization.

4. Discussion

The results of present study can be summarized as follows.
Fig. 6. Hardness change according to hot extrusion and heat treatment.

(i) For S1 and AA7075, the recovery proceeds during the hot extrusion and heat treatment. Abnormal grain growth is evolved around the surface of extruded bar and the hardness abruptly increases during the heat treatment.

(ii) For S2, the recovery proceeds during the hot extrusion but recrystallization takes place during the heat treatment. Abnormal grain growth is rarely observed and the hardness rather slowly increases during the heat treatment.

In this section, authors would like to discuss the influence of the restoration process on abnormal grain growth and hardening behaviors.

4.1. Abnormal grain growth during heat treatments

Fig. 7 shows the representative microstructures of extruded bar after the heat treatment. Compared to rather homogeneous microstructure of S2, the microstructure of S1 clearly shows the rapidly growing grains around the surface. Besides, the microstructure of S1 consists of abnormally growing grains and recovered elongated grains but that of S2 is mainly composed of equiaxed recrystallized grains. It is known that abnormal grain growth often occurs when some grains have growth advantage over other grains so they can preferentially grow consuming their neighbors [8]. The second-phase particles and texture are reported as typical origins for such growth advantage. For present study, to investigate the origin of growth advantage, it is worth paying attention to the microstructure of hot extruded bar in detail according to the position relative to the surface. Fig. 8 shows the microstructure of as-extruded bar of AA7075 at surface region, 1/4 thickness region and central region. One can notice a gradual change of microstructure from the center region to the surface. While the microstructure of the central region consists of recovered elongated grains, many equiaxed grains can be found at the surface region. This microstructural gradient suggests that localized recrystallization can occur at the surface even though the recovery primarily proceeds during the hot extrusion. The friction between die and extruded material tends to increase the temperature and effective strain at the surface and it is thought to be responsible for the evolution of this microstructural gradient. Once the recrystallized grains are evolved locally at the surface region during the hot extrusion, its growth kinetics during the heat treatment is greatly affected by its surrounding microstructure. That is, if the recrystallization no longer proceeds during the heat treatment, the recrystallized grains at surface region, which evolved during hot extrusion, become surrounded by recovered grains and so it will have great advantage for rapid growth. Consequently, the microstructure in Fig 7(a) will be evolved. On the other hand, if recrystallization proceeds during the heat treatment, the recrystallized grains evolved at surface will no longer have
Fig. 8. Microstructure of hot extruded AA7075 (EBSD mapping).

Fig. 9. Two kinds of hardening behavior during heat treatment.
growth advantage over their neighboring equiaxed recrystallized grains and so the rapid growth cannot be observed.

4.2. Hardening behaviors during heat treatments

Previously mentioned two hardening behaviors during the heat treatment are redrawn in Fig. 9. The difference in hardening behavior during the heat treatment means that the precipitation of second-phase particles occurs in different way. For present study, one of the possible origins, which can affect the precipitation behavior, might be the microstructural condition of matrix before precipitation.

For S1 and AA7075, which exhibit rapid increase of hardness, precipitation of second-phase particles proceeds in recovered matrix. Since dislocation substructure, which is usually evolved in recovered condition, is preferential nucleation site for the precipitation of the second-phase particles, it is thought that the precipitates distribution of S1 and AA7075 becomes quite fine and uniform, which can result in the remarkable increase of hardness during the heat treatment. On the contrary, since the recrystallization is thought to precede the precipitation, or at least the recrystallization and precipitation simultaneously proceed for S2, the second-phase particles in S2 will not have enough chances to precipitate as fine and uniform as those in S1 and AA7075 because of the loss of precipitation site. The different hardening behavior of S1 and S2, which contains Sc, is consistent with the results of Jones and Humphreys [7]. They pointed out that Sc content in Al alloy plays an important role in precipitation behavior of second-phase particles. According to their results, in 0.25% Sc alloy, homogeneous precipitation occurred during annealing, therefore, any significant effect of a deformed microstructure on precipitation behavior was not observed. However, in 0.12% Sc alloy, the precipitation appeared to be heterogeneous and hence fine precipitates were preferentially formed at dislocation and boundary. From those results, precipitation behavior of 0.1% Sc alloy in present study is expected to be heterogeneous. Accordingly, for S1 and S2, different microstructural condition of matrix before precipitation will lead to different precipitation behavior of second-phase particle, and further different hardening behaviors during the heat treatment.

5. Conclusion

The microstructural evolution during hot extrusion and heat treatment was investigated for two kind of Al–Zn–Mg–Cu–(Sc) alloy and AA7075. The microstructure of as-extruded bar consists of recovered elongated grains for all alloys. The main restoration processes in recovery during post heat treatment for S1 and AA7075, but recrystallization proceeds during post heat treatment for S2. The restoration process has close relationship with the abnormal grain growth around the surface of extruded bar and hardening behavior during the heat treatment. As long as the recovered elongated grains remain, the locally recrystallized grains, which are evolved around the surface during the hot extrusion, can grow rapidly consuming the recovered elongated grains during post heat treatment. Besides, the substructure of recovered state is thought to be responsible for remarkable increase of hardness during the heat treatment by offering heterogeneous nucleation site for fine and uniform precipitation of second-phase particle.

Acknowledgements

This work is financially supported by R&D program which is funded by Ministry of Commerce, Industry and Energy. Authors would like to thank Hae-Kyung Lee for her experimental assistance.

References