Quantification of Precipitated Phases in 6063 Aluminium Billet by Image Analysis for Improvement of Homogenization Condition.

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Introduction
In producing Aluminium extrusions, constant and reliable properties of the extrusion billet are most especially when more complex die designs are employed to produce thin section complex extrusion profiles. In Thailand, the most common alloy grade used for extrusion is AA6063. Control of AA6063 billet properties is normally ensured by close compositional control and by correct solution treatment of the cast billet to ensure solution of precipitated phases. Control of chemical composition is well established at all billet casters in Thailand but homogenization by solution heat treatment is less well understood and performed in practice. Qualification of acceptable microstructures to avoid later defects during extrusion due to inadequate billet properties is quite often overlooked by billet casters. The paper presents some results from a project to determine the relationships between solution heat treatment temperature, cooling rate and precipitated phases in the homogenized microstructures before extrusion.

AA6063 billet
A medium-strength-aluminium alloy grade 6063 is the normal preferred extrusion alloy for architectural and general applications in Thailand, due to its good surface finish, high corrosion resistance and ease of anodizing. The general chemical composition for AA6063 is shown in Table 1.

Table 1. Chemical composition (% by weight) of AA6063 in this project compared with standard AA6063 (architectural use grade) AA6063A and AA6061 (higher strength structural use grade).

<table>
<thead>
<tr>
<th></th>
<th>6063 (project)</th>
<th>6063 (standard)</th>
<th>6063A</th>
<th>6061</th>
</tr>
</thead>
<tbody>
<tr>
<td>Si</td>
<td>0.364</td>
<td>0.2-0.6</td>
<td>0.3-0.6</td>
<td>0.4-0.8</td>
</tr>
<tr>
<td>Fe</td>
<td>0.218</td>
<td>0.35 max</td>
<td>0.15-0.35</td>
<td>0.7 max</td>
</tr>
<tr>
<td>Cu</td>
<td>0.019</td>
<td>0.1 max</td>
<td>0.1 max</td>
<td>0.15-0.4</td>
</tr>
<tr>
<td>Mn</td>
<td>0.014</td>
<td>0.1 max</td>
<td>0.15 max</td>
<td>0.15 max</td>
</tr>
<tr>
<td>Mg</td>
<td>0.481</td>
<td>0.45-0.9</td>
<td>0.6-0.9</td>
<td>0.8-1.2</td>
</tr>
<tr>
<td>Zn</td>
<td>0.024</td>
<td>0.1 max</td>
<td>0.15 max</td>
<td>0.25 max</td>
</tr>
<tr>
<td>Ti</td>
<td>0.008</td>
<td>0.1 max</td>
<td>0.1 max</td>
<td>0.15 max</td>
</tr>
<tr>
<td>Cr</td>
<td>0.006</td>
<td>0.1 max</td>
<td>0.05 max</td>
<td>0.04-0.35</td>
</tr>
<tr>
<td>Al</td>
<td>98.857</td>
<td>Balance</td>
<td>Balance</td>
<td>Balance</td>
</tr>
</tbody>
</table>

Mg-Si precipitated phase
Typically, Mg2Si (precipitated Mg-Si phase) is mostly found as small particles in the interdendritic areas of AA6063 billet along with other precipitated phases (Al-Fe-Si, Al-Mn-Fe-Si, and Cu-Al) in as-cast microstructures. These small particles effect to reduce the forming ability of billet during extrusion, hence suitable heat treatment is applied dissolve these precipitates to control billet properties before extrusion. Solution treatment or homogenization is involves heating the billet to temperatures of about 510 to 600 degree Celsius with appropriate soaking time and subsequent cooling rate.

Solution treatment
During solidification of a cast billet, primary Aluminium solid solution grows in a dendritic manner to produce a mixture of columnar and equiaxed grains. During subsequent cooling through the solidus line at around 510 °C, magnesium and silicon come out of solution in the solute enriched interdendritic and grain boundary areas to form magnesium-silicon (Mg2Si) precipitates. In AA6063, these Mg2Si and any other precipitates (or non-equilibrium eutectic constituents) must be dissolved by suitable solution treatment as indicated by the phase diagram in Figure 1.

Solution treatment requires the billet to be reheated to a temperature above 520 °C but below the solidus line to avoid incipient melting. Solution treatment also homogenizes the magnesium and silicon contents in the matrix since it allows diffusion to remove the concentration gradients, caused by microsegregation during solidification. Cooling from the solution treatment temperature must be sufficiently fast to prevent re-precipitation since slow cooling will allow Mg2Si to form as relatively coarse incoherent non-strengthening particles (Figure 2). Quenching however will retain the magnesium and silicon atoms in the matrix giving a supersaturated solid solution at ambient temperature with acceptable properties for forming by extrusion.

In other AA6XXX alloy grades with such high iron contents, heat treatment will also transform hard eutectic beta Al-Fe-Si phase to the globular alpha Al-Fe-Si
To study the relationship between temperature, cooling rate and dissolution of \( \text{Mg}_2\text{Si} \), the experiment is designed on the assumption that:

- High billet cooling rate from homogenization temperature reduces \( \text{Mg}_2\text{Si} \) particle size.
- At high heating rates, preheating before extrusion and frictional heating during extrusion can allow complete dissolution of \( \text{Mg}_2\text{Si} \) phase particles before material exits the die.

**Experiment and Results**

To study how solution treatment temperature affects the quantity of retained precipitated phases, the AA6063 aluminium samples were prepared as 25 mm. side cubes. Solution treatment temperatures of 520 to 580 °C were used followed by quenching in water or by forced air. Subsequent microstructures were examined and the amount of precipitated phases determined by an image analysis technique.

Micrographs of the sample analyzed by image analysis are shown in Figure 4 and the plot of the relationship between percentage of retained precipitated phase and solution treating temperature is shown in Figure 5.
Figure 4. Micrograph of aluminium billet after solution treatment image analyzed for precipitated phases quantification. As etched by 10%NaOH (left) and color-contrasted to quantify area (right).

Figure 5. Retained precipitated phase after solution treated by different temperature.

Figure 6. Effect of solution treatment on dissolving precipitated phases comparing between micrographs of (a) before solution treatment and (b) after solution treatment, as etched by 10%NaOH.

Conclusion

The quantification of interdendritic precipitated phases in 6063 aluminium billet by image analysis is useful in predicting the forming ability of AA6063 billet with a well controlled chemical composition. The lowest quantity of precipitated phases is recommended for a more complex extrusion die, while a medium quantity may be considered for general die design, depending on extrusion conditions such as billet diameter, extrusion rate and preheat treating temperature. Most of precipitated phases are Mg-Si and billets can be adequately solution treated by using the correct condition in a batch solution treating furnace. In the case of the precipitated phases that contain iron and copper, their solution must be also considered. However, only solution treatment cannot dissolve all precipitated phases, Mg-Si phase can be small enough to dissolve during the reheating and extrusion operation by solution treatment while Al-Fe-Si can be treated to a better form for extrusion as well. The comparison between cooling by water and air shows no significant differences. The heated billet may be considered for cooling by forced air in case of billet distortion is a concern.

Reference