Impact of Scandium and Zirconium on extrudability, microstructure and hardness of a binary Al-Cu alloy

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Abstract. Scandium additions are known to result in a significant enhancement in the strength of Aluminium alloys. However, the use of Scandium has so far been limited due to the high price and low availability of Scandium. This is on the verge to change with the discovery of the largest Scandium source in the world in Australia and the use of Clean TeQ innovative extraction technology which will bring the Scandium price down and stable in the foreseeable future. The use of Scandium will then become viable, especially in advanced aluminium alloys for the aerospace sector. Optimal strengthening is obtained when Scandium and Zirconium are added to Aluminium. One of the major group of wrought products used in aerospace is as extruded profiles. The impact of Scandium on extruded aluminium products is currently unknown. Most aerospace aluminium alloys (mainly 2xxx and 7xxx grades) have a high Cu content and Zr is commonly used for texture control. Here, we explore the impact of alloying an Al-4wt%Cu model alloy with Sc and Zr. The nominal compositions of the alloys selected for this study are: 1) Al-4wt%Cu, 2) Al-4wt%Cu-0.15wt%Zr and 3) Al-4wt%Cu-0.15wt%Zr-0.1wt%Sc. Scandium is found to have no impact on extrudability. The hardness is found to be almost doubled in the alloy containing both Scandium and Zirconium as compared to the binary alloy. This increment is due to a significant refinement of the grains and precipitates from the scandium additions.
Introduction

Aluminium alloys have been the prime choice of materials for use in airplanes since the start of aviation [1, 2]. Over the past 100 years, intense research and development has allowed the development of Aluminium alloys with superior specific strength (strength/weight) [3, 4]. Nowadays, the main alloys used in aerospace aircrafts are from the 2xxx and 7xxx series and most commonly contain high amounts of Cu. The positive impact of Scandium on Aluminium alloys has been studied in a wide range of alloys [5, 6]. Scandium results in significant improvement in strength with minimum effect on the corrosion resistance and toughness of the alloy. Scandium was found to be the most effective when alloyed together with Zr. In this latter case, the strengthening mainly comes from a refinement of the grains and formation of a fine dispersion of spherical Al₃(Sc,Zr) precipitates [7, 8, 9, 10, 11, 12, 13].

The impact of Sc and Zr additions on the extrudability of Cu containing Aluminium alloys has not yet been studied and this is the subject of the present study. In this study, we use an Al-4wt%Cu model alloy relevant to the modern aerospace alloys. Three compositions were used for the present work as follows: 1) Al-4wt%Cu, 2) Al-4wt%Cu-0.15wt%Zr and 3) Al-4wt%Cu-0.15wt%Zr-0.1wt%Sc. The breakthrough pressure during the extrusion process was monitored in order to assess extrudability. The mechanical properties of the three alloys in the peak aged condition were assessed with Vickers hardness measurements and the microstructures of the peak aged samples were investigated with scanning electron microscopy.

We find that the extrudability is negatively affected by Zr additions but that Sc additions reverses this effect. As such the alloy containing both Sc and Zr had an extrudability comparable to the binary Al-Cu alloy. With the minor Zr and Sc additions, the Vickers hardness is found to nearly double in the peak aged condition. This exceptional increase in hardness is explained by a grain refinement of one order of magnitude and the formation of smaller Cu-rich precipitates.
Casting and extrusion

The alloys selected for this study are listed in Table 1.

Table 1: Nominal compositions of the three alloys under study (in wt%).

<table>
<thead>
<tr>
<th></th>
<th>Al</th>
<th>Cu</th>
<th>Sc</th>
<th>Zr</th>
<th>Fe</th>
<th>Ti</th>
</tr>
</thead>
<tbody>
<tr>
<td>Alloy 1</td>
<td>Bal</td>
<td>4</td>
<td>0</td>
<td>0</td>
<td>0.07</td>
<td>0.005</td>
</tr>
<tr>
<td>Alloy 2</td>
<td>Bal</td>
<td>4</td>
<td>0</td>
<td>0.15</td>
<td>0.07</td>
<td>0.005</td>
</tr>
<tr>
<td>Alloy 3</td>
<td>Bal</td>
<td>4</td>
<td>0.1</td>
<td>0.15</td>
<td>0.07</td>
<td>0.005</td>
</tr>
</tbody>
</table>

The alloys were prepared from Master Alloys to ensure for the targeted compositions to be accurately reached. A small induction furnace was used to melt the metals and casting was conducted in a cylindrical steel mould that comprise a large steel base to ensure directional solidification. A typical cast ingot is shown in Fig. 1. Extrusion billets of 30 mm diameter and 20 mm long were wire cut from the cast ingot.

Figure 1: Photograph of a direct chill cast Al alloy. Length (l) = ~200mm, diameter (d) = 80mm.
Figure 2: (a) Cylindrical rods wire-cut from cast ingots (b) Dimensions of the billet to be extruded.

The homogenization schedule used in this study cannot be revealed for proprietary reasons. After homogenization, extrusion was conducted at 435 °C at a cross head velocity of 10 mm/s. Extrusion was performed on at least four billets per composition to ensure reproducibility of the extrusion pressure measurements. The extruded profiles were essentially defect free, Fig. 3. The extruded billets were then solution treated for 1 h at 500 °C and aged for 10 h at 160 °C.

Figure 3: Typical defect free extruded samples of different compositions. The total length of the extruded product is about 450 mm.

During extrusion, the applied pressure on the ram was monitored as a function of displacement, Fig. 4. The breakthrough pressure is defined as the highest pressure at which extrusion starts, Fig. 5.
Figure 4: Extrusion pressure evolution as a function of displacement for four specimens of alloy 3. The similarity between the four curves demonstrates excellent reproducibility of the measurement.

Figure 5: Breakthrough extrusion pressure as a function of alloy composition
Microstructure

The characterization of the microstructure was performed in a Jeol JSM 7800F scanning electron microscope (SEM) at 20 kV. Secondary electron imaging was used to image the peak aged samples of the three compositions under study, Fig. 4. A significant grain size reduction was observed from Alloy 1 to Alloy 3. In the absence of dispersoids former, Alloy 1 displays a fully recrystallized microstructure with large equiaxed grains. In Alloy 2 and 3, the samples retained the deformation texture thanks to the grain boundary pinning from the Zr and Sc dispersoids. Alloy 2 was observed to be partially recrystallized and alloy 3 was fully unrecrystallised with the smallest grain size.

The presence of the Cu-rich $\theta$ precipitates was then investigated by working at higher magnification, Fig. 7. A refinement of the precipitates was observed when going from alloy 1 to alloy 3. For the binary alloy, the precipitates were clearly visible with sizes ranging from 500 nm to 1 µm. For alloy 2, the precipitates were observed to be slightly smaller between 200 nm and 500 nm. Finally for alloy 3, only the coarser precipitates could be observed and they had size below 100 nm. The size of the latter precipitates is beyond the scope of SEM and requires a more detailed transmission electron microscopy (TEM) investigation, which is the subject of a separate paper. The impact of Sc and Zr on the nucleation and growth of $\theta'$ precipitates will be explored in a separate paper using combined TEM and atom probe tomography (APT).

Figure 6: Example of SEM micrographs of (a) the Al-4wt%Cu alloy, (b) the Al-4wt%Cu-0.15wt%Zr alloy and (c) the Al-4wt%Cu-0.15wt%Zr-0.1wt%Sc alloy. Typical grain size is highlighted in red, the grain size is observed to be reduced by an order of magnitude from addition of Zr and Sc. The image were taken in the same direction with respect to the extrusion direction, as per the white arrow on (b).
Figure 7: Example of high magnification SEM micrographs of (a) the Al-4wt%Cu alloy, (b) the Al-4wt%Cu-0.15wt%Zr alloy and (c) the Al-4wt%Cu-0.15wt%Zr-0.1wt%Sc alloy. Typical $\theta$ precipitate size is highlighted in red. The Sc and Zr content is found to have a clear effect on precipitate size. The image were taken in the same direction with respect to the extrusion direction, as per the white arrow on (b).

Hardness

Vickers micro-hardness measurements were conducted on the peak aged condition for the three alloys using a FM-700 Micro-hardness tester with a 500 g load. Twenty measurements were performed on each sample to ensure to get a reliable average hardness and standard deviation.

The average hardness and related standard deviation are reported in Table 2.

The hardness increase from alloy 1 to alloy 2 comes from the refinement of the grains observed previously. The additional hardness increase observed in alloy 3 comes from the refinement of the $\theta'$ precipitates and precipitation strengthening from the Al3(Sc,Zr) dispersoids. The importance of the different strengthening mechanisms will be explored in a separate paper.

Table 2: Nominal compositions of the three alloys under study (in wt%).

<table>
<thead>
<tr>
<th></th>
<th>Hv</th>
<th>$\sigma$</th>
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<tbody>
<tr>
<td>Alloy 1</td>
<td>66</td>
<td>10</td>
</tr>
<tr>
<td>Alloy 2</td>
<td>90</td>
<td>5</td>
</tr>
<tr>
<td>Alloy 3</td>
<td>123</td>
<td>3</td>
</tr>
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</table>
Conclusions

In the present work, the effect of minor additions of Zr and Sc on extrudability, microstructure and mechanical properties of a Al-4wt%Cu model alloy was investigated. Here are the main findings from this study:

- The extrudability is unaffected by combined additions of low amounts of Sc and Zr. In the case of adding Zr only, the extrudability was negatively affected.
- The addition of both Sc and Zr was found to significantly refine the grain size from ~40 µm to about ~2 µm.
- The addition of both Sc and Zr is also found to significantly refine the θ’ precipitates from ~1 µm in alloy 1 to <100 nm in alloy 3.
- The refinement of grain and precipitate results in additional strengthening contributions. The hardness was almost doubled by adding 0.15wt%Zr and 0.1wt%Sc to an Al-4wt%Cu alloy.

References


