The effect of cold work on the precipitation kinetics of AA6111 aluminum

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In the automotive industry, the drive to meet Corporate Average Fuel Efficiency (CAFE) standards has resulted in the need to reduce vehicular weight in new automobile designs. One effective method has been to use lighter materials such as aluminum alloys in outer body panels. Heat treatable AA6111 is one of the materials being employed in outer body panels due to its unique combination of formability, paint bake strengthening and superior corrosion resistance characteristics. For a formed and painted automobile panel, the final mechanical properties involve a combination of strength components arising from cold work, strain aging, precipitation and recovery. The challenge is to find a method of quantifying each of these strength components.

In this study, the effect of cold work on the precipitation kinetics of AA6111 has been evaluated, by means of tensile testing, differential scanning calorimetry (DSC), and transmission electron microscopy (TEM). The results show a considerable improvement in yield and tensile strength with increasing level of cold work. DSC showed acceleration in the precipitation kinetics of the alloy with increasing level of cold work. The Avrami-Johnson-Mehl approach, further developed by Mittemeijer and co workers was employed to determine the activation energies for dissolution of GPI zones and β″ formation. The activation energy of dissolution of GPI zones is observed to increase with increasing level of cold work while that for formation of β″ decreases with increasing level of cold work. As expected, TEM showed strong interaction of strengthening precipitates with dislocations. The density of dislocation tangles is shown to increase with increasing degree of cold work. © 2004 Kluwer Academic Publishers

1. Introduction

In the automobile industry, the use of aluminum alloys has increased over the years as manufacturers strive to design lighter vehicles as part of an overall goal to meet the North American Corporate Average Fuel Efficiency (C.A.F.E) Standards. These standards seek to improve fuel efficiencies and reduce vehicle emissions. Heat treatable AA6111 has emerged as one of the most prominent materials employed in outer body panels of cars and light trucks due to its unique combination of formability and paint bake strengthening characteristics.

There has always been the desire to have some way of estimating the yield strength of a part following forming and/or heat treatment operations. This is crucial since the dent resistance of a part is proportional to its yield strength and therefore high yield strength may be seen as beneficial. However, during stamping, high yield strength alloys tend to suffer spring back effect and inferior formability. For a formed and painted automobile panel, the yield strength involves combining strength components arising from cold work, strain aging, precipitation, and recovery. The conflicting demands of low yield strength alloy and high yield strength components in the 6xxx series alloys containing Cu are partially addressed through the aging response promoted through the automotive paint bake cycle. Unfortunately, only a small fraction of the full aging potential inherent in the alloy is exploited due to the relatively low temperatures and short duration of most commercial paint bake cycles. Typically, the paint bake curing cycle in the automobile manufacturing process involves: 10–20 min electro-coat (E-coat) curing at 170–185°C, 15–20 min primer curing at 160–170°C, and 15–25 min top/clear coat process at 130–150°C [1, 2]. The need for rapid strengthening response in today’s finishing lines has led researchers to impose preaging treatments as well as other thermo-mechanical histories on 6xxx series aluminum alloys with the view to improving the strengthening characteristics as well as kinetics of these alloys [3–14].

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In the present investigation, the effect of cold work on the precipitation kinetics of AA6111 is evaluated with a view to establishing its contribution to enhancing the paint bake response of the alloy as well as its aging kinetics, using methods that are compatible with industrial thermal practices for optimum results. The evaluation methods employed in the present study are tensile testing, differential scanning calorimetry (DSC), and transmission electron microscopy (TEM).

2. Experimental procedure

The composition limits of the material used in the present study, as received from Alcan International Limited, Kingston, Ontario, is presented in Table I. The material, supplied in the rolled state, was manufactured by a special technique, the details of which are reported in [15].

<table>
<thead>
<tr>
<th>Metal</th>
<th>Cu</th>
<th>Fe</th>
<th>Mg</th>
<th>Mn</th>
<th>Si</th>
<th>Ti</th>
<th>Al</th>
</tr>
</thead>
<tbody>
<tr>
<td>Maximum</td>
<td>0.9</td>
<td>0.40</td>
<td>1.0</td>
<td>0.45</td>
<td>1.1</td>
<td>0.10</td>
<td>Balance</td>
</tr>
<tr>
<td>Minimum</td>
<td>0.5</td>
<td>0</td>
<td>0.5</td>
<td>0.10</td>
<td>0.6</td>
<td>0</td>
<td>0</td>
</tr>
</tbody>
</table>

2.1. Tensile testing

The tensile test specimens were machined from a 1 mm thick stock sheet according to the ASTM E-8 standard, with a gauge length of 50 and 12 mm gauge width. These were then solution heat treated at 560 ± 5°C for 30 min in an air furnace and quenched in 20°C water. Some of the as-quenched samples were given 2 or 5% cold work by stretching. All the specimens were then artificially aged at 180°C for various lengths of time. Subsequently, tensile tests were performed at room temperature using an Instron® screw-driven machine at a constant cross-head speed resulting in an initial strain rate of 0.025 min⁻¹. The yield strength, determined as the 0.2% strain offset, as well as the tensile strength were obtained from the resulting stress-strain plots.

2.2. Differential scanning calorimetry (DSC)

DSC analyses of samples subjected to various levels of cold work (0, 2, and 5%) by stretching were carried out using a temperature-modulated DSC 2910 system (MDSCTM, TA Instrument Inc., USA), incorporating a refrigerated cooling system. The instrument was calibrated for enthalpy and temperature using a high purity elemental indium standard. Three DSC runs were conducted for each level of cold work in order to ensure reproducibility. All DSC runs began at 30°C and ended at 380°C at constant heating rates of 5, 10, 15 and 20°C min⁻¹. In order to correct for the additional heat flow arising from the difference in weight of the sample pan and the reference pan, and also to compensate for any asymmetry in the measuring system, a preliminary experiment was performed with commercially pure aluminum as blank. Thus the heat flow obtained was the difference between the measured and the blank values.

2.3. Transmission electron microscopy (TEM)

TEM samples of AA6111 aluminum at the simulated paint bake cycle of 180°C for 30 min, peak aged for 10 h at 180°C, and simulated DSC runs conducted at 10°C min⁻¹ to the GPI zone dissolution and the first largest exothermic peaks were examined in a Hitachi 2200 FE STEM at 200 kV. Thin foils were prepared by mechanical grinding, followed by an electro-polishing technique in a 30% HNO₃–70% methanol bath at a temperature between −30 to −20°C.

3. Results and discussion

3.1. Tensile results

Figs 1 and 2 show the variation of yield and tensile strengths as a function of aging time at 180°C for various levels of cold work. It can be seen in both graphs that as the aging time increases at each level of cold work, the yield and tensile strengths increase up to the peak value after which they decrease with further aging time. Thus, there is a significant increase in strength with increasing level of cold work. This can be attributed to the increase in dislocation density, due to cold work, piling up to form tangles and hence increasing the strength of the material. The time to reach peak yield strength