Effect of Pre-Deformation on Mechanical Response of an Artificially Aged Al-Mg-Si Alloy*

Michal Kolar¹, Ketill Olav Pedersen², Sverre Gulbrandsen-Dahl¹,³, Katharina Teichmann¹ and Knut Marthinsen¹

¹Norwegian University of Science and Technology (NTNU), Department of Materials Science and Engineering, NO-7491 Trondheim, Norway
²SINTEF Materials and Chemistry, NO-7034, Trondheim, Norway
³SINTEF Raufoss Manufacturing AS, NO-2831 Raufoss, Norway

In order to investigate the effect of deformation on the artificial aging response of an Al-Mg-Si alloy, a series of tensile tests have been designed and carried out on the aluminium alloy AA6060. Extruded and solution heat treated specimens were pre-deformed 0%, 2%, 5%, and 10% (engineering strain) and subsequently artificially aged at three different aging temperatures (150, 175 and 190 °C) for three different times (10, 100 and 300 min). It was observed that the amount of pre-deformation is determining for the final mechanical properties of the short-time artificially aged AA6060 compared to the material aged for longer times when the mechanical properties level out regardless of the introduced pre-deformation. In addition to the hardness measurements and tensile tests, transmission electron microscopy (TEM) and differential scanning calorimetry (DSC) have been used to characterize dislocation evolution, microstructure and precipitation state for various combinations of pre-deformation and aging time. [doi:10.2320/matertrans.L-MZ201127]

(Received October 27, 2010; Accepted February 17, 2011; Published June 25, 2011)

Keywords: aluminium magnesium silicon alloy, artificial aging, pre-deformation, differential scanning calorimetry, transmission electron microscopy, mechanical properties

1. Introduction

The 6000 series (i.e. Al-Mg-Si alloys) of wrought aluminium alloys is widely used in the automotive, food and building industries, e.g. for a variety of structural parts in cars, in railway carriages, in pipelines and for construction purposes. 6000 series alloys are chosen whenever heat-treatable alloys combining medium strength with good corrosion resistance, reasonable weldability and moderate cost are required. Many aluminium products made of 6000 series aluminium alloys are first extruded, then formed, and finally artificially aged, i.e. heat treated at elevated temperatures, to obtain the desired mechanical strength through precipitation hardening. Even without forming, extruded profiles commonly have to be stretched to straighten out a certain degree of buckling and bending of the profiles as they leave the extrusion die. In both cases precipitation takes place in a material which is deformed to various degrees and which may strongly affect the precipitation behaviour and associated aging response. Precipitation and aging behaviour in Al-Mg-Si alloys have been extensively studied and characterized in detail by many groups over the last 10–15 years.¹⁻⁵ However, the complex interplay between deformation, precipitation and aging response is less characterized and understood.⁶⁻⁻⁸ To get a better understanding and quantitative description of the aging behaviour in deformed materials as a function of alloy chemistry and processing conditions a careful and detailed study on this topic is in progress by the present authors.

From a wide variety of the 6000 series of aluminium alloys a commercial AA6060 has been chosen to study the effect of deformation on the artificial aging response. It has been reported⁹⁻¹⁰ that the increase in yield stress due to natural aging is decreased when the pre-deformation is introduced and also that the initial work hardening rate increases when the AA6060 is pre-deformed and naturally aged for long times (e.g. 1 week) compared to a material aged only for short times (e.g. 10 min). These results have served as a basis and reference for further investigations of the combinations of deformation and artificial aging of the AA6060 which are reported in this paper. AA6060 was initially cast, homogenized and extruded according to standard industrial practice. The extruded material was then subjected to a solution heat treatment, deformed to various degrees and subsequently artificially aged. The aging behaviour and associated mechanical properties have been characterized in terms of Vickers hardness and tensile testing (from which the work hardening behaviour has been derived), and the material in different conditions has been characterized with respect to microstructure and precipitation state by a several methods, incl. Differential Scanning Calorimetry (DSC), Scanning Electron Microscopy (SEM) and Transmission Electron Microscopy (TEM), to correlate microstructure and mechanical properties.

2. Experimental

An AA6060 aluminium alloy was produced at Hydro, Norway in the following manner. Molten aluminium tapped from the pots of the electrolysis plant was transported to the cast house where it was alloyed in holding furnaces by the addition of Mg, Si and Mn (according to the alloy specification) and cleaned of oxides and gases. The chemical composition of the investigated alloy is given in Table 1. The melt was poured from the furnace via melt treatment system

---

*The Paper Contains Partial Overlap with the ICAA12 Proceedings by USB under the Permission of the Editorial Committee.
to a DC (direct chill) casting table. Grain refiner (TiB) was used to secure a small and even grain size in the cast metal. Logs with a diameter of 203 mm and a length of 7 m were cast from the melt and subsequently split and machined to billets, 95 mm in diameter and 200 mm in length.

In order to obtain a uniform homogenous composition distribution after casting and machining, i.e. removal of any segregations of Mg and Si and modify the distribution of Fe-containing particles in the metal, the billets were homogenized at 585°C (+/−10°C) for 2 h (+/−15 min) in a continuous homogenisation furnace. The billets were extruded at a ram speed of 3 mm/s into 20 mm rods using an 800 ton instrumented laboratory press at SINTEF in Trondheim. Prior to extrusion the billets were heated to 545°C in an induction furnace, cooled by forced air to 430°C and stabilized at this temperature. The profiles were extruded into a water tank and were thus rapidly quenched to room temperature (RT). This procedure of pre-heating of the extrusion billets is referred to as “overheating” used in order to ensure that all (Mg, Si) particles are dissolved in the profile as it leaves the extrusion die. The 1 meter long section at the back end of an extruded length was cut off in order to avoid a coarse, recrystallized microstructure which tends to form around the periphery of sections that are heavily deformed as the alloy flows through and past the edges of the die.

Hardness measurements were carried out using a Matsuzawa DVK-1S Vickers hardness tester with a load of 5 kg, loading speed of 100 μm/s and 15 s holding time. The reported hardness measurements are based on an average of minimum five hardness indentations on each of the tested samples. For the material with no pre-deformation samples for hardness measurements were cut out of the extruded rod. For the pre-deformed material samples were cut out the deformed tensile test specimen.

Tensile tests were performed at RT after different aging times and different amounts of pre-deformation using a hydraulic MTS 810 testing machine at a constant crosshead speed of 2 mm/min, corresponding to an initial strain rate of 1.3 × 10⁻³ s⁻¹. For each combination of pre-deformation and aging time, a minimum of three parallel round specimens with a gauge diameter of 6 mm were tested in order to verify the experimental results. The tensile direction was parallel to the extrusion direction. For all tests, strain was measured in the reduced section of the specimens using a longitudinal clip-on extensometer with a 25 mm gauge length. The instantaneous work-hardening rate, i.e. \( \frac{d\sigma}{d\varepsilon} \) vs. strain, \( \varepsilon \), was determined by differentiating the true stress-strain curve.

Specimens for transmission electron microscope (TEM) analyses were prepared by cutting 1.5 mm discs normal to the tensile specimens and grinding the specimens to thin sheets prior to conventional electro-polishing with a Tenupol-3 machine. An electrolyte consisting of 1/3 HNO₃ and 2/3 methanol was used to thin the specimens. The TEM investigations were carried out using a Philips CM30 operated at 150 kV.

Differential scanning calorimetry (DSC) tests were carried out in the heat-flux Setaram Sensys TG-DSC instrument under He atmosphere with high purity aluminium used as a reference. Samples for DSC analyses were prepared by cutting 12 mm long cylinders out of the middle part of the tensile specimens. The heating rate was 20°C/min with the temperature range from 25°C to 450°C. The heat effects associated with transformation reactions were then obtained by subtracting a super purity Al (of equal mass as the investigated sample) baseline run from a given heat flow curve. The DSC curves obtained from the experiments were highly reproducible.

### 3. Results and Discussion

The hardness evolution of samples with different pre-deformations (2, 5 and 10%) annealed at 150°C, 175°C, and 190°C, respectively, are presented in Fig. 1. At 150°C we observe almost the same peak hardness for the pre-deformed samples, irrespective of amount of pre-deformation (Fig. 1(a)). The peak hardness is reached after approximately the same aging time of 1000 min (~16h). The initial higher hardness, up to 30 min of artificial aging, of the pre-deformed material is due to the introduced pre-deformation and thereby caused only by a deformation strength addition. Therefore we can assign nearly the same incubation time of 30 min for artificial aging as for in case of natural aging, lower dashed lines in Figs. 1(a)–1(c), and included here as a reference. For the material with no pre-deformation we observe lower peak hardness reached after longer aging times. The hardness evolution follows the values reached in naturally aged material up to 100 min.

At 175°C, as for at the age hardening at 150°C, the same peak hardness (although somewhat lower than for 150°C aging, compare 90 HV to 80 HV) is reached for all the pre-deformed samples, however, the aging time to peak hardness is significantly shorter (~300 min) (Fig. 1(b)). Moreover, a small shift to shorter times to peak hardness with increasing pre-deformation may also be noticed. No obvious incubation time is observed at this temperature as the hardness slightly increases even after short aging times. After the peak hardness is reached, hardness is gradually decreasing with prolonged aging times. For the material with no pre-deformation the hardness evolution follows the values reached in naturally aged material up to 30 min. After that, hardness increases and after an aging time of 500 min reaches the peak hardness which is constant up to ~2500 min. An interesting observation at this aging temperature is that a pre-deformation even as small as 0.5% is sufficient to give a significant strength addition, as compared to no pre-deformation, and almost the same peak hardness as for the samples with higher pre-deformations (2–10%) is reached.

During artificial aging at 190°C we do not observe any incubation time for any of the materials (Fig. 1(c)). For this aging temperature the peak hardness is clearly dependent on the introduced pre-deformation in the sense that a larger pre-deformation leads to higher peak hardness, which is also reached after gradually shorter aging times with increasing

<table>
<thead>
<tr>
<th>Table 1</th>
<th>Chemical composition of the alloy investigated (mass%).</th>
</tr>
</thead>
<tbody>
<tr>
<td>Alloy</td>
<td>Al</td>
</tr>
<tr>
<td>AA6060</td>
<td>0.47</td>
</tr>
</tbody>
</table>

Effect of Pre-Deformation on Mechanical Response of an Artificially Aged Al-Mg-Si Alloy 1357
pre-deformation (compare 76 HV after 120 min for 10% pre-deformed sample to 66 HV after 360 min for sample with no pre-deformation).

Turning to the stress-strain behaviour, the true stress-strain curves for samples with different amount of introduced pre-deformation, i.e. 2%, 5% and 10%, and artificially aged at 190°C for different times (10, 100 and 300 min) are shown in Figs. 2 and 3. Generally, the introduction of pre-deformation and longer artificial aging time increases both yield strength and ultimate tensile strength and slightly decreases uniform strain (Fig. 2) until a maximum stress is reached and the strength decreases with aging time. After artificial aging of 300 min all the curves level out regardless of the pre-deformation. Precipitation is then the leading strengthening mechanism. Pronounced serrated yielding is observed in the stress-strain curves after 10 min of artificial aging; it is almost negligible but still present after 100 min of aging and finally completely diminishes after 300 min of aging. Serrated yielding is a manifest of the Portevin-Le Chatelier\(^{12}\) effect and follows from alternating pinning and unpinning of moving dislocations during plastic deformation by solute atoms. The gradual disappearance of the serrations with

---

Fig. 1 Vickers hardness response with a load of 5 kg of AA6060 pre-deformed to different levels and artificially aged at (a) 150°C, (b) 175°C and (c) 190°C.

Fig. 2 True stress-strain curves for AA6060 with different amount of introduced pre-deformation (a) 2%, (b) 5% and (c) 10% artificially aged at 190°C for different times (10, 100 and 300 min).
increasing aging time indicates a corresponding reduction of the solute level of alloying elements.

In order to illustrate the components of the total yield strength in the different conditions of combined amounts of pre-deformation and aging time, three different strength additions are indicated in Fig. 3:

$$\sigma_y = \Delta\sigma_{ss} + \Delta\sigma_d + \Delta\sigma_p \quad (1)$$

where $\sigma_y$ is the yield strength, $\Delta\sigma_{ss}$ is the solid solution strength addition, $\Delta\sigma_d$ is the deformation strength addition and $\Delta\sigma_p$ is the precipitation strength addition. The friction stress is small in aluminium and therefore neglected. The different contributions to the initial yield stress for both alloys are given in Table 2.

The relative strength addition from precipitation has greater effect on the yield strength in the material artificially aged for 300 min with no pre-deformation ($\Delta\sigma_p/\sigma_y \cong 84\%$) compared to the material aged for the same time with 10% pre-deformation ($\Delta\sigma_p/\sigma_y \cong 43\%$). The relative strength addition from precipitation has the same effect, although to a lower extent, when comparing materials aged for shorter time and/or with smaller pre-deformation. Generally, increasing aging time increases the effect of precipitation strength addition. On the other hand, increasing pre-deformation decreases this effect, but it is compensated by the effect of deformation strength addition resulting in maximum yield strength in the material with 10% pre-deformation. The evolution of yield strength with increasing pre-deformation for the samples annealed at 190°C can be seen in Fig. 4(a). Figure 4(b) shows different additions from precipitation and deformation with respect to introduced pre-deformation. By comparing these two plots, it can be observed that $\Delta\sigma_p$ has a very small effect on yield strength for short time aging (10 min) regardless of the introduced pre-deformation. Yield strength continuously builds up with increasing pre-deformation in the materials aged for 100 min, where $\Delta\sigma_p$ is nearly constant and independent on pre-deformation. Finally, the yield strength is not varying very much in the material aged for 300 min with the amount of pre-deformation introduced, which is caused by an increasing $\Delta\sigma_d$ on the account of a decreasing $\Delta\sigma_p$. Despite this decreasing tendency, the effect of $\Delta\sigma_p$ is still higher compared to the material aged for shorter times.

From the stress-strain curves we can observed an apparent yield point for all the pre-deformed samples (except for the 2% pre-deformed sample aged for 10 min) compared to a smooth transition between elastic and plastic behaviour for...
the samples with no pre-deformation. This can be clearly seen in the work-hardening rate versus strain curves where the apparent yield point is represented by the increase in the work-hardening rate after a very rapid decrease in the slope of the stress-strain curve, which is associated with the elastoplastic transition (Fig. 5). Contrary to the natural aging work-hardening rate behaviour, the work-hardening rate does not decrease more rapidly for pre-deformed samples artificially aged for the same times.

The effect of different levels of pre-deformation on the kinetics of the precipitation sequence is demonstrated by the evolution in DSC curves shown in Fig. 6(a). It is evident that an increasing dislocation density accelerates precipitation kinetics which is represented by a shift to lower temperatures of the exothermic peak associated with the formation of \(\beta''/\beta'\) precipitates. The exothermic \(\beta''/\beta'\) peak for the material with no pre-deformation reaches its maximum above a temperature of 300°C compared to a maximum at 260°C for the material with 10% pre-deformation. The second exothermic peak associated with the equilibrium \(\beta/\beta'\) phase can be consistently identified at 430°C in all DSC curves. While DSC curves in Fig. 6(a) were carried out for freshly solutionized material and eventually pre-deformed, Fig. 6(b) shows the evolution in DSC curves for the material naturally aged for 1 week after SHT and then pre-deformed before aging. As in the previous case, the pre-deformation shifts the exothermic \(\beta''/\beta'\) peak to lower temperatures while the \(\beta\) peak can be found at almost the same temperature in all curves. However, one distinctive difference can be found in DSC curves when comparing freshly solutionized material and naturally aged materials. The DSC curves for the freshly solutionized samples display no trough at the beginning of the exothermic \(\beta''/\beta'\) peak and pre-deformation does not change this observation. On the other hand, in the DSC curve for the naturally aged sample with no introduced pre-deformation, a significant trough can be observed. This trough is most probably associated with the dissolution of Mg-Si co-clusters which have formed during natural aging. Consequently, a slight shift in the exothermic \(\beta''/\beta'\) peak to higher temperatures can be detected by DSC analysis due to a slowed down hardening process in the naturally aged sample, as compared to the sample without any room temperature storage before artificially aging. Pre-deformed samples do not display any of these features. This indicates retained formation of Mg-Si co-clusters due to the annihilation of quenched-in vacancies by dislocations.

Figure 7 shows a comparison of the 0% pre-deformed sample artificially aged at 190°C for 300 min and the 10% pre-deformed sample artificially aged at equal conditions. It can be observed that in the pre-deformed sample the precipitation occurs preferentially along dislocation lines, while in the non-deformed sample the precipitation is more homogeneous. Further TEM investigations of the precipitation behaviour, by Teichmann et al., have shown that most of the microstructure in the deformed condition was coarser, with a higher volume fraction as compared to the undeformed material. The faster over-aging of some deformed conditions (during aging at 190°C), as compared to the non-deformed ones, may therefore be explained by the rapid precipitate coarsening along dislocations. At the same time this observation of a coarser precipitate structure may also explain the decreased strength addition from the precipitates with increasing pre-deformation as shown in Fig. 4(b) for 300 min aging. Any recovery of the dislocations which may occur during aging at 190°C has not been considered in this investigation.
4. Conclusions

Nearly the same peak hardness for the pre-deformed AA6060 is reached during artificial aging at 150°C and 175°C regardless of the introduced pre-deformation. Moreover, the peak-hardness condition is obtained after almost the same time of annealing, although a small shift to shorter times to peak hardness may be noticed at 175°C. At 175°C it was also observed that even such a small pre-deformation as 0.5% is sufficient to promote precipitate formation to such an extent that almost the same peak hardness is reached as for the samples with larger pre-deformation (2–10%). At 190°C there is a distinct increase in the peak hardness at the same time as the peak condition is reached after short times. In general, the peak hardness for the artificially aged pre-deformed material is significantly higher compared to that of the material with no pre-deformation. This is combined effect of strength additions from strain hardening (dislocations) and precipitate hardening, which relative contributions changes with pre-deformation and aging time.

Concerning the stress-strain behaviour it was found that both the yield strength and ultimate tensile strength generally increases with increasing pre-deformation. However, after artificial aging at 190°C for 300 min the stress-strain curves level out regardless the introduced pre-deformation, resulting in almost the same yield and ultimate tensile strength for all pre-deformations. In this latter case the increasing dislocation strength contribution with pre-deformation is counter-balanced by a decrease in the precipitate hardening contribution, which can be understood by changes in the precipitation kinetics and behaviour.

Increasing pre-deformation accelerates the kinetics of the precipitation sequence, as documented by DSC. It is shown that the exothermic peak associated with the formation of $\beta''/\beta$ precipitates is shifted to lower temperatures with increasing pre-deformation. Moreover, it is shown that the endothermic trough which may be associated with the dissolution of Mg-Si co-clusters was identified solely for the undeformed material naturally aged for 1 week. For the freshly solutionized undeformed sample and all pre-deformed samples, this trough do not show up; indicating that introduction of dislocations prior to any aging suppresses the Mg-Si co-clusters formation during subsequent aging.

Finally, the decrease in the precipitate hardening contribution with pre-deformation after aging at 190°C may also be understood in view of TEM observations of non-deformed
and 10% pre-deformed samples aged for 300 min at 190°C, briefly exemplified here, and further detailed by Teichmann et al. In their work it is found that the precipitates in the pre-deformed condition are coarser and fewer as compared to the un-deformed condition.

Acknowledgement

Hydro, Steertec Raufoss AS, and the Research Council of Norway (RCN) are gratefully acknowledged for financial support through the BIP Project No. 176816.

REFERENCES