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Stabilization of 7xxx aluminium alloys

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ABSTRACT

High strength Al-Zn-Mg(-Cu) alloys are rarely used in the automotive industry due to pronounced natural ageing after quenching and resulting poor formability at room temperature. Furthermore, the paint bake response is often suboptimal. To overcome these challenges, we performed various low temperature pre-ageing treatments followed by storage at room temperature and a short high temperature heat treatment simulating industrial paint baking. The intermediate and final mechanical properties were assessed and the nanostructure, precipitation, and local chemistries of the samples were studied by atom probe tomography and differential scanning calorimetry. It was found that a properly designed stabilization treatment can inhibit the natural ageing process in AA7021 resulting in a stable condition with a lower hardness than samples naturally aged for longer than approximately one week. Atom probe tomography revealed that the stabilization leads to co-precipitation of Mg and Zn (indicating GP-zones) hindering the further increase in hardness over the course of three weeks. Furthermore, the paint bake response of AA7075 was improved resulting in yield strength only 2% short of a peak aged sample.

1. Introduction

B-pillars and other key automotive parts require a high specific strength to satisfy the roof crush and side impact test standards while keeping the weight low. High strength Al alloys of the 7xxx series could fulfil these requirements; however, mainly due to their poor formability at room temperature, 7xxx alloys are still mostly used in the aircraft industry and up to this time have only found limited use as automotive parts [1,2].

The final mechanical properties of the parts are a measure for process chain's quality. It was found in many investigations that the final strength of the parts was much lower than the sheet's maximum possible strength (i.e., T6). The main reasons were lower quenching rates than the critical cooling rate and incomplete ageing during paint baking. In one investigation, AA7075-T6 sheet was processed via various routes, however, it was found that the initial as-received mechanical properties of the sheet could not be matched [3]. Kumar el al. [1] investigated the warm forming of AA7020-T6; it was found that as a result of warm forming and paint baking, the yield strength and ultimate tensile strength were reduced by 9% and 13%, respectively. To recover the strength, an intermediate heat treatment before paint baking was proposed. Such an approach was investigated by Bardelcik et al. [4] who studied the effect of an intermediate heat treatment of 100 °C for 3 h on paint bake response of AA7075. Although paint bake response was improved, the mechanical properties achieved by a peak ageing were not matched. In a patent application [5] a variety of pre-ageing treatments with the intent to increase the yield strength after paint baking are disclosed. It is demonstrated that by applying two- or three-step heat treatments, mechanical properties similar to the peak aged state can be achieved in much shorter time.

In this paper, different intermediate heat treatments were applied on sheet material of AA7021 and AA7075 in T4 temper. Such heat treatments are commonly called stabilization due to the fact that thereafter the precipitation of hardening phases should not occur during storage at RT (i.e., no, or reduced, natural ageing). To assess the efficacy of the stabilization treatments, the natural ageing behaviour of as-quenched and stabilized samples was studied over 22 days. In the automotive industry, a material with

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defined mechanical properties before paint baking is vital for the design of joining operations such as self-pierce riveting; furthermore, the paint bake response should be equal or better as in the non-stabilized material. Therefore, the effect of stabilization on the intermediate and final mechanical properties (i.e., after paint baking or peak ageing) is reported. The effects of the various heat treatments on the nano- and microstructure were investigated by means of atom probe tomography (APT) and differential scanning calorimetry (DSC).

2. Experimental

AA7075-T6 and an AA7021-T4 type alloy (Structurlite® 400, provided by Aleris) with a thickness of 2 mm each were used; the chemical compositions in weight percent as determined by optical emission spectroscopy (for AA7075) and provided by Aleris (for the AA7021 type alloy) are shown in Table 1. Although the Mg and Zn contents of the AA7021 type alloy slightly exceed the standard composition limits of AA7021 (i.e., a maximum Mg and Zn content of 1.8% and 6.0%, respectively) it will be referred to as AA7021 below.

Both sheets were heat treated according to the schemes shown in Fig. 1. For the stabilization heat treatment, the sheets were held at 90 °C, 120 °C, and 150 °C for 1 h, 2 h, 4 h, and 5 h. The samples were stored for different times at RT to determine the influence of natural ageing (na) and then subjected to a paint baking (pb) heat treatment of 185 °C/20 min or a peak ageing (pa or T6) heat treatment of 120 °C/24 h.

DSC and APT measurements (for AA7021) as well as hardness and tensile tests were carried out to study the precipitation evolution, nano- and microstructure, and the resulting mechanical properties. Vickers micro-hardness (HV0.1) was measured five times per sample using a load of 100 g. Tensile tests were conducted in triplicate on a Zwick Z100 materials testing machine according to EN ISO 6892-1. Specimens were cut parallel to the rolling direction. They had a width of 12.5 mm, a reduced section length of 75 mm and a gauge length of 50 mm.

APT specimens were produced by cutting matchstick shaped samples with edge dimensions of 0.5 mm × 0.5 mm × 10 mm, mechanically polishing them to an equal width of approximately 0.3 mm, and electropolishing a sharp tip with a solution of 10% perchloric acid in ethanol using an ElectroPointer device at room temperature [6]. The potential was gradually decreased from 20 V to 5 V AC during the electropolishing process which took approximately 15–20 min per sample. The samples were then inspected, sharpened, and cleaned in a scanning electron microscope equipped with a Ga focussed ion beam (FEI FIB-SEM Helios 600i). The FIB was operated at 30 kV for initial annular milling and 5 kV for final cleaning to minimize Ga implantation. APT analysis was performed on a Cameca LEAP 4000X HR atom probe system at a temperature of 34 K. The measurements were performed in voltage pulse mode with a pulse fraction of 0.2, a pulse frequency of 200 kHz, and a detection rate of 0.5%. For reconstruction of APT datasets, the software IVAS 3.6.14 was used.

Differential scanning calorimetry (DSC) experiments were carried out using a Netzsch-DSC 204 F1 differential scanning calorimeter with a heating rate of 10 K min^{-1}.

3. Results and discussion

3.1. Influence of stabilization on the mechanical properties

The response of the solution heat treated and quenched sheets to the stabilization heat treatments is shown in Fig. 2; the hardness values of the two alloys increase with stabilization heat treatment time. The hardness of all samples was monitored for 22 days after stabilization and it was found that for all stabilization variants no significant changes in hardness occurred, with the exception of 90 °C/1 h and 120 °C/1 h for AA7075 where, after 22 days, an increase in HV0.1 from 139 ± 2 to 151 ± 2 (Fig. 3) and 142 ± 2 to 157 ± 3, respectively, was found. It can be concluded that all other stabilization treatments are suitable to inhibit the natural ageing process in the alloys in the surveyed time frame of 22 days.

In Fig. 3 the hardness evolution due to natural ageing over the course of 22 days is shown for the sheets in W-temper as well as in peak aged condition (for reference). As expected, there is a considerable increase in hardness over time for both alloys in W-temper. Furthermore, the natural ageing behaviour of the samples stabilized at 90 °C for one or two hours are shown; the above-mentioned increase in hardness for AA7075 stabilized at 90 °C/1 h can be seen.

Stabilizing AA7021 at 90 °C/1 h yields a stable condition with hardness lower than 125 HV0.1 whereas the hardness after 22 days of natural ageing was 130 ± 1 HV0.1. This is of great interest because the lower hardness is beneficial for subsequent manufacturing operations. For example, we found that proper self-pierce riveting of the sheets is only possible up to a hardness of approximately 125 HV0.1 (using a standard C H4 type rivet by Böllhoff). This value is surpassed by natural ageing of AA7021 after approximately one week while the stabilized sheet maintains a lower hardness. It has been reported for other 7xxx series alloys that GP(II)-zones are only formed by ageing above 70 °C while GP(I)-zones are formed at RT as well as at higher temperatures [7,8]. Therefore, a possible explanation for the observed stabilization by pre-ageing at 90 °C could be the formation of stable GP(II)-zones which inhibit subsequent formation of GP(I)-zones and the associated increase in hardness at room temperature. However, others have reported that no GP(II)-zones were found after ageing AA7050 for 1.5 h at 120 °C [9]. We performed atom probe tomography to obtain further insight into these early stage precipitation processes (see section 3.3).

For AA 7075, a stable condition with hardness lower than 125 HV0.1 or lower than the hardness after three weeks of natural ageing could not be achieved. Since 90 °C for 1 h and 120 °C for 1 h yielded unstable conditions a reduction of ageing time seems not a promising approach. However, stabilizing at lower temperature for longer time might be worth exploring.

Fig. 4 shows the results of tensile tests performed on sheets in different tempers. While the effect of one week of natural ageing is clearly visible for both alloys, the strength of the stabilized samples did not change. Furthermore, there is no significant difference in the mechanical properties whether the stabilization treatment was applied directly after quenching or after one week of natural ageing. Expectedly, the values for the latter samples stayed constant also after another week of natural ageing following stabilization (data not shown). Likewise, after paint baking no difference could be

Table 1
Chemical composition of the used alloys (in wt. %).

Alloys	Al	Si	Fe	Cu	Mn	Mg	Zn	Cr	Ti	Zr	Others
AA7075-T6	rest	0.19	0.11	1.50	0.04	2.64	6.06	0.18	0.04	0.02	max. 0.03
AA7021-T4	rest	max. 0.25	max. 0.40	max. 0.16	max. 0.10	1.60–2.10	6.00–6.80	max. 0.05	max. 0.1	max. 0.18	max. 0.15