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Effect of quenching rate on microstructure and mechanical properties of commercial AA7108 aluminium alloy

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Abstract: A commercial AA7108 aluminium alloy subjected to different quenching procedures from the solid solution temperature of 480 °C was considered. Both continuously cooling and quenching to intermediate temperatures (400, 300 and 200 °C) followed by different holding times were applied. All the materials were subsequently subjected to the same industrial two-step artificial ageing procedure to obtain a maximum strength (T6). Tensile testing of the different quenched materials in the T6 temper reveals a large deviation in strength dependent on the cooling/holding time, compared with the reference sample. A collection of the different quenched materials was chosen for further investigation of the precipitate structure by transmission electron microscopy (TEM) to explain the differences in mechanical properties.

Key words: aluminium; microstructure; precipitate free zone; mechanical properties

1 Introduction

In the heat-treatable aluminium alloys, the mechanical properties are strongly dependent on the microstructure obtained during the thermo-mechanical process. The strength is mainly gained from the formation of nano-sized precipitates distributed in the matrix. To ensure the final strength of a component, it is, however, important to control that the precipitates form and develop in a beneficial way. The 7××× alloys are prone to formation of precipitate free zones (PFZ), which typically appear along the grain boundaries and thus also weaken the overall strength of the material [1-5]. The formation and width of the PFZ are controlled by the diffusivity of vacancies and alloying elements in solid solution [6,7]. Hence, the quenching time from the solid solution temperature is of great importance. This work considers how different quenching procedures from the solid solution heat treatment affect the tensile properties after artificial ageing to the T6 temper of a commercial aluminium alloy. For comparison microstructure and mechanical properties, a selection of different quenched materials was also further investigated by transmission electron microscopy (TEM).

The work is motivated by the fact that during industrial operation unintended stops may take place which may interrupt the quenching after solution heat treatment and leave the material at a certain intermediate temperature for a shorter or longer time, before subsequent artificial ageing. It is important to investigate and quantify how and to which extent this may influence the final mechanical properties.

2 Experimental

A commercial AA7108 aluminium alloy (Al-5.5 Zn-1.2 Mg-0.15Zr + trace elements of Fe, Si, Cu and Mn) was provided by Benteler Aluminium Systems Norway AS as-extruded plates of 3 mm in thickness and 200 mm in width. A preliminary study using numerous quenching/cooling methods from the solid solution temperature, where the hardness and electrical conductivity were measured after artificial ageing, was performed on small test samples before six cooling

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methods were selected for further investigation in addition to a reference sample. All the samples were first solid solution heat treated at 480 °C for 30 min. The reference sample was directly water quenched to room temperature, whereas the six others were exposed to the following cooling procedures:

- 1) Quenched to 400 °C and held for 120 min and subsequently water quenched to room temperature;
- 2) Quenched to and held at 300 °C for 10 min and then water quenched to room temperature;
- 3) Two materials were quenched to 200 °C and held at the temperature for 10 and 120 min, respectively, before water quenching;
- 4) Continuous cooling from 480 °C to 200 °C within 10 min (28 °C/min) followed by water quenching;
- 5) Continuous cooling from 480 °C to 200 °C within 120 min (2.3 °C/min) followed by water quenching.

All the materials were subsequently stored at room temperature for 24 h and then artificially aged to peak temper (T6) using a two-step heat treatment, where at the first step materials were held at 100 °C for 5 h followed by 6 h at 150 °C.

Standard tensile specimens were made in the 0° , 45° and 90° directions with respect to the extrusion direction and subjected to the given heat treatments. The reference sample was, however, only tested in the 0° direction. Tensile testing was carried out at a constant nominal strain rate of $0.01~\text{s}^{-1}$ in an MTS810 hydraulic test machine.

Specimens of the six materials in the T6 temper were prepared for TEM investigations by mechanically grinding and punching 3 mm discs followed by electropolishing in a double-jet Tenupol by a 33% nitric acid solution in methanol operated at -25 °C and 15V. The samples were investigated in a JEOL JEM-2010 TEM operated at a nominal voltage of 200 kV.

3 Results and discussion

Since the mechanical properties are strongly related to the microstructure, it is important to understand and compare the precipitate structure with the mechanical performance. In this part the tensile properties will first be presented, before the results from the TEM investigations of the microstructures will be presented and discussed in relation to the mechanical properties.

In Fig. 1, the true stress—strain curves for the different cooled materials are shown after artificial ageing to peak temper. All the materials show approximately the same properties in the 0° and 90° directions, whereas the 45° direction is lower in strength

as expected for an extruded aluminium alloy with the texture of a fibrous microstructure [8,9]. In Fig. 1(a), the alloy was quenching and held at 400 °C for 120 min before water quenching to room temperature followed by artificial ageing. Only a slight decrease in strength compared with the reference sample is observed for this material. In Figs. 1(b) and (c), the materials were quenched to 300 °C and 200 °C, respectively, and held at the temperature for 10 min before quenching to room temperature and subsequent artificial ageing. Both of these materials show a 10% decrease in strength compared with the reference sample. However, if quenched to 200 °C and the holding time is prolonged to 120 min (see Fig. 1(d)), the influence on the mechanical properties is really detrimental as the strength decreases by ~40% in the T6 temper compared with the reference sample. Figs. 1(e) and (f) show the materials that have been continuously cooled from 480 °C to 200 °C within 10 and 120 min, respectively. The samples continuously cooled within 10 min show a similar behaviour as those quenched to intermediate temperatures and held for the same period of time, i.e. ~10% loss of strength. When continuously cooled within 120 min, a similar 40% loss of strength is observed as for the samples kept at 200 °C for 120 min. Both of these materials show a different work-hardening behaviour more similar to what is expected for a softer and strongly over-aged temper [5], where the material is work-hardening more within a much larger strain interval after yielding compared with the reference sample.

The reason for the observed loss of strength for the different cooled materials in Fig. 1 should be reflected by the difference in the microstructure. In Fig. 2 and Fig. 3 the microstructures of the materials quenched to intermediate temperatures and the continuous cooled materials are presented, respectively, after artificial ageing to peak temper. Figure 2(a) shows the reference sample with a narrow PFZ and a few grain boundary precipitates.

In Fig. 2(b), the material kept at 400 °C for 120 min is presented. The solvus temperature of this alloy is known to be ~350 °C [10], and hence above this temperature the alloying elements are still expected to be in solid solution before being quenched to room temperature. However, an increased number of grain boundary precipitates are shown, and these will lead to a slightly lower amount of alloying elements in solid solution and, hence, a lower hardening potential. Also, the number of quenched-in vacancies is expected to be higher when quenching from 480 °C compared with 400 °C, and this may also promote a more effective hardening precipitate nucleation after artificial ageing.

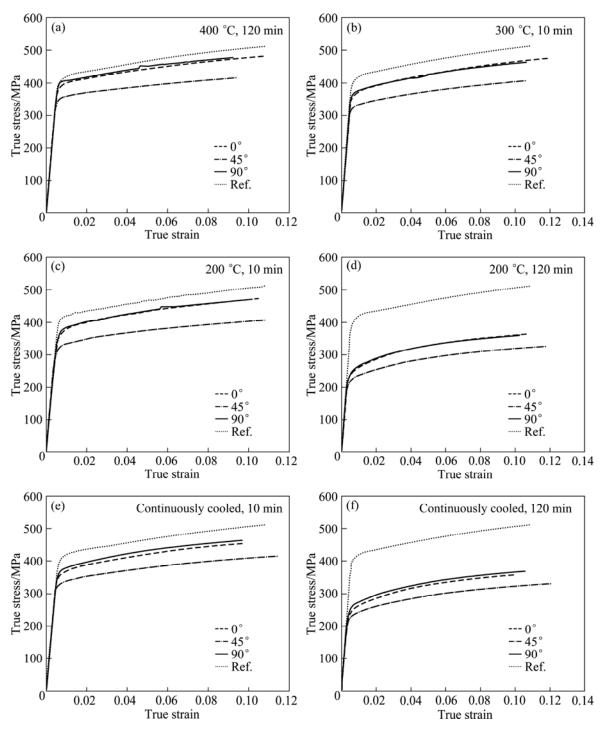
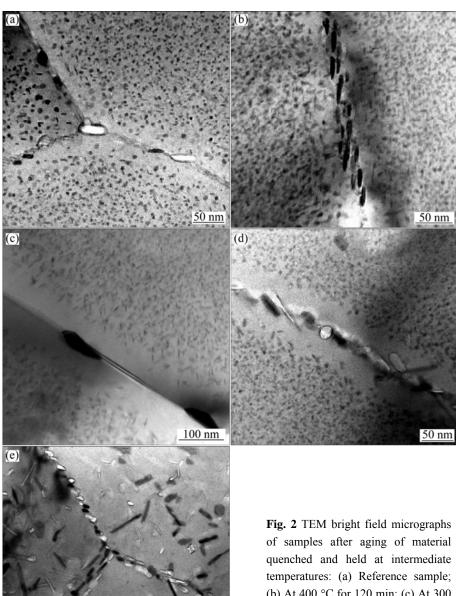


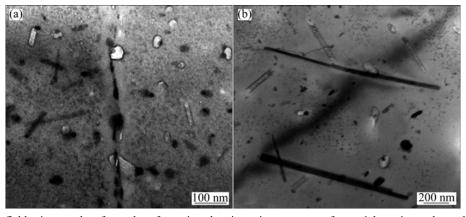
Fig. 1 True stress—strain curves of different cooled materials in 0°, 45° and 90° directions: (a)–(d) Quenched to intermediate temperatures and held for either 10 min or 120 min; (e), (f) Continuously cooled from 480 °C to 200 °C within 10 min and 120 min, respectively (The reference sample in 0° direction is shown in all the figures for comparison.)

Consequently, a slight decrease in strength is observed (Fig. 1(a)). When the material is kept temporarily below the solvus temperature during quenching (Figs. 2(c) and (d)), there will be a competition between particles forming and growing on the grain boundaries and particles nucleating in the supersaturated matrix. After 10 min only the grain boundary particles are growing, depleting the zone around the grain boundaries (Figs. 2(c)

and (d)). No clear evidence of matrix particles formed at 200 °C or 300 °C is found for the materials kept at these temperatures for only 10 min. This observation is supported by the fact that approximately the same drop in strength (10%) is observed after 10 min at both 200 and 300 °C. After 120 min holding time, however, a large number of matrix particles (equilibrium phase η) formed at 200 °C is observed, and apparently a quite



(b) At 400 °C for 120 min; (c) At 300 °C for 10 min; (d) At 200 °C for 10 min; (e) At 200 °C for 120 min



200 nm

Fig. 3 TEM bright field micrographs of samples after aging showing microstructure of material continuously cooled from 480 °C to 200 °C within 10 min (a) and 120 min (b)

reduced number of age hardening particles are formed in the subsequent ageing leaving the material with limited or no hardening potential. This results in the unfavourable loss of strength observed in Fig. 1(d). This also explains the difference observed in work-hardening behaviour.

During continuous cooling from the solid solution temperature down to 200 °C, the nucleation conditions for matrix precipitation of the η phase are promoted when the temperature drops below the solvus temperature, as shown in Fig. 3(a), where larger needle shaped particles have started to form leaving a very inhomogeneous microstructure with most probably both η' , η , grain boundary precipitates and a distinct PFZ. The precipitation of η is, however, not very extensive at the high cooling rate (10 min), and the age hardening potential is only reduced by about 10%. By lowering the cooling rate (120 min), the η -formation during cooling becomes much more pronounced, leaving only a limited amount of solute for η' -precipitation during age hardening, as shown in Fig. 3(b). DESCHAMPS and BRÉCHET [2] have observed formation of needleshaped η_2 particles that have nucleated and grown on Al₃Zr dispersoids when the material is slowly quenched. A closer look at the precipitates in Fig. 4 indicates that this might be the case here as well. The small round particles marked with D in Fig. 4 are the dispersoids, and the large particles which seem to grow are the precipitates.

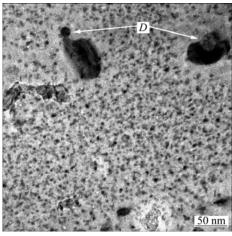


Fig. 4 TEM bright field image of sample continuously cooled within 10 min showing formation of precipitates on existing dispersoids

When the material is continuously cooled within 120 min before quenching to room temperature and artificial ageing (Fig. 3(b)), the large particles have grown to μm size. In the vicinity of the particles, PFZs are observed, and between the needles, clusters of small

precipitates are seen. The PFZs around the particles are due to diffusion restrictions. As the particles nucleate and start to grow, the matrix in the vicinity gets depleted with respect to alloying elements. This again hinders formation of GP zones in this area and thus also formation of η' precipitates. The large particles do not contribute to the strength, and the clusters of small precipitates are not able to compensate the strength loss caused by the PFZs leaving the material poor mechanical properties as seen from Fig. 1(f).

4 Conclusions

- 1) If the material is quenched from the solid solution temperature of 480 °C to a temperature above the solvus temperature (in this case quenched to 400 °C) no specific change in microstructure and only a slight decrease in mechanical properties are observed independent of the holding time. The decrease is attributed to more grain boundary precipitation than by directly quenching from 480 °C or less effective η' -precipitation during age hardening due to reduced number quenched in vacancies, or a combination of the two.
- 2) When the material is quenched to temperature below the solvus temperature and held for a shorter time (10 min), a large PFZ and approximately 10% decrease in strength compared with the reference sample is observed. The reduced strength is attributed to the PFZs themselves or the less effective η' -precipitation during age hardening, or a combination of the two.
- 3) When held for a longer time at intermediate temperatures under the solvus temperature during quenching, a microstructure with coarse, but homogeneously distributed lath-shaped precipitates formed during holding at the intermediate temperature is observed. The tensile properties show an approximate 40% decrease in strength compared with the reference sample. Moreover, the work-hardening behaviour is changed.
- 4) If continuously cooled down to 200 °C, an inhomogeneous precipitate structure was observed. Large lath-shaped precipitates, which have formed in the matrix during cooling (on dispersoids or other heterogenities), as well as small ellipsoidal precipitates are present at the same time. After a longer cooling time, PFZs are observed in the vicinity of the large lath-shaped particles, which have depleted the matrix and prevented GP zones from forming. For cooling time of 10 min and 120 min, the decrease in yield strength is found to be ~10% and ~40% compared with the reference sample, respectively.

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淬火速率对 **AA7108** 铝合金的 微观结构和力学性能的影响

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摘 要:在 480 ℃ 固溶温度对 AA7108 铝合金进行不同工艺的淬火。将合金连续冷却、淬火至中间温度(400,300 和 200 ℃)并保温不同时间,再进行工业两步人工时效处理,以获得最大强度(T6)。T6 态淬火材料的拉伸试验表明,材料的强度很大程度上取决于冷却/保温时间。通过透射电子显微镜(TEM)观察材料中沉淀物结构来解释材料的力学性能差异。

关键词:铝合金;微观结构;无析出带;力学性能

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