THE ROLE OF PRE-DEFORMATION HISTORY IN CRACK INITIATION DURING THE HIGH TEMPERATURE SOAKING TREATMENT OF Al-Mg-Mn ALLOY TYPE-5454

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ABSTRACT

Slabs produced by hot rolling may receive a high temperature soaking treatment before they are finally formed. In this work, 28-mm thick slabs, produced in the VAW Aluminum Company, Bonn, Germany, were homogenized at different homogenization temperatures in the range of 550-620°C for 1 hr. The heating process was carried out with different rates whereas cooling was achieved either with the same rates or faster by quenching into water to room temperature. Localized cracks in the one-quarter thickness positions of these slabs were clearly observed after receiving the 600°C treatment when heating and cooling took place with rates ≥ 20°C/min only. The severity of these cracks increases with increasing cooling rate and they spread over the whole slab thickness when the homogenization temperature is increased. Crack initiation and formation were observed using the optical (OM) and the scanning electron microscope (SEM). Concurrently, differential thermal analysis and thermo-mechanical analysis were carried out to measure respectively the phase transformations and thermal expansion taking place during the homogenization treatment.

KEYWORDS: AA5454, homogenisation, precipitate distribution, pre-deformation, localized cracking, thermal expansion.

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INTRODUCTION

Although the continuous casting process provides high quality finished aluminum plates and/or sheets, most production comes from rolling of as-cast ingots. These ingots are hot rolled, to reduce the required deformation forces, after having been homogenized at high temperature to eliminate the macro-segregation that takes place during the solidification process. In some cases, the material is supplied to the manufacturer in inadequate dimensions so that the slab is repeatedly rolled to reach the required dimensions. In others, the manufacturer needs thicker slabs on which to perform his own treatment to obtain the required microstructure and properties. However, in all cases the material is re-homogenized before it is rolled. Few publications were found concerning this particular alloy (type-5454). Zaidi and Sheppard [1] have studied the effect of homogenization treatment on the recrystallization kinetics of this alloy as well as the present authors in previous work [2,3]. Their work focused on establishing "optimum treatment schedules" to satisfy either the economical restrictions or to avoid the deleterious effects of the over heating exposure since these treatments are always performed at high temperatures very near to the solidus. Their experiments were carried out on the as-cast material where small specimens were homogenized at different temperatures for elongated periods (~24 hr) to subside the effect of the soaking intervals. Enhanced recrystallization rates were observed after the material was homogenized at high temperatures (~600°C). In this temperature range the primary intermetallic particles may transform to more stable phases accompanied by either heat evolution or absorption. Depending on the quantity and morphology of the transformed particles and consequently on the amount of the evolved heat during the reaction, the material may melt locally. Other authors [4] studied the transformation behaviour of the particle species present, but in different alloys. They found that these species go through a partial/complete transformation at temperatures very near to 600°C, depending on the soaking period. In this work, the investigated material is a pre-deformed slab that has received the 15 reverse-rolling passes (as rough-rolled condition). The microstructure of the as rough-rolled slab was observed along its thickness and correlated to the primary constituent distribution. Variation of the observed microstructure has a remarkable effect on the measured macro-hardness across the thickness. Phase transformation, which takes place during the homogenization treatment, accelerates crack initiation whereas its formation is enhanced by the different thermal expansion coefficient at different positions between the slab surfaces.
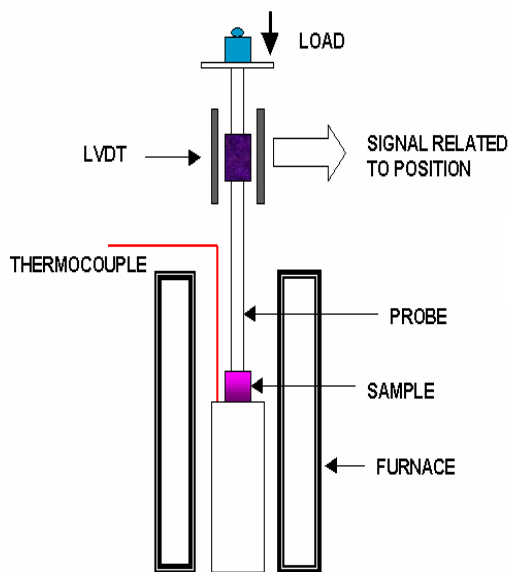
MATERIAL AND EXPERIMENTAL METHODS

The present investigation was carried out on a commercial Al-Mg-Mn alloy type-5454. This material was produced by the VAW Aluminum Company, Bonn, Germany and supplied in the form of a hot rolled 28-mm thick slab. To reach this thickness, the original cast ingot was rolled in 15 reverse-rolling passes over a wide temperature range 520-450°C. Before the rolling process, the material was homogenized at 540°C for 6 hours and air cooled to the rolling temperature. Table 1 shows the chemical composition of the investigated alloy.
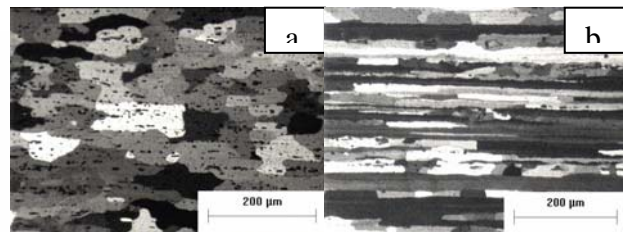
Table 1. Chemical composition of the used aluminium alloy type-5454.

| Element	Mg	Mn	Fe	Si	Al
Percentages (wt %)	2.9	0.83	0.367	0.15	Rem.

All the microstructure observations were carried out on the longitudinal rolling plane (LR) of the supplied slab. The material was electro-etched in Barker's reagent with a current of 20 mA and then investigated with a *Nikon Epiphot 300* optical microscope equipped with a polarized light system. To measure the thermal expansion characteristics of the material, specimens in the form of 5-mm cubes were machined and treated in an inert atmosphere with controlled heating and cooling rates. Figure 1 shows a schematic drawing for the Thermal Mechanical Analyzer (TMA). An argon flow rate of 50 cm^3 per min was used to reduce the chance of oxidization and prevent acquisition of false values. An alumina probe in contact with the sample surface transfers the signal to the attached data acquisition module. The material response was displayed on a monitor and recorded as a computer file. To measure exactly the critical temperatures for the phase transformation reactions that take place during the homogenization treatment a sample of 30mg of fine chips of the material was put in a small alumina crucible and heated in the furnace of the differential thermal analyzer (DTA) (model Thermal Sciences 1640) up to the pre-determined temperature. Both the investigated sample and a standard one (does not have any phase transformation in the proposed temperature range) were heated in the same chamber. Because of the different reactions that might take place during the homogenization process a temperature difference between the two samples is expected. This difference is displayed and recorded for presentation purposes. In this method a downward peak indicates an endothermic reaction whereas an upward one indicates an exothermic reaction.

Fig.2. Microstructure of the as-rough rolled slab at (a) surface and (b) centre positions.

Fig.1. Schematic drawing showing the Thermo-Mechanical-Analyzer (TMA).
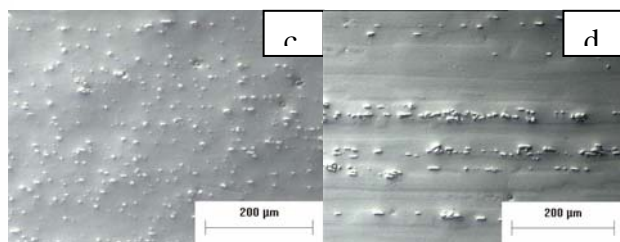
Fig.3. Precipitate distribution in the same positions of the microstructure shown in Fig.2. respectively.

RESULTS AND DISCUSSION

Evaluation of the as-rough rolled slab

Figure 2 shows the microstructure of the as-rough rolled material in the surface and center of the slab. A microstructure gradient was clearly observed through its thickness where completely equiaxed recrystallized grains were observed in the surface position and severely deformed ones in the center. In between these deformed grains, bands of either completely or partially recrystallized grains were observed near the aligned primary particles. Analyzing these precipitates showed that they are mainly $Al_6(Mn,Fe)$ and $Al_{13}(Mn,Fe)_4$. Figure 3 shows the precipitate distribution in the same positions observed and shown in Fig 2. In this case, the specimens were only mechanically prepared (ground and polished) without being chemically etched. In the surface positions, the primary coarse precipitates are homogeneously distributed whereas those in the center are aligned into bands parallel to the rolling plane. Variation is not only observed in the precipitate distribution but also in its morphology. Close to the slab surfaces, the precipitates are nearly spherical whereas they adopt an elongated shape in positions near to the center.

Effect of the homogenization treatment on the grain size of the as-rough rolled slab

Small specimens from the as-rough rolled slab were heated in a muffle furnace to the predetermined temperatures 500, 550, 570, 580, 590, 600, 610 and 620°C. For each homogenization temperature, a series of specimens was heated up with different heating rates 5, 10, 15, 20 and 25°C/min. They were held for 1 hr and then cooled to room temperature with the same rates as those used during heating. These specimens were subsequently prepared for optical observations and the grain size was measured using the standard circle method. To reveal the maximum difference, the microstructure was analyzed in the center and the surface positions only. Figure 4 shows the size variation as a function of the homogenization temperature. It increases from 40 and 50 µm in the center and surface respectively to reach 65µm at 600°C. Homogenization at 610°C increases the grain size to 125µm.
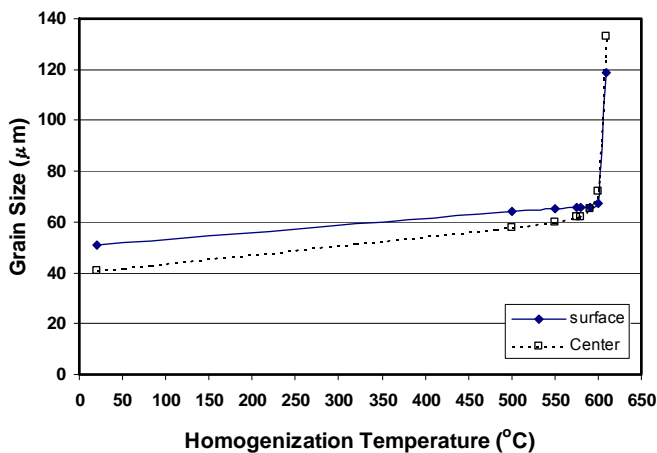
Fig.4. Grain size variation w.r.t homogenization temperature.
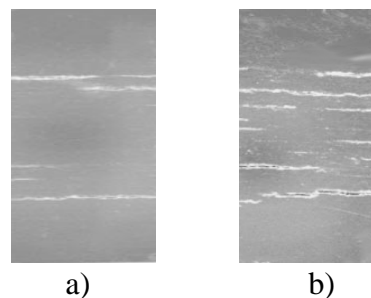
Fig.5. Crack distribution in specimens held for 1 (hr) at a) 600°C   b) 620°C.

Effect of the heating/cooling rates

Although changing the heating rate was expected to have an impact on changing the grain size of the material after receiving the homogenization treatment, its effect was outside the scope of this work. Only with heating and cooling rates $\geq 20°C/min$ and with temperatures $\geq 600°C$ did the specimens show localized cracking. These observed cracks are parallel to the slab surface at one-quarter of the slab thickness, Fig 5-a. Increasing the soaking temperature and the heating/cooling rates used increases the probability of crack formation and causes them to spread over the whole slab core and their width to increase. The maximum severity of these cracks is observed after quenching the homogenized material from 620°C to room temperature, Fig 5-b. All the formed cracks are inter-granular in nature and follow the aligned precipitate bands.

Crack investigation

To characterize the cracking phenomenon, a sample of 30mg of fine chips from the same position as the observed cracks (one-quarter of the slab thickness) was analyzed in the DTA furnace to a temperature very near to the melting temperature. Figure 6 shows the DTA curve representing the reactions, which take place up to a temperature of 600°C. During the heating process, the sample shows an endothermic reaction over a wide range of temperature with a peak at 556°C, position (P1). This reaction is believed to be a dissolution reaction, which dissolves some of the secondary fine precipitates $\leq 1\mu m$ in diameter. At 595°C, an exothermic reaction representing the transformation of the intermetallic particle species $Al_6(Mn,Fe)$ as studied in [5-7] or even the $Al_{13}(Mn,Fe)_4$ as reported in [8] to the α phases ($Al_{12}(Mn,Fe)_3Si$ or $Al_{15}(Mn,Fe)_3Si_2$) is observed in position (P2). This reaction is known to be dependent on the Si content. Exactly at 600°C, the material starts melting and goes through an endothermic reaction. During cooling, the samples show an exothermic reaction in position (P3) representing the re-precipitation of the secondary phases during the solidification process. According to the thermodynamic laws of solidification the re-precipitation process is expected to take place at a temperature lower than that shown in position (P1) but because of the difference between the solidification cooling rate of the original cast and that used during the DTA observation the re-precipitated species are different [9-11]. Consequently, the stoichiometry of these phases and their formation temperatures are different.
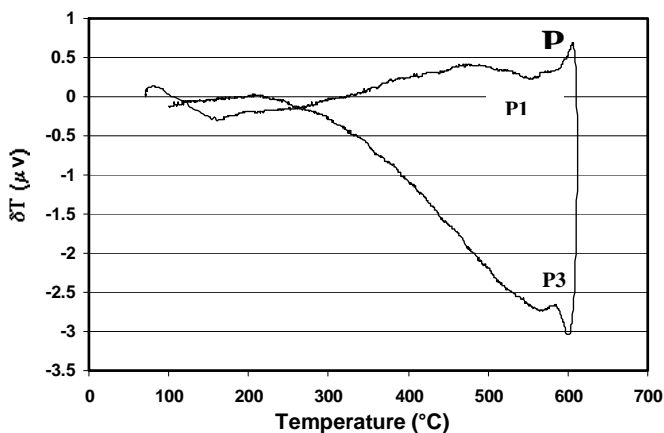

Fig.6. DTA curve shows the critical temperatures for the phase transformations during the homogenization treatment.


Fig.7. Crack initiation at the triple point occupied with the transforming intermetallic particles.

Observing the microstructure of the cracked region under the scanning electron microscope (SEM) with high magnification reveals that the cracks are always initiated at the coarse particles on the triple point and propagate along the grain boundaries Fig 7.

The variations of the thermal properties of the investigated material through the slab thickness were observed using the TMA. Three cubic samples (5 mm in length) were machined from the positions shown in Fig 8 and analysed. The automatic controlled TMA furnace allowed the exact simulation of the same treatment applied in the muffle furnace but with a maximum cooling/heating rate of 30°C/min. Figure 8 shows the behaviour of the material expansion in the Z direction during the simulated homogenization treatment (heating/cooling rate is 20°C/min). All the specimens that were cut from position A showed the same behaviour in Fig 8(a) where the material expands gradually up to a temperature very near to the adjusted furnace temperature. At about 595°C, the transformation of the primary particles evolves heat that causes the material to expand with a higher rate and shows a sudden peak. At 600°C, the observed material goes into an endothermic reaction as a result of a partial melting process. This reaction absorbs heat and decreases the specimen temperature below that of the furnace where the sample contracts and shows a sharp drop. During the holding period, the high temperature surroundings compensate for the heat difference between the sample and the furnace to reach equilibrium. Melted precipitates exert an internal pressure on the cavity walls pushing the material to expand in all directions. As a result of the heat compensation and the molten metal expansion, the specimen dimensions recover (in the Z direction) and reach the original value before the temperature drop. In position B and C, the material showed almost the same behaviour observed in position A but with different recovery percentages, Fig 8(b) and c. Both positions contract with three times that percentage in position A and recover to about 50 and 0% respectively relative to the contraction value.

Calculating the thermal expansion coefficients through both the heating or the cooling process reveals that the material does not behave reversibly. Figure 9 shows that the calculated coefficients change as the distance from the plate surface changes. Positions in between the plate surface and its center have the largest measured variation. As a result, the cooled specimens always have the largest residual stresses in those positions very near to one-quarter of the plate thickness. The irreversible behaviour shown in Fig 9 has a correlation with the precipitate size distribution as shown in Fig 3. In position A, the precipitates are fine and the measured thermal expansion coefficient is the average value for both the matrix and the precipitates where they could be treated as a metal-metal composite. On the contrary, in position C the precipitates are much coarser so that both the matrix and the precipitates were treated as discrete phases. Therefore, the material in position C has a different thermal expansion coefficient from that observed in position A. This distribution not only causes the variation of the thermal expansion coefficients but also has a large contribution to the variation of the amount of recovery observed in the Z direction. In position A, the fine precipitates have a high surface area to volume ratio so that diffusion of atoms from the particles into the surrounding matrix is enhanced. This decreases the composition gradient between the matrix and the precipitates and consequently decreases the probability of their melting, which allows the approximation of dealing with the material as a metal-metal composite. In position

C, the precipitates are very large and have a relatively small surface area to volume ratio. Thus, the persisting high composition gradient enhances precipitate melting especially with high heating rates [8]. Melting of these relatively large volumes of precipitates exerts larger internal pressure than melting of the fine precipitates. As these coarse precipitates in position C are always aligned on the boundaries of the deformed grains it is much easier for the molten metal to spread along the grain boundaries by capillary action.   Therefore, it reduces the exerted pressure and enhances the melting of more material, which decreases the overall temperature of the investigated material. Consequently, it prevents recovery in the Z direction after cooling the material as shown in Fig 8(c). Partial recovery observed in position B is a result of a combination of the coarse and the fine precipitates.
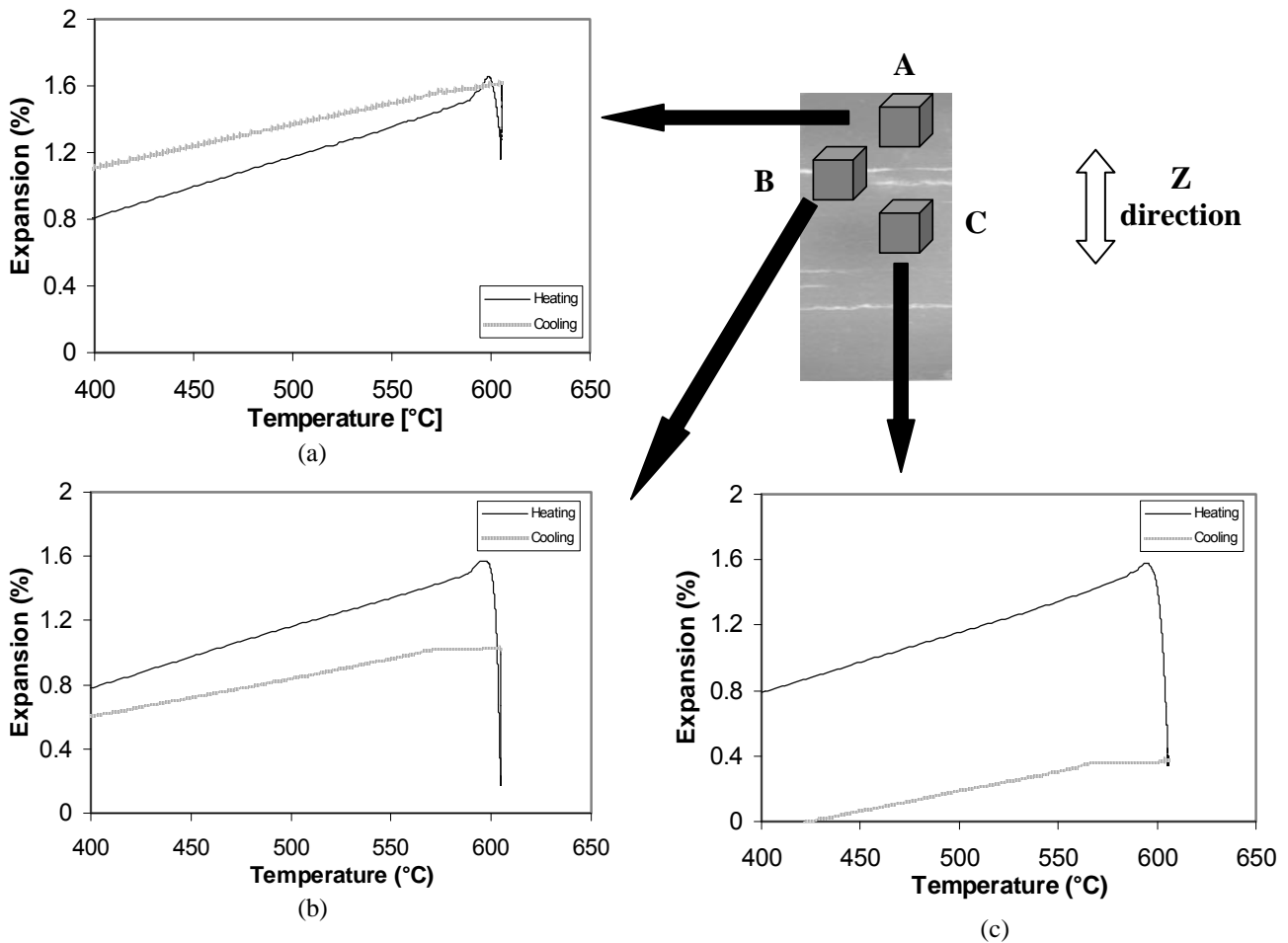
Fig 8 TMA curves along the slab thickness for positions A, B and C respectively.
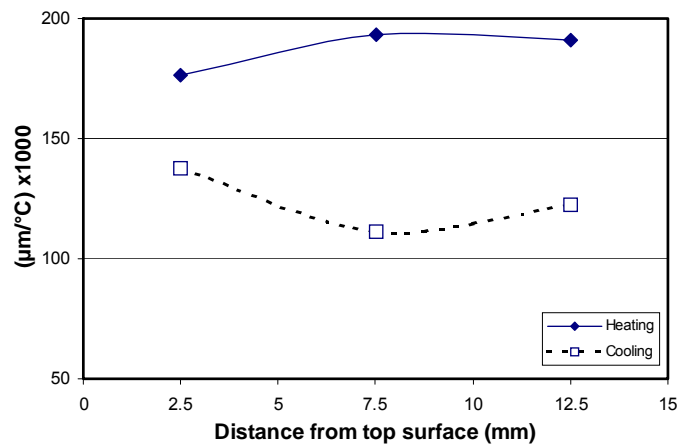
**Fig 9 Variation of the expansion coefficients
with distance from the slab surface.**

Reasons for crack localization

Crack localization in the one-quarter position of the as-rough-rolled plate thickness can be explained with the help of the previous work [12]. The author showed that during rolling the maximum shear strain generally occurs very near to one quarter of the slab thickness depending on the strip geometry and the roll diameter. High shear strain parallel to the rolling direction aids retention of primary precipitates of relatively large size that act as preferred nucleation sites for recrystallization [13]. During heating, new recrystallized grains are formed beside the coarse precipitates. As a result, the one-quarter position accommodates a thin layer parallel to the rolling plane with coarse precipitates and high-density grain boundaries. Therefore, the probability of having a transforming coarse particle in these boundaries and consequently the initiation of the cracks is much larger in this region. Moreover, the difference between the coefficients of expansion during heating and cooling is a maximum at this position as shown in Fig 9. Thus, the residual stresses created in the one-quarter position of the slab after quenching together with the high probability of initiating grain boundary melting act to enhance the formation of cracks in the same position.

CONCLUSION

As a result of the presented previous investigations, the crack phenomenon can be explained as follows: During heating, there are two processes taking place at the same time 1) dissolution of the fine secondary particles over a very wide temperature range (P1 in Fig 6) and 2) transformation of the coarse constituents $Al_6(Mn,Fe)$ and $Al_{13}(Mn,Fe)_4$ to the α phases ($Al_{12}(Mn,Fe)_3Si$ or $Al_{15}(Mn,Fe)_3Si_2$) (P2 in Fig 6). The transformation process is an exothermic reaction so that having the transforming particles on the grain boundary initiates grain boundary melting. Continuing the heating process or holding the material at constant temperatures for longer times accelerates melting. With capillary action, the molten material spreads along the grain boundaries and enhances the crack formation.   During the rough rolling process, maximum shear strain is expected in the one-quarter position of the slab. This strain accelerates the recrystallization process in this particular position and increases the grain boundary density. Cracks are always formed first in this region

because of the high probability of the grain boundary melting during the transformation of the coarse intermetallic particles and the high residual stress created from the differences observed in thermal expansion coefficients.

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