

INFLUENCE OF COLD-ROLLING DEFORMATION AND ANNEALING TEMPERATURE ON THE GRAIN GROWTH OF Al-4%Mg-Mn ALLOY

VPLIV DEFORMACIJE MED HLADNIM VALJANJEM IN TEMPERATURO ŽARENJA NA RAST ZRN ZLITINE Al-4%Mg-Mn

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The propensity of an alloy toward an abnormal grain growth (AGG) severely limits the annealing temperature range, capability for hot forming, and deteriorates mechanical properties. In this work, the results of a study of the effect of thermo-mechanical processing (TMP) of Al-Mg alloy AA5182 on the AGG are reported. TMP included cold rolling with a reduction range of 40–85 % followed by isochronal annealing (1 h) in a temperature range of 350–520 °C or isothermal treatment at 480 °C for different periods of time. The microstructure was characterized using optical microscopy in polarized light and an FEG SEM. The results showed that an increase in the degree of reduction lowers the annealing temperature for the onset of the AGG, while an increase in the annealing temperature increases the extent of the AGG for a given cold reduction. The abnormal grains tended to form in the region close to the surface of the plates, and with extended annealing bands, developed parallel to the surfaces in the rolling direction. However, no appreciable grain growth occurred in the plate center. The AGG and grain-boundary mobility showed a strong anisotropy with a much faster lateral growth than the motion of the growth front normal to the plate surface. Such anisotropy was attributed to the rod-like shape and alignment of Al₆Mn dispersoids through Zener pinning.

Keywords: abnormal grain growth, thermo-mechanical processing, Al-Mg-Mn alloy

Nagnjenost zlitin na osnovi aluminija k abnormalni rasti kristalnih zrn (AGG) močno omejuje temperaturno območje, v katerem se lahko izvaja njeno rekristalizacijsko žarjenje in sposobnost za njeno preoblikovanje v vročem, ker vse skupaj poslabšuje njene mehanske lastnosti. V prispevku avtorji predstavljajo študijo vpliva termo-mehanske obdelave (TMP) na abnormalno rast kristalnih zrn Al-Mg zlitine vrste AA5182. TMP je bila sestavljena iz hladnega valjanja s stopnjo redukcije med 40 % in 85 % in eno-urnega izohronnega žarjenja v temperaturnem območju med 350 °C in 520 °C ali različno dolge izotermne obdelave pri 480 °C. Mikrostrukturo zlitine so preiskovali z optičnim mikroskopom v polarizirani svetlobi in v FEG SEM. Rezultati kažejo, da povečana stopnja redukcije znižuje temperaturo žarjenja za začetek AGG medtem, ko zvišanje temperature žarjenja povečuje obseg AGG za dano stopnjo redukcije. Abnormalna kristalna zrna nastajajo pod površino plošč in s podaljševanjem časa žarjenja nastajajo njihovi trakovi vzporedno s površino v smeri valjanja. Vendar pa niso zaznali znatne rasti zrn v sredini plošče oz. vzorca. AGG in mobilnost mej kristalnih zrn sta močno anizotropni z mnogo hitrejšo bočno rastjo kot je hitrost rasti čela pravokotno na površino plošče. Takšno anizotropijo lahko pripišemo Zenerjevemu pripenjanju usmerjenih paličastih izločkov Al₆Mn.

Ključne besede: abnormalna rast zrn, termo-mehanska obdelava, zlitina na osnovi Al-Mg-Mn

1 INTRODUCTION

Grain growth was the subject of a copious studies for over half a century, but there is still an ongoing research interest in the fundamental aspects of the phenomenon as well as in its practical aspect realized through the development of new processing routes.¹⁻⁵ Some key issues remain unresolved, one of them being the initiation and mechanisms of abnormal grain growth (AGG)¹. AGG represents a development of the microstructure of large grains surrounded by finer, i.e., bimodal grains. It is generally accepted that in metals, AGG takes place when a normal grain growth is inhibited by particles, texture or surface effects, and certain grains have a growth advantage over the neighboring grains.⁶ AGG is unwanted as it severely deteriorates the mechanical pro-

erties of alloys, with the exception of Fe-Si alloys, used for electrical applications.⁶ The propensity of an alloy toward AGG at certain temperatures severely limits the annealing-temperature range as well as the capability of an alloy for hot forming.⁷

In spite of the susceptibility of aluminum alloys to AGG, the number of studies on the conditions for its occurrence is limited.⁸⁻¹⁵ Studies of particle-containing Al-Mg-Mn,^{8,9} Al-Mn¹⁰ and Al-Cu¹¹ alloys showed that the occurrence of AGG strongly depends on the annealing temperature. It takes place at temperatures sufficiently high to provide for the grain-boundary mobility, but below the solvus, and at which the volume fraction of pinning particles is small. Though diminishing Zener pinning is believed to have a critical role,⁸⁻¹¹ some of the studies showed that the texture and special grain boundaries might play important parts.^{8,10} The texture importance is stressed in the studies on

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low-alloyed Al-alloys with an insignificant dispersoid fraction^{12–14}. The AGG in AA5182 is reported to take place at temperatures above 480 °C, albeit with different kinetics.^{8,9} According to both studies, dispersoid coarsening and dissolution were the causes of the AGG, whereas its initiation was attributed to the size advantage of the grains with a certain texture.⁸

The aim of this study was a systematic investigation of the influence of cold work and annealing temperature on the occurrence of AGG. Due to the detrimental effect of AGG on mechanical properties, a strategy for avoiding it during high-temperature annealing was examined.

2 EXPERIMENTAL PART

The material used for the study was a commercial AA5182 aluminum alloy provided by Impol Seval Aluminium Mills. The industrial processing of the alloy, whose chemical composition is shown in **Table 1**, included DC casting, homogenization at 550 °C and hot rolling.

Thermo-mechanical processing (TMP) of the as-received hot band included cold rolling up to reductions of 40–85 % followed by annealing at 350–520 °C for 1 h. In addition to the isochronal annealing, specimens cold-rolled to a 64-% reduction were isothermally annealed at 480 °C for different periods; the exact conditions are shown in **Table 2**.

The microstructure of the as-received and thermo-mechanically processed material was characterized with optical microscopy in polarized light and scanning electron microscopy using a FEG SEM Tescan Mira at 20 kV. Specimens in the RD-ND orientation were mechanically polished and electrolytically etched in Barker's reagent. Composite images of large specimen areas were created by digitally stitching together individual micrographs. Image processing and an analysis were conducted using the ImageJ software.

3 RESULTS

The study of the effect of the annealing temperature on recrystallization and grain growth showed that annealing up to 450 °C resulted in a recrystallized microstructure with equiaxed grains. There is a grain size gradient through the plate thickness: finer grains are closer to the surface and coarser grains are in the middle of the plate. A very small, if any, change in the grain size

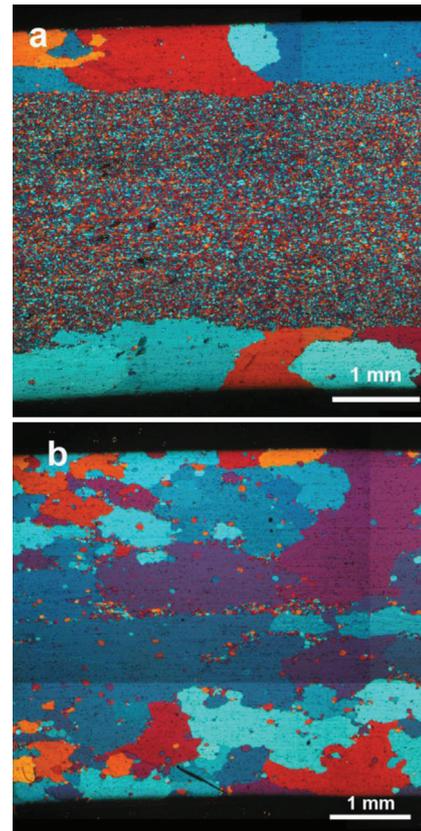


Figure 1: Effect of the temperature on AGG during isochronal annealing (1h): a) 470 °C, b) 520 °C

Table 1: Chemical composition of the AA5182 alloy (w/%)

| Mg | Mn | Si | Fe | Ti | Cu | Zn | Cr | Zn | Other |
|------|-------|--------|-------|--------|-------|--------|-------|--------|--------|
| 4.04 | 0.371 | 0.0732 | 0.186 | 0.0019 | 0.011 | 0.0397 | 0.011 | 0.0223 | 0.0082 |

Table 2: Annealing conditions for the specimens cold rolled to a 64-% reduction

| Isochronal (1 h) t, °C | 350 | 400 | 440 | 470 | 480 | 500 | 520 |
|----------------------------------|-----|-----|-----|-----|-----|-----|-----|
| Isothermal (480 °C) τ , min | 0 | 5 | 15 | 30 | 45 | 60 | 180 |

Table 3: Grain-size and shape parameters of abnormal grains after isochronal annealing (1 h) in the temperature range of 470–520 °C

| Ann. T, °C | Normalized area of AGG*, mm ² /mm | Eq. diameter, μ m | Roundness | Aspect ratio |
|------------|--|-----------------------|-----------|--------------|
| 470 °C | 0.72 | 443 | 0.53 | 2.3 |
| 480 °C | 1.63 | 743 | 0.45 | 2.7 |
| 500 °C | 2.03 | 703 | 0.47 | 2.5 |
| 520 °C | 2.2 | 435 | 0.50 | 2.4 |

*The normalized area was calculated as the area covered by abnormal grains per unit length in RD. It corresponds to the mean length of the propagation front of AGG in ND.

and shape was observed in the specimens annealed at a temperature of 350–450 °C. However, an increase in the annealing temperature to 470 °C gave rise to AGG (**Figure 1a**). Here, we have a sandwich-like microstructure with a strip of fine recrystallized grains between the two bands of abnormally large grains next to the plate surfaces parallel to the rolling direction (RD).

The area transformed by AGG increased with a further increase in the annealing temperature, but the mean size of abnormal grains showed a more complex behavior. The initial increase in the size was followed by a decline since more abnormal grains were nucleated as the temperature rose (**Table 3**). Abnormal grains formed throughout the specimen at 520 °C (**Figure 1b**).

The microstructure upon reaching 480 °C, the temperature of the isothermal-annealing experiments, showed recrystallized, equiaxed grains without evidence of

AGG. After only 5 min of the annealing, grains larger than average were observed in the region up to 700 μm from the plate edge (**Figure 2a**). The smallest of the incipient abnormal grains tend to have a more equiaxed shape while the larger grains are elongated (**Figure 2a**), the tendency confirmed by the size-shape distribution plot (**Figure 2b**). The abnormal grains remain elongated after forming a continuous band (**Figure 2c**), with the longer axis parallel to RD and the mean aspect ratio increasing from ≈ 2 to ≈ 2.7 .

Apparently, the growth rate and grain-boundary mobility are anisotropic, i.e., the grain-boundary migration is faster in the direction parallel to the RD than in ND. If the only force influencing the grain growth decreases in the interface area, i. e. capillary force, such a trend would be unexpected; the migration of the boundaries of the abnormal grains in the ND decreases the total grain-boundary area since the smaller grains are consumed, while the motion of the boundaries in the RD leaves the total boundary area effectively unchanged until a complete collapse of the intermediate grains. It is likely that the observed anisotropy of the grain mobility is caused by the shape and anisotropic alignment of the Al_6Mn particles (**Figure 3**) that can limit the mobility through Zener pinning.¹⁶

In contrast to the dramatic changes to the outer regions during annealing, the grain size in the center is steadier, with the mean grain size changing from 14.5 μm after 5 min to 17.7 μm after 1 h at 480 °C.

In addition to the annealing temperature, the propensity toward abnormal grain growth strongly depends on the cold-rolling reduction as illustrated in **Figure 4**. The specimen annealed at 480 °C for 1 h, having undergone a 40-% reduction, exhibited recrystallized equiaxed grains (**Figure 4a**), but an increase in the reduction to 50 % resulted in a few abnormal grains in the microstructure near the plate surface (**Figure 4b**). A further increase in the reduction to 65 %, under the same annealing conditions, gave rise to two bands of abnormal grains (**Figure 4c**). In the case of a cold

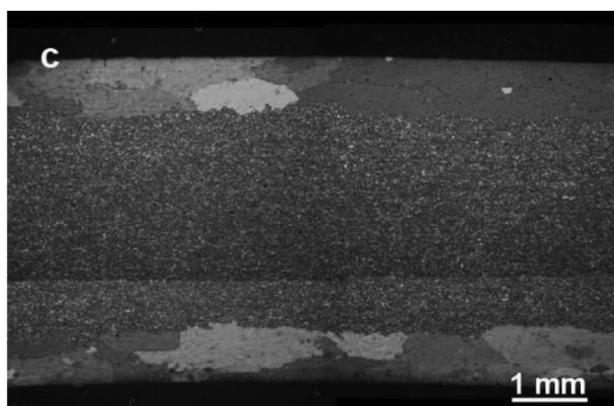
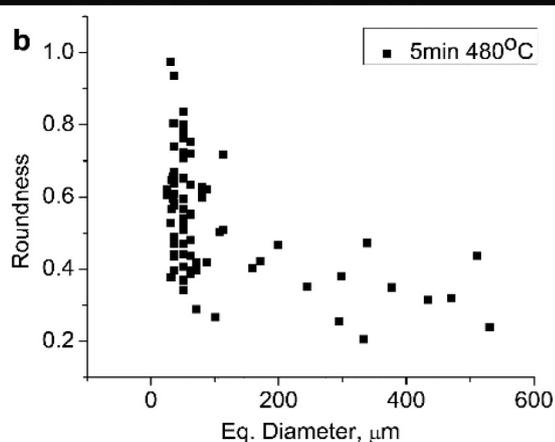
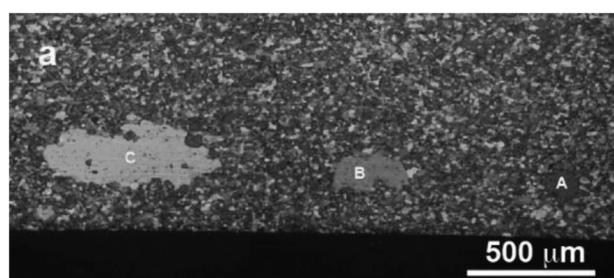


Figure 2: Effect of the annealing time on AGG during annealing at 480 °C: a) 5 min, b) size (eq. diameter) vs shape (roundness) of abnormal grains after 5-min annealing, c) 15-min annealing

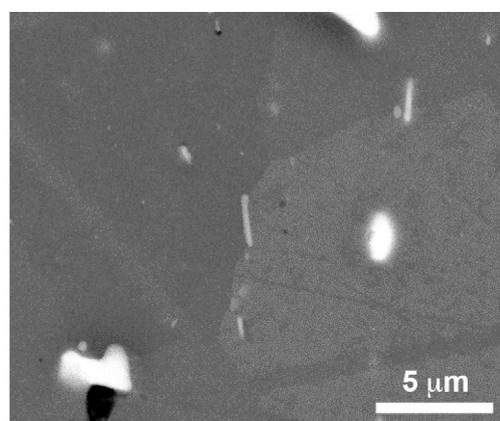


Figure 3: Rod-like Al_6Mn particles are more efficient at pinning grain boundaries in the RD due to the longer axis aligned parallel to the RD (5 min/480 °C)

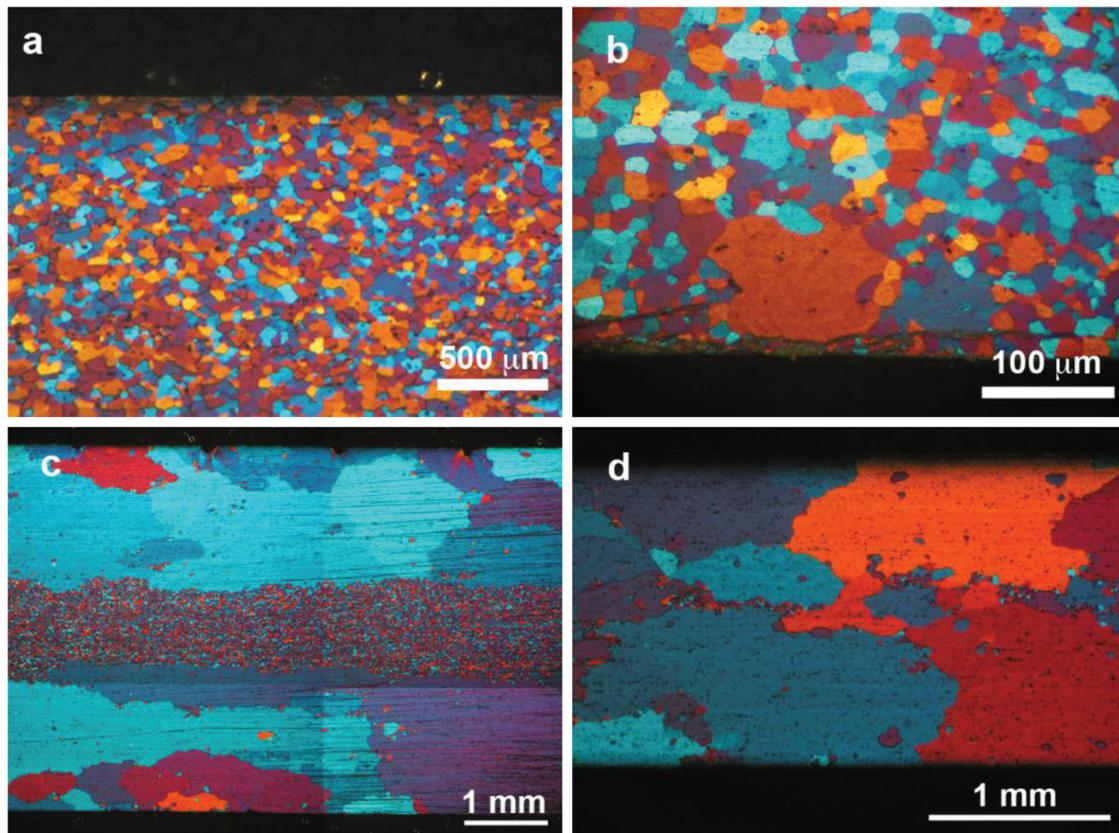


Figure 4: Effect of the reduction on the grain growth during annealing at 480 °C for 1 h: (a) 40 %; (b) 50 %; (c) 64 %; (d) 85 %

reduction of 85 %, abnormal grains covered the plate, although some finer grains were left behind (**Figure 4d**). There is an inverse relationship between the reduction degree and the onset temperature for AGG: the temperature of 440 °C is required for the reduction of 85 %, while lowering the reduction to 40 % requires 500 °C for

the start. Clearly, an increase in the reduction degree promotes AGG. Furthermore, as the reduction increases, incipient abnormal grains form deeper, close to the plate center. For example, abnormal grains are confined to the region near the surface when deformed by 50 % (**Figure 4b**), but after a 64-% reduction, they form as far as 1.5 mm from the edge.

The complexity of the factors inducing AGG is demonstrated by a two-stage-annealing experiment. In contrast to single-stage annealing at 480 °C (**Figure 4c**), low-temperature annealing for 48 h at 220 °C followed by high-temperature annealing for 1 h at 480 °C gave rise to a microstructure without an abnormal grain growth (**Figure 5**).



Figure 5: AGG is absent after a 65-% deformation and two-stage annealing: 48 h/220 °C + 1 h/480 °C

4 DISCUSSION

The effect of the cold-rolling reduction and annealing temperature on the grain growth is summarized in the plot of **Figure 6**.

The results show that AGG can occur at a lower temperature, 440 °C, than previously reported for AA5182, ≤ 480 °C.^{8,9} The lower temperature questions the attribution of AGG to diminishing Zener pinning pressure due to the coarsening and dissolution of dispersoids since the dissolution temperature is reported to be close to 500 °C.^{8,9} The particle coarsening/dissolution is not critical for AGG and it is suggested, based on the obser-

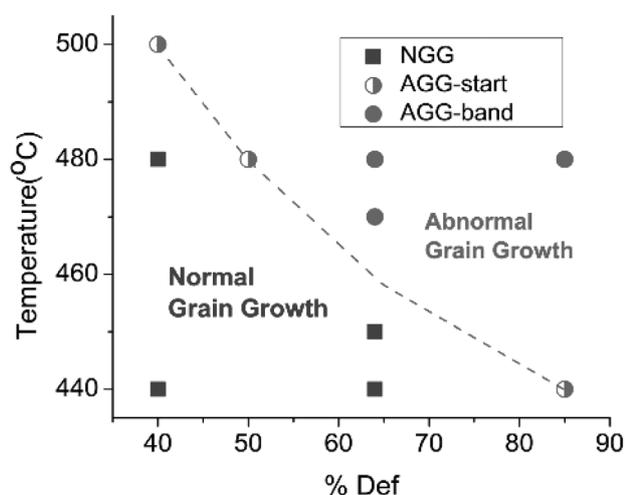


Figure 6: Effect of the TMP, degree of reduction and annealing temperature on the grain microstructure and the onset of AGG

variations, that AGG occurs after a 64-% reduction and not after a 40-% reduction under the same annealing conditions. It is evident (**Figure 6**) that the degree of cold reduction has a strong influence on the temperature of the onset of AGG. To the best of the authors' knowledge, there is no published work that investigates the effect of cold deformation on the temperature, at which AGG occurs in aluminum alloys. For a low-alloyed steel, a similar dependence of the AGG onset temperature versus the reduction degree was reported¹⁷ with an interpretation in terms of a higher dislocation density providing fast diffusion paths for a dissolution. However, in the reduction range of interest to this work, i.e., over 40 %, a saturation with dislocations is expected to be reached, so it is unlikely that a further increase in deformation would significantly boost diffusion. Indeed, even for steel, the onset temperature remains nearly constant in a reduction range of 40–75 %.¹⁷ Moreover, our characterization showed that the microstructure is already recrystallized upon reaching the annealing temperature, so the grains should be nearly dislocation free. The presence of recrystallized grains also rules out the "pinch-off" mechanism proposed by Taleff's group.⁷

The texture and special grain boundaries with a high mobility are also considered as the key factors for AGG.^{6,10} The grain-size gradient across the plate, the band of abnormal grains at the plate surface and the increasing depth of incipient abnormal grains with reduction as well as stable grains in the center suggest that the shear texture and texture gradient are responsible for the onset of AGG in this work, albeit without a texture measurement.

The suppression of AGG in the two-stage annealing experiment (**Figure 6**) highlights a possible role of subgrain boundaries. The activation of low-temperature recovery mechanisms during the annealing at 220 °C can prevent a high-temperature recovery and recrystallization due to the subgrain growth.^{5,18} As a result, during the

annealing at 480 °C, the recrystallization due to the nucleation and growth may take place allowing a more random texture⁵ and the absence of AGG. Hence, the AGG during the one-stage annealing could be a consequence of a discontinuous subgrain growth.¹⁸ The mechanism of AGG, based on energy considerations and solid-state wetting,^{19,20} assumes that low-angle subgrain boundaries of abnormal grains penetrate into the matrix by means of repeated events of triple-junction wetting. Although morphological features such as small island grains and small grain clusters within abnormal grains (**Figures 1** and **4**) resemble the morphology typical for triple-junction wetting,¹⁹ they are commonly observed during AGG^{9–11,19} and ascribed to dispersoid pinning.¹¹ Whether the critical factor for AGG is the grain-boundary mobility, i.e., grains with mobile high-angle boundaries consuming the surrounding grains with low-angle low-mobility boundaries,¹⁰ or the energy criterion and solid-state wetting²⁰ at this point remains a conjecture.

5 CONCLUSIONS

A systematic investigation of the influence of TMP parameters on the occurrence of abnormal grain growth (AGG) showed that an increase in cold deformation and annealing temperature increases the propensity toward AGG. The results show that lowering the reduction degree or two-stage annealing temperature can suppress AGG during annealing in a high-temperature range.

AGG appears to be affected by multiple factors. A confinement of incipient abnormal grains to the region close to the plate's surface and an absence of AGG in the center indicate that the initiation of AGG is controlled by the texture and grain-boundary character gradients. The anisotropy of the growth of abnormal grains is related to the shape and anisotropic alignment of Al₆Mn particles due to Zener pinning, while the absence of AGG in two-stage annealing points at the role of recovery processes.

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