

HOMOGENIZATION OF AN Al-Mg ALLOY AND ALLIGATORING FAILURE: INFLUENCE OF THE MICROSTRUCTURE

HOMOGENIZACIJA Al-Mg ZLITINE IN KROKODILJENJE: VPLIV MIKROSTRUKTURE

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In this study the influence of the microstructure of Al-5.1Mg-0.7Mn alloy on the propensity towards an alligating failure was evaluated. The morphology of the constituent particles appears to be the key factor, with dissolution and fragmentation of fibrous Mg-Si-Sr and transformation of the $Al_6(Fe,Mn)$ into more compact shapes taking place at homogenization temperatures above 520 °C, contributing to improved ductility and diminished propensity towards alligating. Homogenization at temperatures below 500 °C gave rise to a non-uniform precipitation of the dispersoids. Such a microstructure favored localized slip that induced stress concentration at the grain boundaries and triggered grain boundary embrittlement.

Keywords: Al-Mg alloy, homogenization, microstructure, localized slip, alligating

V študiji je bil ocenjen vpliv mikrostrukture zlitine Al-5,1Mg-0,7Mn na pojav krokodiljenja. Izgleda, da je morfologija delcev ključna, ker sta raztapljanje in drobljenje vlaknastih Mg-Si-Sr ter pretvorba $Al_6(Fe,Mn)$ v bolj kompaktno oblike, kar se dogaja med homogenizacijo pri temperaturah nad 520 °C, prispevala k izboljšanju duktilnosti in k odpravi krokodiljenja. Homogenizacija pri temperaturah pod 500 °C povzroči povečanje neenakomernega izločanja delcev. Taka mikrostruktura pospešuje lokalno drsenje ki povzroči koncentracijo napetosti na mejah zrn in sproži pojav interkristalne krhkosti.

Ključne besede: zlitina Al-Mg, homogenizacija, mikrostruktura, lokalno drsenje, krokodiljenje

1 INTRODUCTION

Fabrication of high strength Al-Mg sheet products requires careful design of the thermo-mechanical process to avoid hot fracture.¹ In the first part of this study, we report on the effect of homogenization temperature on failure by alligating during hot rolling of the Al-5.1Mg-0.7Mn alloy.²

Alligating has been studied mostly from the aspect of rolling process optimization¹ while microstructural factors influencing the occurrence of alligating are far less understood. However, there is evidence that the microstructure contributes to a propensity to alligating. Cold rolling of spheroidized 1020, 1045 and 1090 steels failed due to the alligating only in the last alloy, which has greatest density and length of linear inclusions.³ Another study on a steel⁴ showed that the occurrence of the alligating was influenced by the presence of MnS inclusions. The high density of coarse Mn-based constitutive particles increased the susceptibility to alligating in Al-Mg alloys.⁵

The aim of this work was to evaluate the effect of homogenization temperature and resultant microstructures on the occurrence of alligating in Al-5.1Mg-0.7Mn alloy during hot rolling experiments.

2 EXPERIMENTAL WORK

The Al-5.1Mg-0.7Mn alloy studied had higher Mg and Zn contents compared to the standard AA 5083 alloy. The exact chemical composition was given in ².

The industrial cast alloy was hot rolled in the laboratory. Prior to hot rolling, the plates were homogenized. Three homogenization regimes were applied, with a difference in the homogenization temperature. Regime I corresponded to the homogenization temperature of 460 °C, Regime II to 520 °C and Regime III to 550 °C. Details of the homogenization treatment and hot rolling procedure were given in ².

Microstructural characterization involved light microscopy and Scanning Electron Microscopy (SEM). Specimens were prepared by fine mechanical polishing of sections of interest. SEM characterization was conducted in JEOL JSM-6610LV at 20 kV equipped with an Energy-Dispersive X-ray Spectroscopy (EDS) detector. Specimens for light microscopy were examined in the as-polished state and after electrolytic etching with Barker's reagent.

3 RESULTS

3.1 Microstructure around the alligator crack tip

A cross-section of an alligator crack tip in the fractured plate interior is shown in **Figure 1a**. A number of cracks and micro-cracks was observed in its vicinity (**Figures 1** and **1b**). The micro-cracks were associated with a fracture (**Figures 1c** and **1d**) of the constituent particles. The alligator crack propagated in the intergranular manner (**Figure 2a**), but some deviations, most likely following the slip bands, were observed.

In a number of grains, bands with coloration different from the surrounding matrix were observed (**Figure 2b**). Barker's etch has a sensitivity toward changes in the crystallographic orientation, so the bands were most likely slip bands introduced by intensive localized slip. The bands were present in plates homogenized at 460 °C

(Regime I) and 520 °C (Regime II), but in the latter case they were finer and more evenly distributed.

3.2 Microstructure

Microstructure of the as-cast state consisted of constituent particles and Al matrix.⁶ Products of solidification, coarse constituent particles were mostly situated along the dendrite boundaries that, to a large extent, overlapped with grain boundaries. The constituent particles were identified as $Al_6(Fe,Mn)$, Mg_2Si and Mg-Si phase enriched in Sr (Sr is present as a trace element in the alloy)^{2,6} and $Al_6(Fe,Mn)$ phase with a Chinese script morphology. Thin, fibrous Mg-Si-Sr phase with attached rectangular Mg_2Si lined the grain boundaries. Homogenization induced rounding of the sharp facets of the constitutive particles.⁶

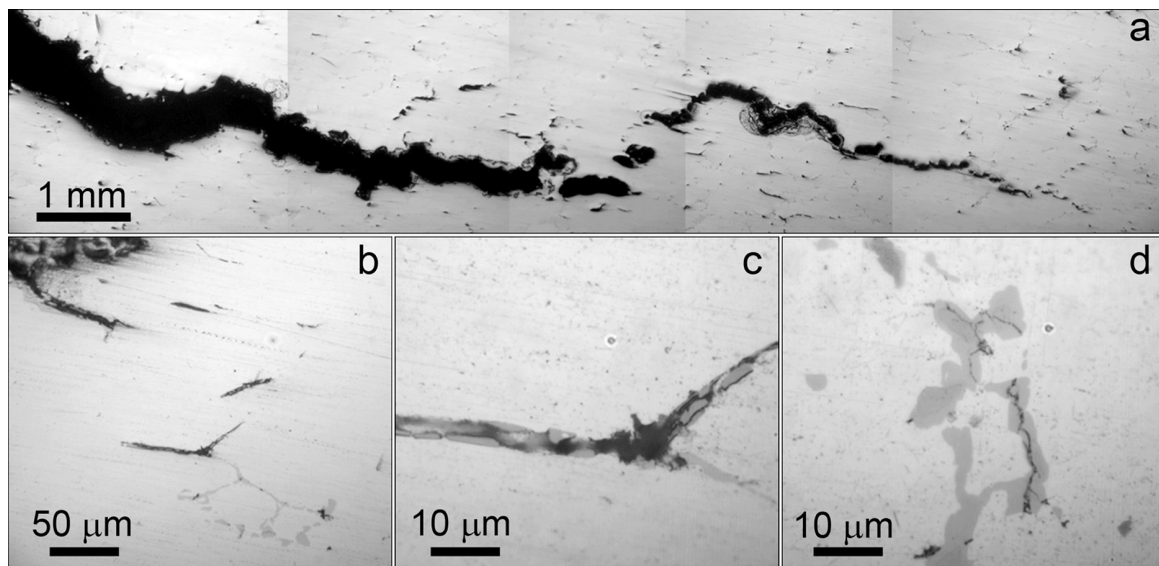


Figure 1: Light micrographs of: a) the tip of alligator crack, b) to d) microcracks and fractured constitutive particles in a plate homogenized at 460 °C

Slika 1: Svetlobni posnetki: a) vrh krokodilje razpoke, od b) do d) mikrorazpoke in prelom delcev v plošči, homogenizirani pri 460 °C

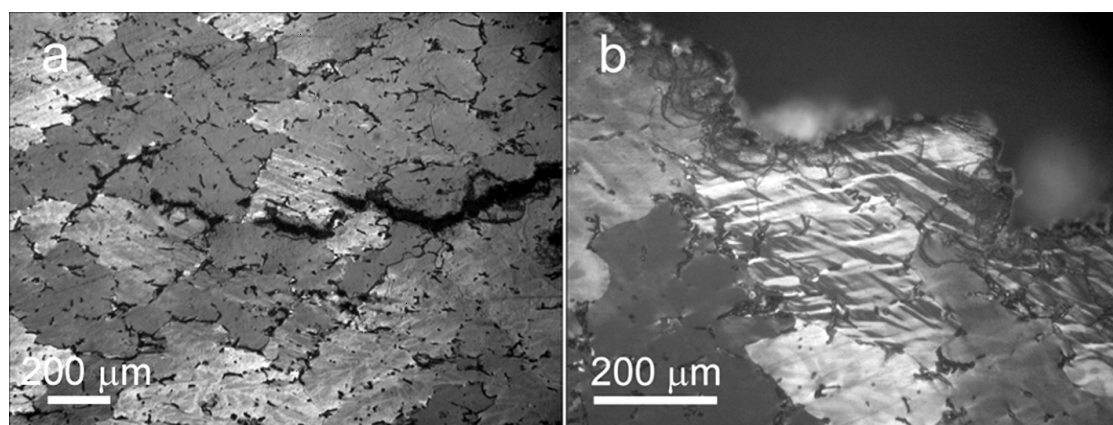


Figure 2: Light micrographs of the plates etched with Barker's reagent: a) intergranular propagation of the alligator crack (homogenization at 520 °C - Regime II), b) slip bands (homogenization at 460 °C - Regime I)

Slika 2: Svetlobna posnetka plošče, jedkane z Barker jedkalom: a) napredovanje aligatorske razpoke med zrni (homogenizacija pri 520 °C - Režim II), b) trakovi drsenja (homogenizacija pri 460 °C - Režim I)

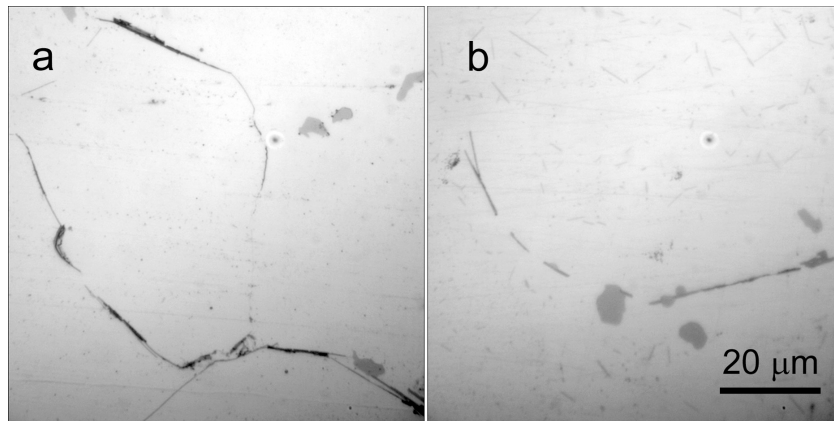


Figure 3: Light micrographs of the fibrous Mg-Si-Sr/Mg₂Si constitutive particles: a) 460 °C (Regime I), b) 550 °C (Regime III)
Slika 3: Svetlobna posnetka vlaknastih delcev Mg-Si-Sr/Mg₂Si: a) 460 °C (Režim I), b) 550 °C (Režim III)

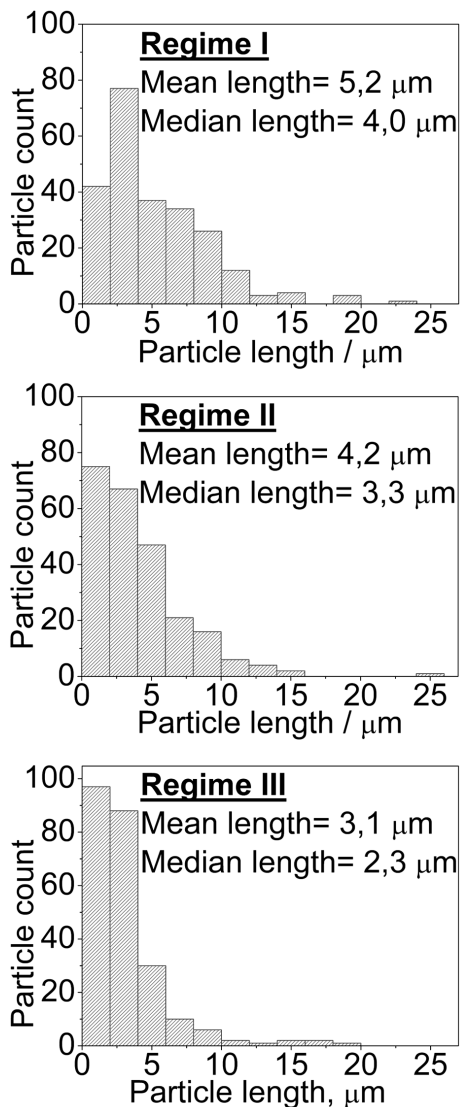


Figure 4: Size distribution (length) of fibrous Mg-Si-Sr constitutive particles. The total number of measured particles (240), was the same for each histogram.

Slika 4: Razporeditev velikosti (dolžine) vlaknastih Mg-Si-Sr delcev. Celotno število izmerjenih delcev (240), je bilo enako za vsak histogram

During homogenization at 460 °C (Regime I), growth and precipitation of Mg₂Si phase took place at interfaces of the constitutive particles with the aluminum matrix (**Figure 3a**).

At homogenization temperatures above 500 °C, Mg-Si rich phases appear to dissolve as the fibers shortened (**Figure 4**). Clusters of the fibers delineating grain boundaries were still present after the homogenization at 520 °C (Regime II), but had almost completely vanished

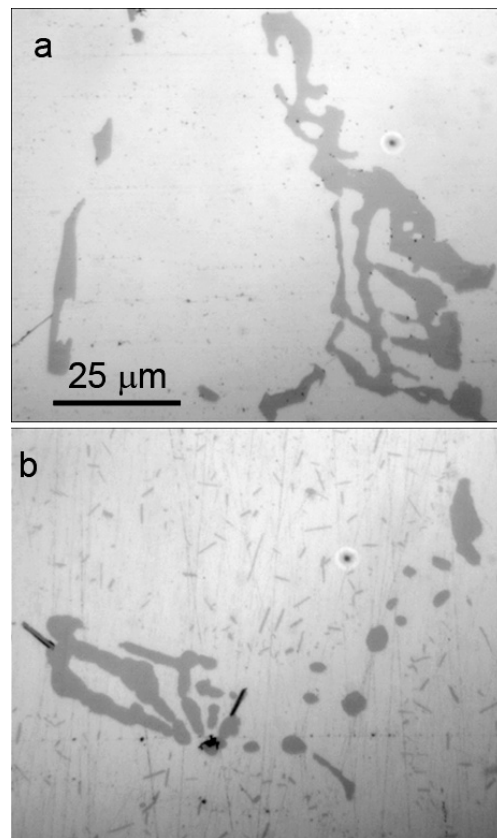


Figure 5: Light micrographs of the Al₆(Fe,Mn) constitutive particles: a) 460 °C (Regime I), b) 550 °C (Regime III)

Slika 5: Svetlobna posnetka delcev Al₆(Fe,Mn): a) 460 °C (Regime I), b) 550 °C (Regime III)

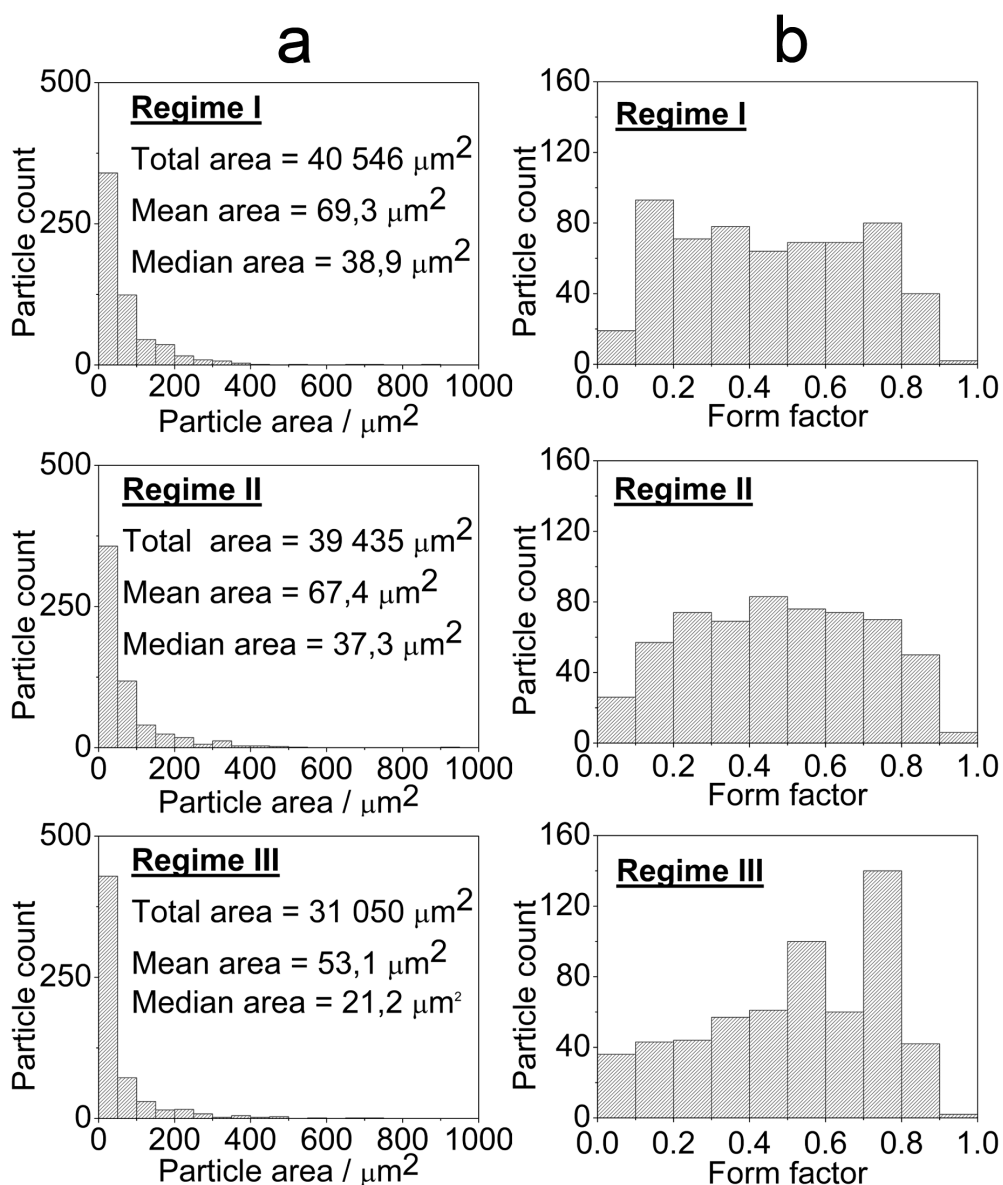


Figure 6: a) Size and b) form factor distribution of $\text{Al}_6(\text{Fe},\text{Mn})$ constitutive particles. The form factor is defined as $F = 4\pi \cdot \text{Area} / \text{Perimeter}^2$ and reflects the particle shape. It is equal to 1 for a perfect circle and < 1 for less regular shapes. The total number of measured particles (585), was the same for each histogram.

Slika 6: a) Velikost in b) faktor oblike razporeditve delcev. Faktor oblike je določen kot: $F = 4\pi \cdot \text{površina} / \text{obseg}^2$ in odraža obliko delcev. Enak je 1 pri popolnoma okroglih delcih in je < 1 pri manj pravilnih oblikah. Celotno število delcev (585), je bilo enako pri vseh histogramih.

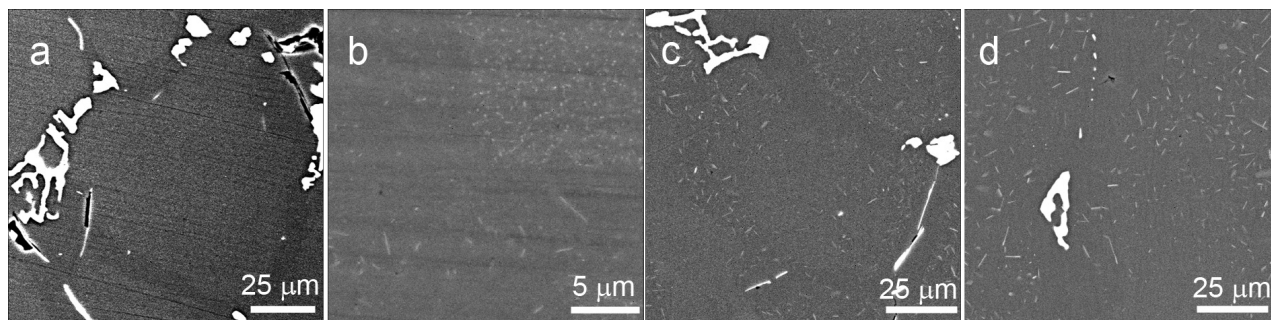


Figure 7: Backscattered SEM electron images of the microstructure after the homogenization anneal: a), b) 460 °C (Regime I), c) 520 °C (Regime II) and d) 550 °C (Regime III)

Slika 7: SEM-posnetek, s povratno sipanimi elektroni, mikrostrukture po homogenizacijskem žarjenju: a), b) 460 °C (Režim I), c) 520 °C (Režim II) in d) 550 °C (Režim III)

after homogenization at 550 °C (Regime III) while the few remaining fibers were fragmented (**Figure 3b**).

Annealing at higher temperatures (Regimes II and III) resulted in a partial dissolution and break down of the branches of the $Al_6(Fe,Mn)$ phase to more compact forms. The process was particularly advanced after the homogenization at 550 °C (Regime III) as can be seen from **Figures 5** and **6**.

Homogenization in a temperature range of 430–500 °C resulted in the precipitation of dispersoids in the dendrite cores (**Figure 7a**). Dendrite cores, with a high density of dispersoids, were surrounded by channels that were almost dispersoid free (**Figure 7b**). The width of the channels was in the range of 4–25 μm .

Precipitation of coarser, rod- and plate-shaped Al-Mn-Fe particles took place during homogenization above 500 °C. The precipitation occurred in the "precipitate free" channels formed during the first homogenization stage (annealing at 430 °C) of Regimes II and III. After homogenization at 520 °C (Regime II), the distinction between the regions was preserved (**Figure 7c**). The homogenization at 550 °C (Regime III) resulted in more uniform precipitation of coarser Al-Mn-Fe particles (**Figure 7d**).

Cooling down to the hot rolling onset temperature (500 °C) caused further precipitation of dispersoids.

4 DISCUSSION

Non-heat treatable Al-Mg alloys contain two types of the particles, dispersoids and constitutive particles. The domination of ductile intergranular fracture in the alligatored plates points out the important role of constitutive particles in the failure. The tendency toward break-up of the constitutive particles is dependent on their morphology, i.e. complex and plate-like shapes are more prone to break-up than compact morphologies.⁷ In this study, the increase in the homogenization temperature led to changes in the constitutive particles that improved the ductility of the alloy. Thin plates of Mg-Si-Sr phase enveloping grains comprised the majority of the broken particles filling the dimples of the fractured plates which had been homogenized at 460 °C (Regime I). Their fraction decreased with a partial dissolution at 520 °C (Regime II). Homogenization at the highest temperature, 550 °C, lead not only to almost complete dissolution of the Mg-Si-Sr phase, but also to a change in the size and shape of the $Al_6(Fe,Mn)$ phase. $Al_6(Fe,Mn)$ transformed into more compact morphologies (**Figures 5** and **6**) resulting in an alloy with a good hot ductility that did not alligator during the hot deformation.

The primary function of the other type of particles in the alloy, dispersoids, is to limit grain growth during hot deformation. It is also known that they can contribute to the homogenization of slip⁸ and to reduction of the tendency toward intergranular embrittlement in Al-Mg-Si alloys.^{9,10} However, in the studied alloy, homogenization

below 500 °C resulted in non-uniform precipitation of the dispersoids and formation of broad precipitate free channels (**Figure 7b**). Such a microstructure strongly promoted localized slip. Impingement of the dislocation pile-ups at the grain boundaries or large constituent particles could create significant local stresses triggering grain boundary decohesion and embrittlement² as well as break-up of the constituent particles (**Figure 1**). Absence of the brittle intergranular fracture in the plates homogenized at 520 °C (Regime II)² indicates that precipitation of the rod-like dispersoids in the "precipitate free" channels decreased the extent of the slip localization that was the cause of the grain boundary decohesion.

Apparently, homogenization affected both types of particles present in the Al-5,1Mg-0,7Mn alloy. The increase in the homogenization temperature led to a more uniform distribution of the dispersoids and, hence, more uniform slip, as well as the reversal of the morphology of the constituent particles toward a more break-up resistant shape. The result was that the microstructure developed during the homogenization Regime III showed high resistance toward intergranular fracture and the absence of alligating.

5 CONCLUSION

The microstructure of the plates homogenized at 460 °C (Regime I) with well-defined precipitate rich and free zones promoted localized slip and inhomogeneous deformation resulting in brittle and ductile intergranular fracture. Precipitation of rod-like dispersoids into the precipitate free channels during homogenization at 520 °C (Regime II) contributed to the more uniform distribution of slip and only the ductile variant of intergranular fracture was observed. During homogenization at 550 °C (Regime III), fairly uniform distribution of the dispersoids, dissolution of Mg-Si-Sr phase and reversal of $Al_6(Fe,Mn)$ constituent particles toward more compact morphology contributed to the development of a ductile material that did not show a proclivity toward alligating.

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6 REFERENCES

- ¹ J. G. Lenard, Workability and Process Design in Rolling, In: G. Dieter, H. A. Kuhn, S. L. Semiatin (eds.), Handbook of Workability and Process Design, ASM International, Materials Park, Ohio, USA 2003, 258–277, doi:10.1361/hwpd2003p258
- ² E. Romhanji, T. Radetić, M. Popović, Homogenization of an Al-Mg alloy and alligating failure Part I: alloy ductility and fracture, Mater. Tehnol. 50 (2016) 3, 403–407, doi:10.17222/mit.2015.110

- ³ L. Xu, G. S. Daehn, Alligatoring and Damage in the cold rolling of spheroidized steels, *Metallurgical and Materials Transactions A*, 25 (1994) 3, 589–598, doi:10.1007/BF02651600
- ⁴ H. Kim, M. Kang, S. Y. Shin, S. Lee, Alligatoring phenomenon occurring during hot rolling of free-machining steel wire rods, *Materials Science and Engineering A*, 568 (2013), 8–19, doi:10.1016/j.msea.2013.01.018
- ⁵ M. R. Cappucci, An investigation of the workability of Al-8.5% Mg alloys, Report MTL TR 89-33, U.S. Army Materials Technology Laboratory, Watertown, Massachusetts, <http://www.dtic.mil/cgi-bin/GetTRDoc?AD=ADA209451>
- ⁶ A. Halap, T. Radetić, M. Popović, E. Romhanji, Study of homogenization treatments of cast 5xxx series Al-Mg-Mn alloy modified with Zn, In: C. E. Suarez (Ed.), *Light Metals 2012*, John Wiley & Sons, Inc., Hoboken, NJ, USA 2012, 387–392, doi:10.1002/9781118359259.ch65
- ⁷ N. Moulin, E. Parra-Denis, D. Jeulin, C. Ducottet, A. Bigot, E. Boller, E. Maire, C. Barat, H. Klöcker, Constituent particle break-up during hot rolling of AA 5182, *Advanced Engineering Materials*, 12 (2010) 1–2, 20–29, doi:10.1002/adem.200900241
- ⁸ J. M. Dowling, J. W. Martin, The influence of Mn additions on the deformation behaviour of Al-Mg-Si alloy, *Acta Metallurgica*, 24 (1976) 12, 1147–1153, doi:10.1016/00016160(76)90032-8
- ⁹ K. C. Prince, J. W. Martin, The effect of dispersoids on micro-mechanisms of crack propagation, *Acta Metallurgica*, 27 (1979) 8, 1401–1408, doi:10.1016/0001-6160(79)90209-8
- ¹⁰ J. D. Evensen, N. Ryum, J. D. Embury, The intergranular fracture of Al-Mg-Si alloys, *Materials Science and Engineering*, 18 (1975) 2, 221–229, doi:10.1016/0025-5416(75)90173-1