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# MICROSTRUCTURE EVOLUTION OF THE HOT-ROLLED MODIFIED AA 5083 ALLOYS DURING THE TWO STAGE THERMAL TREATMENT

Tamara Radetić[1 *], Miljana Popović[1], Endre Romhanji[1], Branislava Milović[2], Radomir Dodok[2]

[1]*Department of Metallurgical Engineering, Faculty of Technology and Metallurgy, University of Belgrade, Karnegijeva 4, Belgrade, Serbia*
[2]*IMPOL SEVAL Rolling Mill, Prvomajska bb, Sevojno, Serbia*

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## Abstract

The effects of the two stage heat treatment on the structure of two hot-rolled 5083 aluminum alloys without and with 0.12% Zr addition were investigated. Combination of the metallographic and transmission electron microscopy (TEM) was carried on in order characterize grain structure as well as precipitates type and morphology. During the homogenization annealing recovery processes and grain structure polygonization were accomplished. Further annealing at the 460°C had little effect on the grain morphology. Two populations of the Mn-based secondary phases were identified: coarser particles containing Fe, while the Mn particles alloyed with Cr were finer. Zr did not precipitate in a form of the fine, coherent precipitate. However, in the alloy containing Zr the recovery appear to be easier and more advanced than in the other alloy.

*Key words: Al alloys, pancake structure, continuous recrystallization*

## Introduction

An exploitation of aluminum and aluminum alloys in marine applications has a long history. Recent research and development in the field have been directed toward increase of a ratio between strength and specific weight of the alloys as well as at increasing a corrosion resistance and strength of a weld. Such requirements make the AA 5083 alloy particulary attractive in marine applications. This alloy drives its

* Corresponding author: Tamara Radetić, tamara.m.radetic@gmail.com

strength from the solution strengthening due to the Mg [1]. Other alloying elements, like Mn, Cr, Zr are added to control grain size and stability of the grain structure [1, 2], or like a Zn to improve corrosion resistance [3].

In this work we investigated the effect of the two-stage thermal treatment on the micrustructure of the industrially hot-rolled commercial AA 5083. In order to evaluate an effect of Zr on the recrystallization and grain size ruination, we characterized two batches of the AA 5083: one with and another one without Zr addition.

## Experimental procedure

Alloys used for this study correspond to an AA designation 5083; however one of the alloys had Zr addition. Compositions of the alloys are given in the Table 1.

The materials were supplied by IMPOL SEVAL Company in the form of 10.2 mm and 11.3 mm plates for the alloy without and with Zr addition, respectively. The alloys were fabricated by DC casting at 680±5°C followed by the two-stage homogenization annealing. The annealing of the cast blocks at 430˚C for 14 h was conducted in order to gradually dissolve Mg-based eutectics. From 430°C the blocks were heated to 540±10°C and further homogenized for 12h. The hot rolling was conducted in a four-high reverse rolling mill in the temperature range starting at 500°C and ending at 300°C.

*Table 1. Chemical composition of the studied AA 5083 alloy, wt. %.*

| Mg | Mn | Cu | Fe | Si | Zn | Cr | Na | Ti | Zr |
|---|---|---|---|---|---|---|---|---|---|
| 4.43 | 0.80 | 0.013 | 0.288 | 0.099 | 0.144 | 0.11 | 0.0003 | 0.0078 | 0.0007 |
| 4.85 | 0.77 | 0.0078 | 0.3 | 0.113 | 0.120 | 0.106 | 0.0003 | 0.0073 | *0.121* |

In order to investigate the effect of the thermal processing and a small amount of Zr addition on the microstructure evolution, the specimens from the industrially hot rolled plates were further heat treated and characterized in the laboratory. The heat treatment was conducted in an air-circulating furnace and involved two stages: homogenization for 6h at 565°C was followed by a furnace cooling to 460°C and annealing for 6h.

Microstructural characterization was carried on by an optical and transmission electron microscopy (TEM). Planar orientation of the specimens for the microstructural characterization corresponded to a longitudinal-transverse (LT) plane. The primary objective of metallography was to establish the degree of recrystallization and determine the as-recrystallized grain size. The specimens were mechanically polished and electropolished and etched in Barker's reagent. Specimens for TEM were mechanically thinned to 100 μm and then electropolished in $CH_3OH:HNO_3=3:1$ solution in Fishione TwoJet Polisher. The electropolishing conditions were U=10V, I=21mA and T=-35°C. TEM characterization and microanalysis were conducted in a JEOL 200CX and Philips CM200 microscopes at 200kV.

## Results and Discussion

The microstructure of the hot rolled 5083 alloy studied in this investigation is shown in Figure 1. In the plate, non-uniform grain structure perpendicular to the rolling direction was developed (Figure 1a). Fine, polygonal grains are observed in regions close to the plate edge, however the central part of the plate is characterized by elongated, recovered grains aligned in the bands separated by the highly deformed lamellar grains parallel to the rolling direction. Such non-uniform structure can be related to the non-homogeneous deformation characteristic for rolling as well as the processing conditions. The hot-rolling was carried on in the temperature range 500-300ºC, so the temperature of a final reduction was not sufficient to complete recrystallization.

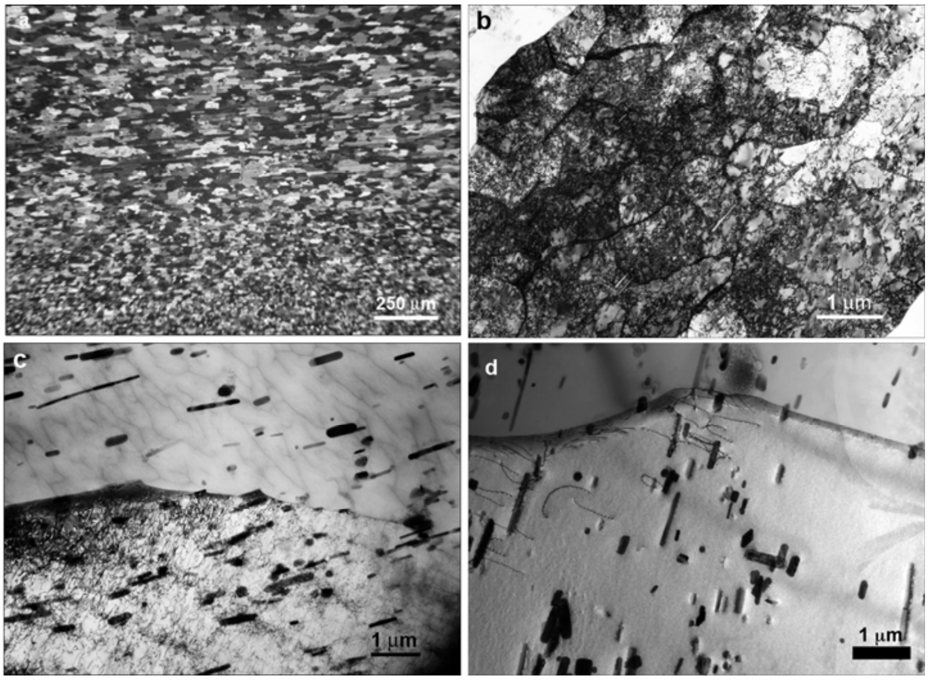


*Figure 1. (a) Optical micrograph of the grain microstructure of the hot rolled 5083 alloy. (b) Bright field TEM micrograph of the substructure of the specimen prepared from center of the plate of the alloy without Zr addition characterized by the elongated grains. c) Bright field TEM micrograph of the substructure of the specimen prepared from a region close to the edge of the plate of the alloy without Zr addition characterized by the equiaxed grains. (d) Bright field TEM micrograph of the substructure of the specimen prepared from center of the plate of the alloy with Zr addition characterized by the elongated grains.*

TEM characterization of specimen prepared from a central part of the plate revealed a presence of well-defined cell bands, 3-10 μm in width (Figure 1b). Such width corresponds well to the measured width of the recovered, elongated grains observed by an optical microscope. Within cell bands, there are cells in a 1-2.5 μm size range. The substructure is characterized by a strong strain contrast and high dislocation density (Figure 1b). However, high dislocation density was also observed in the specimens prepared from material close to the edge of the plate (Figure 1c). Such result was unexpected, as polygonal grains like ones observed in the outer region of the plate are usually indicator of advanced recovery/recrystallization.

During a single stage homogenization, poligonization and homogenization of the grain structure occurred, however grains preserve some degree of elongation in the rolling direction (Figure 2a). Two-stage homogenization did not significantly affect the grain size and morphology, although the mean aspect ratio decreased from 1.8 to 1.5 (Figure 2b). Homogenization anneal at 565°C (the single-stage homogenization) produced substructure with a lower dislocation density, which further decreased during the second homogenization stage.

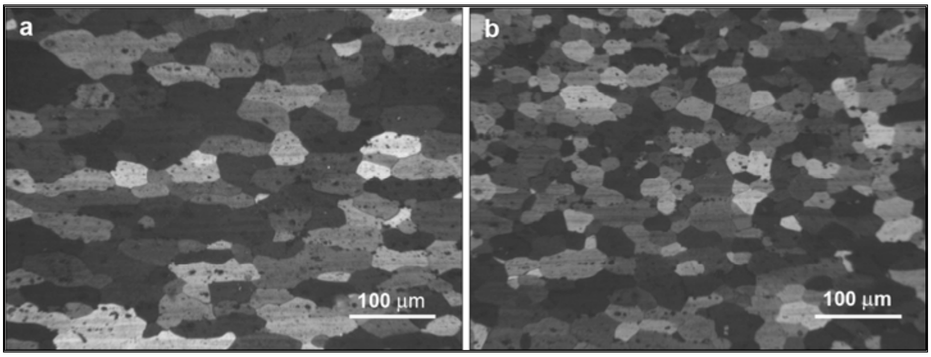


*Figure 2. Grain structure in the central part of the plate of the 5083 alloy: (a) After the single stage homogenization; (b) After the two-stage homogenization.*

Reported observations point out that the likely mechanism of the recovery/recrystallization is a continuous recrystallization [4,5]. The continuous recrystallization, often termed geometric dynamic recrystallization [6], is observed to occur in a number of aluminum alloys [4]. In the case of rolling, an alloy is deformed in a plane strain compression that results in elongation of grains and formation of lamellar so-called "pancake" structure. While the grains stretch in the rolling direction, they contract perpendicular to it and grain boundaries are pushed toward each other. At a certain strain level adjacent grain boundaries can collapse and form new grains. Depending the on the grain boundary mobility as well as amount of strain newly formed grains can have elongated or equiaxed shape. Decrease in a mean aspect ratio of the grains from a center of the plate toward edges is in direct correlation to the fact that periphery of the plate undergoes higher level of strain than the center. Nature of the continuous recrystallization, i.e. new grains are not formed by the classical nucleation & growth process, does not imply formation of dislocation free grains. Hence, as the temperature of final reduction in this study is fairly low for complete recovery, even newly formed polygonal grains can have high dislocation density.

Metallographic characterization of the alloy with Zr addition indicated similar general features of the grain microstructure, however, TEM investigation revealed a number of equiaxed, dislocation free grains even in the specimens prepared from the central part of the hot rolled plate (Figure 1d). Closer examination of the grain structure revealed the presence of such grains in the central regions of the plates. Such grains are associated with clusters of large, undeformed second phase particles that can act as a nucleation site for the new grains activating a discontinuous recrystallization. Results of the metallographic examination of the size and distribution of the second phase particles showed that there is a difference between two alloys. The differences are probably related to the prior processing. In the alloy without Zr the second phase particles are fairly uniformly distributed, while in the alloy containing Zr addition, the non-uniform distribution of coarse second phase particles was observed.

In addition to the large second phase particles detectable by an optical microscope, TEM investigation revealed presence of finer precipitates in both alloys. The as-received, hot rolled state is characterized by the rod shaped particles. Morphology of the particles is determined by the mechanical processing, i.e. hot rolling, as orientation of the longer axis of the rods is independent of a grain orientation and is parallel to the rolling direction. The precipitate density is not uniform as regions densely populated by the particles are separated by the regions without particles. Such distribution originates from the chemical and structural inhomogenieties during the casting that were not removed by the prior thermo-mechanical processing of the alloys. Particle size varies in a wide range from 50 nm to a few μm. Homogenization treatments resulted in the precipitate rounding as reflected through an increase in the form factor from 0.63 to 0.79 after the single stage homogenization, however nonuniform precipitates distribution was not removed. Finer precipitates tend to adopt the plate like or rounded shape, however large precipitates (>1 μm) preserve rod-like morphology as well as orientation of the long axis parallel to the rolling direction. Due to the uneven distribution of the precipitate it was not possible to quantify volume fraction, but an impression is that the precipitate density is lower after the single-stage homogenization than in the other states.

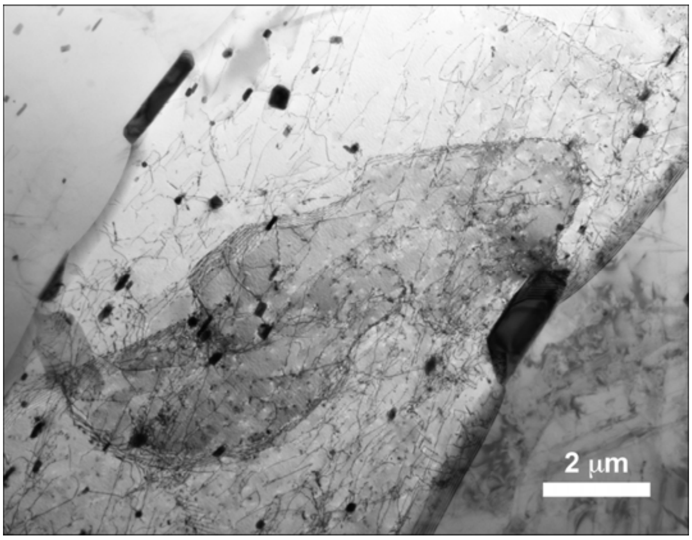


*Figure 3. Bright field TEM micrograph showing an anisotropic grain boundary pinning by large particles resulting in an elongated shape of recovered grains.*

Chemical composition of the particles does not significantly alter during the thermal treatments. The composition appears to be size dependant. Large rod-shaped particles over 1 μm in length are Mn-based but contain also Fe. Rest of the particles also tend to be Mn-based but alloyed with Cr. Preliminary results indicate that there might be difference in a crystal structure among the precipitates. The larger particles alloyed with Fe might adopt hexagonal $MnAl_4$ crystal structure [7], while particles alloyed with Cr have $MnAl_6$ crystal structure [7]. However, this topic needs further investigation.

The effect of the particles on the recrystallization and grain boundary pinning very much depends on the particle shape and size. While the large, undeformed particles aid formation of new grains [4, 6], it was observed that the rod like particles in a similar size range, possessing a high degree of anisotropy due deformation by rolling, pin grain boundaries leading to preservation of the elongated grains (Figure 3) Finer precipitates (<1 μm) can be particularly effective in preventing the growth of the small nuclei of the recrystallized grains.

Although it was expected that a Zr addition would increase resistance toward the recovery and recrystallization of the alloy [8], that did not happened. The issue appears to be that the most of Zr precipitated as a stable, non-coherent phase during the casting. Only negligible fraction of the Zr might precipitate as a desired, metastable $L1_2$ phase along the grain boundaries. Zr-based particles are rod shaped after hot-rolling, but homogenization treatment led to their coagulation (Figure 4). These observations point out that in order to achieve desired effect by the Zr addition, the key is the casting process.

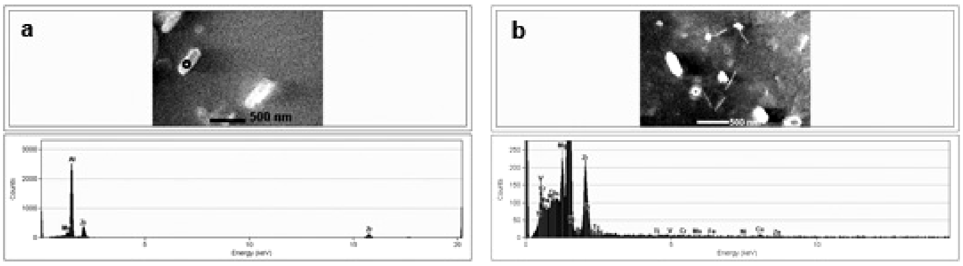


*Figure 4. EDS of the Zr-based particles: (a) As-received state; (b) Two-stage homogenization.*

## Summary

Present investigation has shown that the recrystallization process of hot rolled AA 5083 alloys proceeds via continuous recrystallization. During the two stage homogenization treatment, the recovery of the alloys is completed and the anisotropy of the grain shape decreased. Second phase particles size and deformability play significant role in the extent and recrystallization mechanism. While the large, undeformed particles aid recrystallization processes, deformable particles of similar size can effectively pin the grain boundaries and sustain undesirable “pancake” structure. The results of microstructural characterization point toward the importance of prior thermo-mechanical treatment on microstructure evolution.

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