UDK 669.715:620.19
Original scientific article/Izvirni znanstveni članek

ISSN 1580-2949
MTAEC9, 48(4)479(2014)

INFLUENCE OF THE THERMO-MECHANICAL TREATMENT ON THE EXFOLIATION AND PITTING CORROSION OF AN AA5083-TYPE ALLOY

VPLIV TERMO-MEHANSKE OBDELAVE ZLITINE AA5083 NA LUŠČENJE IN JAMIČASTO KOROZIJO

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Prejem rokopisa – received: 2013-07-18; sprejem za objavo – accepted for publication: 2013-10-04

Exfoliation and pitting corrosion were tested on an AA5083-type alloy sheet, after applying different thermo-mechanical treatments. Hot-rolled plates were laboratory cold rolled with 16–54 % reductions, and subsequently annealed for 2 h in the temperature range from 220 °C to 280 °C. The performed tests have shown that an unacceptable exfoliation and a corrosion susceptibility were developed after the 54 % deformation and low-temperature annealing at 220 °C. However, no severe exfoliation was observed after the cold rolling with low deformations, independent of the applied annealing temperature. The pitting corrosion was less agressive, and the highest degree of the pitting attack was not experienced for the tested alloy. A slight tendency to pitting was detected after the high cold deformation and high-temperature annealing for the samples with recrystallized but significantly flattened grain structures.

Keywords: AA5083 alloy, thermo-mechanical treatment, exfoliation, pitting

Luščenje in jamičasta korozija sta bili preizkušeni na pločevini iz zlitine AA5083 po različnih termo-mehanskih obdelavah. Vroče valjane plošče so bile v laboratoriju hladno valjane z redukcijo 16–54 %, nato žarjene 2 h v območju temperature od 220 °C do 280 °C. Preizkusi so pokazali nesprejemljivo luščenje in občutljivost za korozijo po 54-odstotni deformaciji in nizki temperaturi žarjenja 220 °C. Močnejšega luščenja ni bilo opaziti po hladnem valjanju z majhno deformacijo, neodvisno od uporabljene temperature žarjenja. Jamičasta korozija je bila manj agresivna in močne jamičaste korozije ni bilo pri preizkušeni zlitini. Rahla tendenca po nastajanju jamic se je pokazala pri veliki hladni deformaciji in visoki temperaturi žarjenja pri vzorcih, ki so bili rekristalizirani in so imeli močno sploščeno strukturo.

Ključne besede: zlitina AA5083, termo-mehanska obdelava, luščenje, jamica

1 INTRODUCTION

Al-Mg alloys found a wide range of applications, especially in the construction of transportation means due to the attractive strength-to-weight ratio, good weldability and formability as well as high corrosion resistance.[1–4] However, the Al-Mg alloys with more than 3 % Mg, due to a limited room-temperature solubility of Mg in an Al matrix (w(Mg) = 1.9 %), can be sensitized due to the formation of a continuous film of the β-phase (Mg_2Al_3) at the grain boundaries, and become susceptible to intergranular corrosion (IGC).[5–9] Generally, the susceptibility to the IGC is influenced, in a complex manner, by the structure features of a material,[10,11] and an optimization of the overall thermo-mechanical treatment is the key point in producing a corrosion-resistant material.[10] One of the manifestations of the IGC is a specific lamellar type of corrosion, with a blister-surface appearance, known as exfoliation. It is a form of the IGC that occurs on the surfaces of the wrought aluminum alloys[12–14] with highly flattened grain structures. Another common corrosion type, also associated with a specific surface appearance, is pitting corrosion. It is a form of an extremely localized corrosion[15] that leads to a creation of small holes or pits in the structure, usually covered by the corrosion products. These types of corrosion were often observed and analyzed on 2xxx and 7xxx type alloys, used in aircraft industries, and due to their importance a major attention was paid to the investigation of these alloys.[16,17]

The objective of this work was to study the exfoliation and pitting corrosion on a moderate-strength non-heat treatable AA5083-type-alloy sheet, after applying different thermo-mechanical treatments (TMTs). Exfoliation and pitting corrosion appeared to be very important manifestations of a sea-water attack in Al-Mg-based alloys,[18] therefore, marine-grade alloys should be subjected to the corrosion testing defined in ASTM B 928/B 928M-04a standard,[19] including intergranular, exfoliation and pitting corrosion assesments.

2 EXPERIMENTAL WORK

Material. The material used in this study was an industrially manufactured hot-rolled thick plate 7.4 mm, supplied by Impol-Seval Aluminium Rolling Mill. The chemical composition is listed in **Table 1**.

The as-received hot-rolled plates were laboratory cold rolled to 3.5 mm and inter-annealed for 3 h at 350 °C. These samples were further cold rolled with different degrees of deformation ranging from 16 % to 54 %, and then finally annealed for 2 h in the temperature range of 220–280 °C and air cooled.

Table 1: Chemical composition of the tested alloy in mass fractions (*w*/%)

Tabela 1: Kemijska sestava preizkusne zlitine v masnih deležih (*w*/%)

Mg	Mn	Cu	Fe	Si	Zn	Cr	Ti	Sr
5.13	0.718	0.013	0.337	0.11	0.513	0.008	0.0254	0.003

Corrosion testing. Susceptibility to exfoliation and pitting corrosion was determined with a visual inspection using the ASSET method (the method for a visual assessment of the exfoliation-corrosion susceptibility of AA5xxx-series Al alloys), described in ASTM G66 standard.

Brinell hardness was measured on the samples processed in the manner described above, as well as the grain aspect ratio, calculated after revealing the grain structure using Barker's etchant and a microstructure analysis with an optical microscope.

3 RESULTS

Exfoliation and pitting-corrosion susceptibility were ranked after a visual inspection according to ASTM G66 standard. The following letters were used to describe the degrees of pitting corrosion: N – no appreciable attack, or A, B and C levels of attack (A and B denote acceptable corrosion, C denotes susceptibility to corrosion). In case of exfoliation (besides N) the ratings are as follows: A, B, C and D levels of attack (A and B denote acceptable exfoliation, C and D denote susceptibility to exfoliation). A combination of two letters was used in the case when the surface morphology could not be described with only one letter due to a mixed- or boundary-type surface appearance like the ones on the photographs given in ASTM G66 standard.

The ratings of the exfoliation and pitting-corrosion-attack dependence on the applied TMTs, i.e., the cold-rolling reductions and annealing temperatures, are shown in **Figures 1a** and **1b**. The results for the general IGC susceptibility of the tested alloy (expressed as a mass loss per unit area, according to ASTM G67 standard), considered elsewhere,[20] are also attached. The results shown in **Figure 1a** indicate that the C level denoting unacceptable exfoliation appeared after the 54 % cold deformation and low-temperature annealing at 220 °C. However, after a prior low deformation of 16 %, the exfoliation was absent, even after a low-temperature annealing at 220 °C, and not related to the IGC susceptibility that was very pronounced in this case, as shown in **Figure 1a**.

The most detrimental pitting-corrosion susceptibility, described as the C-type surface appearance, was not recognized for the tested alloy. However, after a higher degree of the cold-rolling reduction, 54 %, and the final annealing at 260 °C and 280 °C, the B level of the pitting susceptibility was identified (**Figure 1b**). The same B level of the pitting attack was noticed after the 30 % deformation and the annealing at the highest temperature of 280 °C.

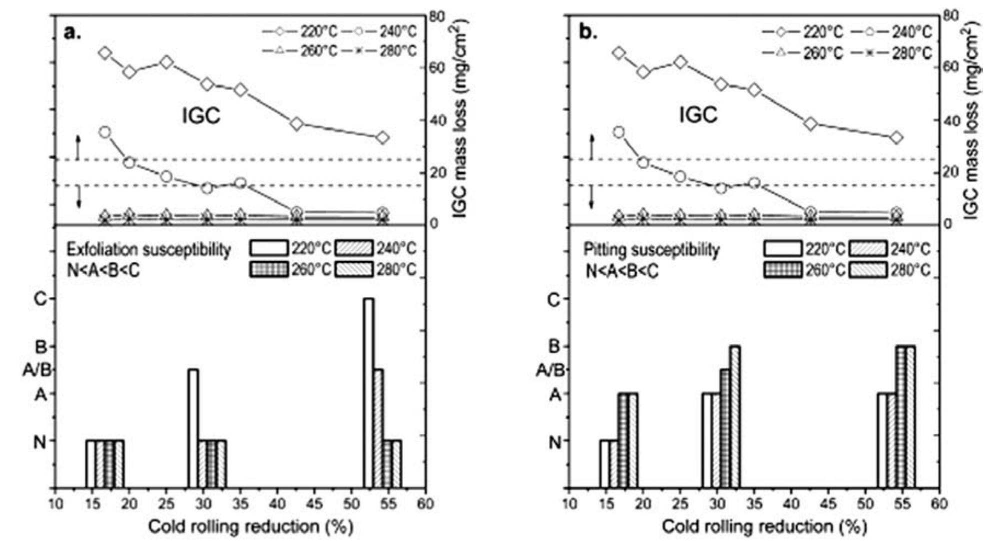

Figure 1: Ratings of the: a) exfoliation and b) pitting corrosion levels (according to ASTM G66 standard) for the cold rolled (16–54 %) and subsequently annealed (220–280 °C) specimens. The appropriate data for the IGC susceptibility is also shown. The values below the lower dashed line (IGC = 15 mg/cm^2) indicate the IGC resistance, while the ones above the upper line (IGC = 25 mg/cm^2) indicate the IGC susceptibility.

Slika 1: Ocena: a) luščenja in b) jamičaste korozije (skladno z ASTM G66-standardom) za hladno valjane (16–54 %) in kasneje žarjene (220–280 °C) vzorce. Prikazani so tudi ustrezni podatki za občutljivost IGC. Vrednosti pod spodnjo črtkano črto (IGC = 15 mg/cm^2) izkazujejo odpornost proti IGC, medtem ko vrednosti nad zgornjo črto (IGC = 25 mg/cm^2) izkazujejo občutljivost za IGC.

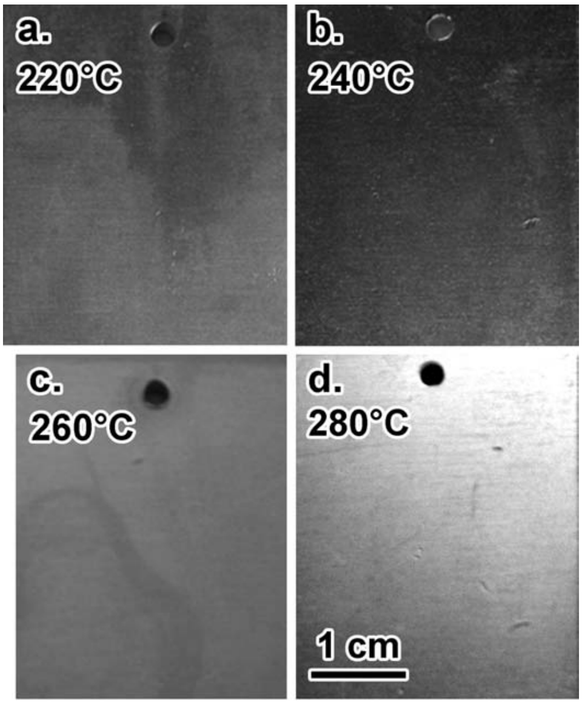

Figure 2: Surface appearance for the samples deformed with the 16 % reduction and annealed for 2 h at different temperatures
Slika 2: Videz površine vzorcev, deformiranih s 16-odstotno redukcijo in žarjenih 2 h pri različnih temperaturah

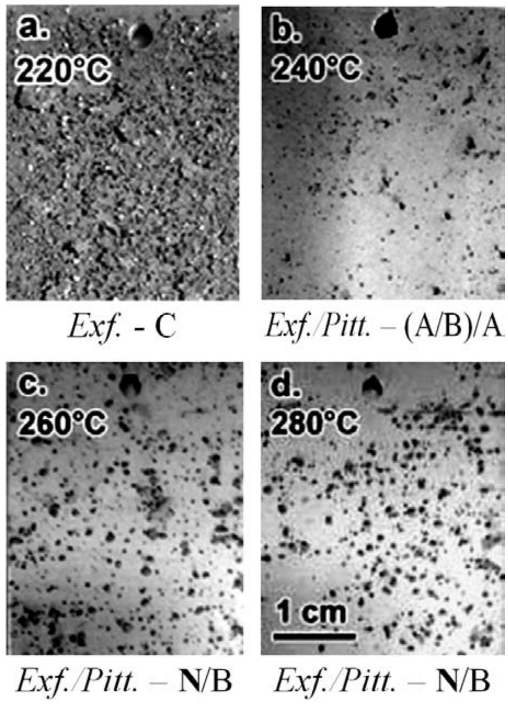

Figure 3: Surface appearance for the samples deformed with the 54 % reduction and annealed for 2 h at different temperatures
Slika 3: Videz površine vzorcev deformiranih s 54-odstotno redukcijo in žarjenih 2 h pri različnih temperaturah

Illustrations of the surface appearances for the samples subjected to the ASSET test, used for the exfoliation and pitting assesment, are shown on **Figures 2** and **3**. **Figure 2** shows the surfaces of the specimens cold rolled with a low-degree deformation, showing no corrosion and no influences of the final annealing temperature.

However, a severe C-type exfoliation was detected on the surfaces of highly deformed samples (54 %) subsequently annealed at a low temperature (220 °C), as shown in **Figure 3a**. The B-level pitting corrosion was observed after the 54 % deformation and annealing at 260 °C or 280 °C as shown in **Figures 3c** and **3d**.

The results of the hardness measurements shown in **Figure 4** revealed that an increase in the cold-rolling reduction up to ≈ 35 % caused a hardness increase. In the cases of higher cold-rolling reductions of > 35 %, the hardness level stayed almost unchanged (ranging from 85–100 HB), except for the samples annealed at 280 °C. For those samples, a steep decrease in the hardness was observed after a 20 % pre-deformation. However, after the annealing at 260 °C a less steep but continuous drop in the hardness occurred when the prior cold deformation was increased to over 30 %.

In order to correlate the corrosion behavior and the microstructure of the tested alloy, the grain structures of the samples cold rolled with 16 % and 54 % reductions and annealed at 220 °C 2 h and 280 °C 2 h, were revealed as shown in **Figures 5a** to **5d**. In the case of the prior 54 % cold deformation, the grains were elongated after the annealing at both temperatures, independently of the recrystallization processes that occurred after the annealing at 280 °C 2 h (the recrystallization is indicated by the hardness drop in **Figure 4**).

The grain aspect ratio was calculated for the prior cold deformations of (16, 30 and 54) %, for the whole range of the annealing temperatures, as shown in **Figure 6**. It was found that the appropriate grain aspect ratios were around 5.5 for the sample annealed at 220 °C 2 h and 5.7 for the sample annealed at 280 °C 2 h after the 54 % deformation.

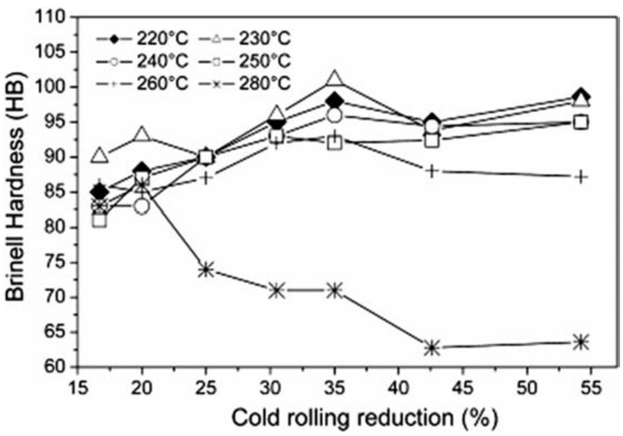

Figure 4: Influence of cold-rolling reductions on the hardness of the samples subsequently annealed for 2 h in the temperature range of 220–280 °C
Slika 4: Vpliv redukcije pri hladnem valjanju na trdoto vzorcev, kasneje žarjenih 2 h v območju 220–280 °C

After the 16 % deformation the grains were less elongated, with the grain aspect ratio being 1.7 for the samples annealed at 220 °C 2 h and 3.5 for the samples annealed at 280 °C 2 h . After the 30 % pre-deformation the grain aspect ratio was from 2.3 to 4.4 for the tested range of temperatures. Generally, the grain aspect ratio significantly increased with the increase in the annealing temperature.

4 DISCUSSION

Previous studies[12–14,21] have shown that the grain structure developed under different thermo-mechanical treatments (TMTs) can have an important role in developing the exfoliation corrosion of the high-strength alloys such as 2xxx or 7xxx series of Al alloys. The basic influence of TMTs is related to the grain flatness or grain aspect ratio enabling a specific long surface path for the IGC, and a formation of corrosion products on a few surface planes with a higher specific volume. In this way the expansion tendency creating the compressive stress can lift the surface grains and form surface blisters. On the other hand, it is already known that the general IGC susceptibility is mostly controlled with the alloy composition and the distribution of grain-boundary precipitates.[22]

In this work, the harmful C level of exfoliation was experienced only after the 54 % cold-rolling deformation and the subsequent low-temperature annealing at 220 °C (**Figures 1a** and **3a**). In this case the recovery degree was supposed to be low, as it was indicated by the hardness level after the annealing at 220 °C (**Figure 4**). The microstructural observations of these samples showed that the grains were highly elongated (**Figure 5c**) with the grain aspect ratio of ≈ 5.7 (**Figure 6**). For the samples with the lower grain aspect ratio of ≈ 2.3 after the 30 %

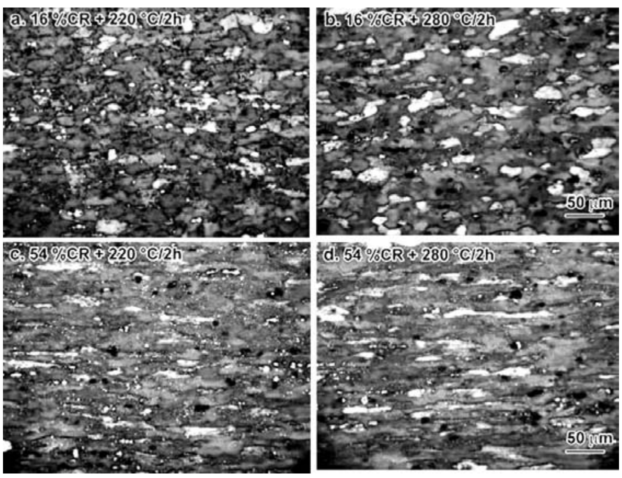

Figure 5: Grain structures of the samples deformed with the: a), b) 16 % and c), d) 54 % cold-rolling reductions and annealed for 2 h at: a), c) 220 °C and b), d) 280 °C

Slika 5: Zrnata struktura vzorcev po hladnem valjanju, deformiranih s: a), b) 16 % in c), d) 54 % redukcijo ter žarjenih 2 h pri: a), c) 220 °C in b), d) 280 °C

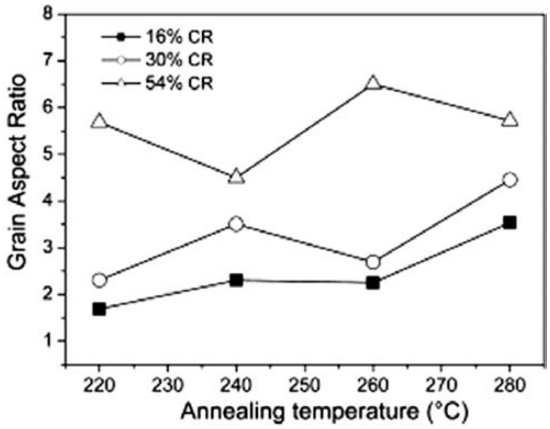

Figure 6: Grain aspect ratios for the samples cold rolled with (16, 30 and 54) % reductions and subsequently annealed for 2 h at 220 °C and 280 °C

Slika 6: Razmerje dolžine proti višini zrn pri hladno valjanih vzorcih z redukcijo (16, 30 in 54) % ter kasneje žarjenih 2 h pri temperaturah 120 °C in 280 °C

deformation, or ≈ 1.7 after the 16 % deformation, no unacceptable exfoliation level of attack was observed or it was completely absent (**Figure 1a**). This indicates the grain-flatness importance in the case of the exfoliation-type corrosion. It should also be emphasized that for the 16 % deformed samples, the general IGC susceptibility was at the highest level (**Figure 1a**), but obviously the grain flatness was low and exfoliation could not develop at all. The grain aspect ratio seemed to increase with the increasing annealing temperature (**Figure 6**), but in spite of this, no detrimental level of the exfoliation attack took place (**Figure 1a**). It was assumed that the general IGC susceptibility was reduced to such an extent that it suppressed exfoliation, i.e., any type of intercrystalline corrosion. Therefore, after the low-temperature annealing, the 54 % deformed sample with a non-recrystallized structure and the grain aspect ratio of 5.5 was prone to exfoliation, while the recrystallized structure with the grain aspect ratio of 5.7 (after the annealing at 280 °C) was completely resistant to exfoliation, as the IGC was suppressed too. It was supposed that an improvement of the corrosion properties with the annealing-temperature variations can be basically related to the β-phase precipitation, i.e., the change of a continuous film after the low-temperature annealing to a discontinuous discrete β-phase morphology after the high-temperature annealing at 260 °C and 280 °C, as discussed elsewhere.[20] Therefore, in practice, the exfoliation-corrosion control should be considered in a limited range of the structure states that are also fully resistant to the IGC. However, according to our results the tendency to the exfoliation-type corrosion in all the circumstances was related to a flattened-grain structure, i.e, it was suppressed in the case of the structure with a grain aspect ratio below a certain critical value.

On the other hand, in the case of a lower grain aspect ratio or a structure with less elongated grains, the pitting-type corrosion is thought to be the active process of the intercrystalline corrosion.[12,13] In this experiment, no unacceptable C-level pitting was detected (**Figures 1b**, **3c** and **3d**) according to the fact that under the applied cold deformations and heat treatments no equiaxed grain structure was developed (**Figure 5**). The appearance of a severe but not harmful B-level pitting corrosion, observed after the annealing at 260 °C and 280 °C of the 54 % deformed samples (**Figures 1b** and **3b**), could be related to a high degree of the recovery or recrystallization process. This is in a good agreement with the hardness drop (**Figure 4**) and cannot be related to the development of a less flattened or equiaxed grain structure enabling the pitting corrosion with the progress in the intercrystalline corrosion over the shorter paths around the grains.[12,13]

Using the results shown in **Figure 1b** it was difficult to correlate the degree of the pitting-corrosion attack and the general intergranular-corrosion (IGC) susceptibility as the pitting was even stronger for the samples showing a high IGC resistance (indicated by a very low mass loss in **Figure 1b**).

5 CONCLUSION

The exfoliation and pitting corrosion of an AA5083-type alloy sheet were considered after applying different thermo-mechanical treatments (TMTs): cold-rolling deformations from 16 % to 54 % and the final annealing for 2 h at 220–280 °C.

The results of the corrosion testing, performed according to ASTM G66 standard, showed that the exfoliation-type corrosion can be significantly affected by varying the applied TMTs. A detrimental or unacceptable exfoliation-corrosion level was experienced after high cold-rolling deformations (≈ 50 %) and low-temperature annealing (at 220 °C) together with a highly elongated grain structure. However, in the case of the high-temperature annealing and a certain grain flatness, the exfoliation could not develop as the general IGC was suppressed too.

No harmful level of pitting corrosion was detected as the equiaxed grain structure was not developed after applying different TMTs. However, a moderate pitting attack was noticed after the annealing at high temperatures on the samples with recrystallized but highly flattened grain structures and with a very low IGC susceptibility.

Acknowledgements

The authors are grateful to the Ministry of Education, Science and Technological Development, Republic of Serbia, and Impol-Seval Aluminium Mill, Sevojno, for supporting this research under contract TR 34018.

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