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## **INTERGRANULAR CORROSION SUSCEPTIBILITY OF AN AA5083 Al-Mg ALLOY PROCESSED BY ACCUMULATIVE ROLL BONDING (ARB)**

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### **Abstract**

Intergranular corrosion (IGC) susceptibility of an AA5083 Al-Mg alloy sheets, highly deformed by accumulative roll-bonding (ARB), was investigated. It was shown that ARB processed specimens were resistant to IGC as the mass loss was  $<15 \text{ mg/cm}^2$ . The degree of plastic strain during ARB processing did not affect IGC susceptibility and a value of corrosion potential. After sensitization treatment at  $150^\circ\text{C}$ , ARB processed specimens stayed resistant to IGC, in spite of  $\beta$ -phase precipitation along grain boundaries and within the structure. A high degree of deformation achieved by ARB processing, and a large amount of  $\beta$ -phase precipitated during the sensitization, did not cause an increase in IGC susceptibility due to favorable morphology and a distribution of  $\beta$ -phase in the structure.

*Keywords: Intergranular Corrosion, Accumulative Roll Bonding, Al-Mg alloy*

### **Introduction**

Accumulative roll-bonding (ARB) is one of the severe plastic deformation (SPD) processing techniques, developed to obtain ultrafine-grained (UFG) structures in bulk and sheet materials. ARB process was originally proposed by Saito et al. [1,2], as an innovative process of plastic deformation for manufacturing of multilayered sheet materials with refined microstructure and a high strength level. Microstructure refinement and mechanical properties of Al-Mg alloys subjected to ARB processing had been extensively investigated [2–7]. However, a general need for improving the strength parameters is usually followed by a requirement for the preservation of corrosion resistance of these alloys. Intergranular corrosion (IGC) is one of the most critical corrosion processes in Al-Mg alloys. It occurs in the grain boundary region, due to a localized decomposition of solid solution or a presence of anodic  $\beta$ -phase particles [8]. Currently, a little is known about corrosion behavior of the Al-alloys fabricated by ARB process and other SPD novel processing techniques. A certain number of studies [9–11] have shown that SPD processes can either increase or decrease corrosion resistance of various Al alloys. Due to a great potential of ARB processing to be introduced into

industrial practice [2,12], there is a great interest in the examination of the corrosion behavior of ARB processed Al-Mg alloys.

The aim of this study was to investigate the influence of high plastic deformation imposed by ARB processing on the intergranular corrosion (IGC) susceptibility of an AA5083 Al-Mg alloy multilayered sheets.

### Experimental work

Material used in this study was industrially produced. It was supplied by Impol-Seval Aluminium Mill (Sevojno, Serbia) as hot rolled 10.6 mm thick plates of an AA5083 type Al-Mg alloy, with chemical composition given in Table 1.

Table 1. Chemical composition of the studied and standard AA5083 type alloys (wt.%)

Alloy	Mg	Mn	Cu	Si	Fe	Cr	Zn	Ti
Studied	4.16	0.47	0.0076	0.149	0.288	0.0599	0.0182	0.0086
AA5083	4.0-4.9	0.4-1.0	<0.1	<0.4	<0.4	0.05-0.25	<0.25	<0.15

Hot rolled plates were further laboratory processed by cold rolling and inter-annealing up to 1 mm in thickness. Final annealing was performed at 320 °C/3h. 1 mm thick specimens were used for ARB processing (Fig.1) that was performed at room temperature. It was possible to achieve maximum 6 ARB cycles with short annealing between ARB cycles at 320 °C for 5 min. ARB processed sheets were characterized by resistivity measurements, corrosion testing and optical microscopy.

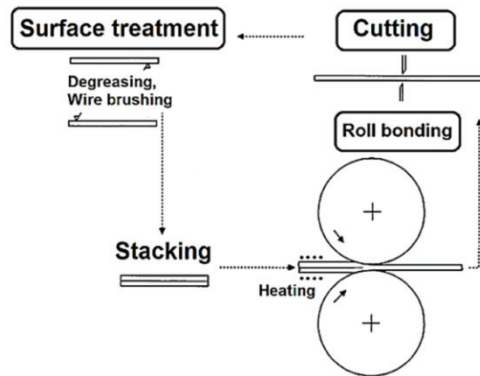


Fig. 1. Schematic illustration of accumulative roll-bonding (ARB) process [1].

The conductivity measurements were performed using Sigmatest 2.069 equipment, at operating frequency of  $f = 240$  kHz and conductivity was converted to resistivity values. For the corrosion testing purposes, one group of ARB processed specimens was sensitized at 150 °C for 7 days. The susceptibility to intergranular corrosion (IGC) was determined on the ARB processed

(unsensitized) and sensitized specimens by NAMLT test (Nitric Acid Mass Loss Test) according to ASTM G67 standard. It consists of the immersion of the tested specimens with dimensions of 50×6 mm, in concentrated HNO<sub>3</sub> at 30 °C for 24 h, and the measurements of mass loss during testing. Specimens susceptible to IGC lose 25-75 mg/cm<sup>2</sup>, while the specimens resistant to IGC lose between 1-15 mg/cm<sup>2</sup>. Corrosion potential of the ARB processed specimens was measured by FLUKE77 multimeter according to ASTM G69. Testing was performed at room temperature using a solution that contains 58.5±0.1 g NaCl and 9±1 ml 30% H<sub>2</sub>O<sub>2</sub>. The reference electrode was a saturated calomel electrode (SCE) with a potential of 0.244 V at 25 °C. Characterization of the microstructure after sensitization treatment was conducted by means of light optical microscopy (LOM) with a Reichert-Jung MeF3 optical microscope. Specimens for LOM were prepared by etching in 10% H<sub>3</sub>PO<sub>4</sub> at 50 °C for 30 s, in order to reveal β-phase precipitates in the structure.

### **Results and Discussion**

Starting material for ARB processing was 1 mm thick fully annealed Al-Mg alloy sheet. Microstructure of the initial state was completely recrystallized before ARB deformation with an average grain size of ~23 μm [13]. ARB processed specimens with 1 mm in thickness consisted of 2 layers after the first ARB cycle and up to 64 layers after 6 ARB cycles. The thickness of each layer was ~500 μm after first ARB cycle and approximately 15 μm, after 6 ARB cycles. ARB processing refined the microstructure. The grains within layers became elongated in the rolling direction, and a significant reduction in grain thickness occurred, from ~ 23 μm, before ARB process, to ~ 3 μm after 6 ARB cycles [13]. Mechanical properties of ARB processed specimens are given in Table 2. They were significantly improved after ARB processing as the maximum value of hardness became greater twice, and yield strength was increased almost three times, in comparison with the initial state of material before ARB.

*Table 2. Mechanical properties of accumulative roll bonded AA5083 Al-Mg alloy*

Mechanical properties	Initial state – before ARB	Number of ARB cycles					
		1	2	3	4	5	6
Brinell hardness, HB	68	91	105	112	112	105	101
Yield strength, YS (MPa)	132	260	342	366	368	357	/

It is well known that Al-Mg alloys with high Mg content (> 3% Mg) may become prone toward intergranular corrosion (IGC), as supersaturated solid solution decomposes and the excess of Mg atoms precipitate out in the form of anodic β-phase (Mg<sub>5</sub>Al<sub>8</sub>) along the grain boundaries [14]. Precipitation of electrochemically active β-phase occurs slowly even at room temperature, and could be significantly accelerated at high temperatures (> 65 °C) rendering grain boundaries susceptible to intergranular corrosion [15]. The extent of IGC susceptibility depends on many factors, such as material chemistry, environment

and thermo mechanical treatment [12,15]. In the present study, influence of high plastic deformation imposed by ARB process, and a presence of interfaces between the layers, on the IGC susceptibility of Al-Mg alloy laminates was investigated. In order to accelerate precipitation of  $\beta$ -phase in ARB processed specimens, a sensitization annealing at 150 °C for 7 days was performed.

Figure 2 shows the results of NAML test and a mass loss of ARB processed specimens before and after sensitization treatment. According to ASTM G67 standard, it is considered that both groups of specimens are resistant to IGC, as the mass loss was below 15 mg/cm<sup>2</sup>. For ARB processed specimens, a mass loss was less than 4 mg/cm<sup>2</sup>, and large plastic deformation during ARB processing (up to  $\epsilon=4.8$  after 6 ARB cycles) did not affect IGC susceptibility. Sensitization of the specimens in initial state and after 1 ARB cycle brought a mass loss of ~6.5 mg/cm<sup>2</sup>, while in case of specimens processed with 2 up to 6 ARB cycles a mass loss was almost unchanged after sensitization treatment. Measurements of the corrosion potential of ARB processed specimens, performed according to ASTM G69 standard, are shown in Figure 3. It is obvious that number of ARB cycles, i.e. a large deformation imposed by ARB processing, did not affect the value of corrosion potential of the tested Al-Mg alloy.

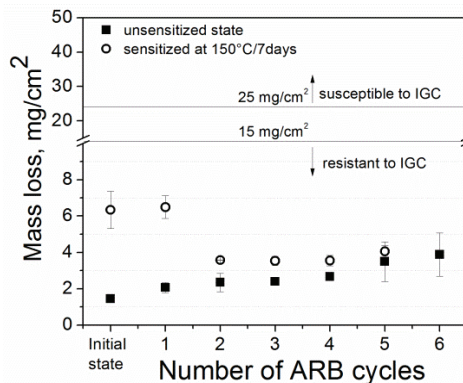


Fig. 2. IGC susceptibility of ARB processed specimens before and after sensitization treatment at 150 °C.

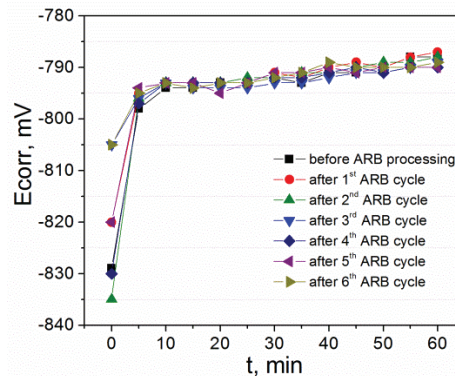


Fig. 3. Influence of the number of ARB cycles on the corrosion potential of tested Al-Mg alloy.

Since IGC occurs due to  $\beta$ -phase precipitation at grain boundaries or inside the grains, a variation in Mg concentration in solid solution (precipitation/dissolution of Mg) was followed by resistivity measurements. Figure 4 shows that the electrical resistivity of ARB processed specimens slightly increases (not more than 0.1  $\square\Omega\text{cm}$ ), while after sensitization the electrical resistivity of ARB processed specimens decreases significantly. Increase in resistivity with increasing the number of ARB cycles can be attributed to the gradual increase in dislocation density during ARB deformation, while decrease in resistivity after sensitization occurs due to  $\beta$ -phase precipitation and a reduction of Mg solute atoms in solid solution.

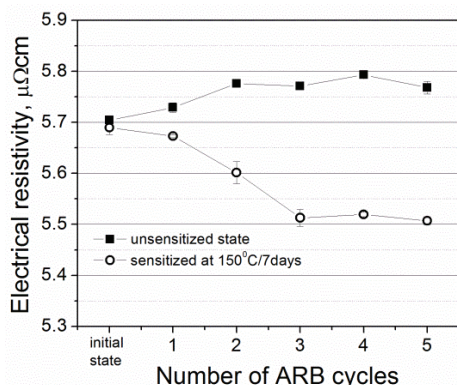


Fig. 4. Variation in electrical resistivity of ARB processed specimens after sensitization treatment at 150°C.

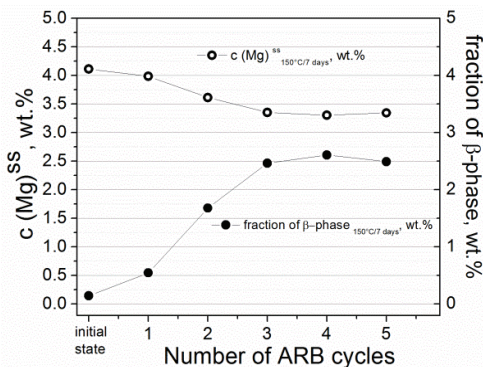
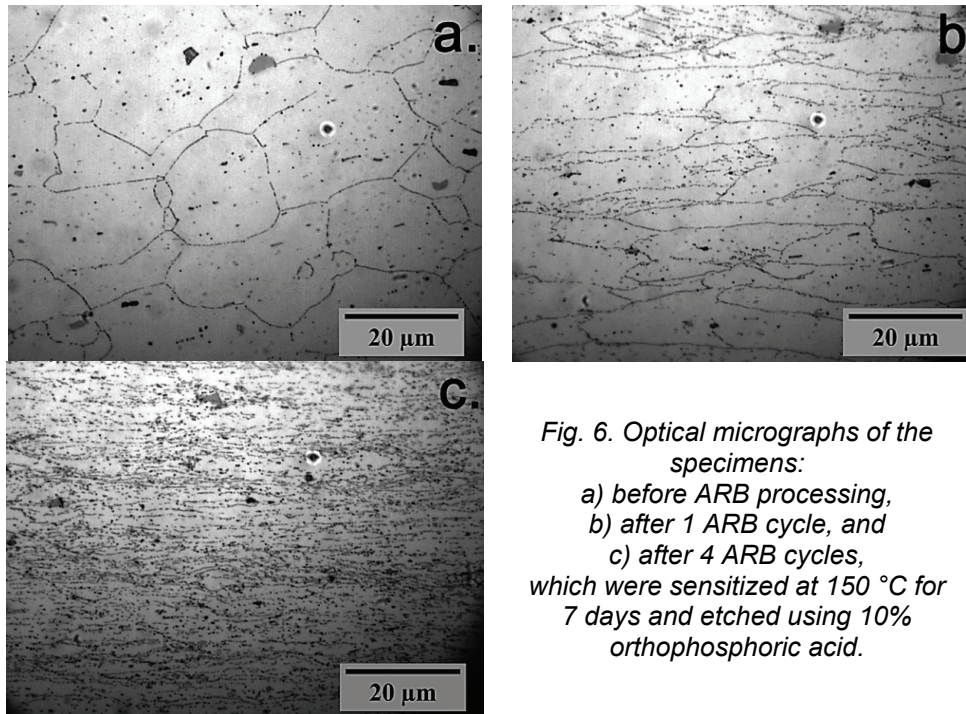


Fig. 5. Influence of sensitization treatment on Mg concentration change in the solid solution and a fraction of  $\beta$ -phase particles in the structure of ARB processed specimens.

Electrical resistivity of the initial state (before ARB processing) was slightly decreased, while in case of ARB processed specimens a significant decrease in electrical resistance was observed after sensitization treatment. Maximum drop in resistivity of approximately 0.27  $\mu\Omega\text{cm}$  was attained after sensitization of ARB specimens processed with 3-5 ARB cycles, as shown in Fig. 4. Decreasing in resistivity values was correlated with a change of Mg solute atoms concentration in solid solution and a precipitation of  $\beta$ -phase in the structure. Based on the resistivity change values that occurred due to sensitization treatment, a concentration of Mg solute in solid solution was calculated, and the amount of  $\beta$ -phase precipitates was determined by a lever rule. Fig. 5 shows that Mg concentration in solid solution was gradually decreased, from 4.16 to 3.3 wt% Mg, while the fraction of  $\beta$ -phase precipitates was increased up to 2.6 wt.% with increasing the number of ARB cycles. We suppose that increasing the number of ARB cycles increases a density of dislocations in a deformed structure which present favorable places for the precipitation of  $\beta$ -phase particles. Influence of the number of ARB cycles on the shape and distribution of  $\beta$ -phase precipitates was revealed by metallographic etching in 10% orthophosphoric acid of sensitized specimens. Fig. 6 shows optical micrographs of the specimens after sensitization treatment as follows: initial state (Fig. 6a), specimen after 1 ARB cycle (Fig. 6b), and specimen after 4 ARB cycles (Fig. 6c).



*Fig. 6. Optical micrographs of the specimens:  
a) before ARB processing,  
b) after 1 ARB cycle, and  
c) after 4 ARB cycles,  
which were sensitized at 150 °C for  
7 days and etched using 10%  
orthophosphoric acid.*

Fig. 6a shows that during sensitization of the specimens in the initial state and processed by 1 ARB cycle,  $\beta$ -phase precipitated along the grain boundaries. However, in the initial state grains were polygonal while after being processed by 1 ARB cycle, grains become elongated (Fig. 6b). The amount of  $\beta$ -phase precipitates in these two cases was 0.15, and 0.55 wt.%, respectively (Fig. 5). In case of highly deformed specimens, with more than 3 ARB cycles,  $\beta$ -phase precipitates are distributed homogeneously throughout the structure, as Fig. 6c shows for the specimen after 4 ARB cycles. The fraction of  $\beta$ -phase particles precipitated in the structure of this specimen was approximately 2.6 wt.% (Fig. 5). However, a mass loss was smaller, and IGC resistance better, than for the specimen in the initial state and after 1 ARB cycle (Fig. 2). In spite of the small amount of  $\beta$ -phase precipitates (0.15 and 0.55 wt.%), a mass loss for these specimens was about  $\sim 6.5$  mg/cm<sup>2</sup>. That can be attributed to the localized precipitation of  $\beta$ -phase along/at the grain boundaries, which cause a higher mass loss. However, the specimens were still resistant to IGC as  $\beta$ -phase precipitates were formed semi-continuous coverage of grain boundaries.

### Conclusion

In this study, the effect of accumulative roll bonding (ARB) process on the susceptibility to intergranular corrosion (IGC) of an AA5083 Al-Mg alloy was investigated. It was shown that:



ARB processed specimens were resistant to IGC as the mass loss was less than 15 mg/cm<sup>2</sup> even after sensitization treatment at 150 °C for 7 days;

The degree of plastic strain during ARB processing did not affect IGC susceptibility and a value of corrosion potential. Increasing the number of ARB cycles increases the amount of  $\beta$ -phase precipitates but the material stayed resistant to IGC;

The results showed that the distribution of the  $\beta$ -phase precipitates had greater influence on the IGC susceptibility than the precipitated amount.

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