

Electrochemical Behavior of Alumix 321 PM and AA6061 Alloys in 3.5 wt% NaCl Solution by Electrochemical Impedance Spectroscopy Measurements

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Abstract

Powder metallurgy (PM) is a very interesting metal manufacturing technique in the production of automotive components of a net or near net shape. In this research, the electrochemical corrosion behavior of a commercially available aluminum powder alloy known as Alumix 321 was investigated and compared to wrought alloy AA6061 in 3.5 wt% NaCl solution using Electrochemical Impedance Spectroscopy Measurements (EIS). Alumix 321 alloy samples were prepared by pressing the powder at pressures ranging from 50 to 500 MPa and subsequently sintering them for 30 min at 630°C. It was found that the presence of pores and their morphology strongly affect the corrosion behavior the PM samples.

Keywords

Aluminum, Powder Metallurgy, Aluminum Alloys, Corrosion, Electrochemical Impedance Spectroscopy, AA6061, Alumix 321

1. Introduction

Aluminum and its alloys find many applications in several technological applications due to their light weight, corrosion resistance and good thermal and electrical conductivities. These alloys are produced as wrought alloys and as powder metallurgy (PM) alloys and exhibit distinguished properties. In addition to the main corrosion mechanisms observed in the ingot metallurgy (IM) aluminum alloys such as pitting, crevice, stress corrosion cracking (SCC), and ex-foliation corrosion the presence of porosity in sintered products add more limi-

tations on using PM. Normally, sintered aluminum parts have some retained porosity. The effect of porous structure on mechanical properties has been studied extensively by many researchers [1] [2] [3]. A limited number of work has been done to investigate the effect of powder processing parameters, such as sintering time, temperature, and sintering atmosphere [4] [5] [6] [7], on PM aluminum alloys corrosion behavior. Also, a comparative corrosion study, on the behavior of Alumix 321 PM alloy and its equivalent commercial counterpart AA6061 regarding processing parameters, was previously conducted at the Advanced Corrosion Laboratory, Dalhousie University [8]. In the present work the behavior of both alloys samples, passivated in 3.5 wt% NaCl solution, was studied by electrochemical impedance spectroscopy (EIS).

2. Experimental Procedure

2.1. Materials

The powder material used in this research is a commercially available alloy known as Alumix 321. The raw material is obtained in a form of powder and was provided by Ecka Granules Inc. Alumix 321 is the powder commercial equivalent to AA6061 aluminum alloy. The powder in its as received condition has been characterized prior to use. Initially, chemical analysis was performed by Atomic Adsorption Spectroscopy (AA). The results of the as received Alumix 321 powder are given in **Table 1**. The commercial equivalent (IM) alloy AA6061 was available from previous experiments [8] in a T1 condition as a rod.

2.2. Processing Powder and Sintering

Powder was first blended to ensure complete mixing and homogenization. The powder was then uniaxially pressed using an Instron SATEC model 5594-200 HVL 1 MN. Samples were pressed at pressures ranging from 50 - 500 MPa. Following pressing, sintering was performed by first heating the samples isothermally to 390°C for 30 min to remove the pre-blended Licowax, this was followed by raising the temperature to 630°C and holding it for 30 min to achieve the desired density, finally samples were allowed to cool to room temperature. All steps were performed in an ultra-high purity nitrogen (>99.999%) atmosphere. Samples were then heat treated to T1 and T6 conditions. Samples treatments and compaction pressures are shown in **Table 2**.

Alumix 321 samples were first ground using 240, 360, 400, and 600 grit SiC paper, and then polished using 9, 6, 3 and 1 µm, diamond paste. Samples were then cleaned and dried. Only distilled water was used to clean the samples and no chemical solvents were involved. Sintered samples with a mirror like surface were then tested for corrosion performance using electrochemical impedance

Table 1. Chemical composition of as received Alumix 321 raw powder.

Element	Mg	Si	Cu	Fe	Bi	Sn	V	Al
W-%	1.31	0.5	0.32	0.10	0.01	0.03	0.01	Bal

Table 2. Samples treatments and compaction pressure.

Samples	Treatments	Pressure
Alumix 321-T6	Sintered	50
		200
		300
		400
		500
Alumix 321-T1	Sintered	50
		200
		300
		400
		500
AA 6061	NA	NA

spectroscopy (EIS). All experiments were conducted in a three-electrode flat cell containing a 3.5 wt% NaCl solution and connected to an EG & G model 273A potentiostat paired with a Model 1255 Solartron frequency response analyzer. Both instruments were controlled by Scribner Z-Plot electrochemical software. The test solution was prepared from analytical grade reagents and distilled water and kept at constant temperature of $22^{\circ}\text{C} \pm 1^{\circ}\text{C}$. Following EIS testing, scanning electron microscopy (SEM) was used to characterize the morphology of the corroded samples.

3. Results and Discussion

3.1. Electrochemical Impedance Spectroscopy (EIS)

In the current work, EIS measurements were performed using a frequency range of 100 kHz to 10 mHz and an amplitude of 10 mV ac signals at the open circuit potential. Before experiments, the working electrode was first immersed into the test solution for 4 h to establish steady state open circuit potential.

To investigate the corrosion behavior, the samples were immersed in a 3.5 wt% NaCl solution for 96 hours, and then SEM and EDS studies were performed.

EIS data were plotted on a Nyquist plot, shown in **Figure 1** and analyzed by fitting them into an equivalent circuit, shown in **Figure 2**. Only the ohmic resistance of the electrolyte (R_s), the capacitance of the surface layer (CPE), and is the resistance of the surface layer (R_p). CPE was used for the surface layer as an ideal capacitor could not be used to the roughness of the layer. As expected, the values of R_s are similar as this is the resistance attributed to the electrolyte, which is the same for all samples. On the other hand, R_p increases with increasing compaction pressure, indicating a significant improvement in the corrosion resistance of the Alumix alloys as the larger the value of R_p at low frequencies

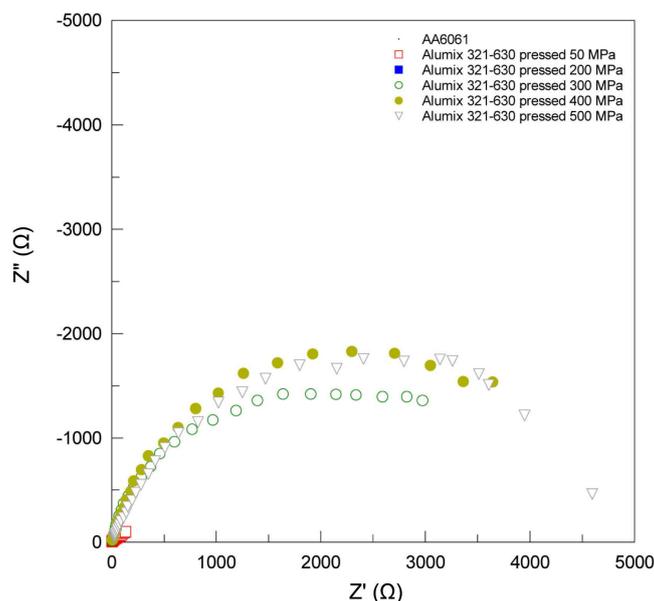


Figure 1. Nyquist plots for Alumix 321-T1 pressed at various pressures.

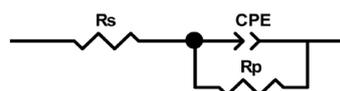


Figure 2. Equivalent electric circuit for Alumix 321-T1.

the better the corrosion resistance is. This is most likely attributed to the decrease in the porosity with increasing compaction pressure, which results in a more continuous oxide layer. The values of components of the model circuit are presented in **Table 3**.

EIS data on Alumix samples pressed at various pressures and sintered T6, were made following the same previous procedure. The Nyquist plot, equivalent circuit and the data are shown in **Figure 3**, **Figure 4**, and **Table 4**, respectively. As can be seen from the values of R_p , compaction pressure played the most noticeable role in affecting the properties of the protective oxide layer.

3.2. Characterization of Corroded Samples by SEM and EDS

Corroded AA6061-T1 and Alumix 321 samples, compacted at pressures varying from 100 to 500 MPa, were examined. The examination involved SEM and EDS. **Figure 5** shows the corrosion surface of (a) AA6061 and its equivalent (b) Alumix 321 after exposure to 3.5 wt% NaCl solution for 48 hours. Even though, both corroded surfaces have very similar morphology, the Alumix 321 PM alloy surface shows evidence of corrosion attacks in the vicinity of the second phase inclusions and the interconnected pores. An EDS analysis indicated that these inclusions are composed mainly of aluminum, iron, and silicon, which most likely cause them to be cathodic to the aluminum matrix.

Figure 6 shows the SEM micrographs of the corroded surface of two Alumix 321 samples pressed at 50 and 500 MPa. Alumix 321 samples pressed at lower

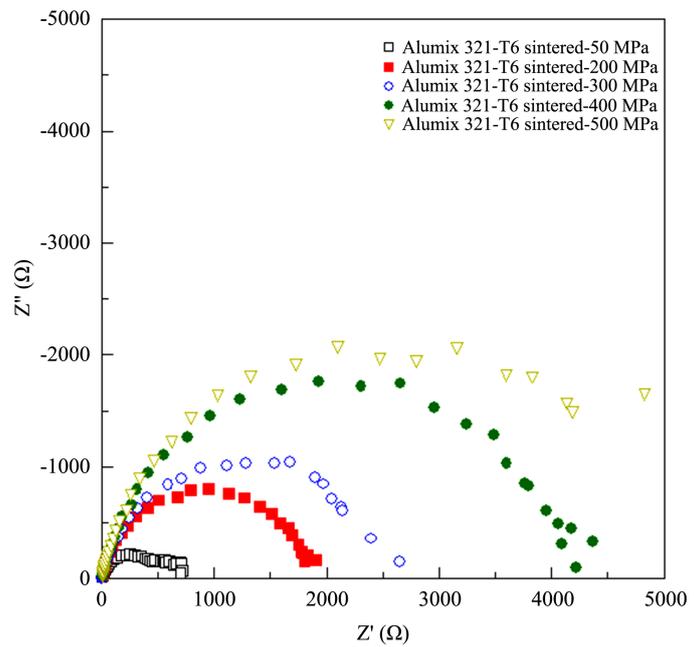


Figure 3. Nyquist plots for Alumix 321-T6 pressed at various pressures.

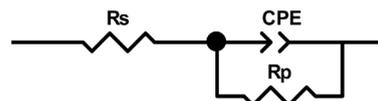


Figure 4. Equivalent electric circuit for Alumix 321-T6.

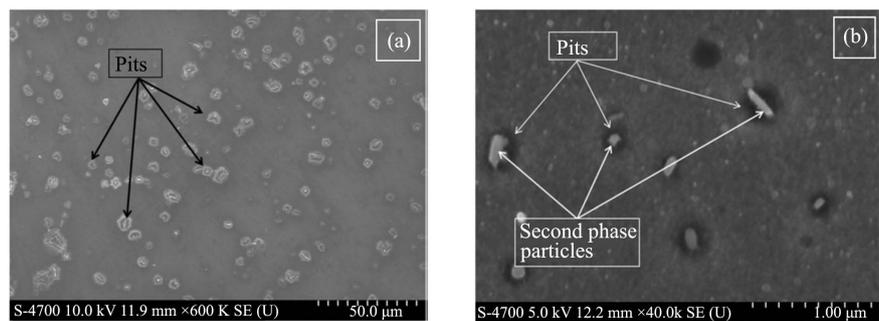


Figure 5. SEM micrographs showing the corrosion morphology of (a) AA6061; (b) Alumix 321 pressed at 500 MPa, after exposure to 3.5 wt% NaCl.

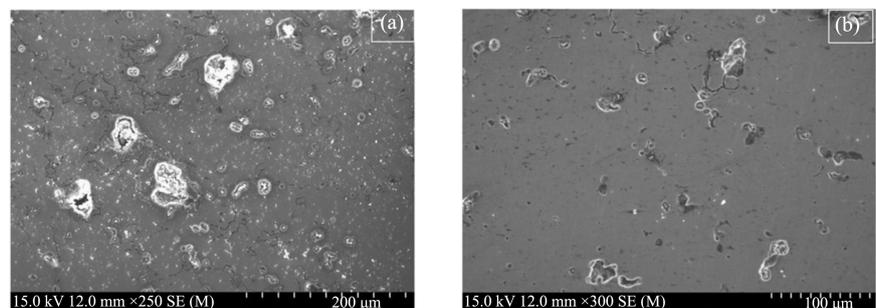


Figure 6. SEM micrographs of the corrosion morphology of Alumix 321 after exposure to 3.5 wt% NaCl (a) compaction pressure of 50 MPa; (b) compaction pressure of 500 MPa.

Table 3. Parameter values of equivalent circuits of all Alumix 321-T1.

Sample	Rs (Ω)	CPE (F)	n	Rp (Ω)
AA 6061	0.771	11.6×10^{-4}	0.94	0.05×10^3
Alumix 321 - 50 MPa	0.498	8.52×10^{-4}	0.52	0.08×10^3
Alumix 321 - 200 MPa	0.501	5.32×10^{-5}	0.75	1.205×10^3
Alumix 321 - 300 MPa	0.481	2.71×10^{-5}	0.85	3.431×10^3
Alumix 321 - 400 MPa	0.489	1.76×10^{-5}	0.84	4.717×10^3
Alumix 321 - 500 MPa	0.6	1.43×10^{-5}	0.78	5.093×10^3

Table 4. Parameter values of equivalent circuits of all Alumix 321-T6.

Sample	Rs (Ω)	CPE (F)	n	Rp (Ω)
Alumix 321 - Sintered T6 - 50 MPa	0.424	2.69×10^{-5}	0.82	0.583×10^3
Alumix 321 - Sintered T6 - 200 MPa	0.357	1.04×10^{-5}	0.89	1.855×10^3
Alumix 321 - Sintered T6 - 300 MPa	0.379	6.74×10^{-5}	0.86	2.631×10^3
Alumix 321 - Sintered T6 - 400 MPa	0.552	1.32×10^{-5}	0.87	4.295×10^3
Alumix 321 - Sintered T6 - 500 MPa	0.696	1.47×10^{-5}	0.87	5.042×10^3

pressure (50 MPa) showed severe corrosion attacks at the interconnected large pores. In addition, some pitting and intergranular corrosion were also observed. On the other hand, samples pressed at higher pressure (500 MPa) showed much less corrosion attack. However, there is some evidence of corrosion in the interconnected pores but to a much less degree compared to the 50 MPa sample. The morphology of the corrosion surface also shows signs of pitting that took place on the surface. There were also indications of intergranular corrosion but to a much lesser extent and severity. The presence of severe intergranular corrosion in Alumix samples pressed at low pressure raises the question of the ability of these alloys to resist stress corrosion cracking.

4. Conclusions

1) In comparison to AA6061 wrought alloy, Alumix 321 PM alloy corroded samples showed pitting, crevice, and intergranular corrosion. However, wrought AA6061 showed only pitting corrosion. On the other hand, for both systems wrought and PM alloys, iron rich particles work as pitting initiation sites. The only different observation is the migration of these particles to the grain boundaries in the case of the Alumix 321 PM alloy; this observation was not seen in the case of wrought AA6061.

2) Alumix 321 alloys showed an increase in corrosion resistance with increasing compaction pressure. In addition, the microstructure of corroded samples pressed at 100 MPa (lowest density) showed severe attacks at the interconnected pores and grain boundaries, which raises a question about the resistance of the sintered alloys against stress corrosion cracking.

3) AA6061, even though it was pitted severely. The alloy is known for its ability to resist SCC, while its equivalent Alumix 321 (low compaction pressures) alloy shows noticeable attack along the grain boundaries and hence decreases the SCC resistance.

5. Future Work

As porosity plays a major role in the corrosion behavior of Alumix 321, it is recommended that it is studied in-depth using focused ion beam (FIB) technique. This technique will allow the investigation of the internal surfaces of pores before and after exposure to the sodium chloride solution. There is also the need to use a technique for the surface area measurement to get representative corrosion parameters. Shot peening post sintering should also be investigated as it is inexpensive, easy to be applied, efficient and effective at closing off the pores.

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Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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