Electrochemical Impedance Characteristics of 7075 Aluminum Alloys Produced by Conventional and Powder Extruded Methods under SSRT Test

Satoshi Sunada¹, Syun Kanayama², Norio Nunomura³ and Kazuhiko Majima¹

¹ Graduate School of Science and Engineering for Research, University of Toyama, 3190 Gofuku, Toyama, 930-8555, Japan.

² Undergraduate Student, University of Toyama, [Present Address: Sankyo-Tateyama Holdings, Inc., 70 Hayakawa, Takaoka, 933-8610, Japan.]

³ Information Technology Center, University of Toyama, 3190 Gofuku, Toyama, 930-8555, Japan.

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Two types of 7075 aluminum alloys: one is fabricated by the conventional ingot metallurgy (I/M) process and the other is fabricated by the powder extruded (P/E) process, were used in this experiment. The electrochemical impedance spectroscopy (EIS) measurement under slow strain rate tensile (SSRT) test was carried out in order to clarify the corrosion behavior under stress for the P/E specimen compared with the I/M specimen. The stress-strain curve (σ - ε curve) obtained from SSRT test showed that the maximum stress of the I/M specimen decreased remarkably in the sulfuric acid solution of 2.5 kmol/m³ H₂SO₄ (hereafter denoted as sulfuric acid solution) compared to that in the ionized water while the maximum elongation of the P/E specimen indicated the similar decrement phenomenon in the sulfuric acid solution. The charge transfer resistance (R_{ct}) during SSRT test decreased with time for both of I/M and P/M specimens.

1. Introduction

The 7000 series aluminum alloys are widely used in the aerospace industry due to the high strength and low density, and, recently, the P/E aluminum alloys have received considerable attention because of their fine and homogeneous structure. Though several papers have been published on the mechanical properties of the aluminum products fabricated by the P/E process, very few studies have been reported regarding their corrosion behavior. In this study, the electrochemical impedance spectroscopy (EIS) measurement [1-6] under slow strain rate tensile (SSRT) test was carried out in order to clarify the corrosion behavior under stress for the P/E specimen process compared with the



Fig.1 Shape of specimen.

I/M process and the sulfuric acid solution was used for the corrosion test solution. The impedance spectroscopy test was performed in the frequency rage from 100 kHz to 10 mHz.

2. Experimental procedure

In this experiment, two kinds of specimens fabricated by I/M and P/E process were used. Their shape and dimensions were indicated in Fig.1, where the gage length is 20mm as shown by GL in the figure, and their chemical compositions were listed in Table 1.

The specimen surface was wet-polished with a series of water-proof Emery papers up to No.2000 followed by washing in alcohol and ultrasonic rinsing in acetone. The specimen electrode was coated with silicon rubber leaving an uncoated area of 1×10^{-4} m². The specimens thus obtained were subjected to SSRT test in the deionized water and the sulfuric acid solution at 303K, where the strain rate of SSRT was kept at 2.5×10^{-6} /s. The schematic illustration of the SSRT test apparatus was shown in Fig.2.

The test solution in the electrochemical cell shown by H was deoxidized through N_2 gas flowing and kept at 303 K. All of the potential values were shown based on Ag/AgCl reference electrode. The electrochemical impedance spectroscopy (EIS) measurement was conducted through frequency

specimen	Si	Fe	Cu	Mn	Mg	Cr	Zn	Ti	Zr	Al
I/M specimen	0.103	0.175	1.50	0.032	2.45	0.231	5.69	0.046	0.0233	Bal.
P/E specimen	0.03	0.01	1.54	Tr.	2.37	0.21	5.53	Tr.	-	Bal.
$(\mathbf{Tr} \cdot < 0.01\%)$										

Table 1Chemical composition (mass%) of A7075 alloys.

analyzer (FRA) indicated by C controlling electrochemical potential by B of potentiostat as shown in Fig.2. The apparatus

for impedance measurement consists of thermostatic bath, H-shape electrolytic cell with anodic and cathodic chambers of 2×10^{-4} m³ in volume, intermediate cell Ag/AgCl (3.33 kmol/m³ KCl) as a reference electrode, potentiostat, AC impedance meter and a personal computer for the



A : Computer B : Potentiostat C : FRA D : Counter electrode(Pt) E : Thermo couple F : Specimen G : Heater H : Electrochemical cell I : Bridge J : Intermediate beaker K : Salt Bridge L : Saturated potassium chloride solution M : Reference electrode

Fig.2 Experimental apparatus for SCC test equipped with computer controlled impedance measuring system.

measurement control. The reference electrode was placed outside the cell and connected to the specimen surface by Luggin capillary and a salt bridge. A natural electrode potential with perturbation AC potential, amplitude of which was 10 mV, was applied to the specimen in AC impedance measurements. Analyzing frequency range was from 100 kHz to 10 mHz. All of the potential values were indicated based on the Ag/AgCl reference electrode.

3. Results and discussion

Fig.3 shows the σ - ε curves obtained by SSRT test in the deionized water and the sulfuric acid solution of 2.5 kmol/m³ H₂SO₄. The I/M specimen showed the maximum stress decrement from



Fig. 3 Stress-strain curves measured in various solutions at 303K for the conventional and powder extruded A7075 (P/E) specimens.

On the other hand, the P/E specimen showed the former decrement from 374MPa and the 389MPa to latter decrement from 0.189 to 0.136. This result implies that the maximum stress of the I/M specimen decreased remarkably in the sulfuric acid solution compared to that in the deionized water while the maximum elongation of the P/E specimen indicated the similar decrement phenomenon in the sulfuric acid solution. Fig.4 (a) shows the σ - ε curve obtained by SSRT test in the sulfuric acid solution for the P/E specimen where EIS test was performed at A to F points in the figure, and Fig.4 (b) shows Nyquist plot



Fig.4 The σ - ε curve obtained by SSRT test in the sulfuric acid solution of 2.5 kmol/m³ H₂SO₄ for the P/E specimen (a) and Nyquist plot diagrams obtained by EIS test performed at A to F points (b) under tensile stress and non stress.

545MPa to 514MPa and the maximum elongation decrement from 0.190 to 0.180 when the test solution was changed from the deionized water to the sulfuric acid solution of 2.5 kmol/m³ H₂SO₄.



Fig.5 Nyquist plot diagram obtained at A point shown in Fig.4 .



Fig.6 Comparison of the two Nyquist plot diagrams obtained at A and F shown in Fig.4.

diagrams obtained from EIS test at each testing point.

Fig.5 shows the Nyquist plot diagram obtained at A as the typical one. It is clarified from Fig.5 that the Nyquist plot curve indicates the favorable semicircle in the first quadrant. The diameter of the semicircle in the direction of the abscissa represented as Re Z interprets the charge transfer resistance, hereafter shortened as $\boldsymbol{R}_{\mathrm{ct.}}$ drawing After capacity semicircle in the first quadrant, the Nyquist plot diagram is followed by the induction semicircle in the fourth quadrant, and then it indicates the capacitive behavior once again. It is obvious from Figs.4 and 5 that the R_{ct} decreases as the strain becomes larger. This result interprets that the corrosion resistance decreases with an increase of strain. The comparison of the two Nyquist plot diagrams at A and F are shown in Fig.6 under the same scale, where the R_{ct} at A point is 8 times as large as that at F point. The similar Nyquist plot diagram by EIS was carried out in the deionized water, and it was also performed for the I/M specimen in both of the two test solutions. All

of the results thus obtained are presented in Fig.7 under time-dependent change during SSRT test. The \mathbf{R}_{ct} of the P/E specimen is smaller than that of the I/M specimen in both of the two test solutions at any point during SSRT test. It is also recognized that the \mathbf{R}_{ct} of the P/E specimen decreases in the sulfuric acid solution compared to the deionized water even in the elastic region though the \mathbf{R}_{ct} of I/M specimen shows the almost the same tendency between the two test solutions and that both specimens show larger \mathbf{R}_{ct} decrement in the sulfuric acid solution compared to the deionized water in the plastic region.



Fig.7 Time dependence of charge transfer resistance R_{ct} for I/M and P/E specimens.

4. Conclusions

The results obtained are summarized as follows: The maximum stress of I/M specimen in the σ - ε curve obtained from SSRT test decreased remarkably in the sulfuric acid solution compared to that in the deionized water while the maximum elongation of the P/E specimen indicated similar the decrement phenomenon in the sulfuric acid solution. The R_{ct} of the P/E specimen was smaller than that of the I/M specimen in both of the two test solutions at any testing time during SSRT test. The decrement of the P/E specimen in the sulfuric acid solution was larger compared to that in the deionized water even in the

elastic region though the \mathbf{R}_{ct} of I/M specimen showed the almost the same tendency between the two test solutions in this elastic region and both specimens show larger \mathbf{R}_{ct} decrement in the sulfuric acid solution compared to the deionized water in the plastic region. The fact that the \mathbf{R}_{ct} of I/M specimen is larger than that of P/E specimen suggests that the corrosion resistance of I/M specimen is better than that of P/E specimen.

Reference

[1] S. Sunada, T. Yamamoto, K. Majima and N. Nunomura: *J. Jpn. Soc. Powder Powder Metallurgy*, Vol. 52, No. 7(2005), pp. 551-561.

[2] S. Sunada, K. Majima, S. Matsumoto, T. Yamamoto and N. Nunomura: *J. Jpn. Inst. Metals*, Vol. 70, No. 4(2006), pp. 389-394.

[3] S. Sunada, T. Yamamoto, S. Tanaka, N. Kada and K. Majima: J. Jpn. Soc. Powder Powder Metallurgy, Vol. 54, No. 5(2007), pp. 336-344.

[4] T. Kawamura, S. Sunada, H. Notoya, K. Kondo and K. Majima: *J. Jpn. Inst. Metals*, Vol. 72, No. 3(2008), pp. 216-223.

[5] T. Kawamura, S. Sunada, K. Kondo, H. Notoya and K. Majima: *J. Jpn. Inst. Metals*, Vol. 72, No. 9(2008), pp. 667-673.

[6] T. Kawamura, S. Sunada, K. Kondo and K. Majima: J. Jpn. Inst. Metals, Vol. 73, No. 3(2009), pp. 189-197.