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AC Impedance Responses of the Inconel 718 Alloy During Electropolishing in Perchloric-Acetic Mixed Acids

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The electropolishing behavior of Inconel 718 alloy was studied with an alternating current (AC) impedance test at 25°C in perchloric-acetic mixed acids with different $HClO_4$ contents. The AC impedance test was conducted by polarizing a potential located in the limiting-current plateau with four potential amplitudes of 20, 200, 500, and 1000 mV. Experimental results show that the electropolishing behavior of Inconel 718 alloy depends strongly on the $HClO_4$ content in the mixed acids. Leveling and brightening of the surface was obtained when Inconel 718 was polished in mixed acids with 30 and 40 volume percents of $HClO_4$, while leveling without brightening of the surface is achieved when Inconel 718 was polished in mixed acids with 10 and 20 vol% $HClO_4$. The electropolishing behavior of Inconel 718 alloy in mixed acids can be recognized from its AC impedance response. Based on the results of AC impedance test, the electropolishing mechanism of Inconel 718 in the mixed acids follows the salt-film precipitated mechanism. A high potential amplitude from 200 to 500 mV is recommended for obtaining a stable AC impedance response based on our experimental results, those contrast the generally accepted potential amplitudes lower than 20 mV used in the AC impedance test. Differences in stable and unstable AC impedance responses with respect to the potential amplitude are discussed in this study.

Keywords: Inconel 718 alloy, electropolishing, AC impedance test, potential amplitude

1. INTRODUCTION

Inconel 718 alloy is a ubiquitously used Ni-based superalloy that has superior mechanical properties and corrosion resistance at high temperatures. Therefore, Inconel 718 alloy is commonly used as a primary construction material in the components used at high temperatures, such as gas turbine engines and aircraft blades [1-6]. It is well-known that the leveling and brightening of the surface of a metallic component can improve its fatigue strength or even corrosion resistance [7]. The leveled and brightened surface of the component can be easily accomplished by electrochemical polishing (EP), regardless of its high strength and toughness.

EP is defined by the American Society for Testing and Materials (ASTM) as the improvement of the surface finish of a metallic component by making it as the anode in an electrolyte solution [8,9]. Several researchers [9-12] have pointed out that two processes, leveling and brightening, are taken place during EP. Both processes occur simultaneously or sometimes independently [9]. A leveled surface has a surface roughness greater than 1 μm, while a brightened surface has a surface roughness less than 1 μm [9]. EP with a metallic component is conducted by an anodic dissolution under mass-transfer controlled limitation. Despite different phases, crystalline imperfections, and inclusions in the metallic component, leveling and brightening of a surface can be achieved by EP. The mass-transfer controlled mechanism is significantly dependent on both the metallic electrode and electrolyte [9,13-18]. Several researchers [9,19,20] have proposed two mechanisms, the acceptor-limited condition and the precipitation of a salt film or viscous layer, to explain EP behavior of metallic components under mass-transfer controlled limitation.

The alternating current (AC) impedance test, or named electrochemical impedance spectroscopy (EIS) test, has been successfully applied to the investigation of various electrochemical processes for the past thirty years [19,20]. This technique has found increasing application in the investigation of anodic dissolution behavior because of the possibility of obtaining the chemical reaction mechanisms involved. The results of AC impedance tests in the frequency domain can be simulated by an equivalent circuit for the electrochemical process. In this study, the EP behavior of Inconel 718 alloy is studied in $HClO_4$-CH_3COOH mixed acids with different $HClO_4$ contents by using a rotating disc electrode (RDE), which has been found to be a useful tool for studying electrochemical processes under mass transport to and from the electrode. In the AC impedance test, four potential amplitudes, 20, 200, 500, and 1000 mV, are used in the limiting-current plateau. The anodic dissolution behavior of the Inconel 718 alloy is explained based on its AC impedance response. Meanwhile, the justification for using of a large potential amplitude for the AC impedance test will be discussed in this work.

2. EXPERIMENTAL PROCEDURE

As-rolled Inconel 718 sheets with a dimension of 120 x 40 x 3 mm^3 were used in this study, and its chemical composition is listed in Table 1. Disc-shaped specimens with a diameter of 6 mm were cut from the as-rolled Inconel 718 sheet using wire electrical discharge machining to make the RDE for the electrochemical polishing and testing investigated in this study.

Table 1. Chemical composition of as-rolled Inconel 718 alloy used in this study.

Element	Ni	Fe	Cr	Nb	Mo	Ti	Al	Mn	Co	Si
wt.%	Bal.	19.5	17.65	5.14	2.94	0.99	0.60	0.1	0.14	0.15

The surface of the Inconel 718 RDE was mechanically ground with 600 grit emery paper, cleaned with distilled water, dried with a cold air blaster, and then prepared for electrochemical polishing and test.

The electrochemical test was conducted in a typical electrochemical three-electrode cell by using a potentialstat/galvanostat (EG&G Model 2263A). The Inconel 718 RDE was used as the working electrode. A platinum plate with an exposed area of 0.28 cm^2 and an Ag/AgCl electrode in saturated KCl solution were used as counter and reference electrodes, respectively. Analytical grade 70 wt% perchloric acid and 99.7 wt% acetic acid were mixed in various volume ratios for the electrochemical polishing and tests. The temperature for the electrochemical polishing and tests was kept at 25 ± 1^oC. The anodic polarization curve of the Inconel 718 RDE was measured potentiodynamically with a scan rate of 5 mV s^{-1} starting from -250 mV (*vs.* open-circuit potential) to 9 V (*vs.* $Ag/AgCl_{sat.}$). According to the anodic polarization behavior, the potential range corresponding to the limiting-current plateau was determined, and the subsequent AC impedance test and the potentiostatic polishing were conducted at this plateau.

The same pretreatment for the anodic polarization measurement of the Inconel 718 RDE was conducted for the AC impedance test. In the AC impedance test, four potential amplitudes, 20, 200, 500 and 1000 mV, were applied, respectively, to obtain the AC impedance responses of the Inconel 718 RDE polarized in the limiting-current plateau. The frequency range of the AC impedance test was varied from 0.001 Hz to 1 MHz.

After electrochemical polishing and the AC impedance test, the Inconel 718 RDE was cleaned with distilled water, acoustically cleaned in an ethanol bath for 5 min, dried with a cold air blaster, and then prepared for examination with a scanning electron microscope (SEM, Hitachi S3500N).

3. RESULTS AND DISCUSSION

3.1 Potentiostatic etching in the limiting-current plateau

Fig. 1 shows the anodic polarization curves of the Inconel 718 RDE in the mixed acids with various $HClO_4$ concentrations. It can be seen that a limiting-current plateau can be determined from each anodic polarization curve. In the mixed acids with 10 and 20 vol% $HClO_4$, the limiting-current densities were found at an anodic potential higher than 4.2 V. In the mixed acid with 30 vol% $HClO_4$, the limiting current density was detected at an anodic potential higher than 4 V. The limiting-current density in the mixed acids with 40 and 50 vol% $HClO_4$ were found in a potential range between 3.5 V and 4.5 V.

To study the anodic dissolution behavior under mass-transfer controlled limitation, potentiostatic etching of the Inconel 718 RDE was carried out in the limiting-current plateau. According to the anodic polarization curves shown in Fig. 1, potentiostatic etching was conducted at 7 V in the mixed acids with 10 and 20 vol% $HClO_4$, and at 4 V for the mixed acids with 30, 40, and 50 vol% $HClO_4$.

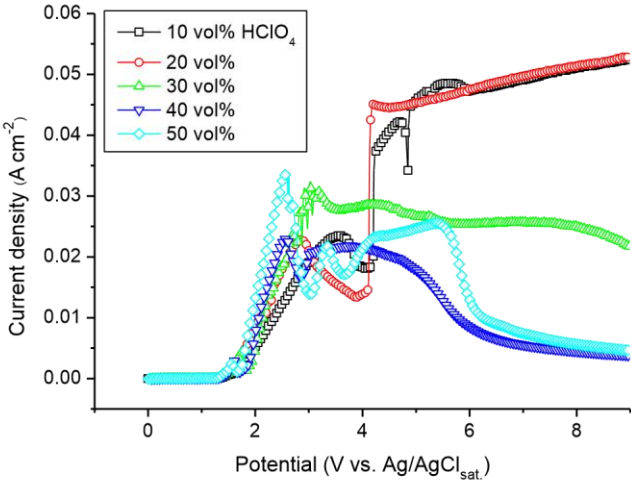
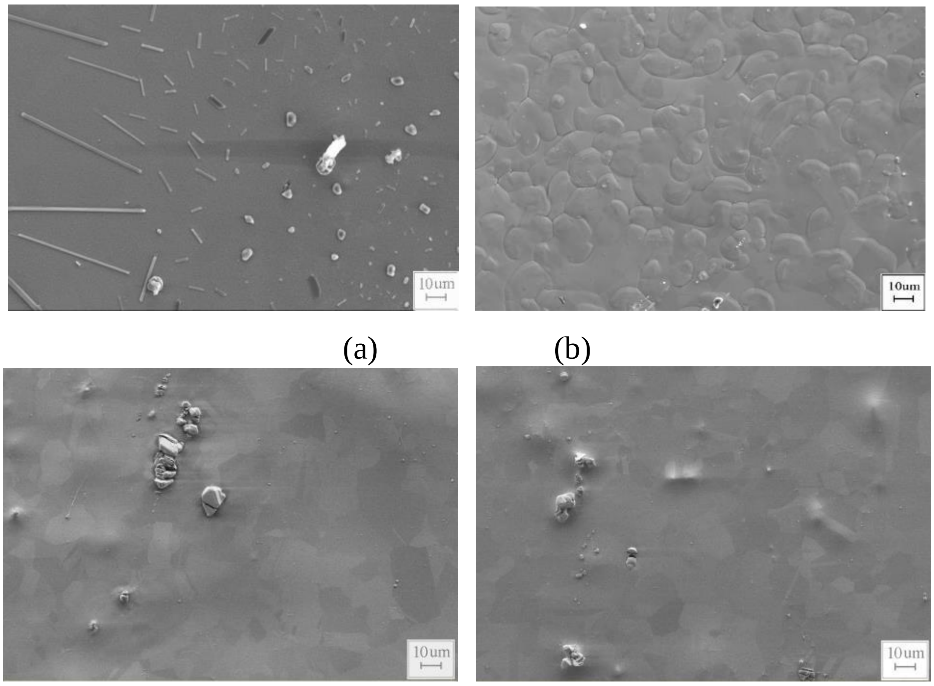


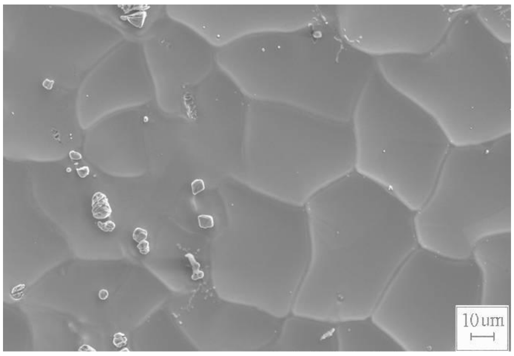
Figure 1. Anodic polarization curves of Inconel 718 specimens in the $HClO_4$-CH_3COOH mixed acids with various $HClO_4$ concentrations at 25℃.

After potentiostatic etching, the resultant surface morphologies of the Inconel 718 RDEs are presented in Figs. 2 (a)-(e). From the etched surfaces shown in Fig. 2, three types of anodic dissolution behavior can be characterized as a function of the $HClO_4$ content in the mixed acids. Leveling without brightening of the surface is the first behavior, which was observed from EP in mixed acids with 10 and 20 vol% $HClO_4$, as shown in Figs. 2 (a) and (b). The second behavior of anodic dissolution is observed from EP in mixed acids with 30% and 40 vol% $HClO_4$, wherein leveling and brightening of the surface was achieved (see Figs. 2 (c) and (d)). That is, a good EP effect on the Inconel 718 RDE was obtained in the second type of anodic dissolution. The final behavior is regarded as an irregular dissolution, and was observed from EP in mixed acids with 50 vol % $HClO_4$.



(a) (b)

(c) (d)



(e)

Figure 2. SEM micrographs of the surface morphologies of Inconel 718 RDEs after electrochemical polishing in the mixed acids with (a) 10, (b) 20, (c) 30, (d) 40 and (e) 50 vol% $HClO_4$.

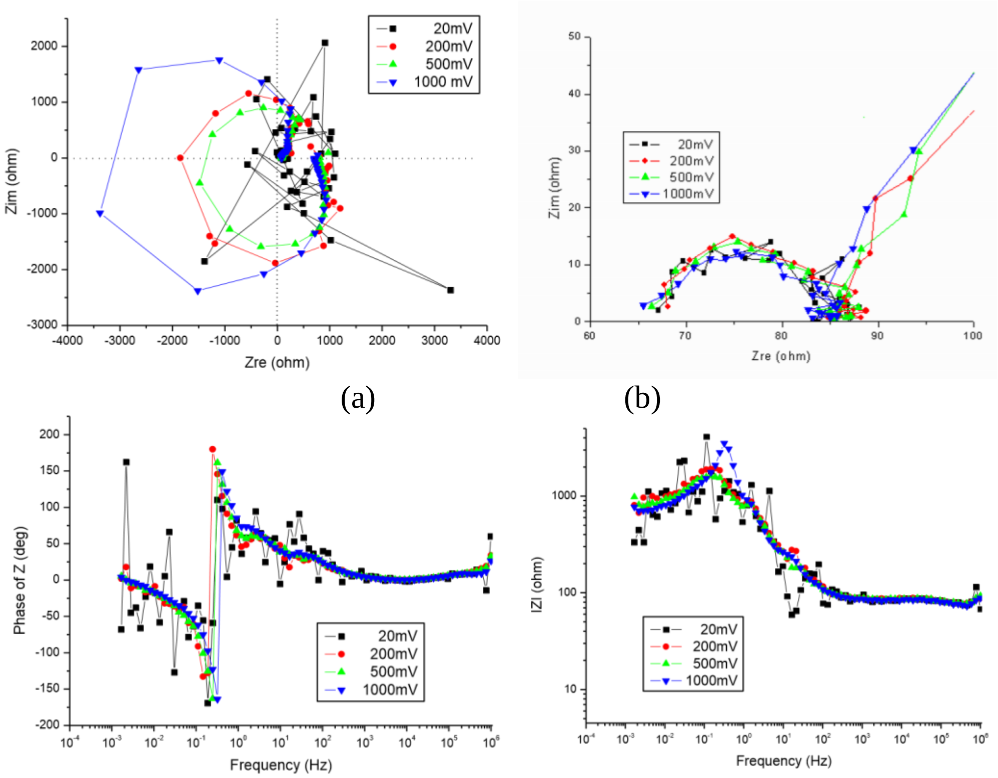
In this anodic dissolution type, a matte surface was achieved, as shown in Fig. 2 (e). A matte surface was also achieved when potentiostatic etching in mixed acids with a $HClO_4$ concentration higher than 50 vol%, meaning Inconel 718 alloy cannot be electrochemically polished in mixed acids with a 50 vol.% of $HClO_4$ or more.

3.2 AC impedance test

Based on three aforementioned anodic dissolution types during potentiostatic etching in mixed acids, the AC impedance test of the Inconel 718 RDE was conducted by polarizing the same potential used for potentiostatic etching in the limiting-current plateau in a selected mixed acid. In order to achieve a stable AC impedance response, the Inconel 718 RDE was potentiostatically etched for 600 s until a dynamically stable reaction between the RDE and electrolyte was established. The AC impedance test was then carried out at the same potential without interruption. The AC impedance test of the Inconel 718 RDE in a selected mixed acid adopted four potential amplitudes of 20, 200, 500, and 1000 mV. That is, four AC impedance responses were obtained for the EP behavior of an Inconel 718 RDE in a selected mixed acid. The AC impedance responses are expressed in the forms of Nyquist and Bode diagrams, respectively.

3.2.1 AC impedance responses in mixed acids with 10 and 20 vol% $HClO_4$

Figs. 3 and 4 show the AC impedance responses in mixed acids with 10 and 20 vol% $HClO_4$, respectively. Regardless of having the same EP effect of leveling without brightening, their AC impedance responses are quite different. Fig. 3(a) shows the Nyquist diagram of the Inconel 718 RDE in the mixed acid with 10 vol% $HClO_4$. A small Randle's circuit with a charge-transfer resistance of 20 Ohm can be identified in a relatively high frequency domain [see Fig. 3 (b)]. As presented in Fig. 3(c), a transition frequency of 0.3 Hz was detected, at which an abrupt change of phase angle from -180 to 180^o was measured. In the frequency domain lower than the transition frequency, a negative Randle's loop across the 2nd, 3rd, and 4th quadrants was identified. The small Randle's circuit is considered an anodic dissolution under the precipitation of the salt or viscous film mechanism [9,19,20]. A negative Randle's circuit is often referred to as a transpassive dissolution, which has been evidenced by Gabrielli et al. [21].

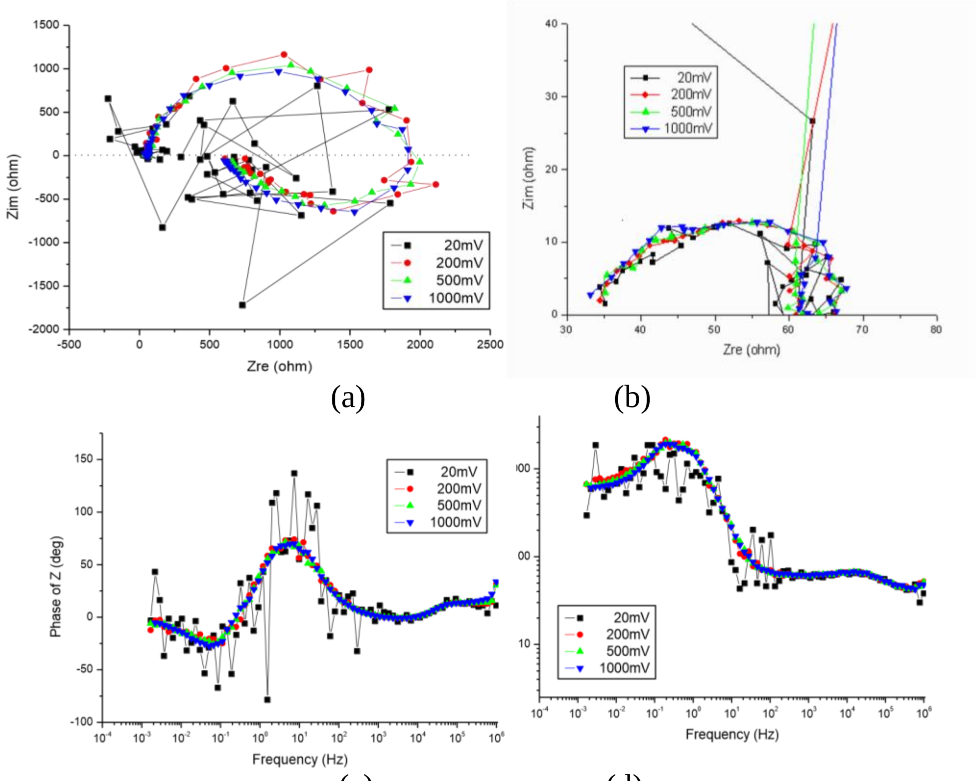


(a) (b)

(c) (d)

Figure 3. The AC impedance responses of Inconel 718 RDE tested with potential amplitudes of 20, 200, 500 and 1000 mV in the mixed acid with 10 vol% $HClO_4$. The responses are presented with Nyquist diagrams (a) in whole frequency and (b) in high frequency domains, and presented with Bode diagrams in (c) the relation of phase angle vs. frequency and in (d) impedance vs. frequency.

Figs. 4 (a) and (b) show the Nyquist diagrams corresponding to potentiostatic polishing in the mixed acid with 20 vol% $HClO_4$. In the frequency domain between 0.001 Hz and 10 kHz, the Nyquist diagram comprises a Randle's circuit and an inductance loop, which demonstrates anodic dissolution of a partial adsorbed surface [22], degradation behavior of metallic electrode [23], or localized dissolution [24]. In contrast to the AC impedance response in the mixed acid with 10 vol% $HClO_4$, there is no transition frequency in the Bode diagram obtained from the mixed acid with 20 vol% $HClO_4$ (see Figs. 4 (c) and (d)). However, in the high frequency domain (> 10 kHz), a Randle's circuit with a small charge-transfer resistance is observed. That is, two Randle's circuits in series with an inductance loop present the AC impedance response of the Inconel 718 RDE tested in the mixed acid with 20 vol% $HClO_4$. Nyquist plot for two capacitive reactance loops and a inductive impedance loop corresponded to the two kinds of equivalent circuits, as shown in Figure 4(b)(c).



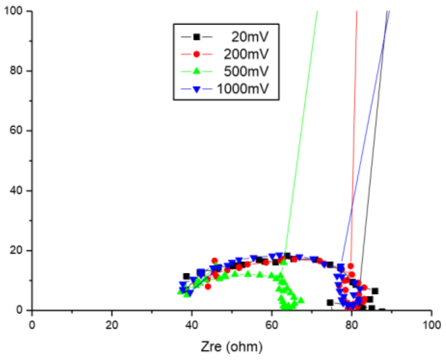
(a) (b)

(c) (d)

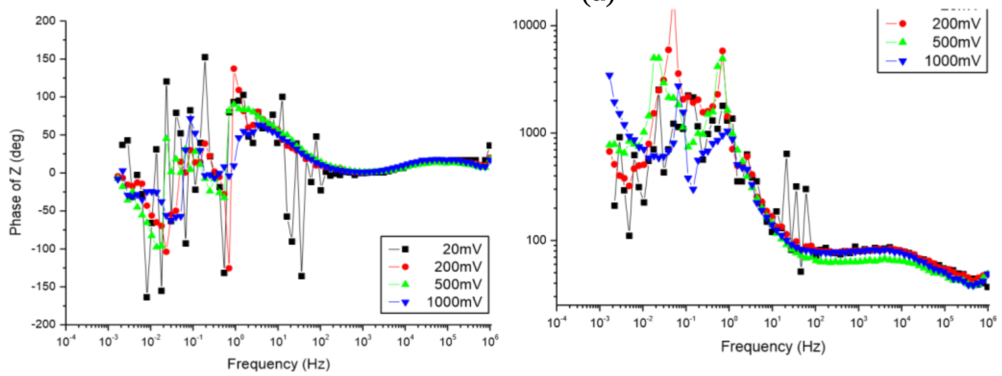
Figure 4. The AC impedance responses of Inconel 718 RDE tested with potential amplitudes of 20, 200, 500 and 1000 mV in the mixed acid with 20 vol% HClO4. The responses are presented with Nyquist diagrams (a) in whole frequency and (b) in high frequency domains, and presented with Bode diagrams in (c) the relation of phase angle vs. frequency and in (d) impedance vs. frequency.

3.2.2 AC impedance responses in the mixed acids with 30 and 40 vol% HClO4

The results of potentiostatic polishing show that leveling and brightening of the Inconel 718 surface were obtained in mixed acids with 30 and 40 vol% $HClO_4$. As shown in Figs 5 (b) and 6 (b), it is obvious that their AC impedance responses in the frequency domain are extremely random for frequencies lower than 1 Hz. At least two transition frequencies are detected from the Bode diagrams generated from mixed acids with 30 and 40 vol% $HClO_4$.

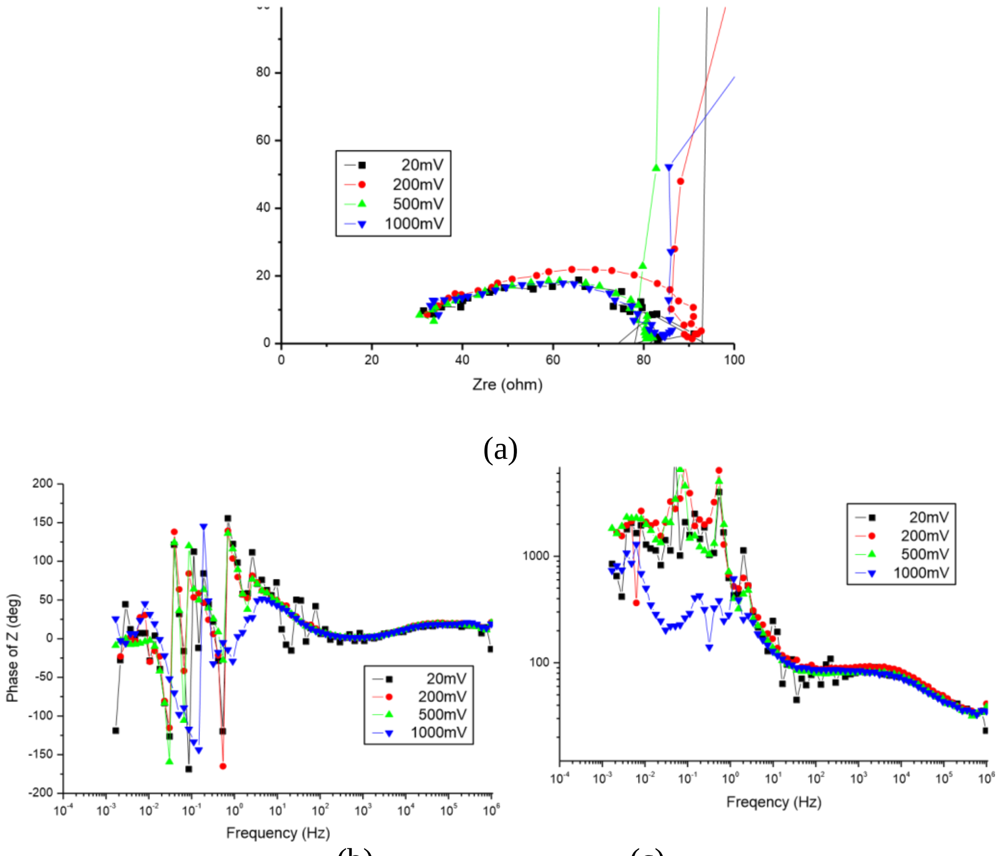


(a)



(b) (c)

Figure 5. The AC impedance responses of Inconel 718 RDE tested with potential amplitudes of 20, 200, 500 and 1000 mV in the mixed acid with 30 vol% $HClO_4$. The responses are presented with Nyquist diagrams in (a) high frequency domains, and presented with Bode diagrams in (b) the relation of phase angle vs. frequency and in (c) impedance *vs.* frequency.



(a)

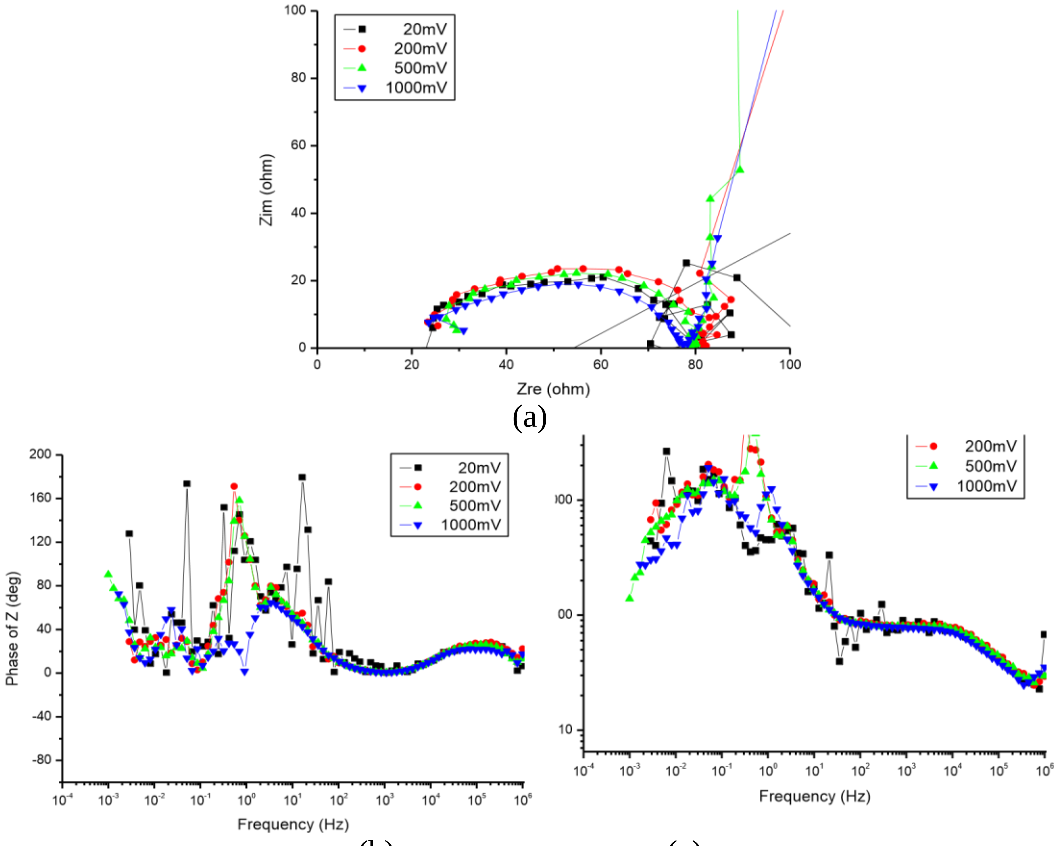
(b) (c)

Figure 6. The AC impedance responses of Inconel 718 RDE tested with potential amplitudes of 20, 200, 500 and 1000 mV in the mixed acid with 40 vol% $HClO_4$. The responses are presented with Nyquist diagrams (a) in high frequency domains, and presented with Bode diagrams in (b) the relation of phase angle vs. frequency and in (c) impedance *vs.* frequency.

Thus, any equivalent circuit cannot be determined from their AC impedance responses in the low frequency region. However, in the high frequency region (> 100 Hz), the Bode diagrams show smooth variation of phase angle, and the Nyquist diagrams present a similar response of a Randle's circuit with a Warburg impedance, as shown in Figs. 5 (a) and 6 (a). Small values of the charge-transfer resistance (Rct) are detected in the Randle's circuits, being 50 Ohm in the mixed acid with 30 vol% $HClO_4$ and 70 Ohm with 40 vol% $HClO_4$.

3.2.3 AC impedance response in the mixed acid with 50 vol% $HClO_4$

As mentioned in the above section, a matte surface of the Inconel 718 RDE was obtained when potentiostatically etched in mixed acids with a 50 vol% or higher $HClO_4$. The impendence responses observed for the potentiostatically etched RDE in acids with 50 vol% or higher $HClO_4$ trended similarly as the impedance responses obtained in the mixed acids with 30 and 40 vol% $HClO_4$. Irregular responses are observed in the low frequency domain ($\leq$ 2 Hz). In the high frequency domain, a Randle's circuit with a Warburg impedance is observed (see Fig. 7 (a)). An R_{ct} of 60 Ohm in the Randle's circuits is observed, as shown in Fig. 7 (b). Furthermore, a transition frequency of 0.6 Hz was observed, in which a change of phase angle from 0° to 180° was found.



(a)

(b) (c)

Figure 7. The AC impedance responses of Inconel 718 RDE tested with potential amplitudes of 20, 200, 500 and 1000 mV in the mixed acid with 50 vol% $HClO_4$. The responses are presented with Nyquist diagrams (a) in high frequency domains, and presented with Bode diagrams in (b) the relation of phase angle vs. frequency and in (c) impedance *vs.* frequency.

Although the EP effect on the Inconel 718 RDE in the mixed acid with 50 vol% $HClO_4$ is quite different from that in 30 or 40 vol% $HClO_4$, their AC impedance responses have the same trend. The AC impedance response has an irregular AC impedance response in the low frequency domain and a Randle's circuit with a Warburg impedance in the high frequency domain. However, the phase-angle change of the transition frequency in the AC impedance response obtained in the mixed acid with 50 vol% $HClO_4$ is quite different from those obtained with 30 and 40 vol% $HClO_4$. The former varies in a region with positive values, whereas the latter change from negative to positive values. This difference can also be realized from their Nyquist diagrams. Testing in the mixed acids with 30 and 40 vol% $HClO_4$, as shown in Figs. 5(a), 6(a) and 7(a), results in a small loop that can be observed in the transition from a semi-cycle of the Randle's circuit to a line relation of the Warburg impedance, whereas, the small loop was not found in the mixed acids with 50 vol% $HClO_4$.

It's well known that the EP mechanism of a metal component could be either salt-film-precipitated or acceptor-limited mechanism [9,26]. The anodic dissolution behavior is a mass-transfer controlled process during EP which is normally conducted in the limiting-current plateau of its anodic polarization curve. According to Matlosz et al. [26,27], the EP mechanism could be recognized from the result of AC impedance test conducted in the limiting-current plateau. As shown in Fig. 5(a), 6(a) and 7(a), the Nyquist diagrams can be expressed with an equivalent circuit of a Randle's circuit with a Warburg impedance. These results are fully in agreement with the salt-film precipitated mechanism for the mass-transfer controlled process proposed by Matlosz et al. [26,27]. In this EP mechanism, a salt film precipitated on the RDE surface during EP, leading to saturated Ni^{2+} cations adjacent to the RDE surface and fully exhausted Ni^{2+} cations within the diffusion layer. In our previous study, a salt film with a thickness about 2 μm was developed on the Inconel 718-RDE surface after potentiostatic EP in the mixed acids [28,29]. The corrosion resistance of Inconel 718 specimen was significantly increased after EP [28]. Increasing the corrosion resistance of a metal component through EP was also evidenced by many researchers [30,31].

In this study, the AC impedance test of the Inconel 718-RDE was polarized in the limiting-current plateau shown in Fig. 1. However, we found that the EP effect on the Inconel 718-RDE depends strongly on the $HClO_4$-content in the mixed acid. Leveling without brightening was found from the RDE surface after EP in the mixed acids with 10 and 20 vol% $HClO_4$. This could be recognized from their AC impedance responses shown in Figs. 3(a) and 4(a) in which a negative Randle's circuit was detected in the low frequency domain. Many researchers have pointed out that uneven etching takes place when a negative Randle's circuit is detected from its AC impedance response [21,23]. Therefore, electropolishing the Inconel 718-RDE in the mixed acids with 10 and 20 vol% $HClO_4$ achieved a rough surface in spite of polarizing in the limiting-current plateau. Interestingly, the EP mechanism of Inconel 718-RDE can be characterized as the salt-film precipitated mechanism in the mixed acid containing a $HClO_4$ content from 30 and 50 vol%. A good EP effect on the Inconel 718-RDE was observed in mixed acids with 30 and 40 vol% $HClO_4$; whereas, a mat surface was seen in the mixed acid with 50 vol% $HClO_4$. This implies that electropolishing the Inconel 718-RDE is not suitable in the mixed acid with 50 vol% $HClO_4$. The reason for this EP effect on the Inconel 718-RDE could be possibly realized from the anodic polarization curves shown in Fig. 1, in which an obvious decrease in the anodic current density was detected when the RDE was polarized at relatively high potential in the mixed acid with 40 vol% $HClO_4$ or more. This implies that RDE tends to be oxidized or passivated at relatively high anodic overpotential in the mixed acid with 40 vol% $HClO_4$ or more. Therefore, passivating the Inconel 718 surface takes place when polarized in the limiting-current plateau in the mixed acid with 50 vol% $HClO_4$, decreasing its anodic current density and achieving a mat surface.

3.3 The effect of the potential amplitude

An important advantage of the AC impedance test over other laboratory techniques is the possibility of using very small potential-amplitude signals without disturbing the electrochemical reaction being measured. Therefore the AC impedance response is often referred as a quasi-stable test. Normally, the AC impedance response of an electrochemical reaction is measured with a potential amplitude smaller than 20 mV [26,27]. However, in this study we confirm that relatively random and dispersive data were obtained when a potential amplitude of 20 mV was used in the AC impedance test, while a stable AC impedance response was obtained with a potential amplitude of 200 mV or 500 mV. The AC impedance response obtained using 20 mV follows approximately those responses obtained with 200 mV or 500 mV, meaning a stable AC impedance response could be achieved with a large potential amplitude when the Inconel 718 RDE was polarized at the potential located in the limiting-current plateau. This can be recognized by their Bode diagrams shown in Figs 3-6.

It is known that the anodic dissolution rate of a metallic electrode is limited by the diffusion rate of the reactive chemical species (acceptor or metallic ions) in the electrolyte when polarized in the limiting-current plateau [9,32]. That is, the anodic dissolution rate or the anodic current density of the electrode is constant in a potential range corresponding to the limiting-current plateau. In this study, we found that the current density increased slightly with an increased anodic potential in the limiting-current plateau. Moreover, a linear relation between potential and current density was detected in the limiting-current plateau of the mixed acid. Therefore, a large potential amplitude within the potential region corresponding to the limiting-current plateau could be reasonably used for the AC impedance test. During EP, a relatively high anodic density was detected by using potentiostatic etching. Fluctuant impedance values were measured by using a small potential amplitude of 20 mV when small variation of anodic current density occurred during potentiostatic etching in the limiting current plateau. Thus, an unstable AC impedance response could be obtained by using a small potential amplitude of 20 mV. On the other hand, relatively stable impedance values were detected by using a large potential amplitude when this variation of anodic current density took place. This suggests that using a large potential amplitude is helpful to achieve a stable AC impedance response in the limiting-current plateau.

Clearly, all AC impedance responses obtained with potential amplitudes of 200 and 500 mV were almost identical. On the other hand, the AC impedance responses obtained with a potential amplitude of 1000 mV in the mixed acids with 40 and 50 vol% $HClO_4$ have an apparent deviation from those responses with 200 and 500 mV in the low frequency domain (see Figs. 6 and 7). This could be attributed to that the potential ranges in their limiting-current plateaus are not wide enough.

Therefore, in testing with a potential amplitude of 1000 mV, the linear relationship between the potential and current density cannot be sustained in the limiting-current plateau. It means that a potential amplitude of 1000 mV is not suitable for obtaining an AC impedance response in the mixed acids.

4. CONCLUSION

The electropolishing behavior of the Inconel 718 alloy was studied with an AC impedance test in the $HClO_4$-CH_3COOH mixed acids with different concentrations of $HClO_4$. The AC impedance test was conducted with four potential amplitudes of 20, 200, 500, and 1000 mV at a potential located in the limiting-current plateau region. A relatively unstable AC impedance response was detected by using a small potential amplitude of 20 mV. In contrast, the electropolishing effect on the Inconel 718 alloy can be realized with its AC impedance response tested with relatively large potential amplitudes of 200 and 500 mV.

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References

1. R.G. Thmpson, D.C. Mayo, B. Radhakrishnan, *Metall. Trans.* A 22 (1991) 557.
2. Q. Chen, N. Kawagoishi, H. Nisitani, *Mater. Sci. Eng.* A 277 (2000) 250.
3. L. Nastac, D.M. Stefanescu, *Metall. Mater. Trans*. A 27 (1996) 4075.
4. M. J. Cieslak, T. J. Headley, G. A. Knorovsky, A. D. Romig, T. Kollie, *Metall. Trans*. A 21 (1990) 479.
5. J. Prybylowski, R. Ballinger, *Nat. Assoc. Corrs. Eng*. 43 (1987) 111.
6. C. A. Huang, T. H. Wang, C. H. Lee, W. C. Han, *Mater. Sci. Eng*. A 398 (2005) 275.
7. D. Wallinder, J. Pan, C. Leygraf, A. Delblanc-Bauer, *Corros. Sci*. 42 (2000) 1457.
8. *Annual book of ASTM Standards, Part 9 B 374,* ASTM. Philadelphia, PA , 1979
9. D. Landolt, *Electrochim. Acta*, 32 (1987) 1.
10. P. A. Jacquet, *Nature,* 135 (1935) 1076.
11. T. P. Hoar, D. C. Mears, G. P. Rothwell, *Corros. Sci.,* 5 (1965) 279.
12. T. P. Hoar, G. P. Rothwell, *Electrochim. Acta,* 9 (1964) 135.
13. S. Pednehar, S. Smialowska, *Corrosion* 36 (10) (1980) 565.
14. S. C. H. Huang, Y. Pan, *Corros. Sci*. 48 (1992) 594.
15. P. Chung, S. S. Smialowska, *Corrosion* 37 (1981) 39.
16. V. B Singh, Archana Gupta, *Mater. Chem. Phys*. 85 (2004) 12.
17. J. R. Sculllly, R.G. Kelly, *Corrosion* 42 (1986) 537.
18. W. K. Kelly, R. Lyer, W. Pickering, *J. Electrochem. Soc*. 140 (1993) 3134.
19. S. Magaino, M. Matlosz, D. Landlt, *J. Electrochem. Soc*., 140 (1993) 1365.
20. R. Vidal, A.C. West, *J. Electrochem. Soc.,* 145 (1998) 4067.
21. C. Gabrielli, M. Keddam, *J. Electrochem. Soc*., 129 (1982) 2872.

22. W. J. Lorenz, F. Mansfeld, *Corros. Sci*., 21 (1981) 647.
23. A. Pardo, M.C. Merino, A.E. Coy, F. Viejo, R. Arrabal, S. Feliu Jr., *Electrochim. Acta*, 53 (2008) 7890
24. C. A. Huang, Y.-Z. Chang, S. C. Chen, *Corros. Sci.*, 46 (2004) 1501.
25. J. M. Esteban, M. E. Orazem, *J. Electrochem. Soc.*, 138 (1991) 67.
26. M. Matlosz, S. Magaino, D. Landolt, *J. Electrochem. Soc.*, 141 (1994) 410.
27. M. Matlosz, *Electrochim. Acta*, 40 (1995) 393.
28. C. A. Huang, Y. C. Chen, J. H. Chang, *Corros. Sci.*, 50 (2008) 480.
29. C. A. Huang, Y. C. Chen, *Corros. Sci.*, 51 (2009) 1901.
30. M. Sowa, K. Gren, A. I. Kukharenko, D. M. Korotin, J. Michalska, L. Szyk-Warszynska, M. Mosialek, J. Zak, E. Pamula, E.Z. Kurmaev, S. O. Cholakh, W. Simka, *Mater. Sci. Eng. C*, 42 (2014) 529.
31. W. Simka, M. Mosialek, G. Nawrat, P. Nowak, J. Zak, J. Szade, A. Winiarski, A. Maciej, L. Szyk-Warszynska, *Surf. Coat. Tech.*, 213 (2012) 239.
32. C. A. Huang, J. H. Chang, W. J. Zhao, S. Y. Huang, *Mater. Chem. Phys.*, 146 (2014) 230.