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Development of EPR Test Technique for Alloy 800

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The degree of sensitization (DOS) of austenitic stainless steels and some nickel-based alloys (e.g., alloy 600) is evaluated by the electrochemical potentiokinetic reactivation (EPR) test. In this study a number of test solutions based on H_2SO_4 + KSCN composition have been evaluated to establish a reliable EPR test method for alloy 800. Different passivation (vertex) potentials are also tested. It has been shown that dilute test solutions with lower vertex potentials resulted in single loop (SL) and double loop (DL) EPR test methods that distinguished between different sensitized samples and also between sensitized and desensitized samples. It has been shown that an SL-EPR test in 1 M H_2SO_4 + 0.002 M KSCN (de-aerated) at 26 °C at a scan rate of 3 mV/s from a vertex potential of 700 mV_{SCE} (180 s hold time) gave results that matched with the DOS indicated by microstructures and the Huey test results. Similarly, the DL-EPR test in 1 M H_2SO_4 + 0.002 M KSCN (de-aerated) at 26 °C, forward scanning from the OCP to + 700 mV_{SCE} and then backward scanning from there to the OCP at a scan rate of 2 mV/s produced a good measure of DOS as indicated by the Huey test results. The effectiveness of the EPR test was ascertained by testing on alloy 800 containing Ti and Al (alloy 800 HT) and Nb (alloy 800 Nb).

Keywords alloy 800, alloying elements, degree of sensitization, double loop test, electrochemical potentiokinetic reactivation test, Huey test, intergranular corrosion, single loop test, vertex potential

1. Introduction

Alloy 800 is an iron-based alloy with high nickel and high chromium content, which has good resistance to oxidation and carburization at high temperatures. Alloy 800 is used as a construction material for steam generator (SG) tubing in pressurized heavy water reactors (PHWRs) that operate at temperatures around 300 °C.[1,2] Various failure modes observed in SG tubing materials[1-4] are pitting corrosion, and stress corrosion cracking (SCC) of tubes on both primary and secondary side.[4] Alloy 800 is susceptible to sensitization due to precipitation of carbide ($M_{23}C_6$ type) at grain boundaries (GB). The precipitation of carbide at GB leads to formation of chromium depletion zone adjacent to it. This may lead to intergranular corrosion (IGC) and to intergranular stress corrosion cracking (IGSCC). Sensitization can occur in the temperature range of 500-800 °C, during stress relieving or due to slow cooling from high temperature after welding. Isothermal heat treatments in this temperature range cause sensitization but longer-term heat treatments lead to desensitization. This is attributed to high rates of chromium diffusion in the alloy matrix. In addition to this, low temperature sensitization[5] also occurs in this alloy (e.g., after heating for 30 000 h at 450 °C).

As sensitization can have a substantial effect on the in-service performance of the alloy 800, it is essential to characterize and quantify the same. The ASTM A 262 Practice A and Practice C are used for evaluation of degree of sensitization (DOS) in stainless steels (SS). These methods can also be used for the evaluation of DOS in alloy 800. Also, electrochemical potentiokinetic reactivation (EPR) method used for the quantification of sensitization in SS, can also be used for alloy 800.[6,7] Edgemon et al.[8] had studied the evaluation of DOS by EPR for alloy 800H by modifying EPR test parameters.

High nickel austenitic steels contain large number of precipitates and solution hardening elements (e.g., titanium, aluminum, and/or niobium) that form secondary phases. This may lead to the presence of a secondary anodic current peak in the EPR test curve. Moreover, general surface activation may lead to an extra peak in EPR curves due to low passivation potentials.[8] At higher potentials in the transpassive region, the segregation of elements like phosphorus, sulphur, and silicon at GB significantly contributes to anodic current.[9] All of these factors affect the acquisition of current during EPR tests for alloy 800 and may lead to spurious results.

The aim of the current study is to modify and optimize EPR test parameters for alloy 800 so that the EPR test can be used for evaluation of DOS of annealed and sensitized specimens. Single loop EPR (SL-EPR) and double loop EPR (DL-EPR) tests were chosen for the present investigation. Specimens were also subjected to anodic polarization and ASTM A 262 Practice C (Huey test). This test is a conventional test for determination of DOS in SS. The results of the Huey test were compared with the results of the EPR tests. Alloy 800 with additions of titanium and aluminum (alloy 800 HT) and niobium (alloy 800 Nb) were also tested to ascertain the effectiveness of the EPR test and the role of alloying additions in this alloy.

2. Experimental

2.1 Materials and Heat Treatment

The material for this investigation was a rolled sheet of 2 mm thickness. The chemical composition of the material is given in the Table 1. Specimens were sealed in silica tubes filled with argon gas and given sensitization heat treatment in the range of 500-850 °C for different durations.

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Table 1 Chemical Composition of Alloy 800, Alloy 800 HT, and Alloy 800 Nb Used in the Study

All in wt.%	C	Ni	Cr	Mn	Si	Ti	Al	Co	N	P	S	Others	Fe
Alloy 800	0.035	30.1	19.5	0.72	0.4	0.5	0.3	0.12	0.02	0.012	0.012	…	Bal
Alloy 800 HT	0.065	30.61	19.15	0.60	0.44	0.86	0.85	…	…	0.014	0.0024	Cu-0.48	Bal
Alloy 800 Nb	0.10	32.87	21.06	1.15	0.57	…	…	…	…	0.026	0.026	Nb-0.86, Mo-0.016	Bal

2.2 Anodic Polarization

Specimens prepared for anodic polarization tests were approximately 1 cm^2 in area and mounted in cold-setting resins. Specimens were polished on different grades of silicon carbides (emery) papers and finally with 1 μm diamond paste to obtain scratch-free, mirror-like surfaces. Specimens were subjected to anodic polarization in 1 M H_2SO_4 at room temperature with the scan rate of 1 mV/s.

2.3 SL-EPR Test

As with anodic polarization tests, specimens were polished to a mirror-like, scratch-free surface for all the EPR tests. The SL-EPR tests were carried out in 1 M H_2SO_4 + 0.005 M KSCN at room temperature. Four different passivation potentials (400, 700, 800, and 1100 mV_{SCE}) were chosen with the scan rate of 2, 3, 2, and 3 mV/s, respectively, for the single loop EPR test. In this test, specimens were held at passivation potentials for 180 s followed by a potential scan towards the open circuit potentials (OCP) at a chosen scan rate. For the SL-EPR, DOS values were calculated using the following formula.

$$P_a = \frac{Q}{GBA} \quad \text{(Eq 1)}$$

Where, Q = total charge consumed during SL-EPR experiment, GBA = effective grain boundary area = A_s 5.09544 × 10^{-3} exp(0.3496X), A_s = specimen area exposed to EPR solution, X = ASTM grain size number of the specimen at 100X.

2.4 DL-EPR Test

The DL-EPR tests were carried out in different electrolytes as summarized in Table 2. All the solutions were prepared fresh from reagent grade chemicals. All EPR tests were carried out using a computer-controlled potentiostat. The DL-EPR tests were carried out at room temperature (26 °C) with the scan rate of 2 mV/s. The surface finish was the same as given in the SL-PER tests.

The results for DL-EPR are reported as ratio of current density (A/cm^2) and charges consumed (C/cm^2) during reactivation scans to that with activation scans.

$$I_r = \frac{I_c}{I_{max}} \times 100 \quad \text{(Eq 2)}$$

$$C_r = \frac{C_c}{C_{max}} \times 100 \quad \text{(Eq 3)}$$

Where, I_r = current ratio, I_c = maximum current density during the reactivation scan, I_{max} = maximum current density during the activation scan, C_r = charge ratio, C_c = charge measured during reactivation scan, and C_{max} = charge measured during activation scan.

Table 2 Composition of Various Test Solutions and the Passivation Potentials for the EPR Test

Sr. No.	H_2SO_4	KSCN	Passivation Potential, mV_{SCE}
A	0.5 M	0.001 M	600
B	1.0 M	0.001 M	600
C	1.0 M	0.002 M	700
D	1.0 M	0.005 M	700
E	1.0 M	0.01 M	700

In addition to this, to study the effect of sensitization treatments on values of DOS, relative values of I_r and C_r of the sensitized specimens to that of annealed specimens were calculated using following equations:

$$I_m = \frac{I_1 - I_0}{I_0} \quad \text{(Eq 4)}$$

$$C_m = \frac{C_1 - C_0}{C_0} \quad \text{(Eq 5)}$$

Where, I_m = modified current ratio, I_1 = current ratio for sensitized sample as calculated by Eq 2, I_0 = current ratio for annealed sample as calculated by Eq 2, C_m = modified charge ratio, C_1 = charge ratio for sensitized sample as calculated by Eq 3, and C_0 = charge ratio for annealed sample as calculated by Eq 3.

Each test solution was de-aerated with commercial purity argon for a minimum of 45 min before and during each experiment. In the DL-EPR test, the potential was scanned from OCP to Passivation Potential (Vertex Potential) and then back to OCP.

2.5 ASTM 262 Practice A and Practice C

All the specimens were subjected to ASTM Practice A 262 practice A for quick characterization of the microstructure of sensitized samples. Also, ASTM A 262 Practice C (Huey test) was carried out to ascertain the validity of the various EPR parameters as this test has been accepted as a conventional test for the determination of DOS values in SS. For this test, specimens were polished on emery papers, dried, cleaned, and weighed before exposure to the test solution. The 65% nitric acid solution was prepared from reagent grade nitric acid, as described in ASTM 262. Specimens were subjected to boiling solutions of 65% nitric acid for three consecutive periods, each of 48 h. After every 48 h, specimens were taken out of solution dried and weighed and again exposed in fresh, boiling 65% nitric acid solution.

2.6 Effects of Alloying Additions on DOS

To study the effects of alloying additions on the DOS and if the developed EPR test technique is able to detect the DOS in alloy 800 with additional alloying elements, two different heats of alloy 800 were used. These were alloy 800 HT and alloy 800 Nb. The chemical composition of these two heats is given in Table 1. Different samples of these alloys were heat treated at 675 °C for 6 h and 24 h. The anodic polarization and the DL-EPR tests were carried out on the annealed and heat treated samples of these alloys as per the procedure mentioned in sections 2.2 and 2.4, respectively. The exposure tests in the solution of practice C, A262, ASTM (as described in section 2.5) were also done on these samples.

3. Results

3.1 Anodic Polarization

The anodic polarization curve, obtained for the annealed sample of the alloy 800 exhibited an active-passive behavior typical of austenitic stainless steels (Fig. 1). However for annealed and sensitized specimens, a second anodic current peak appeared during the anodic polarization. The Flade potentials for sensitized specimens were found to be more positive compared with annealed sample (e.g., for 650 °C/1 h specimen, the Flade potential was 100 mV_{SCE} while that for the annealed sample was −100 mV_{SCE}). These anodic polarization curves for the sensitized samples are also shown in Fig. 1.

3.2 SL-EPR Test

As comparisons were made for the specimens of the *same alloy* (with same grain size and exposed area) but with *different sensitization* treatments, the GBA factor in Eq 1 is ignored. The maximum current density measured and the charge consumed during the single loop EPR test is tabulated in Table 3 and 4, respectively. The modified values for these parameters as calculated using Eq 4 and 5 are also given in these tables.

Table 3 Maximum Current Density (in mA/cm^2) Measured During the Single Loop EPR Test as Calculated by Eq. 1; Values in Bracket-Modified Current Density as Calculated by Eq 4

Materials Condition	400 mV_{SCE}, 2 mV/s	800 mV_{SCE}, 2 mV/s	700 mV_{SCE}, 3 mV/s	1100 mV_{SCE}, 2 mV/s
Annealed	9.549 (0)	0.6042 (0)	0.2475 (0)	9.558×10^{-3} (0)
650 °C/1 h	28.1 (1.94)	19.79 (31.75)	8.541 (33.509)	…
650 °C/6 h	30.79 (2.220)	35.08 (57.06)	11.75 (46.47)	1.327 (137.8)
820 °C/0.5 h	21.93 (1.2965)	8.403 (12.90)	5.832 (22.560)	74.88 (6.834)
820 °C/18 h	12.00 (0.257)	37.41 (5.19)	1.953 (6.89)	4.653×10^{-3} (0.53)

Table 4 Total Charge Measured During Single Loop EPR Test as Calculated by Eq. 1 in C/cm^2, Values in Bracket-Modified Charge Measured as Calculated by Eq 5

Materials Condition	400 mV_{SCE}, 2 mV/s	800 mV_{SCE}, 2 mV/s	700 mV_{SCE}, 3 mV/s	1100 mV_{SCE}, 2 mV/s
Annealed	0.5477 (0)	0.02553 (0)	0.00628 (0)	4.08×10^{-4} (0)
650 °C/1 h	1.504 (1.749)	1.278 (48.475)	0.3623 (56.690)	…
650 °C/6 h	1.955 (2.570)	2.510 (97.315)	0.5795 (91.270)	0.05153 (125.17)
820 °C/0.5 h	1.303 (1.382)	0.5235 (19.505)	0.2049 (31.267)	0.002276 (4.570)
820 °C/18 h	0.6825 (0.247)	0.0374 (0.465)	0.05516 (7.780)	7.813×10^{-4} (0.53)

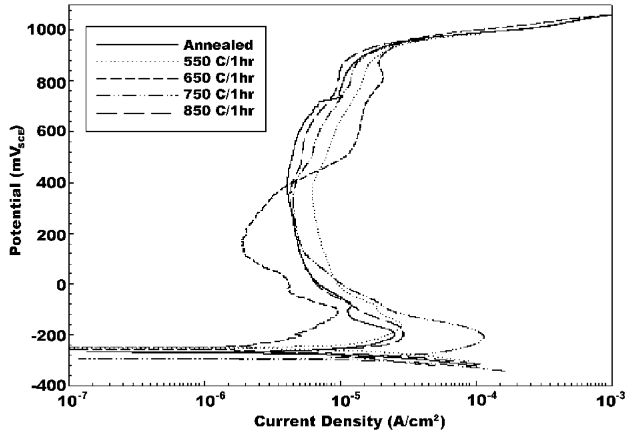

Fig. 1 Anodic polarization curves for different specimens in 1 M H_2SO_4 at room temperature

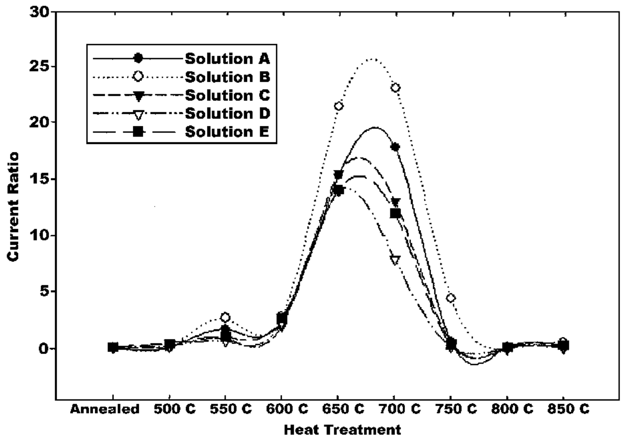

Fig. 2 DOS values, expressed by current ratio, for different EPR test solutions

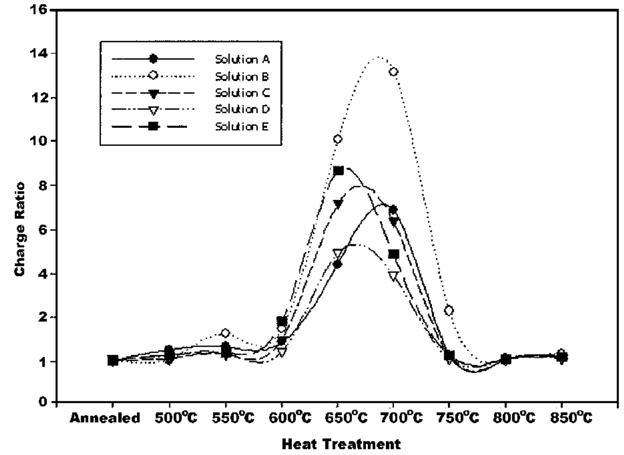

Fig. 3 DOS values, expressed by charge ratio, for different EPR test solutions

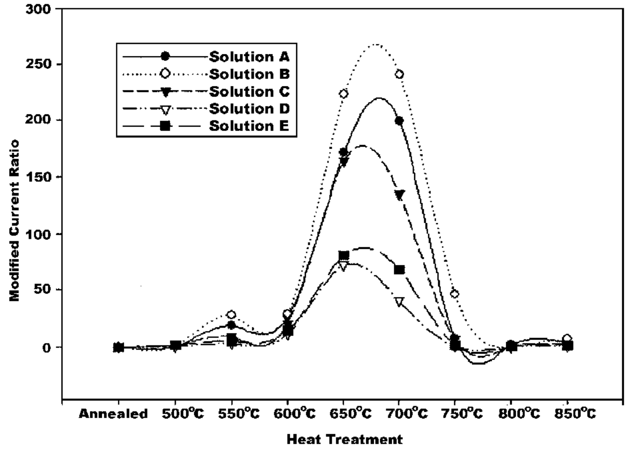

Fig. 4 DOS values, by expressed modified current ratio, for different EPR test solutions

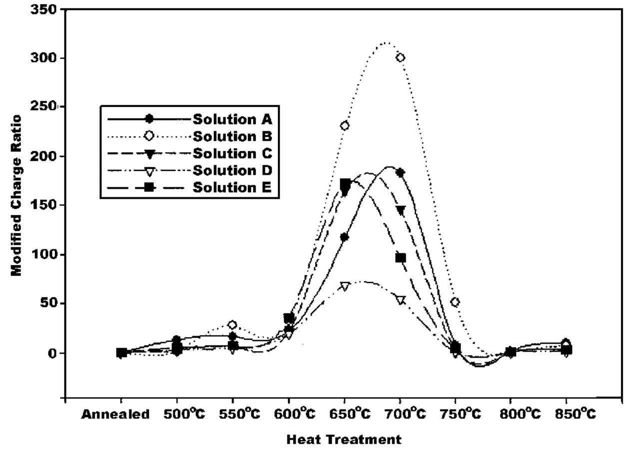

Fig. 5 DOS values, expressed by modified charge ratio, for different EPR test solutions

Table 5 Current Ratio (I_r) and Modified Current Ratio (I_m) for EPR Solution C for Different Sensitization Treatments

Materials Condition	Avg. I_r	I_m
Annealed	0.0943	0
500 °C/1 h	0.1995	1.1155
550 °C/1 h	0.9026	8.5716
600 °C/1 h	2.0483	20.7211
650 °C/1 h	15.5963	164.390
700 °C/1 h	12.8199	134.9480
750 °C/1 h	0.7224	6.6606
800 °C/1 h	0.1508	0.59915
850 °C/1 h	0.2878	2.05196

Table 6 Charge Ratio (C_r) and Modified Charge Ratio (C_m) for EPR Solution C for Different Sensitization Treatments

Materials Condition	Avg. C_r	C_m
Annealed	0.0434	0
500 °C/1 h	0.1155	1.6613
550 °C/1 h	0.3259	6.5092
600 °C/1 h	0.9964	21.9585
650 °C/1 h	7.1852	164.5576
700 °C/1 h	6.3913	146.265
750 °C/1 h	0.2317	4.3387
800 °C/1 h	0.0693	0.5968
850 °C/1 h	0.1390	2.2027

3.3 DL-EPR Test

The results of double loop EPR tests for all the solutions mentioned in Table 2 are summarized in Fig. 2 and 3. The modified current ratios and charge ratios are shown in Fig. 4 and 5. The EPR test results showed that the DOS values are the highest in the temperature range of 650-700 °C. The DOS value for 850 °C/1 h sample was higher than that for 800 °C/1 h sample. This is attributed to precipitation of secondary phases at that temperature.

The difference between DOS values of two samples with nearly equal sensitization treatments (e.g., 500 °C/1 h and 550°/1 h) is sufficiently large when tested in solution C. In addition to this, no loose corrosion products were observed for highly sensitized specimens after DL-EPR tests using solution C. In view of this, solution C was chosen for EPR test and subsequent EPR tests were carried out using this solution. The DL- EPR tests were repeated three times for each specimen to ensure the reproducibility and reliability of DOS values. The results of these tests are given in Table 5 and Table 6 as current ratio and charge ratio. Modified current ratio and charge ratio are given in the last column of respective tables.

To study desensitization, two specimens were sensitized for 24 h and 120 h at 700 °C. Specimens were prepared for the EPR test from these sensitized specimens and EPR tests were carried out as described earlier. I_r and C_r values for 700 °C/24 h sample were 0.44 and 0.314 and that of 700 °C/120 h sample were 1.2055 and 0.8318. Therefore, this set of EPR parameters

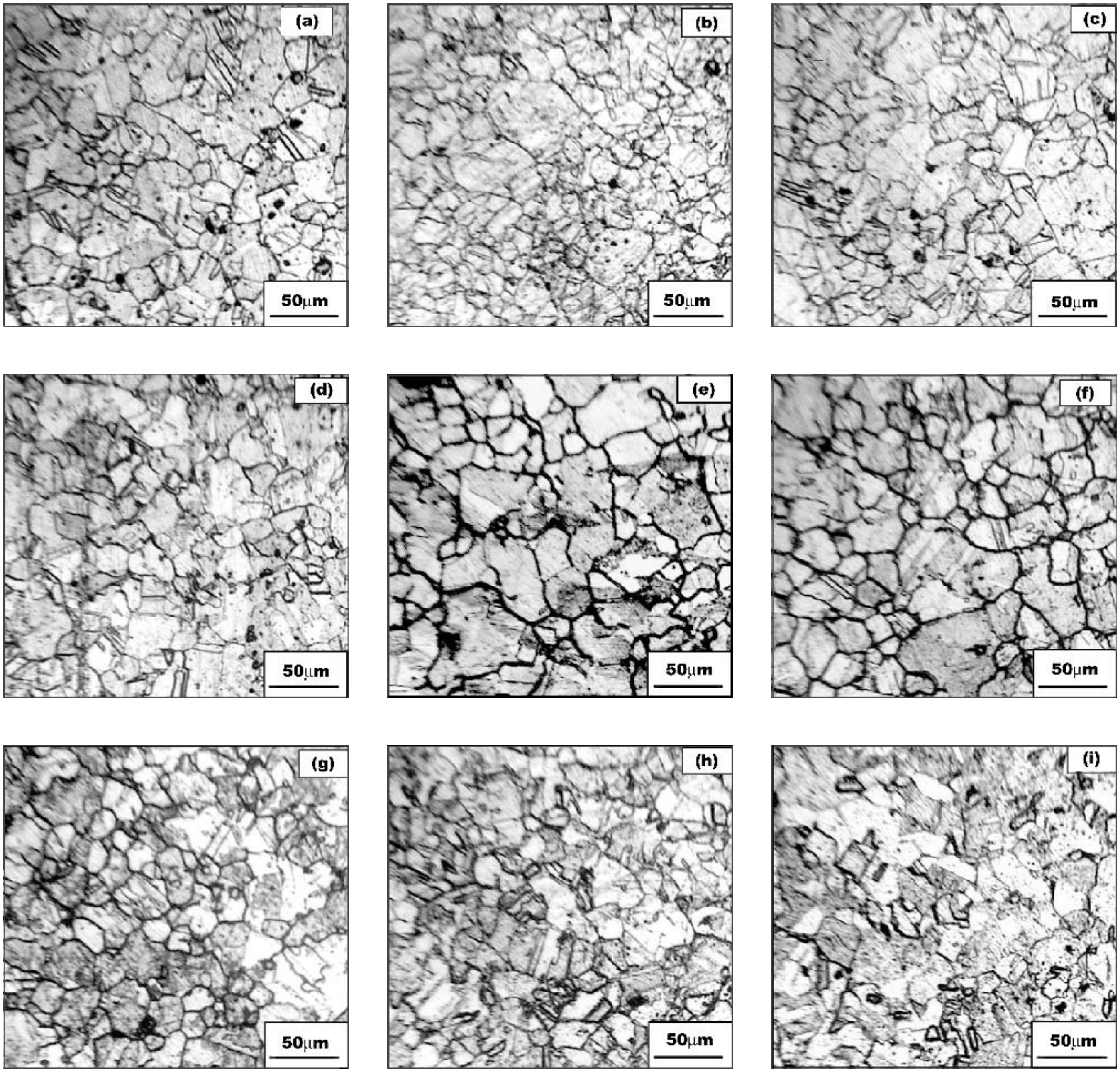

Fig. 6 Microstructure of samples after the DL-EPR test in solution C: **(a)** annealed specimen; **(b)** 500 °C/1 h specimen; **(c)** 550 °C/1 h specimen; **(d)** 600 °C/1 h specimen; **(e)** 650 °C/1 h specimen; **(f)** 700 °C/1 h specimen; **(g)** 750 °C/1 h specimen; **(h)** 800 °C/1 h specimen; and **(i)** 850 °C/1 h specimen

successfully detected desensitization also. It can be also be noticed that I_m and C_m values give better indication of the extent of sensitization as compared with I_r and C_r values.

3.4 Microstructure After EPR Tests

The microstructures of each sample after the EPR test were recorded and shown in Fig. 6a-g. Specimens 650 °C/1 h and 700 °C/1 h exhibited ditch microstructures as indicated by their I_r and C_r values. The annealed sample showed a "step" microstructure. All other specimens showed "dual" type microstructure. Pitting was not observed in any specimen. Also, there was no heavy general corrosion for highly sensitized specimens.

3.5 ASTM A 262 Practice A and Practice C

All specimens were subjected to ASTM A 262 Practice A and it was observed that heat treated specimens 650 °C/1 h and 700 °C/1 h showed "ditch" type microstructure. The annealed specimen showed "step" structure and moderately sensitized

Table 7 Corrosion Rates of Specimens as Measured in the Fifth Period of Practice C, A262, ASTM

Materials Condition	Corrosion Rate mpy, mm/y
Annealed	4.5574 (0.1139)
550 °C/1 h	1.9489 (0.0487)
650 °C/1 h	72.995 (1.8248)
650 °C/6 h	379.42 (9.4855)
650 °C/24 h	55.00 (1.375)
700 °C/1 h	12.541 (0.3135)
750 °C/1 h	4.3325 (0.1038)
750 °C/24 h	20.029 (0.500)
820 °C/0.5 h	4.9542 (0.1238)

mpy = mils per year. 1 mpy = 0.0254 mm/y

specimen like 500 °C/1 h and 550 °C/1 h exhibited "dual" type microstructures.

The results of Huey test are depicted in Table 7. These results confirm the sensitization behavior of alloy 800 as evaluated from the EPR tests. Therefore, it can be said the both SL-EPR and DL-EPR successfully evaluate DOS values for different sensitization treatments.

3.6 Effects of Alloying Additions on DOS

The anodic polarization curves for the alloy 800 HT and alloy 800 Nb are given in Fig. 7(a) and (b), respectively. The DL-EPR test was done in solution C at 2 mV/s at room temperature (26 °C) as described in section 2.4. The DL-EPR ratios are given in Table 8 for both the alloys. The results of the practice C, A262, ASTM test are also given in Table 8. The corrosion rates for alloy 800 Nb were high, therefore the corrosion tests were terminated after the second period of the practice C, A262, ASTM and those corrosion rates are reported for alloy 800 Nb in Table 8.

4. Discussion

4.1 Anodic Polarization Test

As shown in Fig. 1, the secondary peak occurs at around -50 mV_{SCE} for the sensitized specimen. The samples that were heat treated for longer duration showed little variation in secondary peak current density values than that for the samples heat treated for short duration. Also there was not much difference in the secondary peak current density values for the samples heat treated at 650 °C and 820 °C. Therefore, the secondary peak current density is not a good measure of DOS.

4.2 SL-EPR Test

As seen in Table 3 and 4, the specimens heat treated at 650 °C for 1 and 6 h showed higher values of DOS compared with other specimens. The primary objective of the current study was to modify EPR parameters to evaluate DOS values in moderately to highly sensitized specimens. In addition to this, the EPR test must adequately resolve DOS values of samples with nearly similar sensitization treatments. The results of the Huey test (Table 7) showed that the specimen heat treated at 650 °C for 6 h showed the maximum intergranular corrosion rate (hence maximum DOS) and the ranking by DOS as indicated by the intergranular corrosion rates are as follows: 650 °C for 6 h > 650 °C for 1 h > 820 °C for 0.5 h > annealed > 820 °C for 18 h. For the SL-EPR test parameter 400 mV_{SCE} – 2 mV/s, the difference in DOS values of moderately and highly sensitized specimens is very small; therefore it is not sensitive enough to distinguish the extent of sensitization. And for the higher potential of 1100 mV_{SCE}, the ranking of alloys by the EPR test does not match the ranking as per the Huey test. In addition, the segregation of elements like phosphorus, sulphur, and/or silicon is reported[9] to contribute to current and charge values at high (transpassive) potentials. Hence these conditions cannot be used for the evaluation of DOS. In view of this, it can be seen that the EPR test parameters, namely 800 mV_{SCE} – 2 mV/s and 700 mV_{SCE} – 3 mV/s, adequately evaluate DOS values. Because the passivation potential of $800_{mV(SCE)}$ is towards the transpassive region, it was decided to carry out further DL-EPR tests with passivation potential of 700 and with scan rate of 2 mV/s. This also lowers time for EPR tests.

4.3 DL-EPR Test

As mentioned earlier, the primary objective of the current study was to modify EPR parameters such that EPR tests must adequately resolve DOS values of two samples with nearly similar sensitization treatments. Figure 2-5 indicate that the EPR test results using EPR solution A and B did not adequately resolve DOS values for moderately sensitized specimens, e.g., 500 °C/1 h, 550 °C/1 h, and 600 °C/1 h. The same trend was observed for EPR tests using solution D and E. In addition, DL-EPR tests using solution A and solution B showed that 700 °C/1 h specimen has higher values compared with 650 °C/1 h; however, the results of the Huey test and DOS values obtained using all other solutions indicated that this is not the case. Moreover, visual observation of highly sensitized specimens, namely 650 °C/1 h and 700 °C/1 h tested in solutions D and E, revealed the presence of loose thin corrosion products, indicating that the specimens had undergone uniform corrosion during EPR tests.

Note that the results in Fig. 2-5 showed maximum DOS for the specimen heat treated at 650 °C for 1 h. This is confirmed by the microstructure in Fig. 6 that showed higher degree of "ditching" for the specimen sensitized at 650 °C than that for the sample sensitized at 700 °C. Also note that all other solutions (A, B, D, and E) showed higher DOS for the specimen sensitized at 700 °C than that sensitized at 650 °C. Therefore, it can be elicited that EPR solutions A, B, D, and E would not produce optimized DOS values. It can be noticed from Fig. 2-5 and Tables 6-7 that the EPR test solution C did adequately resolve DOS values for moderately sensitized specimens. As seen from Table 5 and 6, all the results for a given sensitization treatment were within a narrow range and the overall reproducibility of EPR test is good for this set of parameters. The results on desensitized specimen (section 3.3) also showed that this test can distinguish between sensitized and desensitized specimen. The results of the DL-EPR in solution C at a scan rate of 3 mV/s match with the microstructural observations reported in Fig. 6(a)-(i).

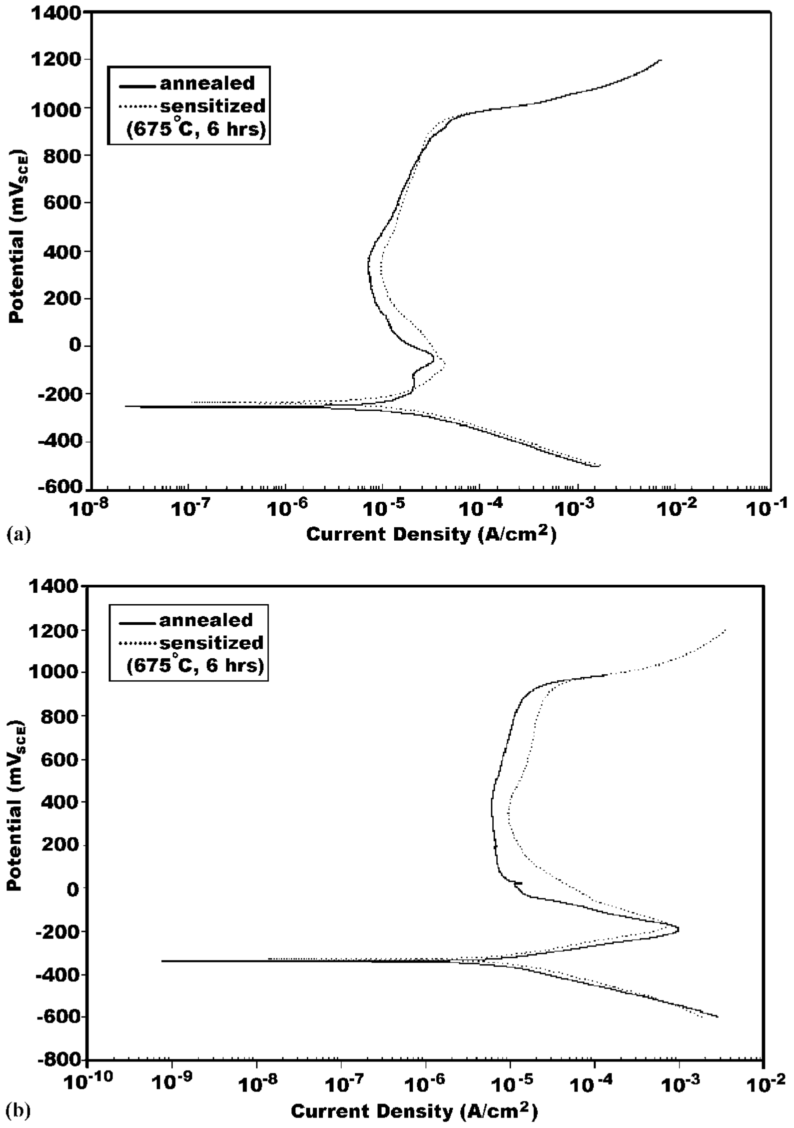

Fig. 7 Anodic polarization curves in 1 M H_2SO_4 at room temperature for **(a)** alloy 800 HT and **(b)** alloy 800 Nb

Figure 2-5 and Table 5 and 6 show that the sensitivity of the test results improve when the modified current ratio (Eq 4) or the modified charge ratios (Eq 5) are used. These ratios also remove the uncertainty in the EPR values in determination of the annealed specimen and do not require measurement of grain size.

4.4 Effects of Alloying Additions on DOS

The alloy 800 HT contained higher alloying additions of titanium and aluminum compared with the conventional alloy 800. This imparts higher strength, resistance to stress rupture, and creep due to gamma prime precipitates. Because these precipitates form during a high temperature annealing (above 1150 °C), the high carbon content of the alloy 800 HT results in sensitization at grain boundaries even in its annealed condition. This is reflected by the DL-EPR values (see Table 8). Upon heating the alloy 800 HT in the sensitization temperature range (675 °C for 6 h), the DOS increases. This is clearly reflected by the increased values of DL-EPR ratio given in Table 8. Further heating at the same temperature brings down

Table 8 DL-EPR Values and Corrosion Rates Measured in the Fifth Period of Practice C, A262, ASTM for Alloy 800 HT and Measured in the Third Period of Practice C, A262, ASTM for Alloy 800 Nb

Alloy Condition	Current Ratio, I_r	Charge Ratio, C_r	Corrosion Rate in Practice C, A262, mpy
Alloy 800 HT			
Annealed	8.38	5.24	3.86
675 °C-6 h	13.10	14.18	928.62
675 °C-24 h	0.634	1.064	70.0
Alloy 800 Nb			
Annealed	0.073	0.035	7.14
675 °C-6 h	1.777	0.856	1721.66
675 °C-24 h	0.256	0.198	317.63

DOS as the diffusion rates of chromium in nickel-based alloys are fast and lead to replenishment of the chromium-depleted regions at grain boundaries. This effect of desensitization is reflected in the DL-EPR values. This is due to the fact that in the EPR test using H_2SO_4 + KSCN solution, the chromium depleted regions are attacked and not the chromium rich carbides at the grain boundaries.[10] This effect is also reflected in the corrosion rate measured in practice C of A 262, ASTM (Table 8). The corrosion rate increases initially when DOS increases. Longer heating at the sensitization temperatures reduces the corrosion rates in practice C. Note that in practice C, A 262, ASTM test the chromium rich carbides as well as the chromium-depleted regions are attacked.

Similar results are obtained on alloy 800 Nb also. This alloy contains Nb and forms niobium carbide (NbC) when the alloy is heated at a temperature where NbC forms and is stable. The alloy 800 Nb used in this study was in a solution-annealed condition, annealed at 1150 °C. The high carbon alloy (carbon 0.1 wt.%) had NbC in the matrix. Therefore the DL-EPR value and the corrosion rate for the alloy in practice C, A262, ASTM were low (Table 8). These increased upon sensitization at 675 °C for 6 h. This heat treatment caused precipitation of chromium rich carbides at grain boundaries and associated chromium depletion regions. Longer heating at the same temperature caused desensitization (replenishment of the chromium depletion at grain boundaries). Therefore these desensitized samples had more carbides at grain boundaries but less chromium depletion. This reflected in the lower DL-EPR values and lower corrosion rates in practice C, A262, ASTM (Table 8).

These results on the alloys 800 HT and 800 Nb showed that the DL-EPR test developed in this study is effective in evaluating the DOS of alloy 800 even when other alloying additions are present. The DL-EPR test essentially attacks the chromium depletion regions and the carbides are not affected. The results from the DL-EPR test can be correlated to the results from practice C, A262, ASTM.

5. Conclusions

The parameters of the SL and DL EPR tests for alloy 800 are established that allow distinction between moderately to highly sensitized conditions. The selected test parameters also allow distinction between sensitized and desensitized conditions. It has been shown that an SL-EPR test in 1 M H_2SO_4 + 0.002 M KSCN (de-aerated) at 26 °C at a scan rate of 3 mV/s from a vertex potential of 700 mV_{SCE} (180 s hold time) gives results that match with the DOS indicated by microstructures and the Huey test results. Similarly, the DL-EPR test in 1 M H_2SO_4 + 0.002 M KSCN (de-aerated) at 26 °C, forward scanning from the OCP to + 700 mV_{SCE} and then backward scanning from there to the OCP at a scan rate of 2 mV/s produces results that allow distinguishing between different sensitized conditions and between sensitized and desensitized conditions. A new parameter, modified current ratio, or modified charge ratio is shown to enhance the sensitivity of the test results for the DL-EPR test and does not require measurement of grain size. It has also been shown that the EPR test remains effective in evaluating the DOS of alloy 800 even when alloying elements are added (Ti and Al in alloy 800 HT and Nb in alloy 800 Nb) to improve its high temperature strength.

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