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Corrosion monitoring of type 304L stainless steel in nuclear near-high level waste by electrochemical noise

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Abstract Electrochemical noise (EN) monitoring of 304L stainless steel (SS) and sensitized 304 SS in 3 N nitric acid and nuclear near-high level waste (HLW) solution was carried out using a three nominally identical electrode configuration under open circuit conditions. EN signals characteristics of passivation process was obtained for 304L SS in 3 N nitric acid throughout the measurement period, while in near-HLW solution, features of passivation and depassivation were observed. Potentiodynamic polarization of 304L SS in 3 N nitric acid and near-HLW showed spontaneous passivation. Noise resistance was evaluated from EN time record and this parameter was used in the present investigation to qualitatively assess the corrosion behavior during the immersion period. The average noise resistance was found to be lower in near-HLW solution when compared to 3 N nitric acid. The results of the investigation are presented in this article.

Keywords 304L · Electrochemical noise · Corrosion monitoring · Near nuclear HLW

1 Introduction

High level liquid waste is the liquid effluent generated from the reprocessing of the spent nuclear fuel [1, 2] and it consists of unrecovered uranium, plutonium transuranics, and other highly radioactive fission products generated during fission. The yield of these fission fragments depends on the type of fissile atom loaded in the reactor and the

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neutron energy for reactor applications. Most of the radioactive isotopes in HLW emit large amounts of radiation and decay heat and have extremely long half lives creating long time periods before the waste will settle to safe levels of radioactivity. The activity of HLW ranges from 3.7×10^9 Bq m^{-3} and above [1, 2]. In spite of having lower volume than low and intermediate level waste, HLW disposal is of great concern for the public. Thus, waste management and immobilization of HLW is required to safeguard the present and future generations. A three-stage strategy for management of HLW has been formulated in India. This involves immobilization of waste oxides in stable and inert solid matrices, also called as vitrification, followed by interim retrievable storage of the conditioned waste under continuous cooling and final disposal in geological repositories [1, 2]. The vitrification facilities are co-located near the reprocessing plants so as to avoid long-term transportation of HLW. HLW is stored in underground waste tank farm, housing high integrity stainless steel type 304L tanks of large capacity (250–500 m^3 each) provided with temperature, pressure, and level monitoring. HLW is transferred from here to vitrification plant through underground piping. Liquid storage is an interim step and requires cooling and continuous surveillance [1–3]. Though liquid storage has been proved to be largely safe and acceptable for few decades, this cannot be considered as the ultimate system of storage and disposal of HLW. Conversion of HLW into a suitable solid matrix for vitrification has been studied extensively in many countries [1–3]. Corrosion and tank integrity are major issues concerning worker and environmental safety. Since corrosion, whether localized or uniform, could initiate in these tanks, monitoring of corrosion is necessary. Several departments of energy sites in USA such as Hanford, Savannah River, Oak Ridge Reservation, and the

Idaho National Engineering and Environmental Laboratory have conducted laboratory studies and field applications to correlate the electrochemical signals, with corrosion mechanisms active in the radioactive waste tanks [4]. Coupon exposure, linear polarization resistance, electrical resistance techniques have been tested to determine the corrosivity of nuclear waste underground storage tanks. They were found to be effective in predicting uniform corrosion, but not suitable to predict pitting and stress corrosion cracking which were the primary modes of corrosion of carbon steel and stainless steel used in radioactive waste tanks. For the first time, initial proof-of-principle laboratory work at Hanford produced the technical basis for using electrochemical noise (EN)-based systems to monitor corrosion in alkaline waste in waste tanks made of carbon steel. A prototype system was deployed in one of the waste tanks in 1966 following which three full scale systems were deployed in the successive years in three other waste tanks of Hanford. At Savannah River site (SRS), the EN technique was combined with Laser Raman spectroscopy for real-time information of waste inhibitor concentrations and corrosion in actual radioactive waste tank environment [4]. Laboratory work performed in SRS waste solution, showed EN more feasible and flexible than other techniques for use in HLW tanks. The SRS probe was designed as a movable probe head on a flexible cable deployed from a real mechanism housed in a confinement chamber on top of the tank. Oak Ridge Reactor (ORR) used EN to monitor waste tanks, made of welded 304L stainless steel plates. At Idaho National Engineering and Environmental Laboratory (INEEL), the results from the preliminary test program showed that 304L SS alloys passivated and showed low currents and no signal features in nitric acid exposure. Laboratory scale studies related to EN measurements of 304L SS as storage material in nuclear HLW medium is of significant importance to understand the system prior to application in nuclear waste storage tanks. In nuclear waste tank infrastructure, problems of sensitization can occur in the heat-affected zones near welds as a result of thermal cycling during welding of thick components. This could lead to corrosion problems during long-term exposure to nitric acid and HLW. The present investigation presents the results of EN monitoring of 304L SS and sensitized 304 SS in near-nuclear HLW composition.

2 Experimental

2.1 Heat treatment

The composition of the AISI type 304L SS and 304 SS used for the EN monitoring is given in Table 1 Type 304L SS and 304 SS were solution annealed at 1050 °C for 30 min and water quenched. Type 304 SS was subsequently subjected to sensitization treatment by heating at 675 °C for 1 h followed by air-cooling [5]. The heat-treated specimens were used for the present investigation.

2.2 Preparation of near-high level waste solution

Oxides and nitrates of the corresponding elements mentioned in Table 2 were taken in the required stoichiometric ratio and were dissolved in 3 N HNO_3. The dissolution process in nitric acid was carried out in a laboratory fume hood for safe handling of natural uranium oxide and nitric acid fumes. The EN cell and the glass wares used for the specific study were isolated. The composition of the near-HLW for EN measurement is given in Table 2. This composition represents one of the stored nuclear waste solutions of natural uranium oxide (UO_2) fuel used in pressurized heavy water reactors in Tarapur, India.

2.3 Potentiodynamic polarization experiment

Solution annealed 304L SS and sensitized 304 SS specimens of dimensions 10 mm × 10 mm × 10 mm were prepared. The specimens were polished to remove the oxide scale, mounted in an araldite resin, and polished till diamond finish. While mounting, proper care was taken to avoid gaps between specimen and resin to prevent crevice attack during polarization experiments. These specimens were immersed in the electrolyte for 30 min at open circuit potential and subsequently subjected to potentiodynamic anodic polarization at room temperature from a potential of 0.25 V below OCP. The potential was measured with respect to saturated calomel electrode (SCE) and a potential scan rate of 0.01 V min^{-1} was maintained. Polarization experiments were stopped around 0.5×10^{-3} A, after the current monotonically increased beyond 0.25×10^{-4} A in the passive region. The experimental details are presented in our earlier work [6].

Table 1 Composition of the stainless steels used for the investigation

Code	C	Si	Mn	Cr	Ni	Mo	S	P	Fe
Concentration in wt %									
304L SS	0.029	0.41	1.66	17.45	8.97	0.46	0.028	0.031	Balance
304 SS	0.08	0.85	0.85	17.7	8.8	<0.170	0.1	0.032	Balance

Table 2 Composition of the HLW solution used for the investigation

Serial no.	Elements	Concentration of elements (g/L)	Corresponding salts taken
1	Fe	0.72	$Fe(NO_3)_3$
2	Cr	0.119	CrO_3
3	Ni	0.107	$Ni(NO_3)_3$
4	Na	5.5	$NaNO_2$
5	K	0.224	KNO_3
6	U	6.34	UO_2 (natural uranium)
7	Sr	0.031	$Sr(NO_3)_3$
8	Zr	0.004	$ZrO(NO_3)_2$
9	Ba	0.064	$Ba(NO_3)_2$
10	La	0.18	La_2O_3
11	Ce	0.06	$Ce(NO_3)_2$
12	Pr	0.09	Pr_6O_{11}
13	Nd	0.12	Nd_2O_3
14	Sm	0.0855	Sm_2O_3
15	Y	0.06	Y_2O_3
16	Cs	0.315	$CsNO_3$
Acidity of nitric acid = 3 N			

2.4 Electrochemical noise experiment

The solution annealed 304L SS and sensitized 304 SS specimens were lathe finished into a cylindrical shape with diameter 8 mm and length 15 mm. The specimens were mounted into a Teflon mount and polished till 2400 grit emery exposing a surface area of 0.5 cm^2. The prepared specimens were used for EN studies.

A schematic of the experimental setup for the EN measurements used for the study is shown in Fig. 1. The detail of the experimental setup is reported in our earlier work [7]. The EN cell comprised a three identical electrode configuration where the electrodes were made from the same material and same surface area of exposure. Two such identical electrodes were made as the working electrodes and the third electrode was made as the pseudoreference electrode. The following combination of electrode environment formed three EN cells from which measurements were carried out.

1. AISI type 304L SS immersed in 3 N HNO_3.
2. AISI type 304L SS in near-HLW.
3. Sensitized 304 SS in near-HLW.

Electrochemical current noise was measured between the two working electrodes, simultaneously, the potential noise was measured between one of the working electrodes and the pseudoreference electrode. The current noise and potential noise were monitored with respect to time under open circuit condition using a multichannel EN system, Autolab PGSTAT 30 custom built with a low noise module and a data management system (Ecochemie make, The Netherlands). Data was acquired sequentially from the three EN cells which were connected to three different channels of the multiplexer unit of the Autolab PGSTAT 30. A sampling interval of 0.5 s was chosen over 8192 consecutive data points. The measurement details are given in Table 3. The frequency domain corresponding to the sampling conditions was evaluated to be between 1 Hz (f_{max}) and 0.2 mHz (f_{min}), from $f_{max} = 1/2 \Delta t$ where Δt is the sampling interval and $f_{min} = 1/N\Delta t$ where N is the total number of data points.

2.4.1 Data analysis

Data analysis was carried out by visual examination of the electrochemical current and potential noise time records and also by statistical evaluation of the time records. The DC trend in the collected EN data was eliminated by linear

Fig. 1 Schematic of the experimental setup for electrochemical noise measurements
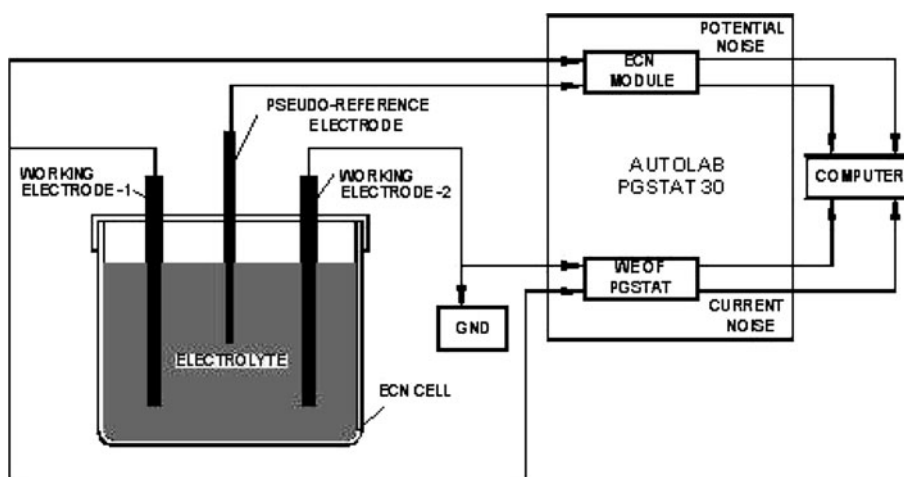
Table 3 Electrochemical noise measurement details

Electrolyte and stainless steel used for measuring electrochemical noise	Duration of EN measurement after immersion of the probe (h)	
	Start of measurement	End of measurement
1. 304L SS + 3 N HNO$_3$	36 h after immersion	170 h after immersion
2. 304L SS in near-HLW	35 h after immersion	169 h after immersion
3. Sensitized 304 SS in near-HLW	34 h after immersion	167 h after immersion

fit method [6] to represent the data in absolute noise unit and this data was used for further analysis. The statistical parameter called noise resistance (R_n) was evaluated from the time record as the ratio of the standard deviation of potential noise (σ_v) to the standard deviation of current noise (σ_i) [7–10]. The statistical calculation and the trend removal were performed using commercial software. Since a three identical electrode configuration was used, the potential noise from each electrode would be the same. The measured potential noise will be the combined noise from both electrodes, but the potential noise from one electrode is the quantity used to calculate R_n. Therefore, σ_v measured is divided by $\sqrt{2}$ to obtain σ_v [7–10]

$$\sigma_{v,m} = \left(\sigma_{v1}^2 + \sigma_{v2}^2\right)^{0.5}$$
$$\sigma_{v1} = \sigma_{v2} = \sigma_v$$
$$\sigma_v = \sigma_{v,m}\big/\sqrt{2}$$

$\sigma_{v,m}$ is the standard deviation of measured potential noise, σ_{v1} is the standard deviation of potential noise from first electrode, σ_{v2} is the standard deviation of potential noise from second electrode, and σ_v is the standard deviation of potential noise used to evaluate R_n.

2.5 Optical microscopy

The microstructure of 304L SS and 304 SS used for the present investigation was examined by etching in oxalic acid as per ASTM A 262 practice A [5]. Etching was carried out by immersing the specimen in 10% oxalic acid at 6 V for 90 s. The specimens were subsequently observed under optical microscope.

3 Results and discussions

### 3.1 Microstructural analysis

Microstructure of the solution annealed 304L SS, and 304 SS subjected to sensitization heat treatment is shown in Fig. 2a, b. 304L SS shows a step microstructure and 304 SS shows carbide precipitation along the grain boundaries which can be conferred to a "ditch" structure as per ASTM A 262 practice A.

3.2 Potentiodynamic polarization

Figure 3 shows the potentiodynamic anodic polarization curves for 304L SS in 3 N HNO$_3$ and near-HLW solution at ambient temperature. From the polarization curves, it is observed that 304L SS has spontaneously passivated in 3 N HNO$_3$ and near-HLW, and in both solutions, the polarization curves were characterized by a well-defined passive region prior to transpassive breakdown which occurred almost at a similar transpassive potential (T_p). The passive
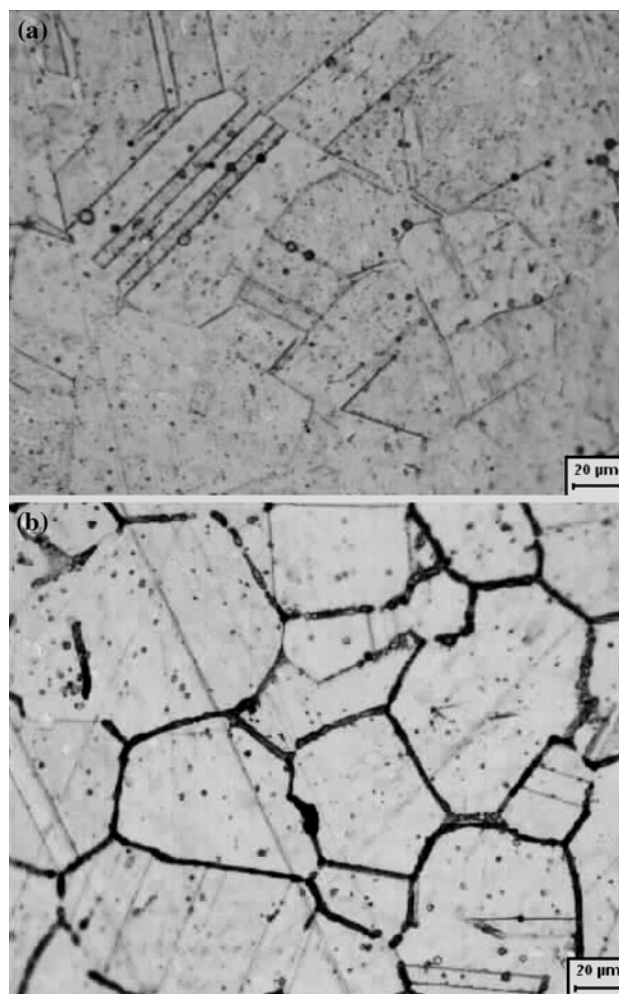
Fig. 2 Micrograph of the specimens used for the electrochemical noise investigation. **a** 304L SS showing step microstructure and **b** 304 SS showing ditch microstructure
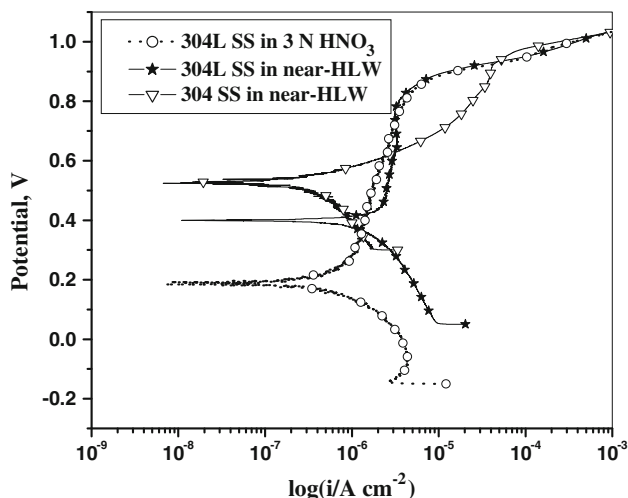
Fig. 3 Potentiodynamic polarization curves for 304L SS in 3 N HNO₃ and near-HLW showing spontaneous passivation

range for 304L SS in 3 N HNO$_3$ was found to be 0.57 V, ranging from a potential of 0.26 V to 0.84 V (T_p). In near-HLW, the passive range was found to be lower than in 3 N HNO$_3$ (0.408 V) ranging from a potential of 0.43 V to 0.84 V (T_p). The lower passive range in near-HLW is a result of a higher corrosion potential which could be attributed to the reduction of the cations present in near-HLW. The passivation current density in 3 N HNO$_3$ and near-HLW was found to be 1.8×10^{-6} A cm^{-2} and 3.2×10^{-6} A cm^{-2}, respectively. Although in both solutions, a well-defined passivation region was observed, the slightly higher passivation current density in near-HLW could be attributed to lower stability of the passive film in near-HLW solution. In sensitized 304 SS, an increased passivation current density ($\sim 20 \times 10^{-6}$ A) was observed which could be attributed to unstable passive film.

3.3 Electrochemical noise

3.3.1 Visual examination of time record

EN time records have been used by researchers to indicate the corrosion processes occurring on a wide range of materials and environments to get mechanistic information about the nature of attack on the metal surface. A simple visual examination of the time record gives distinct noise characteristics for various processes like passivation, uniform and localized corrosion [8, 10–13]. Typical representation of the EN time records taken for AISI type 304L SS in 3 N HNO$_3$, 304L SS, and sensitized 304 SS in near-HLW solution are shown in Figs. 4, 5, and 6, respectively. For 304L SS in 3 N HNO$_3$, the current fluctuations were of very low amplitude, in the range of about 0.015 μA, appearing at high repetition rates and almost white type of

noise throughout the measurement period was observed. The potential noise was very low, about 0.1 mV. These features are typical of passivation phenomenon. In our earlier studies [7, 10] on EN of 304L SS in 0.1% NaOH and 304 SS in 4 N HNO$_3$, similar current and potential time records were obtained which were conferred to passivation process. Legat et al. [14] have also reported similar EN features for passivation phenomenon. The noise monitoring for the entire period of measurement revealed a very stable passive film on the specimen. Typical representation of the time records is shown in Fig. 4a, b. The EN time record for 304L SS in near-HLW solution showed low-level current and potential fluctuations with an amplitude of 0.01 μA in the current noise. A white noise feature was observed similar to that observed in 3 N HNO$_3$, which depicts passivation process. At certain immersion times, the current noise time record for 304L SS in near-HLW solution depicted low amplitude fluctuations followed by a sudden burst of current noise with the amplitude being almost ten times that during the passivation process. This could be attributed to two competent phenomenons. 3 N nitric acid is a spontaneously passivating medium and therefore passivation of 304L SS has resulted in white noise feature in the current noise time record. As near-HLW has innumerable cations and anions, these could get adsorbed on the surface of the passive film thus destabilizing the film leading to sudden burst of current noise. However, after the experiment, the samples were observed visually and no features of uniform or localized attack were noticed. For sensitized 304 SS in near-HLW, the features of passivation was observed as above, with the amplitude in the current noise being about 0.02 μA and potential noise being around 0.4 mV. The amplitude of current noise for the sensitized 304 SS in near-HLW was found to be similar to that for 304L SS in 3 N HNO$_3$ and 304L SS in near-HLW during passivation process. The potential noise is marginally higher when compared to the other systems. A typical representation is shown in Fig. 6a, b.

The features of passivation process as observed from the EN time records is in concurrence with the results obtained from potentiodynamic polarization experiments which showed a well-defined passivation region for 304L SS in the solutions studied and also depicting lower stability of the passive film in near-HLW medium when compared to 3 N HNO$_3$.

3.3.2 Statistical analysis

3.3.2.1 Trend removal
The potential and current noise raw data contains noise component superimposed on a direct current (DC) trend. The DC drift or trend is defined as the variation of the mean current or potential divided by time and is not useful for corrosion phenomenon. Since the

Fig. 4 Electrochemical noise time records for 304L SS in 3 N HNO$_3$ taken after **a** 85 h, **b** 125 h of immersion
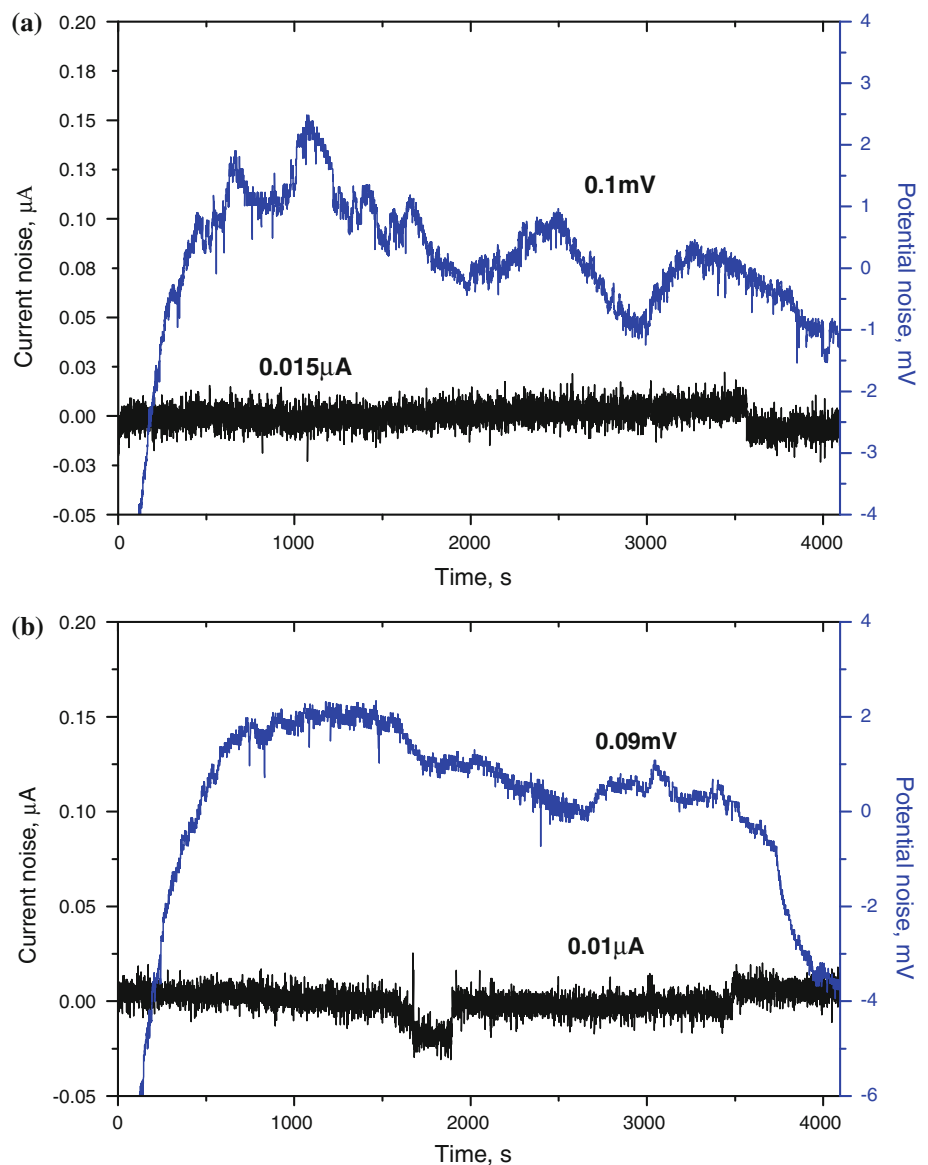
noise signals are measured between two nominally identical electrodes, the mean value of the difference of potential and current flowing between them is expected to be zero. The EN signals should appear as quasi-random fluctuations around zero. Among the various methods available in literature [15, 16], the most effective method to eliminate the trend is the linear method that is based on the assumption that the drift follows a linear relationship. To remove the trend, the experimental raw data was fit by linear method and the linear regression functions generated for the potential and current time records were subtracted from the raw data. Trend removal was carried out for every time record of the potential and current noise. It was found that the data after trend removal had the mean of potential and current noise around zero. The mean of data after trend removal for each sample is given in Table 4.

3.3.2.2 Electrochemical noise resistance Several investigators [17–23] have found that the noise resistance (R_n) from EN analysis is equivalent to polarization resistance (R_p) from linear polarization resistance method and hence R_n can replace R_p in Stearn Geary equation to determine the corrosion rates. It has also been emphasized to remove the DC components from the potential and current noise time record for the calculation of noise resistance. Riechert [24] has found that the corrosion rates calculated from R_n are effective for systems exhibiting general corrosion rates below 1.3 mm year^{-1} and a good agreement between R_n and R_p was found for carbon steels and stainless steels in different environments. Noise resistance has been used to indicate the onset of localized corrosion or the formation of faults on coated material and an inverse relationship between noise resistance and corrosion rate has been

Fig. 5 Electrochemical noise time records for 304L SS in near-HLW taken after **a** 124 h, **b** 144 h of immersion
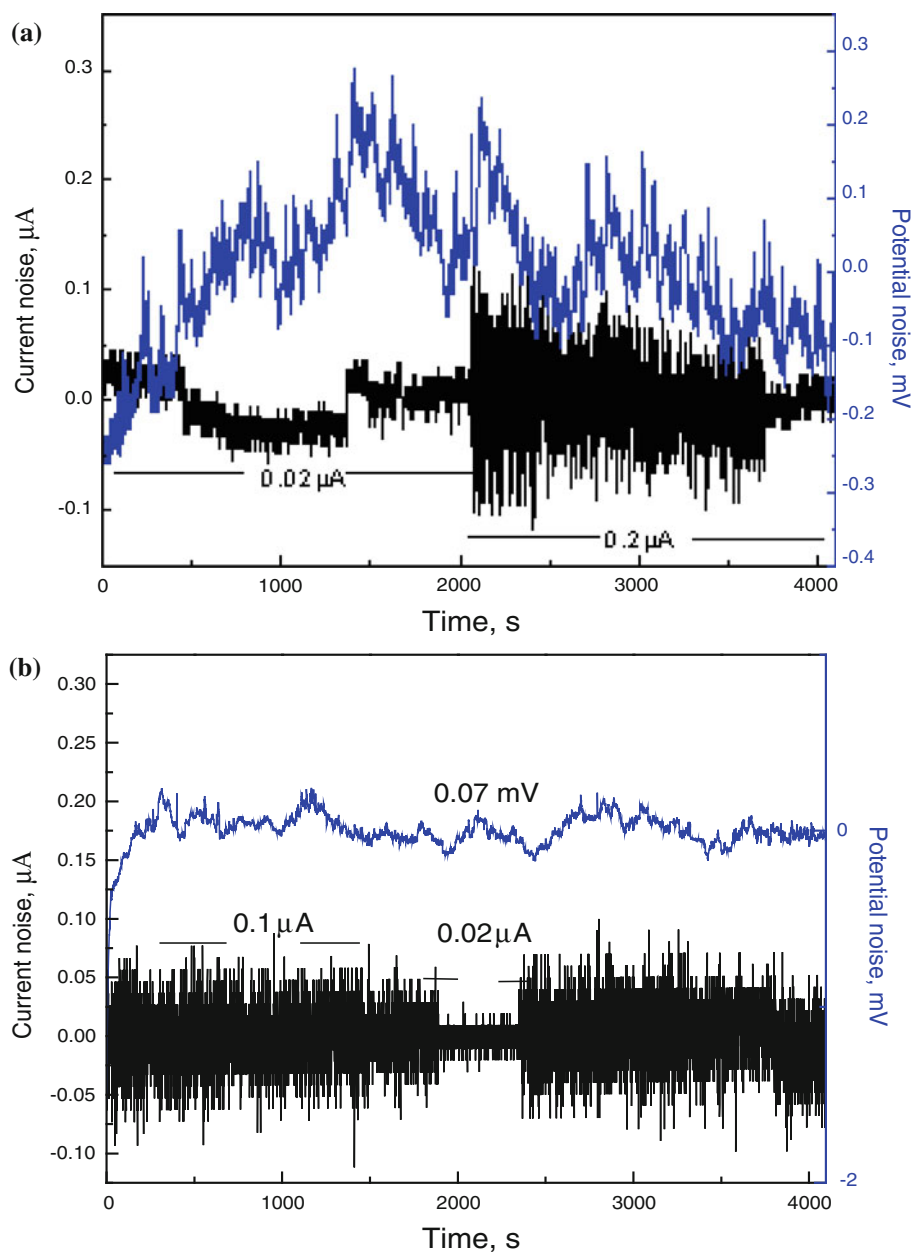
observed [25]. In our earlier study [7], an inverse relationship between noise resistance and corrosion rate was established. This parameter was used to evaluate the corrosion resistance of AISI type 304 SS in different concentrations and temperatures of nitric acid and it was found that, with increase in concentration and temperature of nitric acid, the noise resistance decreased implying higher corrosion rates at higher concentrations and temperature. In this study, this inverse relationship between EN resistance and corrosion rate has been used to qualitatively assess the corrosion behavior 304L SS in 3 N HNO₃ and near-HLW and 304 SS in HLW during monitoring. Noise resistance was evaluated from the detrended time records as the ratio of standard deviation of potential noise to standard

deviation of current noise. The calculations were done at certain periods of immersion and the plot of noise resistance as a function of time is presented in Fig. 7. It was observed that for 304L SS in 3 N HNO₃, the noise resistance was high and in the range of 1.5×10^5 to $5 \times 10^5 \ \Omega \ cm^{-2}$ to obtain an average of $2.967 \times 10^5 \ \Omega \ cm^{-2}$ over a time interval of 100 h of data acquisition. The high and constant noise resistance is attributed to good passive behavior, which existed throughout the measurement period. Since noise resistance is inversely proportional to the corrosion rate it could be noted that the corrosion resistance of 304L SS in 3 N HNO₃ remained high throughout the measurement time. For 304L SS in near-HLW, the noise resistance was found

Fig. 6 Electrochemical noise time records for 304 SS in near-HLW taken after **a** 102 h, **b** 122 h of immersion
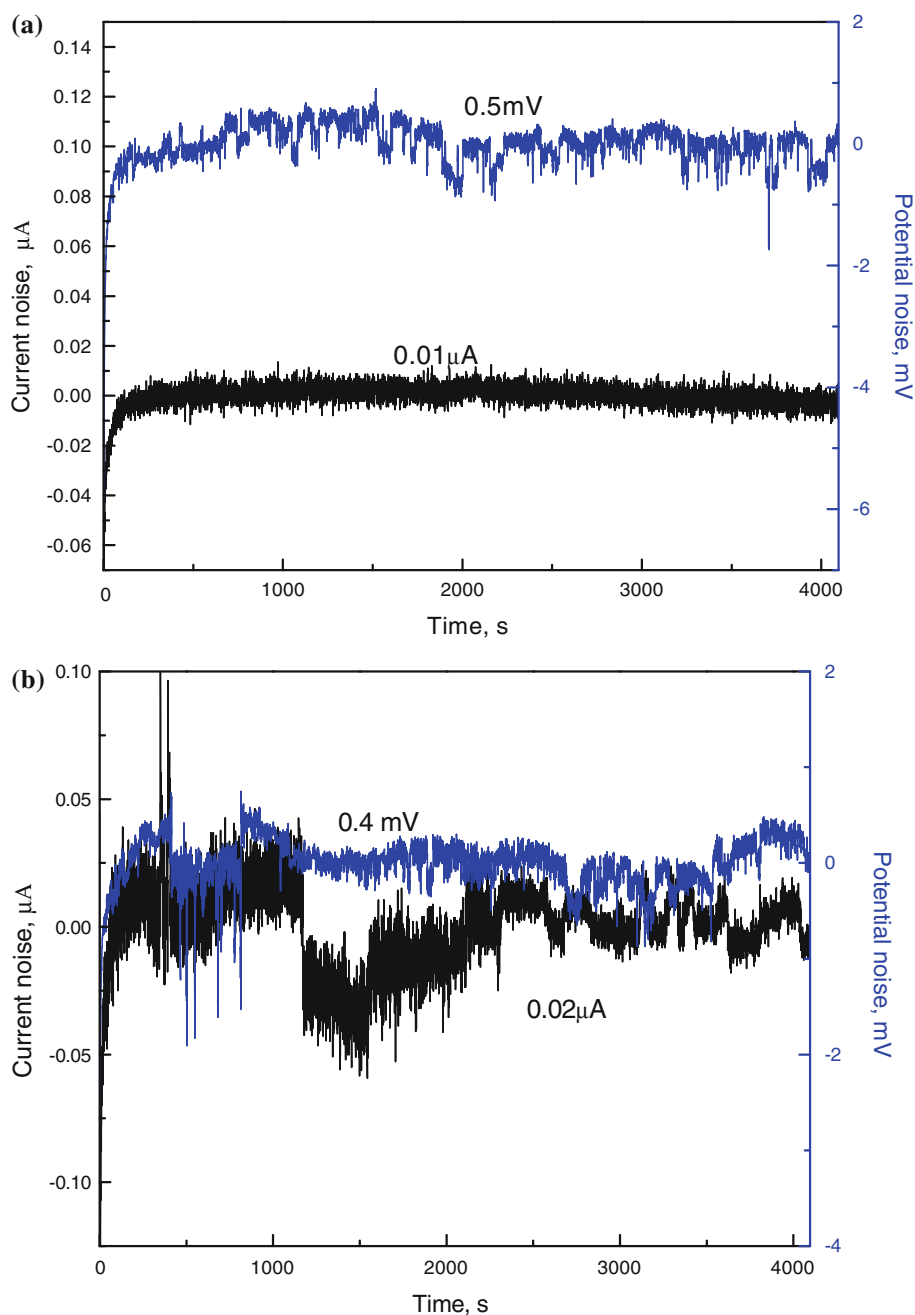
Table 4 Mean of the electrochemical noise data after trend removal

Stainless steel and electrolyte	Time after immersion (h)	Mean potential (V)	Mean current (A)
1. 304L SS in 3 N HNO$_3$	36	-1.2329×10^{-18}	-1.8144×10^{-21}
	98	8.1286×10^{-21}	-2.273×10^{-21}
2. 304L SS in near-HLW	35	-1.634×10^{-19}	-5.13×10^{-21}
	97	-1.11×10^{-21}	3.78×10^{-18}
3. 304 SS in near-HLW	34	-1.685×10^{-19}	6.0255×10^{-22}
	167	-1.257×10^{-21}	5.547×10^{-18}
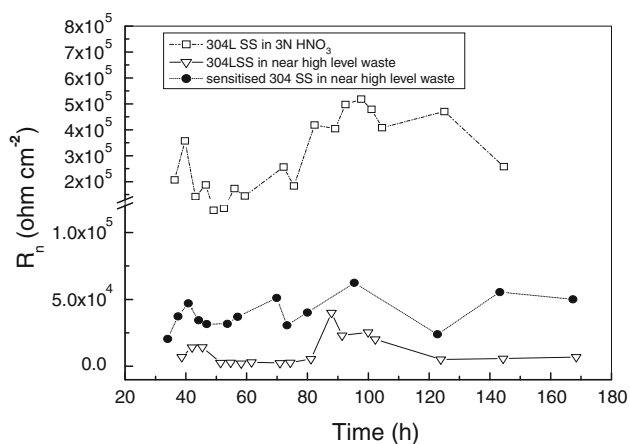
Fig. 7 Plot of noise resistance versus time

to be fluctuating in the range of 2.5×10^3 to $6.9 \times 10^3 \ \Omega \ cm^{-2}$ to $2.5 \times 10^4 \ \Omega \ cm^{-2}$ with an average of $10,800 \ \Omega \ cm^{-2}$. For 304 SS in near-HLW, the noise resistance was found to be in the range of 2×10^4 to $9 \times 10^4 \ \Omega \ cm^{-2}$ with an average of $60950 \ \Omega \ cm^{-2}$. A drop in noise resistance was observed for 304L and 304 SS in near-HLW when compared to 304L in 3 N HNO_3, which implies that corrosion resistance of the material in near-HLW is lowered. This could be attributed to the instability of the passive film of the material caused by the cations present in near-HLW. It was also observed that sensitization of the specimen did not have any profound effect on the corrosion rate during the period of measurement.

4 Conclusion

EN signals acquired from 304L SS in 3 N nitric acid showed features of passivation process. The current–time record showed highly repetitive and low amplitude fluctuations and the potential fluctuations were also very feeble. In near-HLW solution, EN signals depicted two competing phenomenon, namely, passivation process by 3 N HNO_3 and destabilization of the passive film by the ions present in the near-HLW solution. Also, 304 SS showed passivation during the course of measurement. Potentiodynamic polarization studies showed a well-defined passivation region for 304L SS in the solutions studied and lower passive film stability in near-HLW when compared to 3 N nitric acid. The noise resistance evaluated from the time record showed higher values for 304L SS in 3 N HNO_3 compared to 304L SS and sensitized 304 SS in near-HLW, implying higher corrosion resistance for the former. From the EN resistance measurements for 304L SS and 304 SS which were found to be in close proximity in

near-HLW, it was found that under open circuit potentials, both the materials exhibited similar corrosion resistance during the period of monitoring (~ 170 h).

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