Corrosion studies of stainless steel 304 L in nitric acid in the presence of uranyl nitrate: effect of temperature and nitric acid concentration

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Abstract

In order to better understand the role of uranium on the corrosion behavior of stainless in aqueous nuclear fuel reprocessing and decontamination operations, 304L stainless steel coupons were treated with nitric acid media at 45 °C and 90 °C in the presence of 0.3 M of uranyl nitrate. The impact of time, temperature, nitric acid concentration and uranyl nitrate concentration has been studied over a 183 days period and corrosion rates determined. After 183 days, corrosion rates ($\mu\text{m y}^{-1}$) at 90 °C are: 33.7 ± 3.4 for 8 M HNO_3 with $[\text{U}] = 0$ M; 39.7 ± 0.1 for 8 M HNO_3 with $[\text{U}] = 0.3$ M; 5.3 ± 0.1 for 3.5 M HNO_3 with $[\text{U}] = 0$ M and 5.9 ± 0.2 for 3.5 M HNO_3 with $[\text{U}] = 0.3$ M. After 183 days, the corrosion rates at 45 °C are all below $2 \mu\text{m y}^{-1}$ for the nitric acid concentration and uranyl nitrate concentration studied. Analysis of the data indicate that the corrosion domain remains passive, no uranium uptake onto stainless steel has been observed but only an increase of corrosion rate with temperature (major effect) and nitric acid concentration (minor effect) were reported. When both temperature and nitric acid concentration increased and drive the system into a transpassive corrosion domain, the presence of uranium promote the increase of corrosion rate after 3 months. Analyses of the coupon by scanning electron microscopy indicates that at the first stage low homogeneous dissolution of the steel occurs; intergranular corrosion appears at 10 days and finally grain loss is observed after 6 months treatment.

Keywords Stainless steel · Uranium · Corrosion · Nitric acid

1 Introduction

Nuclear fuel reprocessing facilities use a wide range of metallic materials for spent fuel treatment. Under reprocessing conditions, those metallic materials are in contact with highly corrosive nitric acid and the understanding of corrosion of those materials is crucial for the reprocessing facilities stakeholders and represents challenging issues for scientists [1].

Especially, the Plutonium Uranium Redox Extraction (PUREX) process is widely used in the nuclear industry for uranium and plutonium treatment-recycling of the nuclear spent fuel. The conditions used in the PUREX process, such as temperature (up to 121.9 °C), nitric acid concentration

(up to HNO_3 azeotropic level, 15.2 M) varies throughout the different industry engineering designers [1, 2]. Generally, two different nitric acid concentrations have been reported in PUREX: 3.5 M, that is one of the most cited concentration for the U/Pu TBP-solvent extraction step [3] and 8 M that is a typical concentration for spent fuel dissolution [4]; the concentration of 8 M is also the limit of use of the austenitic stainless steel 304L (304 L SS) [1].

Stainless steel 304L is commonly used in vessels in the PUREX system [1, 5] and its corrosion behavior in nitric acid has been documented [1, 6–8]. Its anodic behavior will remain in the passive corrosion domain and a few nanometers thick passive layer principally composed of Cr_2O_3 will form on its surface in moderately oxidizing solutions. Over

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its limits of use, intergranular corrosion can occur in the transpassive domain, especially in presence of oxidizing species in the solution [1, 9, 10].

In PUREX process, the nitric acid solution can contain concentrations of uranium up to 2 M [11] and only few studies on the effect of actinides on corrosion of stainless steel in PUREX conditions have been reported. Previous electrochemical studies [6] investigated the concentration of uranium, nitric acid and temperature effect on austenitic steel and showed that those parameters have a keyrole in the corrosion via a modification of redox conditions but a better understanding of long-term exposition effect is needed (e.g. months to year time scale). The presence of others actinides in nitric acid such as plutonium [12, 13], neptunium [12, 14] or americium [12] were found to shift electrochemical potentials of studied systems and accordingly to modify the corrosion rate of stainless steel.

A recent study [15] has shown that the corrosion of 304 L SS in uranium-containing nitric acid solutions increases with exposure time and has highlighted uranium contamination within intergranular regions of the material, however parameters in this study remains limited (only one temperature has been studied and blank coupons have not been studied). A review [16] on the corrosion behavior of several stainless steels in nitric acid solution indicates that all the corrosion rates reported are below $100 \mu\text{m y}^{-1}$. Moreover, no existing data on combining effect of long-term experiment, nitric acid concentration, and high uranium concentration below 100°C on the corrosion behavior of 304 L SS have been reported.

In this context, concerning the corrosion rate of 304 L SS in HNO_3 in the presence of uranium, new data which are representative to industrial reprocessing conditions are needed. Here, over a 6 months period, we investigate the corrosion behavior of 304 L SS coupons in HNO_3 (3.5 M and 8 M) in the presence of uranyl nitrate (0.3 M) as a function of the temperature (45°C and 90°C) in view to report corrosion rates and to investigate the corrosion by scanning electron microscopy.

2 Materials and methods

2.1 Materials

Uranyl nitrate hexahydrate (UNH), from laboratory stock was purified after treatment [17] of 100 g batches in 10 M HNO_3 at 70°C and cooled to -10°C in a freezer for 12 h.

HNO_3 (ACS reagent, 68–70%) was purchased from Sigma Aldrich. Deionized water ($18.2 \text{ M}\Omega\cdot\text{cm}$) was used for the dilution. HF used for removal of corrosion products procedure was reagent grade (48–51%).

A stock solution of uranyl nitrate (0.85 M) was obtained after dissolution of solid UNH in 2 M HNO_3 . The concentration of this stock solution was ascertained by UV–Visible spectroscopy. The stock solution was used to prepare the solutions of uranyl nitrate (0.3 M, 310 mL) in 8 M and 3.5 M HNO_3 investigated in this study. Blank solution of 3.5 and 8 M HNO_3 was used to investigate the effect of uranium of the corrosion behavior of the 304 L stainless steel coupons. Perfluoroalkoxy alkanes (PFA) cell were ($V=473 \text{ mL}$) were purchased from Saint-Gobain Chemware. Grade 304L stainless steel coupons ($20 \times 10 \times 1.9 \text{ mm}$) were received from Rolled Alloys (wt% elements composition in Table 1).

2.2 Methods

Procedure ASTM G31-72 [18] was used to design the experimental setup. All coupons were polished on their six faces using SiC paper (from P400 to P4000 followed by $3 \mu\text{m}$ and $1 \mu\text{m}$ with diamond spray) with ethanol as lubricant. Before weighting, all the coupons were cleaned in acetone and gently wiped with a Kimtech paper then stored in a desiccator under vacuum.

Coupons were then placed into a homemade PFA sample holder to keep a vertical position and avoid confinement during the treatment. In each PFA cell (473 mL), three coupons were immersed into solution just before treatment. After the corrosion experiment, two coupons in each cell were used for corrosion rate measurement and one for SEM–EDX analysis.

Two ovens set at 45°C and 90°C ($\pm 1^\circ\text{C}$) were used for this study and all the PFA cells were put simultaneously in the oven after each term. All the cells were open on the same day after at least 2 h cool down at 4, 10, 31, 92 and 183 days. Coupons were immediately soaked and rinsed into an acetone bath and gently wiped with a Kimtech paper before mass measurement after treatment.

Following ASTM G1-03 procedure [19] for corrosion rate measurement via mass loss, all coupons were weighted before and after treatment with a balance of $1 \pm 0.1 \text{ mg}$ precision range. After treatment, coupons were treated with 20 mL of a solution of 1% HF and 7% HNO_3 with cycle of 2, 3, 10, 45, 300 s to estimate the mass loss of metal involved in the corrosion process and to calculate the corrosion rate (Eq. 1). Error scale calculation involved the

Table 1 Elemental composition of 304L stainless steel

Element	C	Mn	P	S	Si	Cr	Ni	Mo	Cu	N	Fe
wt%	0.021	1.70	0.033	0.0003	0.39	18.23	8.10	0.45	0.44	0.08	Balance

average of the 2 coupons mass loss for each treatment solution and the error of the balance.

$$R = \frac{8.76 \times 10^7 \times m}{A \times T \times D} \quad (1)$$

Equation (1): corrosion rate (R) calculation in $\mu\text{m y}^{-1}$; m is the mass loss in grams; T is the time exposure (hours); A is the area of the coupon (cm^2) and D is the density of the 304L steel (7.94 g cm^{-3}),

SEM–EDX analyses were performed using a JEOL jsm5600 with an Oxford EDX system with the following parameter: tension acceleration of 15 kV, spot size of 35 nm, working distance of 20 mm and BSED detector. EDX analyses were made on a representative area of each coupon of $420 \times 340 \mu\text{m}$ and using INCA program.

3 Results and discussion

The corrosion rates of 304L SS coupon in 3 and 8 M HNO_3 at 45 °C and 90 °C as a function of the uranyl nitrate (UN) concentration are presented in Table 2. Analysis of Table 2 indicates that after 4 and 10 days of immersion, the mass of some of coupons did not change and the value of corrosion rates calculated was equal to the standard deviation.

The new data of average corrosion rate obtained here on 304L SS are consistent with previous literature data in similar condition [15, 16]. In order to better understand the corrosion mechanism, all coupons were analyzed with SEM–EDX.

At 45 °C for both nitric acid concentrations and the corrosion rates values remains relatively low (few $\mu\text{m y}^{-1}$) and the coupons are in a stable passive corrosion domain. Figure 1 shows the surface of the coupon at this temperature after 6 months treatment and shows that no intergranular attack occurs.

At 90 °C at 3.5 M HNO_3 media, corrosion rates are slightly higher and appear constant through time. Figure 2 shows the evolution of the morphology of surface of the coupons between 92 and 183 days for this parameters without UN

Fig. 1 SEM picture of the morphology of surface of 304L SS coupon exposed to 8 M HNO_3 at 45 °C with 0.3 M UN after 183 days (magnification $\times 1000$, image represent an area of $84 \times 104 \mu\text{m}$)

(morphology with UN are equivalent). The early stage of a shift from passive domain into a transitional transpassive domain is observed here via the appearance of weak intergranular corrosion phenomena. No influence of uranyl nitrate on corrosion rate is observed for these experimental conditions (at 45 °C for all nitric acid concentration and for 90 °C at 3.5 M HNO_3).

At 90 °C in 8 M HNO_3 , corrosion rate are relatively stable between 4 days and 3 months then increases between 3 and 6 months, going from a passive domain to a transpassive domain [10] and this increase is promoted by the presence of 0.3 M uranyl nitrate. The transition into a transpassive domain is observed with the SEM microscopy of the coupon (see Fig. 3), going from a low homogeneous dissolution to a relatively severe intergranular corrosion with grain loss at the end of the experiment. The SEM analysis indicates that only the coupons treated at 90 °C in 8 M HNO_3 exhibits major morphology change. Figure 3 shows the evolution of morphology of surfaces of 304L SS coupons from 4 to 183 days in 8 M HNO_3 at 90 °C with and

Table 2 Average corrosion rate ($\mu\text{m y}^{-1}$) from 4 to 183 days of 304L SS coupon at 45 °C and 90 °C; [HNO_3] of 3.5 M and 8 M and [UN] of 0 M and 0.3 M

Time (days)	T = 45 °C				T = 90 °C			
	[HNO_3] = 3.5 M		[HNO_3] = 8 M		[HNO_3] = 3.5 M		[HNO_3] = 8 M	
	[UN] = 0 M	[UN] = 0.3 M	[UN] = 0 M	[UN] = 0.3 M	[UN] = 0 M	[UN] = 0.3 M	[UN] = 0 M	[UN] = 0.3 M
4	2.3 ± 2.3	4.3 ± 2.2	2.4 ± 2.4	2.3 ± 2.3	4.5 ± 4.5	6.6 ± 2.7	17.4 ± 3.9	18.1 ± 12.5
10	0.9 ± 0.9	1.9 ± 0.9	2.5 ± 1.0	2.8 ± 0.9	7.5 ± 2.1	10.6 ± 1.6	20.6 ± 2.2	21.4 ± 1.4
31	1.2 ± 0.3	1.5 ± 0.5	2.0 ± 0.6	1.8 ± 0.7	6.9 ± 0.7	6.9 ± 1.0	15.4 ± 1.6	18.0 ± 2.5
92	0.6 ± 0.2	0.7 ± 0.2	1.7 ± 0.2	1.5 ± 0.1	5.7 ± 0.2	5.7 ± 0.6	20.3 ± 1.5	18.2 ± 0.3
183	0.6 ± 0.1	0.7 ± 0.1	1.9 ± 0.1	1.6 ± 0.1	5.3 ± 0.1	5.9 ± 0.2	33.7 ± 3.4	39.7 ± 0.1

Fig. 2 SEM pictures of the morphology of surface of 304L SS coupon exposed to 3.5 M HNO₃ at 90 °C without UN after 92 days (left) and 183 days (right) (magnification $\times 1000$, image represent an area of $84 \times 104 \mu\text{m}$)

without UN. No discernable difference were observed for the coupon treated with UN = 0.3 M and UN = 0 M. Only the exposure time has an effect on the morphology change; at 4 days, low homogeneous dissolution of the steel occurs; intergranular corrosion appears at 10 days and continues up to 6 months; and finally grain loss was observed after 6 months treatment. In term of corrosion domain, the switch from passive into transpassive domain happens between 10 and 31 days (appearance of IGC attack) and become significant after 91 days. No discernable differences in elemental compositions of the steel were observed by EDX between all treatments.

Previous studies on corrosion of 304L reported that corrosion rates depend on experimental parameters such as temperature, time and concentration of nitric acid. A literature review with similar experimental conditions for corrosion rate of 304L SS are in accordance with the results presented here (below $100 \mu\text{m y}^{-1}$) [16]. No long term studies in 3.5 and 8 M HNO₃ at 45 and 90 °C have been reported. Recently, Kerry et al. [15] studied the corrosion of 304L SS coupons at 50 °C for 155 days with 1.17 mM U with 12 M nitric acid media and reported a rate of about $42 \mu\text{m y}^{-1}$. Unlike previous work [15], uranium is not detected at the surface by EDX spectroscopy. In the following section, we will discuss more details the effect of temperature, HNO₃ and UN on the corrosion of 304 L stainless steel.

3.1 Effect of Temperature

Corrosion rates of the coupons in 3.5 M HNO₃ as a function of the time at 45 °C and 90 °C and as a function of uranyl

nitrate concentration are presented in Fig. 4. We noticed that at 90 °C, corrosion rate is ~ 8 times higher than at 45 °C but no discernable effect on UN on the corrosion rate was observed.

Corrosion rate are higher at 90 °C than 45 °C and this difference could be explained thermodynamically. In case of nitric acid media, the main anodic reactions to consider are the oxidation of Fe, Cr and Ni metal into respectively Fe(III), Cr(VI) and Ni(II) [20]. The cathodic reaction leading the corrosion of the material is the nitric acid reduction reaction into nitrous acid [6, 20, 21] (Eq. 2):

Nernst equation with pure nitric acid (Eq. 3) [21] shows that the redox potential with temperature and allows a decrease of the transpassive potential [22].

$$E = E_0 - \frac{2.303 \times 3 \times RT}{2F} \text{pH} + \frac{RT}{2F} \ln \frac{a(\text{NO}_3^-)}{a(\text{HNO}_2)} \quad (3)$$

3.2 Effect of nitric acid concentration

The corrosion rates of 304L SS at fixed temperature of 45 °C in 3.5 M and 8 M HNO₃ with and without UN are presented in Fig. 5. An increase of nitric acid concentration leads to a low increase of the corrosion rate (factor 3 maximum after 6 months) and corrosion rates remain below $3 \mu\text{m y}^{-1}$ after 1 month and above. There is no effect of UN on corrosion rates observed at 45 °C.

Fig. 3 SEM pictures of the evolution through time of morphology of surface of 304L SS coupon exposed to 8 M HNO_3 at 90 °C without (left) and with (right) UN (magnification $\times 1000$, all images represents an area of $84 \times 104 \mu\text{m}$)

Fig. 4 Average corrosion rate ($\mu\text{m y}^{-1}$) as a function of the time for 304L SS coupons immersed at 45 °C (blue) and 90 °C (red) in 3.5 M HNO_3 for $[\text{UN}] = 0 \text{ M}$ (circle) and 0.3 M (triangle)

At fixed temperature, corrosion rates are slightly higher in 8 M HNO_3 than in 3.5 M HNO_3 ; in our experimental conditions ($[\text{HNO}_3] = 3 \text{ M}$ and 8 M and $T = 45^\circ\text{C}$ and 90°C), nitric acid concentration impact is less efficient than temperature is between 45°C and 90°C .

Previous studies [6, 20] show that corrosion mechanism of stainless steel in nitric acid depends on the acid concentration. Above 8 M HNO_3 , an autocatalytic reduction of nitric acid into oxidant species such as NO_2 occurs (Eq. 4) [6] and promote the transition to transpassive corrosion domain.

Fig. 5 Average corrosion rate ($\mu\text{m y}^{-1}$) as function of time for 304L SS coupons immersed in 3.5 M HNO_3 (blue) and in 8 M HNO_3 (red) at 45 °C for $[\text{UN}] = 0 \text{ M}$ (circle) and 0.3 M (triangle)

3.3 Cumulated effect of time, UN concentration, nitric acid concentration and temperature

The average corrosion rates of coupons treated with 8 M HNO_3 at 90°C are compared to those treated with 3.5 M HNO_3 at 45°C on Fig. 6. Beside the effect of HNO_3 and temperature observed previously, an effect of uranium on corrosion rate is observed after 6 months in 8 M HNO_3 at 90°C .

After 3 and 6 months at 90°C in 8 M HNO_3 , the corrosion rates increase for the coupon in the presence and absence of UN. At 6 months, SEM analyses of the coupon shows severe grain attack at the surface that relate to transpassive corrosion domain [10]. This phenomenon is explained that in highly concentrated acid, the Cr_2O_3 passive layer could not properly stabilize and is dissolved rapidly leading to Cr depletion [10]. The effect of the Cr content is the steel has been described as a keyrole into the corrosion rate value; decreasing Cr content could highly increase the corrosion rate [1]. At that point, the redox potential of the solution will harm the alloy by forcing HNO_3 to oxidizes Cr^{3+} into HCrO_4^- (Eq. 5) that is a Cr^{6+} soluble species and will turn to be an oxidant for the metals in the steel (Eq. 6) [21], hence increasing corrosion rate.

In 8 M HNO_3 , the speciation of uranium is dependent to nitric acid concentration and is mostly dominated by

Fig. 6 Average corrosion rate evolution with time of coupons in 3.5 M HNO_3 at 45°C (blue) and in 8 M HNO_3 at 90°C , with (black) and without (red) uranyl nitrate

U(VI) species (e.g. uranyl dinitrate) [23]. Furthermore, the U (VI)/U(IV) redox couple (+0.33 V vs SHE [24]) is higher than those of the metal in the steel (i.e., $E_0(\text{Fe}^{3+}/\text{Fe}^0) = -0.04 \text{ V}$ vs SHE and $E_0(\text{Cr}^{3+}/\text{Cr}^0) = -0.41 \text{ V}$ vs SHE [25]) then uranyl dinitrate (U^{6+} species) react with metal and be reduced into U^{4+} as Eq. (7), becoming a major oxidizing species of the system:

In such media, U (IV) generated from Eq. (7) will be immediately reoxidised to U (VI) either by HCrO_4^- or HNO_3 , creating an autocatalytic reaction. Therefore, the presence of uranium in HNO_3 should be considerate for the transpassive corrosion domain of 304 L stainless steel.

4 Conclusions

In summary, the effect of uranyl nitrate on the corrosion behavior of 304L SS in 3.5 and 8 M HNO_3 at 45 °C and 90 °C has been studied over a period of 183 days. Results indicate that the increase of the nitric acid concentration and the temperature lead to an increases of corrosion rates. At 90 °C in 3.5 M and 8 M HNO_3 a transition to a transpassive domain was observed respectively between 3 and 6 months and between 10 and 31 days. Despite no direct chemical interaction between uranium and the steel were observed, the average corrosion rate of the coupon reported were higher with uranium only when the corrosion domain switch into an advanced stage of transpassive domain. This impact of uranium should be taken in account to predict the long-term behavior of the stainless steel in spent nuclear fuel reprocessing and decontamination operations. Further studies with other radionuclides with higher redox potential than uranium, such plutonium ($E_0(\text{Pu(IV)/Pu(III)}) = +0.970 \text{ V}$ vs SHE and $E_0(\text{Pu(VI)/Pu(IV)}) = +1.040 \text{ V}$ vs SHE [1]) should be conducted to get a better understating of corrosion process of austenitic stainless steel used in PUREX processes.

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Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

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