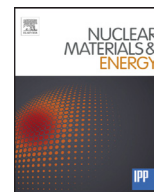




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Comparison of passivation behavior of SS316L with that of SS304 in tritiated water solution

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ABSTRACT

The effects of tritium on the passivation behavior and passive layer formed in tritiated water circumstance for SS316L were investigated by means of an anodic polarization measurement technique and X-ray photoelectron spectroscopy, respectively. The results were compared with those for SS304, since it was predicted from a model of the tritium effects on corrosion suggested in the previous studies that SS316 would be less affective to tritiated water circumstance than SS304. As the results, the passivation inhibitory effect of tritium could not be observed for SS316L, while it was observed for SS304 and the other researched materials so far, as predicted. However, the thickness of the passive layer and the boundary between the passive layer and bulk of SS316L were found affected by tritium; thickened and gradated, respectively. From these results, it was concluded that SS316L would be more sustainable in tritiated water circumstance than SS304, although the corrosion of SS316L would be more or less enhanced in tritiated water circumstance.

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1. Introduction

In fusion reactors, much more tritium than ever before will be used, generated and processed, i.e. even an experimental reactor of ITER requires 15–18 kg of tritium as a total amount [1]. Therefore, it is necessary to consider the tritium safety much more than ever before. Since metal has little radiation susceptibility in general and tritiated water has been thought to be rather stable, the studies about the durability of metal materials in tritiated water has been regarded as less serious. On the other hand, some radiochemical effects, such as enhancement of corrosion, production of unexpected corrosion products, and so on, have been predicted to be induced in tritiated water since the water circumstances would be changed by tritiation of water due to radiolysis of water, generation of ³He and hydrated electron, and so on. Indeed, it was reported so far that pH was changed in tritiated water [2–4]. From this viewpoint some studies about the corrosion behavior of in tritiated water has been reported [5]. However, it is thought to be insufficient to understand the corrosion behavior of metal materials in tritiated water, since the reports seem not to be systematic but sporadic.

In the previous studies, the effects of tritium on passivation behavior of SS304 in 1 N sulfuric acid solution were studied by means of an anodic polarization method, one of the electrochemical techniques. As the results, it was indicated that there would be passivation inhibitory effect in tritiated water, resulting in the enhancement of corrosion. Additionally, the changes of open-circuit potential over time after cathodic treatment were measured to observe the self-passivation behavior of SS304. As the results, there found to be two steps of passivation and tritium affected both steps [6–8]. Additionally, the anodic polarization behavior of both chromium and F82H steel were measured with changing tritium and dissolved oxygen concentrations as in the above studies in 0.01 N sulfuric acid and 1 N sodium sulfate solutions, respectively. The results indicated that the passivation inhibitory effect of tritium was also observed for both samples [9,10]. In the present study, the passivation behavior of SS316L was studied in tritiated solution by means of an anodic polarization method and compared with SS304.

2. Model

A model of the passivation behavior of SS304 and the effect of tritium on it is described in Fig. 1, which is based on the previous experimental results [6–8]. In this model, the passivation of SS304 consists of two steps and concerns three (pseudo-)equilibriums named pre-passive, primary-passive and fully-passive states. At the

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Table 1
Composition ratios (%) of SS316L and SS304 in JIS [12].

	C	Si	Mn	P	S	Ni	Cr	Mo
SS316L	≤0.030	≤1.00	≤2.00	≤0.045	≤0.030	12–15	16–18	2–3
SS304	≤0.08	≤1.00	≤2.00	≤0.045	≤0.030	8–10.5	18–20	–

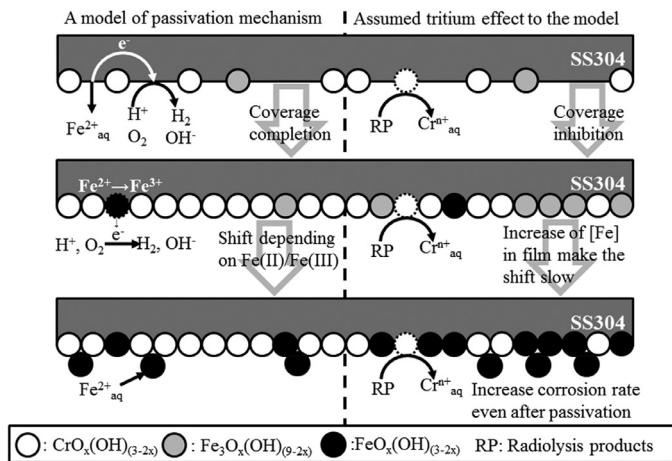


Fig. 1. A model of passivation mechanism of SS304 and assumed effects on it.

pre-passivation state, the constituents of SS304 would be actively eluted into the solution and chromium oxides would condense with the help of dissolved oxygen. It should be noted that the oxides indicated here include not only a simple oxide but also the other oxidized forms such as hydroxide, oxi-hydroxide, and so on, and the passivation is hard to start without oxygen or the other oxidant that helps the formation of oxides. The first step of passivation would be triggered by surface coverage of chromium-rich oxides layer. The primary-passive state is the fully covered one, where oxidation of bivalent iron to trivalent iron would be under pseudo-equilibrium in the primary passive layer. Thereafter, the passivation would be completed (fully-passivated state) by coverage and deposition of trivalent iron oxides which is derived from the oxidation of divalent iron which is in the solution and diffused from the bulk.

The effects of tritium in this model are based on the assumption that further oxidation and following elution of chromium in the passive layer can be induced in tritiated water solution by highly oxidative radiolysis products such as hydroxyl-radical, super-oxide radical and so on. Under this assumption, all of the tritium effects observed so far can be explained.

From this viewpoint, since SS316L is corrosion-protective under the circumstances where chromium can be eluted [11], the effects of tritium is expected to be smaller than that for SS304 and the other materials experimented so far.

3. Materials and methods

Stainless steels of SS316L and SS304 used in the present study are fabricated so as to accord the Japan Industrial Standard (JIS G 4305) of which composition ratio is listed in Table 1 [12]. A cylindrical electrode sample made of SS316L used in the present study was 3 mm in diameter covered by poly-ether-ether-ketone (PEEK) resin except for the electrode surface (detail of this electrode and electrochemical cell can be found in [6]). To avoid the air contamination and for the radiological safety, the air-tight electrochemical cell purchased from ALS Co., Ltd. was used. The counter electrode was sheathed to prevent from the dispersion of O₂ generated during cathodic treatments. The model 1287 potentiostat/galvanostat

manufactured by Solartron Analytical of AMETEK Inc. was employed as the equipment for electrochemical experiments.

The anodic polarization measurements were performed as following sequences; (i) polish the surface of sample electrode, (ii) set the electrochemical cell described above with 11 cm³ of electrolyte of 1 N sulfuric acid solution, (iii) flow the purge gas for 60 min. to remove the dissolved gases derived from air and to equilibrate dissolved oxygen with purge gas, (iv) apply 0.9 V (SHE) for 10 min. called 'cathodic treatment' to remove oxide layers on surface, (v) keep open circuit for 1 h to observe the self-passivation, and (vi) sweep the potential from open circuit potential to 1.25 V (SHE) where the state of sample become trans-passive. It should be noted that no additional heat-treatments were applied onto the samples since the PEEK resin as insulator cannot be heated approximately 600 K and higher, and the surface passive layer was electrically removed as experimental pre-treatment.

The dependence of the anodic polarization behavior of SS316L on tritium concentration, [T], and [DO] were measured. In the present experiments, the range of [DO] was from approximately 0 (<10⁻³) to 43 g-O₂ m⁻³ and the range of [T] was from <0.5 Bq cm⁻³ (without additional tritium) to 10 MBq cm⁻³. Hereafter, the conditions with and without tritium are called 'Hot' and 'Cold', respectively. It should be noted that dissolved oxygen enhance the generation of highly oxidative radiolysis products which would be the key substances of the passivation inhibitory effect of tritium as indicated in Model section, although it generally works as a help of the passivation of stainless steels. The anodic polarization measurements were performed in the same manner as the above experiment.

Additionally, the passive layers for SS316L and SS304 were analyzed by means of X-ray photoelectron spectroscopy (XPS). The XPS apparatus used in the present study is VersaProbeII design and fabricated by ULVAC-PHI Inc. For this measurement, the self-passivation treatments for them were performed with dissolved oxygen condition. The other conditions were the same as the anodic polarization measurements except for open circuit time (sequence (v)), which was changed from 1 to 4 hours. After the anodic polarization treatment, the samples were rinsed in ultrapure water to be detritiated and to remove surface sulfuric acid and dried in vacuum.

4. Results and discussion

The anodic polarization curves of SS316L obtained under Cold and Hot, with and without dissolved oxygen conditions as shown in Fig. 2. It is indicated that a passivation peak is observed in DO-removed for both of Cold and Hot conditions as in the previous studies. On the other hand in the solution with dissolved oxygen, the self-passivation due to dissolved oxygen is observed for both of Cold and Hot conditions and no obvious tritium effects could be observed such as passivation inhibitory effects. As a comparison, the anodic polarization curves for SS304 obtained under similar experimental condition except for [T] (10 and 230 MBq cm⁻³ for SS316L and SS304, respectively) of the present study is shown in Fig. 3, where obvious tritium effects can be seen; passivation inhibitory effect [6]. It should be noted that the passivation inhibitory effect could be observed in the solution with not only 230 MBq cm⁻³ but also rather lower [T]. It is clarified from these

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