

Preparation of Oxide Layer on Inconel-600 for a Decontamination Performance Test

Sang Yoon Park, Jun Young Jung, Hui-Jun Won, Wang-Kyu Choi, Jei-Kwon Moon
Korea Atomic Energy Research Institute, 989-111 Daedeok-daero, Yuseong-gu, Daejeon, 303-353, Korea
*Corresponding author: nsypark@kaeri.re.kr

1. Introduction

About 75% of primary side surface of PWR is composed of Inconel-600 or similar Ni-base alloys whose oxide properties have a critical influence on decontamination performance [1]. Their surface in the cooling circuits may become contaminated with radioactive isotopes during their normal operation. It is well known that corrosion and contamination process in the primary cooling circuit of nuclear reactors are essentially interrelated: the contaminant isotopes are mostly corrosion products activated in the reactor core, and the contamination takes place on the out-core of Inconel-600 surface[1]. This radionuclide uptake takes place up to the inner oxide layer and oxide/metal interface. So, it is necessary to remove inner oxide layer as well as outer oxide layer for excellent decontamination effects. The outer oxide layers are composed of Fe_3O_4 and $NiFe_2O_4$, on the other hand the inner oxide layers are composed of Cr_2O_3 , $(Ni_{1-x}Ni_x)(Cr_{1-y}Fe_y)_2O_4$, and $FeCr_2O_4$ [2]. Because of chromium in the trivalent state which is difficult to dissolve, the oxide layer has an excellent protectiveness and become hard to be decontamination. In order to develop excellent decontamination process, it is necessary to simulate the oxide layers on Inconel-600 surface which have chemical compositions and crystal structure similar to actual protective layers.

This study describes the preparation method of LWR simulated oxide layers on Inconel-600 surface in high temperature aqueous solution. And chemical composition, morphology, microstructure and thickness of the oxide were investigated using scanning electron microscopy (SEM) and transmission electron microscopy (TEM).

2. Experimental Methods

Inconel-600 specimens were in the form of coupons 20 mm by 20 mm by 2mm. The specimens were polished using 600-grit emery paper, degreased with acetone and ethanol, rinsed with deionized water before corrosion tests.

Test solution was 650 ppm H_3BO_3 and 2.2 ppm LiOH which simulating LWR primary water. Table 1 shows testing condition in this work. After corrosion test, some specimens for SEM and TEM analysis were cut from the coupon. The surface morphology of the specimens was examined by scanning electron microscopy (SEM) and detailed cross-section characteristics were investigated using transmission electron microscopy (TEM). Thin-foil specimens for TEM observations were

prepared using a forced ion beam (FIB) with Ga ion sputtering after a protective platinum layer was deposited on the oxide surface.

Table 1. Test Conditions

	Test 1	Test 2	Test 3
DO (ppb)	210	30	15
Temperature ($^{\circ}C$)	350	350	350
Pressure (psi)	2,500	2,500	2,500
Corrosion Time(day)	2	4	6

3. Results

3.1 Surface Oxide Morphology

Fig.1 shows SEM photographs of the oxide films formed on Inconel-600 immersed for different water chemistry.

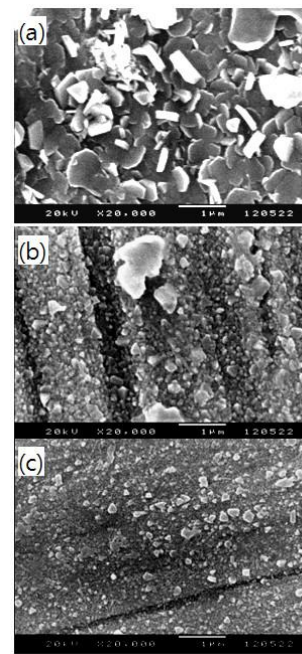


Fig. 1. SEM morphologies of the oxide films formed on specimen after (a) two days of immersion in the test 1 condition, (b) four days of immersion in the test 2 condition, and (c) six days of immersion in the test 3 condition.

It can be found that some deposited oxide particles form on the outmost surface. There are two types of oxide particles which one type is large and faceted oxide particle (disk shape) on the surface of test 1 specimen, the other one is small and irregular shape on the surface of test 2 and 3 specimens. Large and faceted oxide particles covered a whole surface of the specimen immersed in high DO containing solution (test 1), however, small and irregular particles packed on the grown-on oxide surface of the specimen immersed in low DO containing solution (test 2 and 3). Average particle size of faceted oxide particle formed in high DO containing solution was $690^d \times 170^t$ nm and irregular particles formed in 30 and 15 ppb DO containing solution has about 330 nm and 193 nm of diameter.

3.2 Cross-Sectional Properties of Oxide Film

Fig. 2 shows Cross-sectional TEM morphology and line-scanning profiles of a specimen immersed in 210 ppb DO containing solution (test 1). From the line-scanning profiles of nickel, chromium, iron and oxygen, it can be distinguished oxide layer from base alloy. It was found that 195 nm thickness of grown-on oxide layer formed under the 95 nm thickness of outer layer. Table 2 shows oxide properties formed on Inconel-600 surface corroded in test 1, 2, 3 conditions showing the grown-on oxide (inner layer) thickness, shape, size and thickness of deposited oxide particle (outer layer).

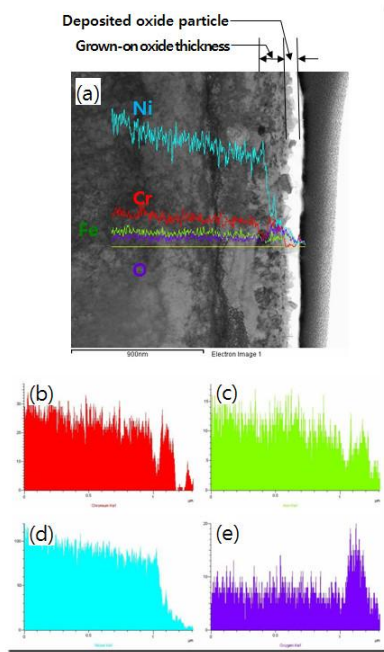


Fig. 2. (a) Cross-sectional TEM morphology of a specimen sampled in test 1 showing the grown-on oxide and deposited oxide particles, and line-scanning profiles for (b) chromium, (c) iron, (d) nickel, (e) oxygen.

Table 2. Physical properties of simulated oxide layer

		Test 1	Test 2	Test 3
Inner Layer	TG* (nm)	195	86	110
Outer Layer	Oxide particle shape	Faceted (disk type)	Irregular shape	Irregular shape
	APD** (nm)	$690^d \times 170^t$	330	193
	Thickness (nm)	95	45	40

* TG : Thickness of grown-on oxide

** APD : Average particle size of deposited oxide

4. Conclusions

This study describes the preparation method and characterization of PWR simulated oxide layers on Inconel-600 surface. Preparation of oxide in several content of dissolved oxygen was conducted and analyzed. Dissolved oxygen content played an important role of the oxide properties such grown-on oxide thickness, size and shape of deposited oxide particles. It was proven that home-made semi-loop is adequate for control of water chemistry in a static autoclave and to preparation of oxide layer similar to PWR cooling circuit's oxide layer.

REFERENCES

- [1] W.E.Berry, R.B.Diegle, Survey of corrosion product generation, transport, and deposition in LWRs, EPRI-NP-522, 1979,
- [2] Y.L.Sandler, Structure of PWR Primary Corrosion Products, Corrosion, 35, 5, 1979.