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In-situ Raman spectroscopic analysis of interfaces of Ni-base alloy/LAS dissimilar metal weld

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1. Introduction

Aging of structural materials in pressurized water reactors (PWRs) has been one of the major issues for plant safety and life extension. While a new alloy (Alloy 690) introduced, some concerns remain unresolved especially deterioration of a dissimilar metal weld. Intergranular stress corrosion cracking (IGSCC) is one of major degradation modes of aging problems that occur in structural materials for steam generators or nozzles in PWRs. The IGSCC of Ni-base alloys in PWR primary water has been extensively studied to establish theoretical and empirical models for predicting the crack initiation in field components[1]. Even though there is no general agreement on the origin of IGSCC, one common assumption of these approaches is that the damage to the alloy substrate can be related to some transport or repair properties of its protective oxide film. Therefore, the oxide film is believed to play a key role in the process of IGSCC on the surface of the Ni-base alloys in the primary water. This study is aimed to characterize the oxide film by in-situ method in order to investigate the effects of aging on the degradation of Ni-base alloy/low alloy steel dissimilar metal weld(DMW). Since the SCC of structural alloys is related to the property of native oxide layers, surface oxides on the aged weld specimens need to be characterized. It is essential to overcome the current limitation of typical ex-situ methods. In this study, therefore, a hydrothermal cell for the in-situ surface oxide analysis using Raman spectroscopy were designed and assembled. It is mainly composed of a water loop for simulated PWR primary water condition, in-situ Raman spectroscopy system, and hydrothermal optical cell for oxide analysis. Using the developed experimental system, the interface region of Ni-base alloy/low alloy steel dissimilar metal weld will be analyzed by acquiring Raman spectrum from DMW specimens exposed to high temperature and high pressure water condition.

2. Experimental Procedure

The dissimilar weld metal made of Alloy690/ Alloy152/A533B used in this study for analysis of the oxide films have been fabricated by Argonne National Laboratory(ANL). A533B was buttered with Alloy152 by shielded metal arc welding(SMAW) followed by post welding heat treatment(PHWT) at 607~635℃ for 3 hours. After processing, The Alloy690 was jointed with A533B by the SMAW with Alloy152fillers.

The hydrothermal cell for the In-situ observation in primary water environments was developed as shown Figure 1.

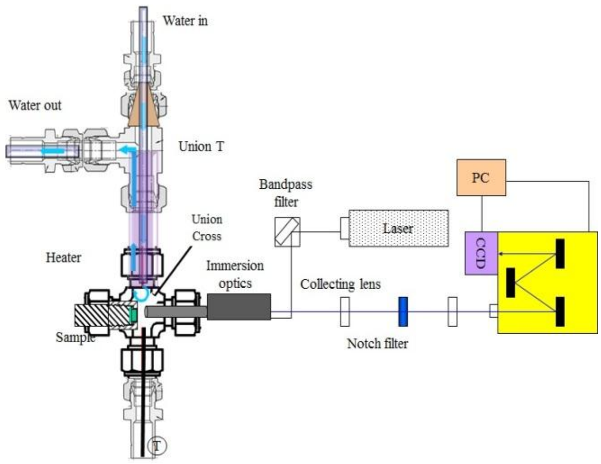


FIG. 1. The schematic diagram of Raman spectroscopy system and hydrothermal cell in this study

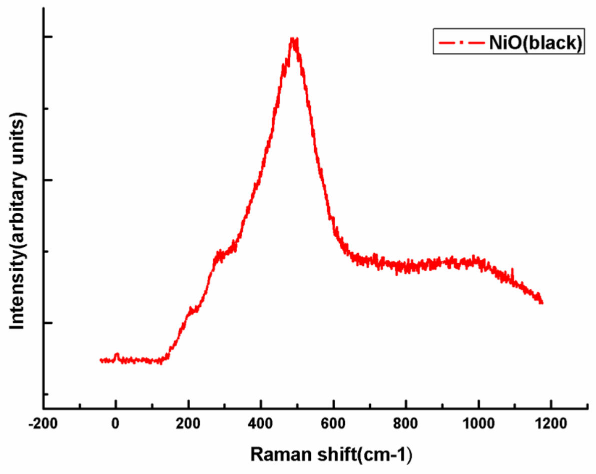
The half-inch union cross of compression fitting made of alloy 600 is used as a main optical cell, which is connected to a half-inch union tee for simulated PWR primary water circulation. The half-inch union tee plays a role of water inlet and outlet with coaxial configuration with 1/4 inch inner tubing. Through the left arm of union cross, the sample holder will be inserted, and the immersion optics will be inserted through the right arm of union cross. The union cross is fully covered by main heater and thermal insulation as described in previous section, thus its temperature should be maintained around 350℃.

Raman spectroscopy system consists of optics generating the laser exited with 514.5nm radiation, filter collecting the wavelength, and CCD detecting the light, as shown in Figure 1. There are two types of optics, immersion and non-contact optics. The former makes the laser contacting with the sample, and the latter does the light transmitting through the window. Since the manufacturing of optical window involves various, technical challenges and optical noise from window materials can be included, immersion optics primarily used in this study.

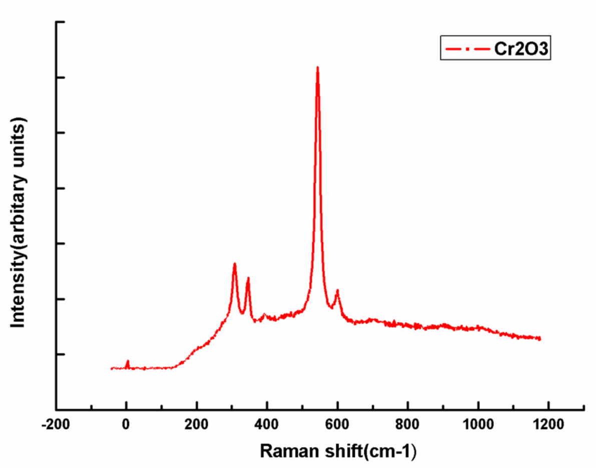
For measurements of ex-situ spectra in the air, the commercial high purity powders were used to obtain reference spectra for corrosion of DMW such as NiO-black(99.98%) and Cr2O3(99.9%) .

3. Results & Discussion

Fig 2, Shows the measured Raman spectrum for NiO and Cr2O3 powder with 532 nm radiation.



(a) NiO (black)



(b) Cr_2O_3

Fig 2. Raman spectroscopy of (a) NiO and (b) Cr_2O_3 Powder

Table 1. Nominal Raman peak wavenumbers of NiO powder

NiO (this work)	NiO-green [2]	NiO-black [2]
	1,074	
	913	
	727	
490	**535**	**490**
	370	

Table 2. Nominal Raman peak wavenumbers of Cr_2O_3 powder

Cr_2O_3 (this work)	Cr_2O_3 [3]
612	**613**
554	**552**
	527
355	**350**
300	**300**

And Table 1 and 2 shows the nominal Raman peak wave-numbers of NiO and Cr_2O_3 powers. The underline means the most intense peak in each spectrum.

The result of Raman spectrum for NiO powder is 490cm^{-1}. This result is well matched with black NiO spectrum found in [2]. The spectrum of Cr_2O_3 powder in this study exhibits the most intense feature at 554cm^{-1} and also sharp peaks at 612 cm^{-1}, 355 cm^{-1} and 300 cm^{-1}.it is also well matched with Maslar's data[3]. But the difference of intensities and locations of Raman shift corresponding to representative peaks between the results in this study and the literature is probably due to that of crystallinity of reference powder and laser wavelength. But, major Raman peaks observed in this work show good agreements with those reported in the literature, this Raman spectrum could be utilized as reference spectrums.

CONCLUSION

This work was aimed to characterize the oxide film by in-situ method in order to investigate the effects of aging on the degradation of Ni-base alloy/low alloy steel dissimilar metal weld. So that a hydrothermal cell for the in-situ surface oxide analysis using Raman spectroscopy were designed and assembled. And the primary test results of the Raman spectroscopy system with reference powders, are good agreement with the other's data. Using the developed experimental system, the interface region of Ni-base alloy/low alloy steel dissimilar metal weld will be analyzed by acquiring Raman spectrum in primary water environments.

REFERENCES

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