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Electrochemical Impedance Response of the surface treated FMS in Liquid Sodium Environment

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INTRODUCTION

HT9 and Gr.92 (Ferritic/martensitic steels) are considered as candidates materials of of Sodium-cooled Fast Reactor (SFR). Their compatibility with sodium is one of problems especially dissolution, chemical reaction with impurities, and mechanical properties.

HT9 and Gr.92 are known as compatible in sodium environment because the usual refueling time of SFRs is designed about 54 months. It is very important to investigate the corrosion-related behavior such as surface corrosion rate, carburization, decarburization and mechanical properties for its operation time. SiC and Si_3N_4 CVD coating for decarburization barrier on the surface of FMS is considered in this study.

The decarburization process where dissolved carbon near the specimen surface disused in to the liquid sodium. This process can originate from the difference between dissolved carbon in the material and liquid sodium. A compatibility test the cladding tube revealed that a decrease of the mechanical property instigated by the aging proves governed the whole mechanical property [2].

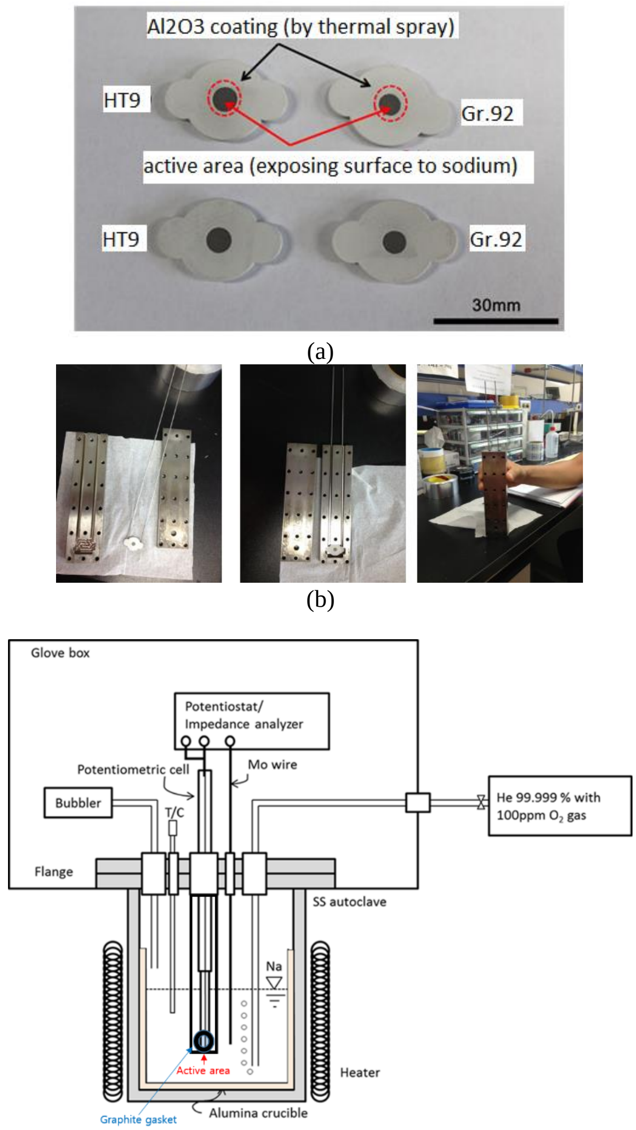
To monitor the corrosion behavior of these candidate materials in sodium environment, Electrochemical Impedance Spectroscopy (EIS) method is first introduced and investigated in this study.

The compatibility of cladding and structural materials with sodium has to be carefully investigated, as sodium could promote corrosion of cladding and structural materials in two ways. One is produced by the dissolution of alloy constituents into the sodium, and the other is produced through a chemical reaction with impurities (especially oxygen and carbon) in the sodium environment [3].

EXPERIMENTS

The chemical composition of Gr.92 is shown in Table. I. Specimen of Gr.92 was coated by thermal spraying method with 100 μm thickness of Al_2O_3 except the active area as shown in Fig. 1 (a). The working electrode (Gr. 92) was pre-oxidized at 650℃ for 48h in air condition, and at 800℃ for 48 h in air condition.

The Electrochemical impedance spectroscopy (EIS) stage was manufactured with SS 316 steel, for containing the working electrode in liquid sodium environment. Between the working electrode and the stage an insulation material was kept.

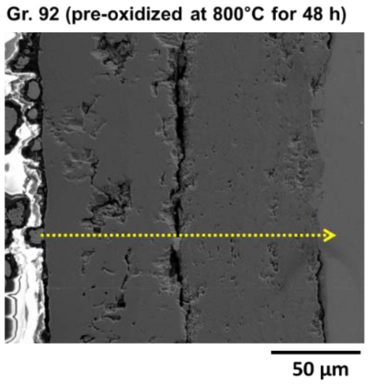


(c)

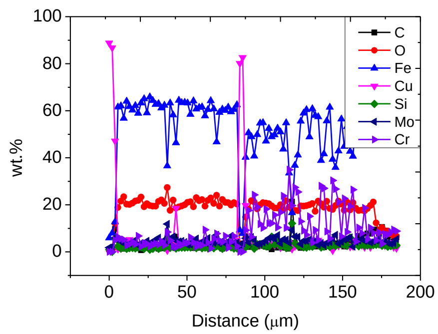
Fig. 1. Electrochemical Impedance Spectroscopy (EIS) test preparation with (a) coated working electrode (Gr.92), (b) EIS stage and (c) experimental set up.

Table I. Chemical composition of the test material

	C	Si	Mn	Cr	Ni	Mo	W	V
Gr.92	0.087	0.21	0.41	8.69	0.13	0.38	1.62	0.18
	C	Si	Mn	Cr	Ni	Mo	W	V
HT9	0.19	0.14	0.49	12.05	0.48	1.00	0.49	0.30



(a)



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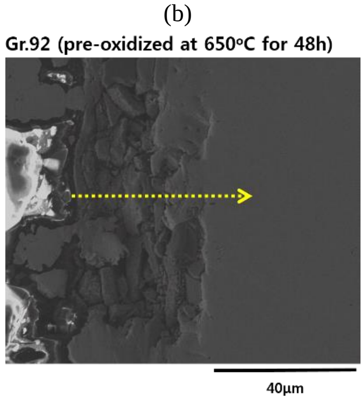
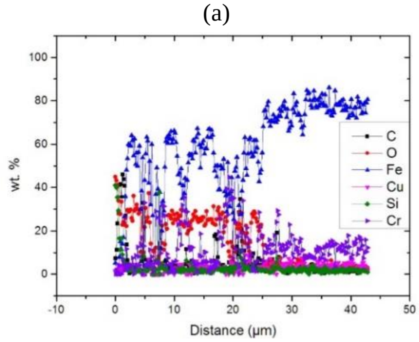


(a)



(b)

Fig. 2. SEM image of cross section of Gr.92 pre-oxidized at 800℃ for 48 h (a) and EDS data (b). SEM image of cross section of Gr.92 pre-oxidized at 650℃ for 48 h (c) and EDS data (d).

RESULTS

Before exposure to sodium, the cross-section of the pre-oxidized specimen was investigated.

The cross-section image of the pre-oxidized at 800℃ for 48 h Gr. 92 is shown in Fig. 2. EDS (energy dispersive spectroscopy) analysis reveals that there are two oxide layers formed on the surface. The outer oxide is mainly composed of Fe and O and the inner oxide is composed of Fe, Cr and O. Phenomenon of crack between inner and outer oxide comes from difference of Pilling-Bedworth ratio (P-B ratio). The spinel thickness is 158.09μm, magnetite thickness is 140.28 μm.

The cross-section image of the pre-oxidized at 650℃ for 48 h Gr. 92 is shown in Fig. 2. EDS analysis reveals that there are one oxide layers formed on the surface. The oxide of pre-oxidized at 650℃ for 48 h specimen is composed of Fe and O. This layer is similar to the outer oxide of pre-oxidized at 800℃ for 48 h specimen. The magnetite thickness is 39 μm. There is not observed spinel section.

According to pre-oxidized temperature, thickness of the cross-section of pre-oxidized specimen was different from 650℃ oxide to 800℃ oxide.

After EIS test with this specimen in sodium environment, the Nyquist and Bode plots were obtained as shown in Fig. 3. From the plots, the resistance of oxide was measured as 175.46 Ω, the resistance of sodium was measured as 19.13 Ω, and the capacitance of oxide was measured as 2.85 μF.

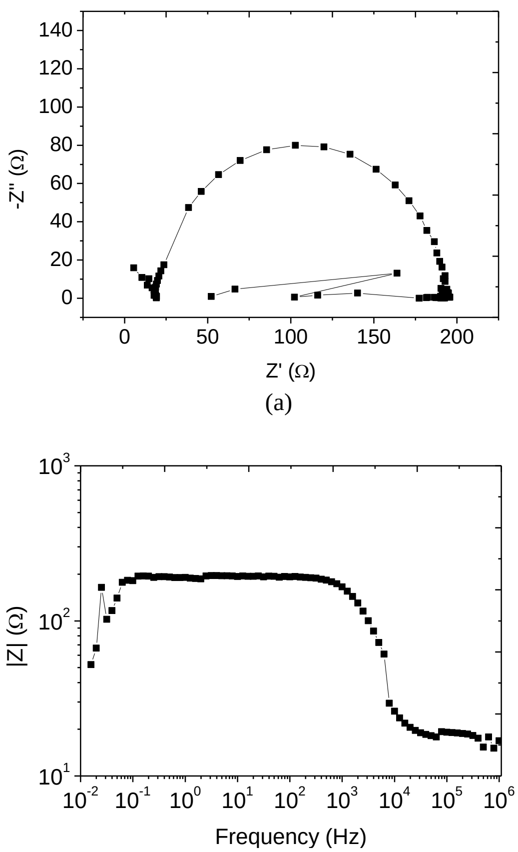
CONCLUSIONS

EIS test with pre-oxidized Gr. 92 specimen in 200℃ liquid sodium environment was carried out in this study. A clear Nyquist and Bode plots were obtained in liquid metal environment and the resistance of sodium and the oxide, and the capacitance of the oxide were measured from this result.

FUTURE WORK

The EIS tests at higher temperature with pre-oxidized HT9 and Gr.92 specimens are under process and also as-received specimens were exposed to oxygen saturated sodium environment for $NaCrO_2$ formation on the surface for further EIS tests.

The specimen of 650℃ pre-oxidized will be tested on same experiment environment which is 200℃ liquid sodium and 1 hour exposure time. The Silicon Carbide (SiC) coating and Silicon Nitride (Si_3N_4) coating will be deposited on the specimen surface, and these will be exposed to oxygen saturated sodium environment. After exposure process, role of SiC and Si3N4 can be analyzed, and difference from corrosion behavior on non-deposition specimen to deposition specimen can be analyzed. The cross-section image of Si_3N_4 coating is shown in Fig.4.



(b)

Fig. 3. Results of electrochemical Impedance Spectroscopy (EIS) test with (a) Nyquist plot and (b) Bode plot.

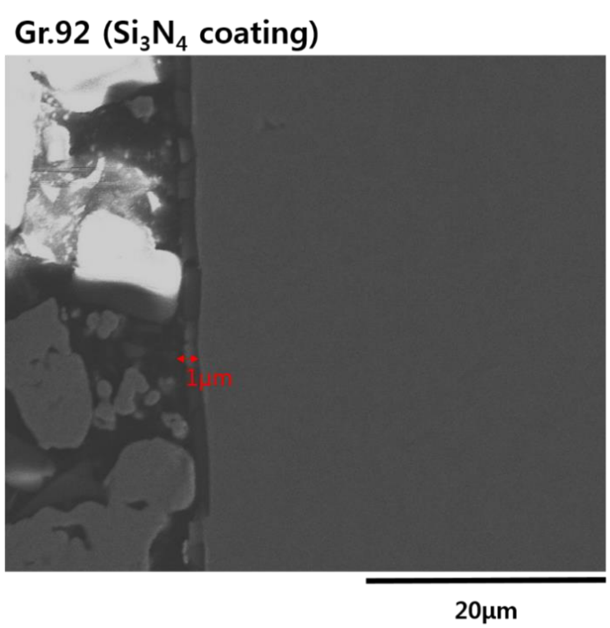


Fig. 4. . SEM image of cross section of Gr.92 Si_3N_4.

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