

§28. Experimental Study on Self-healing Functional Layer for Liquid Breeder Blanket

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The development of functional materials such as an electrical insulator and a tritium permeation barrier are essential technology for the development of fusion blanket system. Several oxides, such as  $ZrO_2$ ,  $Y_2O_3$ ,  $Al_2O_3$  and  $Er_2O_3$ , are thermodynamically stable, and their coating layers have good performance as the hydrogen permeation barrier. These functional layers can be formed by the oxidation of surfaces of Zr, Y, Al and Er metals, which are plated on the structural materials. The electrochemical impedance spectroscopy (EIS) must be a feasible technique to monitor the formation and the deterioration of the functional layers in the liquid breeders. The purpose of the present study is to investigate the oxidation behaviors of Zr, Y and Er metals and the electrical characteristics of these oxide layers around the operation temperature by EIS method.

The specimens used for the pre-oxidation tests are shown in Fig. 1 (a). The pre-oxidation tests were performed in an air atmosphere at 773K. The test durations are 100, 250 and 500 hours. The weight changes of the pre-oxidized specimens were measured using an electro-reading balance with accuracy 0.1mg. The oxide layers formed on the specimen surfaces were analyzed by XRD. Their surfaces and cross sections were metallurgically analyzed by EPMA. Fig. 1 (b) shows the cylindrical type specimen for the EIS analysis. The electrode of Au was plated on the specimen surface by a sputter coating. Figure 2 (a) shows the schematic diagram of the test apparatus for the EIS analysis. Figure 2 (b) shows the detail structure of the test section. The analysis was performed at the temperature up to 773K.

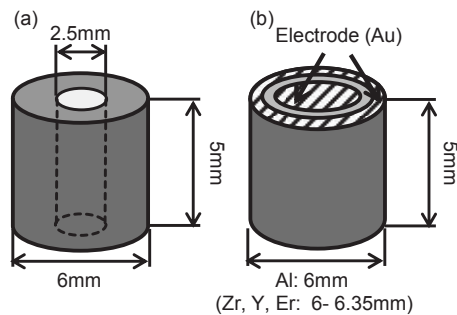


Fig. 1 Specimens (a) pre-oxidation test, (b) EIS measurement after pre-oxidation

The compact oxide layers were formed on the Zr and Y metals in air atmosphere at 773K. The thickness of the layer formed on the specimen surface was evaluated from the results of EPMA cross sectional analysis and summarized in Table 1. The EIS analysis on the Zr and Y specimens were

performed. Their electrical properties were successfully obtained by EIS analysis. Fig. 3 shows the Nyquist plot obtained at 673K with Zr specimen pre-oxidized at 773K. The plots indicated a semi-elliptical shape and the inhomogeneity of the layer structure such as a thickness variation and/or a porosity of the layer. This feature agreed with the thickness variation observed by EPMA analysis. The oxide layer was modeled as a parallel RC circuit with the elements of electrical resistance ( $R$  [ $\Omega$ ]) and capacitance ( $C$  [F])

Table 1 Thickness of oxide layers formed at 773K evaluated from results of EPMA analysis

	Time [hour]	Thickness [ $\mu\text{m}$ ]		
		Variation	Arithmetic mean	Harmonic mean
Zr	100	1.3-3.5	1.9	1.9
	250	2.0-4.8	3.5	3.3
	500	1.6-8.2	4.6	4.2
Y	100	3.2-15.8	9.8	8.0
	250	14.6-43.0	27.8	25.0
	500	17.1-55.1	35.1	32.3

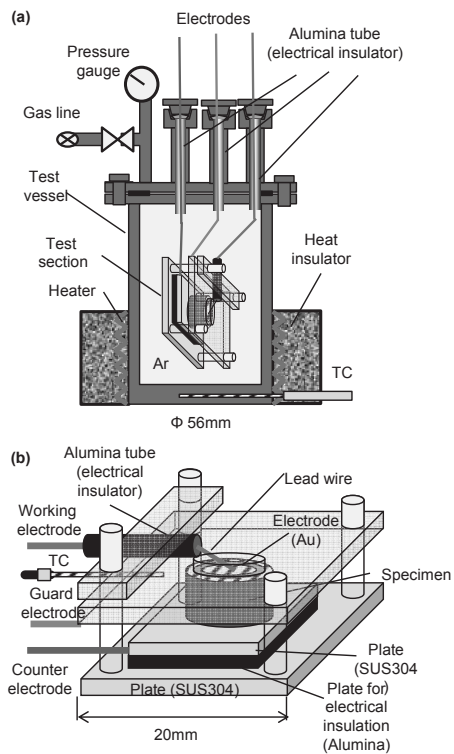


Fig. 2 (a) Test vessel for EIS analysis, (b) test section

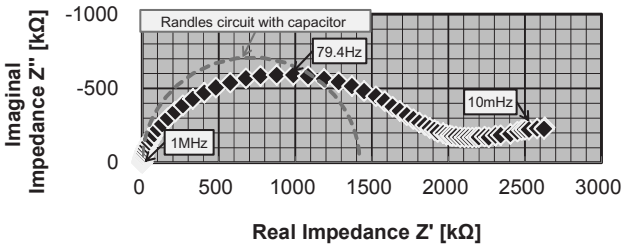


Fig. 3 Nyquist plot obtained at 673K with Zr specimen pre-oxidized at 773K