# Corrosion test with developed ARAA in the Experimental loop for a liquid breeder

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#### **1. Introduction**

Korea has designed various concepts of a breeding blanket for the development of a DEMO fusion reactor. One of them, a helium cooled ceramic reflector (HCCR) test blanket module (TBM) has been developed for testing in ITER, and others have been investigated using domestic R&D programs. Among them, the Korea Atomic Energy Research Institute (KAERI) has participated the HCCR TBM and liquid breeder blanket [1,2]. In the liquid breeder blanket concept, helium (He) was used as a coolant and liquid lithium (Li) or lead lithium (PbLi) was used as breeder material. In addition, ferritic-martensitic steel (FMS) was considered as a structural material. An Experimental Loop for Liquid breeder (ELLI) has been constructed to evaluate the effects of magneto-hydrodynamics (MHD) in a flowing liquid material, Pb-15.7Li, in the test loop, and to perform the compatibility of PbLi using structural materials such as ferritic martensitic steel. After construction of the ELLI, some performance tests for the main components of the ELLI, such as a sump tank, an EM pump, and a magnet, were conducted. The EM pump performance test was carried out in the ELLI. The maximum flow rate was measured at up to 105 kg/min (11.1 lpm) by varying the voltage of the EM pump [3,4]. Long-term operational tests of the EM pump were performed and corrosion tests were carried out by forced convection circulation using an EM pump to investigate the corrosion behavior of FMS in flowing PbLi [5].

In a previous study, a corrosion test using a commercial FMS with grade 91 in the flowing PbLi was performed. Two kinds of specimen, a tubular type and a cylinder type, were fabricated using commercial FMS for the corrosion specimens. The corrosion test was carried out in the ELLI loop by forced convection circulation using an EM pump during three different period [5].

In this study, a corrosion test was performed with Korean FMS ARAA [6] and with a commercial FMS with grade 91 material. For the corrosion test, tubular-type specimens were fabricated. Some of the specimens were coated and replaced with uncoated specimens between the first and second period experiments. The specimens were exposed to the flowing PbLi at a speed of 0.16 m/s at 320 °C for the two different experiments. The corroded specimens were analyzed by measuring the weight change and observing the corroded surfaces, respectively.

## 2. Corrosion Test with developed FMS of ARAA

### 2.1 Test Loop

The Experimental Loop for a Liquid breeder is a forced convection loop by using an electromagnetic pump [3]. The main components of the ELLI consist of an EM pump to circulate the liquid metal, a sump tank to reserve the PbLi and to melt the solid PbLi using heating jackets, a magnet, and heating and control systems. The experimental loop was equipped with a pressure gauge, a level sensor to measure the level of melted PbLi in the sump tank, and thermocouples for diagnostic purposes in the experimental loop. The experimental loop and control systems are shown in Fig. 1.

## 2.2 Experiments

The chemical compositions (wt.%) of the ARAA used were 9.33Cr, 1.05W, 0.46Mn, 0.21V, 0.09Ta, 0.09C, 0.07Si, 0.024N, 0.018Ti, 0.009Zr, and Fe for balance [6]. Tubular-type samples 15 mm in length, with a 10 mm diameter and 1 mm thickness, were fabricated using the ARAA for the corrosion specimens. Some of the samples were surface coated with either Al<sub>2</sub>O<sub>3</sub> (A), ZrO<sub>2</sub>-Y<sub>2</sub>O<sub>3</sub> (ZY), or Ta<sub>2</sub>O<sub>5</sub> (T) using a sol-gel coating method [7]. Precursors for the oxides were prepared from commercial dip coating solutions (Al-03-P, SZ-10-3, Ta-10-P, Kojundo, Japan). The dip coating was performed in air at 120 °C (for drying) to 750 °C (for curing). The samples were then finally heat-treated at 950 °C in a vacuum for 10 min for the crystallization of the coated oxides.

A corrosion test for the ARAA was performed in flowing PbLi. The fabricated tubular-type specimens were fixed to a post using wires, as shown in Fig. 2. Three sets of the corrosion specimens were installed to three test pots. Fig. 3 shows the flow rate measurements of the liquid breeder.



Fig. 1. Schematic diagram of the experimental loop.



Fig. 2. Photographs of the fabricated specimens fixed to posts for the corrosion test (above) and specimens after the corrosion test (below).



Fig. 3. Flow rate measurements during two experiments.

#### 3. Results of the corrosion test

The corrosion specimens were analyzed by the measurement of the weight change and the microscopy of the corroded surfaces. A metallographic analysis was conducted using a scanning electron microscope for the corroded ARAA specimens with and without coating.

## 2.1 Mass change

After a corrosion test in the flowing PbLi at 320 °C for 250 h, the weigh losses of the samples were measured. The initial masses for the fresh ARAA sample and fresh Grade 91 sample at test pot #1 were 3.31847 g and 3.40016 g, respectively. Then, the masses after the corrosion test were obtained as 3.31827 g for ARAA and 3.39985 g for Grade 91. The mass of the samples was decreased about 0.007% on average, which corresponds to 0.26 g/m<sup>2</sup> or 0.033  $\mu$ m of a corrosion attack. Unfortunately, we were unable to compare these weight

losses with previous reports because of the relative low corrosion temperature (320 °C) and short exposure time (250 h). In the case of the coated samples, the weight loss was suppressed to about 0.002% on average. The initial masses for the Al<sub>2</sub>O<sub>3</sub> coated ARAA sample and ZrO<sub>2</sub>- $Y_2O_3$  coated ARAA sample at test pot #2 were 3.3690 g and 3.4180 g, respectively. Then, the masses after the corrosion test at 250 h were decreased to 3.3689 g and 3.4179 g, respectively. This suggests that the coated oxide layers prevented corrosion of the FMS.

## 2.2 Microstructure investigation

Fig. 4 shows the microstructures of the corroded FMS samples without surface coatings. The machining marks along the vertical direction appear on the specimen's surface. Some parts of the sample were damaged while other parts remained intact. In addition, the grains and grain boundaries were observed at the corroded parts. The weak or stress accumulated regions that had deformed during the mechanical machining corroded preferentially, as observed in our previous report [7,8]. The elemental distribution at the corroded surface is also presented in Fig. 4 from an EDS line-scan analysis on the cross sections of the samples. The concentration profile of Fe, Cr, W, Zr, and Mo at the surface had not changed within the resolution limit during 250 h of exposure. The appearance of the corroded microstructure as well as the elemental distribution is similar between the ARAA and Grade 91 sample. Oxides such as Al<sub>2</sub>O<sub>3</sub>, ZrO<sub>2</sub>-Y<sub>2</sub>O<sub>3</sub>, and Ta<sub>2</sub>O<sub>5</sub> were coated on ARAA as a corrosion barrier layer.



Fig. 4. Microstructures along with cross-sectional element analysis of the corrosion sample of (a) ARAA and (b) Grade 91 without surface coating during 250 h.

## 3. Conclusions

The corrosion tests using ARAA and commercial FMS were performed under the conditions of a flowing PbLi in the ELLI. The flowing speed in the loop was 0.16 m/s for 250h during two separated experiments. The effects of the oxide coatings on the prevention of the corrosion progress were also investigated. After the corrosion test, micro-structural observations of the FMS surface and an elemental analysis were conducted. The mass of the samples was decreased by about 0.007 % on average during 250 h, which corresponds to 0.26 g/m<sup>2</sup> or 0.033  $\mu$ m of a corrosion attack. In the case of coated ARAA samples, the weight loss was suppressed to about 0.002% on average. It was observed that the sol-gel derived oxide coatings of Al<sub>2</sub>O<sub>3</sub> and Ta<sub>2</sub>O<sub>5</sub> were protective against PbLi corrosion, well.

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