A study of lead-alloy in static experiment of 600~650°C

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1. Introduction

We try to evaluate the possibility of maintaining the corrosion-resistance of a structural material under the operational temperature and flow velocity of the optimized HYPER [1] (HYbrid Power Extraction Reactor) cooling system. It was attempted to consider the control of the oxygen concentration in the possible ranges of 350~650°C. Lead-Bismuth eutectic (LBE) alloy was determined as a spallation target and coolant material of the HYPER due to its high production rate of neutrons and effective heat removal. However, LBE has a great corrosion problem over more than 400°C because the solubility of Fe, Cr and Ni is high. Thus, the problem has been considered as an important design-factor that limits the operational temperature and flow velocity of the ADS system.

2. Methods and Results

2.1 Experimental Methods

For a long, several methods have been considered to prevent the corrosion problem in LBE, one of the method is to control the oxygen concentration in the liquid LBE [2]. Thus, the corrosion behavior is reduced to form a stable oxide layer on the material surface through an oxygen level control. Another one is to modify the surface of the material or the material compositions. Static corrosion tests are useful to investigate the corrosion properties and modes of various kinds of materials to develop corrosion resistant materials for liquid LBE. KAERI finished the setup of the static corrosion facility in 2003 and started a static corrosion test and systematic research to develop the measuring techniques for the control of the oxygen concentration.

The experiment is performed static corrosion tests with the exposure time of 200~1500 hours. The oxygen contents are both reduced and 10⁻⁶ wt% atmospheres at the temperature of 600~650°C. The test specimens were 316L and some martensitic steels such as T91, HT9.

The dimension of the samples are prepared as 10mmX18mmX2mm. The samples were annealed at 1050°C for 1 hour and the heat treatment was done at 750°C for 2 hour except for the 316L.

2.2 Oxygen Control Methods

The oxygen concentration is controlled by adjusting the H₂ and H₂O vapor ratio. The oxygen concentration of 10⁻⁶wt% is controlled by the chemical equilibrium between the Ar-5%H₂ and the water vapor. Equations (1)–(4) are used to calculate the corresponding oxygen partial pressure. Then Equation (5) is used to determine the pressure ratio of H2 and H2O. The H₂O pressure is set to be 15.94mb. Ar gas is forced to flow with a rate of 100ml/min and the corresponding rate of the Ar-5%H₂ gas is also forced to with a rate of 5ml/min.

\[ a_s = \frac{C_s}{C'_s} = \left( \frac{P_{O_2}}{P_{O_2}^*} \right)^{1/2} \]
\[ \log C'_s = 1.2 - \frac{3400}{T} \]
\[ \log P_{O_2} = 10.55 - \frac{23060}{T} \]
\[ \log P_{O_2} = 2 \log C'_s + 8.16 \frac{16261}{T} \]
\[ P_{O_2} = \frac{P_{O_2}^*}{P_{O_2}^*} \exp\left(\frac{2\Delta G_{O_2}}{RT}\right) \]

\( C_0 \): Oxygen concentration (wt%), \( C'_s \): Solubility of oxygen in Pb-Bi, \( P_{O_2}^* \): Partial pressure saturated (bar), \( T \): Temp. (K)

2.3 The results of Oxygen Control Methods

Fig. 1 shows the SEM/EDX results of the 316L exposed to Pb-Bi at 650°C with the oxygen content of 10⁻⁶wt% for 200 hour. The oxygen layer was not detected. Dissolution attack is clear and the corrosion peak is heterogeneous throughout the sample surface.

Fig. 1 EDX of 316L tested under Oxygen Content 10⁻⁶ wt% at 650°C for 200 hour
Fig. 2 shows that the Point 10 of (a) affected by Pb with the 22.29wt%; Point 10 has clear Dissolution attack due to the solubility of Cr and Ni. Point 3 and 4 regions of (b) appeared Pb-Bi attack of 14.15wt% and 18.61wt%. The (c)~(d) show that the zone at the top, that ends at the original specimen surface, consists of magnetite without appreciable Cr concentration. The layer in middle contains Cr-Fe spinal that roots in the pore belt. In the interior, an oxygen diffusion zone can be observed in which oxides precipitate along the grain boundaries[3]. Also, no clear corrosion attacks were seen in Pb and Pb-Bi.

![Fig. 2 SEM/EDX of 316L tested with a reduced <10^{-8} wt% at 600° for 1500 hour [(a): Pb, (b): Pb-Bi], Specimens exposed Pb under oxygen content 10^{-8} wt% at 600° for 500 hour [(c)~(d)]]

3. Conclusion

The solubility of nickel is more than iron and chromium in LBE. The 316L of austenitic steel has more nickel than the T91. Therefore the corrosion of 316L is severe under the reduced atmosphere due to the solubility of the nickel. And small amount of iron and chromium dissolved near the surface. A partial oxygen layer was found under oxygen control atmosphere at 600° for 200 hour. The other side, a homogeneous oxide layer was formed under oxygen content 10^-8 wt% for 500 hour.

REFERENCES

