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Effect of temperature on the corrosion behaviors of 304 stainless steel in static liquid lithium

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ABSTRACT

The study of the corrosion behaviors of structural materials exposed to liquid lithium (Li) is very important for the application of liquid Li as a potential plasma-facing or blanket material in future fusion device. It was demonstrated that the temperature plays an important role in the corrosion process. The corrosion behaviors of 304 stainless steel (304SS) in static liquid Li at 600 K and 640 K for 1320 h under high vacuum have been compared in the present work. The results show that the weight loss and corrosion depth of the 304SS specimens in static liquid Li at 640 K are about 3 times more than those at 600 K. After exposure to liquid Li, all the surfaces produce non-uniform damage which includes pitting and grain boundary corrosion. The surface damage of 304SS specimens at 640 K is more serious than that at 600 K. The corrosion mechanisms include physical dissolution and chemical corrosion; the effect of the latter on the corrosion behavior at 640 K is much more than that at 600 K.

1. Introduction

Li is a very attractive element in fusion application due to its particular physical and chemical properties, such as low atomic number, low radiating power and strong impurities gettering [1,2]. The melting and boiling temperatures of Li are 454 K and 1616 K, respectively; thus liquid Li has a wide temperature range in liquid state. Liquid Li is not only considered as a potential candidate material for the blanket as a coolant and tritium breeder but also as a plasma-facing components (PFCs) for the inner wall (First Wall) and divertor; therefore, it is being got more attention recently. Blanket concepts using liquid Li are promising for that they can offer a high tritium breeding ratio, high efficiency, low radiation damage for breeders and good thermal transfer [3,4]. Furthermore, in some fusion devices, a dramatic improvement of plasma performance has been observed by using liquid Li surfaces [5–14]. The compatibility of liquid Li with structural materials, especially stainless steels (SSs), is a critical issue because most of the structure materials used are SSs in these systems.

To investigate the compatibility of SSs with liquid Li, a static liquid Li testing device in our group has been built, which can be heated to more than 600 K and pumped to high vacuum. The detailed description of the testing device can be found in our previous paper [15]. The corrosion behaviors of 304SS in static liquid Li at 600 K up to 1548 h were investigated in our previous work [15], where it was found that the weight loss for 304SS specimens increases with the corrosion time and the 304SS specimens suffer from the corrosion by preferential grain boundary attack.

It is known that, aside from the corrosion time, the temperature also plays an important role in the corrosion processes. There are many investigations focused on the compatibility of SSs in liquid Li environments at high temperature (> 773 K). For example, the corrosion behaviors of five different commercial Cr-Mn austenitic SSs (MACR, ICL016, NMF3, Nitronic 32, and Carpenter 18/18 Plus) in static liquid Li at 873 K up to 6000 h were investigated by Ruedl et al. [16], it was observed that all materials showed a penetration of high-angle boundaries by Li. Xu et al. [17,18] found the weight loss of JLF-1 steel at 873 K static liquid Li was larger than that at 773 K, and due to C depletion there was a phase transformation from martensite to ferrite for JLF-1steel at 973 K liquid Li. Li et al. [19] found that the 9Cr-ODS steel exhibited a slight weight loss and decrease in hardness near

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surface and a non-uniform corrosion behavior by preferential grain boundary attack. Its tensile property showed a negligible change and the creep property degraded at 973 K after exposure to liquid Li at 873 K for 250 h.

Despite the numerous experiments regarding the corrosion issues of conventional steels in high temperature liquid Li, the corrosion behaviors or corrosion mechanism of SSs maybe very different at low temperature which is the operational conditions of the liquid Li limiters (LLLs). The experiments of 304SS in static liquid Li at 640 K for 1320 h are carried out and the corrosion behaviors of 304SS in Li at 640 K and 600 K are compared in the present work. The effect of temperature on the corrosion mechanisms of 304SS in LLLs system conditions has been discussed, which is important for further guiding the design and application of components with liquid Li in fusion devices.

2. Experimental conditions

In LLLs systems, the temperature range for liquid Li is between 500 K and 800 K and the vacuum chambers' pressure of fusion devices is less than 10^{-5} Pa [20]. As confirmed by experiments [21,22], the critical wetting temperature range is found to be around 580 K–600 K and the contact angle of liquid Li on SS decreases with an increase of the temperature. When the temperature reaches 640 K, liquid Li has good spread-ability on the surface of SS and the contact angle is about 50°. In 2014, a new flowing liquid Li limiter (FLiLi) with temperature about 640 K based on the concept of a thin flowing film has been successfully tested in the EAST device [14]. Nevertheless, the surface of the FLiLi was damaged by the bombardment of high temperature particles and corrosion of liquid Li. Therefore, the experiments of 304SS in static liquid Li at 640 K for 1320 h under high vacuum (approximately 10^{-5} Pa) are carried out, and the corrosion behaviors of 304SS in Li at 640 K and 600 K are compared in the present work.

3. Experimental setup

The 304SS specimens, Fe-Cr-Ni based austenite SS, are coupon-type ones with the size of 15 mm (Length) × 10 mm (width) × 1 mm (Thickness). The surfaces of the specimens were mechanically polished and cleaned with ultrasound in high purity alcohol before the experiments. Metallic Li was supplied in ingots with high purity of 99.9%, the N content was about 270 ppm. The compositions and ratios of 304SS and Li are listed in Table 1.

The experiments were carried out in a static test facility which has been described in detail previously [15], the experimental procedure was the same as before. The fluctuation range of the temperature was about 5°. Six specimens were dipped into liquid Li (900 g) and the ratio of Li volume V_{Li} (cm^3) to the total surface area S (cm^2) is about 2.14 cm. After being exposing to liquid Li for the expected time, the specimens, which were protected by an Ar atmosphere, were removed from the test vessel when the device cooled to ambient temperature. The remnant Li, covering the specimen surface, quickly lost the metallic luster and became black as a result of reaction with air (forming a complex mixture, Li_3N:Li_2O = 3:1). To avoid the nitride solution, the remnant Li covering the specimens was finally cleaned with high purity alcohol of 99.9%.

The investigations of the specimens were carried out for the weight loss by an electronic balance with an accuracy of 0.01 mg, the morphology and composition by Scanning Electron Microscope (SEM) equipped with an Energy Dispersive X-ray Spectrometer (EDS) and X-ray diffraction (XRD) analysis. For the weight loss, each specimen was measured 5 times and then the average value was obtained.

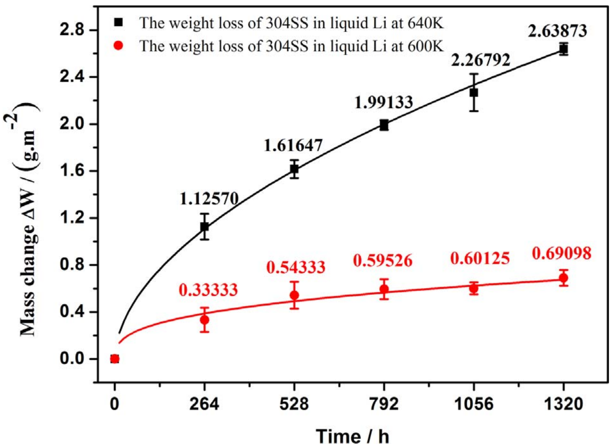

Fig. 1. The weight loss-time curves of 304SS specimens in liquid Li at 600 K and 640 K.

4. The comparison of corrosion behaviors at different temperatures

4.1. Comparison of the weight-loss rate and corrosion depth

The corrosion rate was evaluated by the weight-loss method, where the weight changes for the specimens were obtained before and after exposure to static liquid Li. Fig. 1 shows the weight loss-time curves of 304SS specimens in liquid Li at 600 K and 640 K, respectively. The results show that the weight-loss of 304SS specimens increased slowly with the corrosion time and the weight-loss of 304SS specimens in static liquid Li at 640 K is about 4 times of those in static liquid Li at 600 K. The fitted curves also indicate that the weight loss of 304SS specimens in liquid Li at 600 K and 640 K have an 1/2-power law trend with corrosion time.

4.2. Comparison of the microstructures and compositions

The microstructures and compositions of 304SS specimens before and after being exposed to liquid Li were measured by SEM and XRD, as shown in Figs. 2–4. The 304SS specimen exhibited a metallic luster but many nicks existed on the surface because of the mechanical processing before test, as shown in Fig. 2a. After the remnant Li on the specimens' surfaces was cleaned by water or high purity alcohol, the specimens showed a non-uniform brightness which indicated some inhomogeneous corrosion.

After being exposed into liquid Li at 600 K for 1320 h, the surface of 304SS specimens were slightly damaged. As shown in Fig. 2b, there were a lot of precipitates adhered to the 304SS specimens' surface and the area ratio of precipitates was about 43%. At higher magnification, it was shown that the precipitates were spinel-like with the sizes of 1–2 μm and lots of holes were on the rest of the surface, as shown in Fig. 2c. The EDS and XRD surface analysis showed that the

Table 1
The compositions and ratios of 304SS and Li.

Li	Element	Na	K	Ca	Fe	N	Si	Cl	Al	Ni	Cu
	Content ration%	0.0045	0.0002	0.0015	0.003	0.0027	0.002	0.002	0.001	0.002	0.001
304SS	Composition	Cr	Mo	Mn	Ni	C	Si	Cu	Co	Al	Fe
	Wt%	17.07	2.4	1.0	10.31	0.026	0.34	0.24	0.21	0.045	Bal