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Stress corrosion behavior of T91 steel in static lead–bismuth eutectic at 480 °C

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HIGHLIGHTS

- The stress corrosion test of T91 stressed C-rings in LBE with the constant strain technology was conducted.
- Cracks were not found on the T91 in oxygen saturated LBE due to the oxide prevents the contact between the matrix and LBE.
- The growth of the oxide layer was accelerated by stress, especially for the outer layer.

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ABSTRACT

The corrosion behavior of stressed C-rings made of martensitic steel T91 was investigated through constant strain tests. The specimens with different initial hoop stresses (0 MPa, 150 MPa and 300 MPa) were exposed to static oxygen saturated lead–bismuth eutectic (LBE) at 480 °C for 500 h, 1000 h and 1500 h, respectively. The results showed that no crack was found on the outer surface of all the specimens after exposure; and the microscopic analysis showed that the specimens were covered with two oxide layers, which included a magnetite outer layer and a Fe–Cr spinel inner layer. The transformation of spinel into magnetite at the spinel/magnetite interface might be promoted by stress, which increased the difference between the thickness of the inner and outer layers. Moreover, the steel loss was estimated by the observed oxide layers; it increased rapidly when the stress was above 300 MPa, and was about 1.3 times of when the stress was absent.

1. Introduction

Lead–bismuth eutectic (LBE) has been considered as a primary candidate material for high-power spallation neutron target and coolant in the sub-critical reactor at the accelerator driven systems (ADS) due to its favorable thermal–physical and chemical properties, including the low melting point, low vapor pressure, wide margin to boiling, high spallation neutron yield, low neutron moderation and capture, and especially its chemical inertness resulting in mild reaction with air and water [1]. However, one of the key limitations for design and application in lead based cooled reactors is the resisting corrosion ability of structural materials.

Research and development on China LEAd-based cooled Reactor (CLEAR) for ADS program has been done at the Institute of Nuclear Energy Safety Technology, China Academy of Sciences [2,3]. LBE has been selected as the main coolant material in CLEAR-I [2], which is the first stage of the program. T91 martensitic steel has been proposed as a candidate structural material for some critical components (such as the heat exchanger and the cladding tubes, etc.) in CLEAR. These components are exposed to LBE and also suffered from internal pressure. The study of corrosion behavior of the T91 steel under stress in LBE is an essential issue in the development of CLEAR.

Related experimental results revealed that the service performances of structure materials were reduced by the synergetic effect of LBE and stress. Liquid metal embrittlement (LME) has been studied with slow strain rate test (SSRT) by J. Van den Bosch's and Yong Dai's groups [4–6]. Their research focused on the effects of LBE on the mechanical properties of T91, while studies on long time stress corrosion behavior are limited. Before considering the effect of stress, a lot of studies on the corrosion behavior and mechanism of T91 in oxygen saturated LBE without stress have been conducted by L. Martinelli et al. [7–9]. According to these authors, the oxide layers of T91 steel exposed to LBE have a duplex structure

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composed of an external magnetite layer growing by Fe outward diffusion and an internal Fe–Cr spinel layer growing inside the space kept by the Fe vacancies. Therefore, the inner oxide layer is coupled to the outer layer if there is no significant Fe element dissolution in LBE. However, in the experiment of T91 cladding tube exposed to LBE at 550 °C for 3000 h with the internal pressure of 15 MPa conducted by A. Weisenburger et al. [10] found that the stress obviously increased iron diffusion and enhanced magnetite formation.

In view of the above mentioned facts, it is necessary to investigate the influence of stress on corrosion behavior of T91 steel or other materials. In the present work, experiments on stressed C-rings of the T91 steel in static oxygen saturated LBE at 480 °C were performed to study the stress corrosion cracking (SCC) and interface corrosion behaviors. At room temperature, the initial hoop stresses applied at the center of the C-rings outer surface were 0 MPa, 150 MPa and 300 MPa, respectively, which were much less than the yield strength 415 MPa of T91 steel.

2. Experimental

Commercial T91 steel was used to fabricate the test specimens. Its main chemical compositions are listed in Table 1. The material underwent normalization at 1050 °C for 20 min, followed by air cooling and tempering at 750 °C for 2 h, followed by air cooling.

The stresses were loaded according to the ASME standard G 38–73. The C-ring specimens under different initial hoop stresses were exposed to static liquid LBE to investigate SCC susceptibility. The schematic drawing of the specimen is shown in Fig. 1. The C-ring specimens were machined with an outer diameter of 19 mm and a thickness of 1.5 mm. The outer surface roughness was polished to 0.2 μm. The specimens were cleaned with acetone, and then rinsed with ethanol in an ultrasonic bath. Preparation procedures of the specimens under stress were as follows: The initial tensile hoop stress corresponding to a constant strain was applied at room temperature by changing the outer diameter of a C-ring using a tightening bolt (as shown in Fig. 1). When the outer diameter of the C-ring changed, the hoop stress would be loaded around the C-ring and increased from zero at the position of the nut, to the maximum at the outer surface of the apex of the arch gradually. The change of the outer diameter corresponding to the desired hoop stress (the maximal stress) was calculated with the following equations:

$$D_f = D - \Delta D \tag{1}$$

$$\Delta D = \frac{\sigma \pi d^2}{4EtZ} \tag{2}$$

Where D_f is the outer diameter between the nut and the bolt head of the stressed C-ring (mm), D is the outer diameter of the C-ring before stressing (mm), ΔD is the change of the outer diameter between the nut and the bolt head giving desired stress (mm), σ is the desired stress (MPa), t is the thickness of the C-ring (mm), d is the mean diameter (mm), E is the elasticity modulus (MPa), and Z is 0.943, which is the correction factor for curved beams.

Additionally, in this case, the elasticity modulus E_{T91} at room temperature is about 218 GPa. According to Eqs. (1) and (2), the change of the outer diameter with the desired stress were calculated and listed in Table 2.

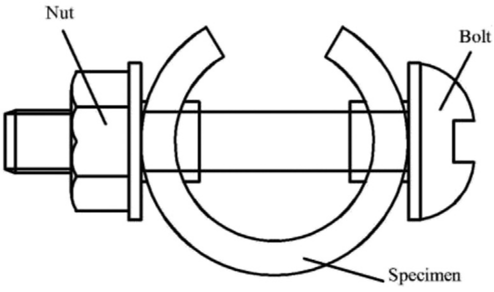

Fig. 1. The shape schematic drawing of the C-ring specimen.

The tests were conducted in a non-oxygen control corrosion device. The detail of the specimen chamber is shown in Fig. 2. Components contacting liquid LBE were made of 316 steel. The C-rings were fixed on the specimen holder with bolts. Before test, the chamber was heated to melt the LBE, and then the specimen holder was installed to insert the C-rings into the liquid metal. At last, the chamber was sealed and heated to 480 °C under Ar. The temperature was detected with a thermocouple during the test. After exposing for 500 h, the first batch of specimens (0 MPa, 150 MPa and 300 MPa) were taken out and replaced with new specimens with the same stresses for subsequent exposure up to 1000 h and 1500 h. Given the short exposing time, the LBE in the chamber was not replaced in-between the exposures.

After the tests, lead oxides were observed on the surface of LBE and the chamber, which indicated the oxygen content in LBE during the test was saturated. According to the relationship of oxygen content vs. temperature as shown in Eq. (3) [11], the oxygen content in the LBE was about 5×10^{-4} *wt%* at 480 °C:

$$\log C_O(wt\%) = 1.2 - \frac{3400}{T} \quad (673\mathrm{K} < T < 973\mathrm{K}) \tag{3}$$

where C_O is the dissolved oxygen concentration in LBE (*wt%*), T is the temperature of LBE (K).

In order to evaluate the cross-section morphology of the maximum stress zone, the C-ring specimens were machined perpendicular to the height of the ring along the center line across the width. Then the cross-sections were polished. The interface morphologies were examined with scanning electron microscope (SEM) and energy dispersive X-ray spectroscopy (EDXS).

3. Results

3.1. Morphological characteristics

No crack was detected on the surfaces of any of the T91 specimen under the present conditions, even for the specimens stressed

Table 2
The outer diameter change with the desired stresses for T91 steel.

Stress/(MPa)	0	150	300
ΔD/(mm)	0	0.12	0.23

Table 1
Chemical compositions of T91 steel (*wt%*).

Element	Cr	Ni	Mn	Mo	C	Si	P	Nb	V	S	Fe
Content	9.25	0.25	0.44	0.85	0.099	0.20	0.015	0.067	0.19	0.0016	Bal.