Influence of High-Density electropulsing treatment on the interface corrosion characteristics of 316L steel in Lead-Bismuth eutectic at 823 K

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Abstract. In order to find an effective method to improve the corrosion resistance properties of the candidate structural materials for lead-alloy cooled fast reactors, the 316L steel was treated by high density single electropulsing. And then the interface corrosion characteristics in stagnant lead-bismuth eutectic (LBE) at 823 K for 1000 h were examined. The results show that electropulsing treatment (EPT) has a significant influence on microcosmic appearance and corrosion depth of the corroded specimens. All the specimens were subjected to dissolution corrosion, but EPT with a current density of 1828 A mm⁻² can improve obviously the corrosion resistance and reduce the extent of local corrosion of 316L in LBE. In addition, the corrosion depth of the 1828 A mm⁻² EPT specimen decreases by 38%, compared with that of the non-EPT specimens.

1 Introduction

Lead-bismuth eutectic (LBE), due to its excellent thermal-physical properties and high neutron economy, is one of the candidate coolants and spallation target materials for application in lead-alloy cooled fast reactors (LFR) and accelerator driven systems (ADS) [1]. However, one of the main issues is the compatibility of the candidate structural materials with LBE, which is one of the lifetime-determining factors for the reactor components, due to the dissolution of steel constituents into LBE at elevated temperatures [2]. In order to inhibit corrosion from LBE and prolong the service life of structural materials, it is necessary to develop some effective methods to improve the corrosion resistance of structural materials.

One promising method to reduce the corrosion rate is the addition of elements (for example Zr, Ti or O) in a proper concentration that inhibit corrosion by forming a protective film [3]. Recently, a special anti-corrosion treatment, called GESA, has been developed. GESA uses a very intense electrons beam to merge the steel at the surface with a protective layer (FeCrAl) applied by the LPPS (Low Pressure Plasma Spraying) technique [4]. In recent years, electropulsing treatment (EPT) has been received much attention due to the special properties it imparts to the treated materials [5, 6]. Previous studies have indicated that an electric current can significantly affect material behaviors such as electromigration, electroplasticity, metal and alloy solidification, amorphous solid structural relaxation, and amorphous crystallization [7-13]. Liu et al. studied effect of EPT on corrosion behavior of nickel base corrosion-resistant alloy [14]. They found that the

intergranular corrosion resistance could be improved substantially without the degradation of tensile properties of the alloy by EPT. Therefore, EPT is attempted to improve the corrosion resistance of the steel matrix in LBE in present work.

The austenitic stainless steel 316L is a widely used engineering material which has the good formability and excellent resistance against corrosion and oxidation [15]. Even so, when exposed to the liquid LBE with dissolved oxygen, the 316L steel is corroded. In present work, the 316L steel was treated with high-density electropulsing to investigate how EPT affects corrosion resistance in stagnant LBE at 823 K for 1000 h.

2 Experimental

2.1 Materials and EPT Condition

The chemical compositions of the investigated steel 316L are given in Table 1. The 316L was annealed in air at 1323 K for 30 min, and then cooled down to room temperature in water. Subsequently, the 316L was machined along the rolling direction to produce a set of thin specimens with dimensions of 70 mm×4.2 mm× 1.2 mm. Before the corrosion test, the specimens were ground using SiC paper up to #600, and then were washed in acetone and ultrasonically cleaned in an acetone bath.

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Table 1. Chemical compositions of the investigated 316L steel (wt. %).

Material	C	Cr	Ni	Mo	Mn	V	Nb	Si	Fe
316L	≤0.03	17.31	10.67	2.47	1.47	–	–	0.65	Bal.

The experimental setup for the EPT and the typical electropulsing waveform are shown in Figure 1 [13]. As shown in Figure 1a, the two ends of each specimen were connected to a discharge circuit through copper electrodes during EPT. The capacitor banks were then discharged to electropulse the specimen in air as follows. A trigger circuit was used to discharge the capacitor banks through the specimen to achieve high-density electropulsing. The waveform of electropulsing was detected to be a damped oscillation wave via a Rogowski coil and a TDS3012 digital storage oscilloscope, as shown in Figure 1b. Although the electropulse waveforms were similar, the maximum current densities (J_m) of EPT were different due to the different load voltage. The pulse duration (about 800 μ s) and the electropulse period ($t_p = 110$ μ s) were almost identical for different waveforms. Each specimen was treated only once by the high-current-density electropulsing.

Figure 1. The schematic diagram of the experimental setup for the EPT and the typical electropulsing waveform: (a) The schematic diagram of the experimental setup and (b) the typical electropulsing waveform.

The EPT conditions used for the corrosion-test specimens are listed in Table 2. After EPT, each of the corrosion-test specimens above-mentioned was cut into several small specimens with dimensions of 10 mm×4.2 mm× 1.2 mm. As shown in Table 2, these small specimens were divided into six groups (L, A, B, C, D and E) depending on the different current densities, and each group consisted of three specimens with identical treatment. The group-L specimens, which were not electropulsed, were used as references.

Table 2. The EPT conditions used for the corrosion-test specimens.

Group	L	A	B	C	D	E
J_m (A/mm ²)	0	1828	2828	3902	4692	5841
U (V)	0	3000	5100	6900	8100	9600
τ_p (μ s)	0	800	800	800	800	800
t_p (μ s)	0	110	110	110	110	110

Note: J_m is the amplitude of current density of electropulsing. U is the charging voltage of the capacitor banks. τ_p is the pulse duration for the electropulsing waveforms. t_p is the electropulse period for the electropulsing waveforms.

2.2 Corrosion Test and Examination of Corrosion Effects

The surfaces of the electropulsed corrosion-test specimens were ground using SiC paper up to #2000 and were then polished to a 1.5- μ m finish. The polished specimens were subsequently washed in acetone and ultrasonically cleaned in an acetone bath. The specimens in the identical group were placed in the identical Mo crucible (Figure 2). LBE was placed in an alumina crucible in a glove box and melted at 473 K under Hi-purity Argon (99.999%) atmosphere. After the LBE was melted, the resulting dross of floating oxides and other impurities were carefully

removed with stainless steel spoon. Each Mo crucible was filled with the melted LBE afterwards. In order to prevent the specimens from floating above the LBE, Molybdenum mesh was used and fixed in the middle of the Mo crucible. After cooling down to room temperature, Mo crucibles were taken out from the glove box and put into a stainless steel container (Figure 2). The stainless steel container was highly evacuated and then filled with

Hi-purity Argon (99.999%) achieving a small pressure to prevent the LBE from excessive oxidation. Then it was heated by an electric strip heater and kept the internal temperature in the container of 823 K for 1000 h. It should be noted, 823K is the maximum temperature in the typical operating temperature range (623K-823K) for the LBE cooled reactor. In the process of LBE operation and corrosion, the LBE was in the Hi-purity Argon protection environment all the time. All the specimens were exposed to the same corrosion test conditions at the same time.

After the corrosion test, the uncleaned corroded specimens were embedded in epoxy resin adhesive and polished to a 1.5- μm finish to examine the cross sections with backscattered electron (BSE) imaging. The chemical compositions of the adherent corrosion product and bulk material were analyzed using energy-dispersive X-ray spectroscopy (EDS).

Figure 2. The schematic diagram of corrosion test setup.

3 Results and Discussion

3.1 Microcosmic Appearance of Corrosion Products

The BSE images of the polished cross-sections of 316L specimen are shown in Figure 3. Based on these images, all specimens show severe corrosion. No oxide scale is found on the surface of each specimen, and a large amount of LBE penetrates into the steel matrix. Meanwhile, a great number of steel matrix particles are also found at the LBE region. Thus, in our experimental environment, the LBE and the steel matrix interpenetrate with each other, and the LBE attacked the steel with dissolving a significant

amount of steel matrix elements. In analyzing the data from the BSE images, the corrosion behavior of Group-L, B-E are very similar, these specimens all present a local corrosion. However, as for Group-A, the corrosion behavior can be characterized by uniform corrosion. These phenomena can be justified by the BSE images in Figure 4 which is displaced in a larger scale. From Figure 4, the interfaces between LBE and the steel matrix of the specimen from Group-A are very flat, indicating that uniform corrosion happens on the Group-A specimens. With regard to the Group-L-1 and the Group-B-1, the interfaces between LBE and the steel matrix are rather roughness, indicating that the steel matrix is subjected to local corrosion. Thus the specimen with EPT at current density of 1828 A mm⁻² has a flat corrosion cross-section morphology when exposed to stagnant LBE.

Figure 3. BSE images of polished cross sections of 316L specimens that were electropulsed at different current densities after the corrosion test at 823 K: (a) $J_m = 0$ for the group-L reference specimen; (b) $J_m = 1828 \text{ A mm}^{-2}$ for the group-A specimen; (c) $J_m = 2828 \text{ A mm}^{-2}$ for the group-B specimen; (d) $J_m = 4692 \text{ A mm}^{-2}$ for the group-D specimen; (f) $J_m = 5841 \text{ A mm}^{-2}$ for the group-E specimen.

Figure.4. The BSE images of polished cross sections of the specimens in a larger scale of BSE.

3.2 Corrosion Depth Measurement

Corrosion depth is an important parameter in evaluating the corrosion resistance of the material exposed to the LBE. Here, the corrosion depth was the Pb and Bi penetration depth measured in BSE images. Based on these depths, the maximum corrosion depth is collected for each specimen tested in stagnant LBE at 823 K for 1000 h. Then the average corrosion depth was calculated as the average of the maximum corrosion depths of three independent specimens in the same group. Figure 5 shows the corrosion depths of the 316L specimen electropulsed at different current densities. The average corrosion depths for Groups-L, A, B, C, D and E are 94 μm , 58 μm , 107 μm , 95 μm , 98 μm and 85 μm , respectively. Clearly, the Group-A specimens have a better corrosion resistance than the others. As can be seen from Figure 4 and Figure 5, EPT is an effective method to influence the interface corrosion characteristics of 316L steel in LBE. This is because EPT is a rapid heating process and the energy provided by such an external source is converted into Joule heating. The resulting transient thermal stress would form a mass of supersaturated point defects, which can increase the vacancy density. Thus, the vacancies could diffuse, which would be beneficial to the diffusion of atoms. Consequently, the diffusion rate at the alloying elements interface would be higher than that in the steel matrix.

Figure.5.EPT conditions used and corrosion depths of the specimens subjected to EPT at different current densities.

3.3 Interactions of Pb, Bi and The Steel Matrix

Figure 6 shows the results of the EDS mapping analysis of the group-A specimen after exposure in LBE for 1000 h. The figure reveals the depletion of Cr and Ni near the steel surface into LBE and the penetration of Pb and Bi into the steel matrix. In addition, the austenite-ferrite phase transition exists in the interaction region between the steel matrix and LBE because Ni preferentially dissolves from the matrix into the LBE. The EDS map analysis indicates that EPT does not affect the dissolution process or elemental redistribution relative to the L specimens that were not electropulsed.

Figure.6.EDS map analysis of the group-A specimen after exposure in LBE for 1000 h.

4 Conclusions

The 316L steel was treated by high density single electropulsing, and the influence of EPT on the interface corrosion characteristics in stagnant LBE at 823 K for 1000 h was investigated. The findings of the present study can be summarized as follows:

- All the 316L specimens were subjected to dissolution corrosion in stagnant LBE at 823 K for 1000 h. However, EPT has a significant influence on microcosmic appearance and corrosion depth of the corroded specimens.
- The average corrosion depth of the 1828 A mm⁻² EPT specimen decreases by 38%, compared with that of the non-EPT specimens. EPT with a current density of 1828 A mm⁻² can improve obviously the corrosion resistance and reduce the extent of local corrosion of 316L in LBE.
- EPT does not affect the dissolution process or elemental redistribution. After EPT, the depletion of Cr and Ni and the penetration of Pb and Bi still occur in the interaction region between the steel matrix and LBE.

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