Influence of LBE Temperatures on the Microstructure and Properties of Crystalline and Amorphous Multiphase Ceramic Coatings

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Abstract: An Al₂O₃–TiO₂ amorphous composite coating with a thickness of 100–120 µm was fabricated on China low activation martensitic steel (CLAM steel) by oxygen acetylene flame spraying technology and the laser in-situ reaction method. We investigated the microstructures and mechanical properties of the coating after liquid lead-bismuth eutectic (LBE) alloy corrosion under different temperatures for 300 h and found that the corrosion temperature of the LBE had no observable effect on the microstructure and chemical phase of the Al₂O₃–TiO₂ amorphous composite coatings. However, the mechanical properties (micro-hardness and shear strength) of the Al₂O₃–TiO₂ multiphase coating deteriorated slightly with the increase in the immersion temperature of the LBE. As a result of oxygen acetylene flame spraying and laser in-situ reaction technology, it was found that the Al₂O₃–TiO₂ amorphous composite coating exhibits an excellent LBE corrosion resistance, which is a candidate structural material for the accelerator-driven subcritical system (ADS) to handle nuclear waste under extreme conditions.

Keywords: multiphase ceramic coatings; temperature of LBE corrosion; LBE corrosion resistance

1. Introduction

The lead-bismuth eutectic (LBE) alloy is the preferred material for reactors and spallation targets of an accelerator-driven subcritical system (ADS) to deal with waste that has a long-term and high-level radioactivity, because of the advantages of high neutron flux density, the strongest irradiation resistance, excellent heat conductivity, and inferior chemical activity [1–4].

China low activation martensitic (CLAM) steel has been considered a candidate for structural materials in future fusion reactor applications because of its excellent swelling resistance [5–7]. However, the mechanical properties of CLAM steel will deteriorate when exposed to the working temperature of 823 K for a long time [8,9]. The creep property of CLAM steel obviously degenerates and the phase also significantly changes after 823 K thermal aging [10,11]. CLAM steel is subjected to severe corrosion by the LBE cooling medium under the working temperature. Hence, the coatings of the CLAM steel substrate surfaces were prepared via a number of modification techniques to improve the surface of the alloys to suit the harsh working conditions of the ADS. In general, coatings fabricated via thermal spraying have the disadvantages of low density, micro-holes, cracks, oxidation inclusion defects, high porosity, and low bond strength, thereby preventing them from widespread application [12,13]. Laser cladding is an effective technology to eliminate defects and enhance the mechanical properties of the coating fabricated via thermal spraying technology [14–16]. The ceramic coating finds multifarious industrial applications because of its excellent mechanical properties and corrosion resistance [17–20].
Here, we apply the laser surface re-melting in-situ reaction technique to fabricate the coatings, aiming to obtain a functional coating layer with a crystalline and amorphous multiphase that can dramatically improve the corrosion resistance and mechanical property of the CLAM steel surfaces. Microstructures and properties of the ceramic coating after immersion in the LBE alloys at different temperatures are also investigated.

2. Experimental

CLAM steel (8.91% Cr, 1.44% W, 0.35% Mn, 0.15% Ta, 0.13% C, and P and Fe surplus) was adopted as the substrate, which was first machined into plates with dimensions of 50 mm × 20 mm × 13 mm. The Cr–Fe, Fe–Al, and Ti powder was mixed in proportion and then went through ball-milling, heating, stirring, crushing, sifting, and other processes, and a CrFeAlTi composite powder with the size of a 150–300 mesh was obtained. We first prefabricated a coating on the CLAM steel surface via oxygen acetylene flame spraying technology, and then adopted the laser in-situ reaction compound technology to remelt the prefabricated coating, where a composite ceramic coating was formed on the surface of the CLAM steel. Details of the coating fabrication are shown in our previous work [15].

The morphology of the samples was observed using a scanning electron microscope (SEM) (S4800, Hitach, Tokyo, Japan) coupled with energy-dispersive X-ray spectroscopy (EDS) (Oxford ISIS, Oxford, UK) to identify the possible phases. Microstructures were characterized by transmission electron microscopy (TEM) (JEOL JEM-2010, Tokyo, Japan). The operation process of SEM and TEM is described in references [21–23]. A new experimental facility (Figure 1) was adopted to measure the bonding strength of the specimen’s interface between the coating and substrate. The Vickers hardness of the specimens was tested under a load of 1.96 N on several regions using a microsclerometer (HXS-1000A, Shanghai, China), where an average value was adopted.

![Figure 1. Schematic diagram of shear test by the self-made fixture.](image)

To examine the corrosion resistance of the coating, the LBE alloy was prepared. The CLAM steel and coated CLAM steel with a size of 5 mm × 5 mm × 13 mm were machined out separately, followed by a final cleaning with acetone before the immersion test. The corrosion tests were carried out by the self-made fixture, as Figure 2 demonstrates. The specimens were immersed into the LBE alloy for 300 h at 725 and 825 K, respectively, and were then taken from the LBE after corrosion and washed with acetone and alcohol under ultrasound. All immersion test processing was under Ar as the protected gas.

As for the wettability test of the heavy metals, the LBE wire was placed on the surface of the samples, and the samples were then slowly heated up until the solid lead melted. The whole process of the experiment was under argon protection.
3. Results and Discussion

Figure 3 shows the microstructure of the coating fabricated by the oxygen acetylene flame spraying technology and laser in-situ reaction method. A coating with a dense structure and uniform thickness was obtained and no significant defects such as cracks or pores were observed, which could result in excellent mechanical and corrosion properties (Figure 3a). TEM and selected-area diffraction pattern (SAED) analyses were employed to gain insights into the structure of the coating. From Figure 3b, we can see that the coating has no pores or cracks. The HR-TEM of Figure 3b reveals that the grain has high crystallinity. Figure 3d shows the SAED pattern of the grain, from which the diffraction spots belonging to Al₂O₃ with an orthorhombic structure are identified. Moreover, diffraction spots with a relatively weak brightness were identified as TiO₂ by further analysis. Figure 3e shows a HRTEM image taken at the upper region of Figure 3b, from which no lattice fringe is found, and the corresponding SAED pattern is identified as amorphous rings (Figure 3f). These suggest that the crystalline and amorphous multiphase ceramic coatings were successfully fabricated by laser in-situ reaction technology.

Figure 4 shows the morphology of the samples without (Figure 4c) and with LBE immersion at different temperatures (Figure 4a,b) for 300 h. The porous structure is formed on the surface of the CLAM steel during immersion in the LBE for 300 h, illustrating that the CLAM steel was subjected to severe corrosion (Figure 4d). Compared to the original coating (Figure 4c), the surface morphology of the coating after LBE immersion has no obvious changes (Figure 4a,b), thereby effectively protecting the CLAM steel.

Figure 5 shows the morphology of a cross-section of CLAM steel specimens corroded by the LBE at 723 and 823 K. The corroded steel appears to be covered with a layer, and as the temperature increases, the layer thickness increases. EDS is used to analyze the element composition of a cross-section of the sample by taking points along the surface in the depth direction, and the corresponding element compositions are shown in Tables 1 and 2. From those two tables, we can infer that the layers covered on the CLAM steel are the oxide layers produced by corrosion; in addition, Pb and Bi elements have been diffused through the oxide layer to the interior of the CLAM steel samples, and the diffusion depth reached 10 µm at 723 K (Table 1) and 15 µm at 823 K (Table 2). It can also be found from the tables that the composition of oxide film can be divided into two layers: The outer layer is rich in Fe, Cr, and Pb (points 1 and 2), and the inner layer is rich in Fe and O (points 3 and 4), illustrating that the double-layer oxide film consisting of an Fe₂O₃ outer layer and (Fe,Cr)₃O₄ inner layer is formed on the steel surface during the corrosion process of the static LBE. However, this loose oxide layer is not enough to resist the penetration of lead-bismuth heavy metals, leading to corrosion of the steel [24]. Diffusion plays a crucial role in the corrosion resistance properties of coatings. The corrosion of steel is caused by the mutual migration of elements in the liquid corrosion medium (Pb and Bi) and in the steel (Fe, Cr, Ni, and so on) [25,26].
Figure 3. Microstructure of the coating: (a) SEM image of cross-section of the sample; (b) TEM image of coating; (c,d) microstructure of crystalline region; (e,f) microstructure of amorphous region.

Figure 4. Morphology of the surface of the samples after LBE corrosion at different temperatures: (a) coating without corrosion, (b) coating corroded by LBE at 723 K for 300 h; (c) coating corroded by LBE at 823 K for 300 h; (d) China low activation martensitic (CLAM) steel corroded by LBE at 823 K for 300 h.

Table 1. Chemical composition of each point in Figure 5

| Sample          | Point | C  | O  | Cr | Fe  | Pb  | Bi  |
|-----------------|-------|----|----|----|-----|-----|-----|
| CLAM steel      | 0     | 13.90 | 2.76 | 4.85 | 56.34 | 8.05 | 14.10 |
|                 | 1     | 15.25 | 1.86 | 9.25 | 64.20 | 4.21 | 5.23 |

Figure 5 shows the morphology of a cross-section of CLAM steel specimens corroded by the LBE at 723 and 823 K. The corroded steel appears to be covered with a layer, and as the temperature increases, the layer thickness increases. EDS is used to analyze the element composition of a cross-section of the sample by taking points along the surface in the depth direction, and the corresponding element compositions are shown in Tables 1 and 2. From those two tables, we can infer that the layers covered on the CLAM steel are the oxide layers produced by corrosion; in addition, Pb and Bi elements have been diffused through the oxide layer to the interior of the CLAM steel samples, and the diffusion depth reached 10 μm at 723 K (Table 1) and 15 μm at 823 K (Table 2). It can also be found from the tables that the composition of oxide film can be divided into two layers: The outer layer is rich in Fe, Cr, and Pb (points 1 and 2), and the inner layer is rich in Fe and O (points 3 and 4), illustrating that the double-layer oxide film consisting of an Fe\textsubscript{3}O\textsubscript{4} outer layer and (Fe,Cr)\textsubscript{3}O\textsubscript{4} inner layer is formed on the steel surface during the corrosion process of the static LBE. However, this loose oxide layer is not enough to resist the penetration of lead-bismuth heavy metals, leading to corrosion of the steel [24]. Diffusion plays a crucial role in the corrosion resistance properties of coatings. The corrosion of steel is caused by the mutual migration of elements in the liquid corrosion medium (Pb and Bi) and in the steel (Fe, Cr, Ni, and so on) [25,26].
Table 1. Chemical composition of each point in Figure 5a.

| Sample                     | Point | C    | O    | Cr   | Fe    | Pb   | Bi   |
|----------------------------|-------|------|------|------|-------|------|------|
| CLAM steel after LBE       | 0     | 13.90| 2.76 | 4.85 | 56.34 | 8.05 | 14.10|
| corrosion at 723 K         | 1     | 15.25| 1.86 | 9.25 | 64.20 | 4.21 | 5.23 |
|                            | 2     | 14.84| 2.00 | 7.45 | 70.20 | 2.47 | 3.03 |
|                            | 3     | 17.00| 1.97 | 6.90 | 73.19 | 0.94 | 0    |
|                            | 4     | 15.69| 1.76 | 8.22 | 74.21 | 0.12 | 0    |
|                            | 5     | 16.32| 1.34 | 9.12 | 73.22 | 0    | 0    |

Table 2. Chemical composition of each point in Figure 5b.

| Sample                     | Point | C    | O    | Cr   | Fe    | Pb   | Bi   |
|----------------------------|-------|------|------|------|-------|------|------|
| CLAM steel after LBE       | 1     | 8.84 | 2.41 | 2.96 | 77.33 | 3.77 | 4.69 |
| corrosion at 823 K         | 2     | 8.60 | 2.28 | 3.49 | 81.29 | 2.65 | 1.69 |
|                            | 3     | 9.24 | 7.73 | 0.17 | 80.98 | 1.85 | 0.03 |
|                            | 4     | 9.47 | 8.16 | 0.57 | 81.39 | 0.41 | 0    |
|                            | 5     | 9.12 | 0    | 7.81 | 82.79 | 0.28 | 0    |
|                            | 6     | 14.64| 0    | 9.60 | 75.43 | 0    | 0    |
|                            | 7     | 15.25| 0    | 9.25 | 73.38 | 0    | 0    |

The diffusion coefficient D can be expressed in terms of the Arrhenius formula as follows:

$$D = D_0 \exp(-Q/RT)$$  \hspace{1cm} (1)

where $D_0$ is a frequency factor, Q is the diffusion activation energy (vacancy diffusion mechanism) or atomic transition activation energy (interstitial diffusion mechanism), $R$ is the gas constant, and $T$ is the temperature. According to the Arrhenius formula, as the temperature increases, the migration of elements such as Fe, Cr, and Ni to the LBE and the migration of elements Pb and Bi to steel accelerate [27,28].
Through EDS analysis of the LBE attached to the CLAM steel surface (point 0 in Figure 5a), it is found that a small amount of Fe, Cr, and other elements are dissolved in the LBE. There is no obvious change in the section morphology of the samples soaked in the LBE (Figure 6). Further EDS analysis on the crystalline and amorphous multiphase ceramic coating after immersion in the LBE alloy at 823 K revealed that the signal of Pb and Bi could not be detected, thereby confirming that the coating shows excellent corrosion resistance properties even at 823 K (Figure 7a). From Figure 7b, we can see that the Pb and Bi signals could not be detected near the surface of the coating, suggesting that the LBE alloy could not be diffused to the coating even at high temperature. The coating fabricated by the laser with a dense structure acts as the diffusion barrier to prevent the diffusion of heavy metals, so it has outstanding LBE corrosion resistance.

**Figure 6.** Morphology of cross-section of the coating without or after LBE corrosion at different temperatures for 300 h: (a) no corrosion; (b) 723 K; (c) 823 K.

**Figure 7.** Energy-dispersive X-ray spectroscopy (EDS) analysis of the coating after LBE corrosion at 823 K: (a) distribution by line scanning, (b) EDS spectrum of the point in (a).

In addition, wettability is another key factor that may affect the mechanism of the corrosion resistance of coatings in the LBE under high temperature. Figures 8 and 9 show the melting process of the LBE on the surface of the CLAM steel and multiphase ceramic coatings. When the LBE melts on the surface of the CLAM steel, it spreads around quickly, paving the surface of the sample (Figure 8), and the wetting angle is ~18° (Figure 10a). Meanwhile, when the LBE melts on the surface of the coating, spherical droplets are formed because of surface tension (Figure 9), and the wetting angle is ~112° (Figure 10b). This is because the coated samples have been remelted by the laser to form a glass ceramic layer with a dense structure, which also has a certain hydrophobic property on liquid heavy metals and can effectively prevent the material from being corroded by liquid media.
When the LBE melts, the multiphase ceramic coatings are stable while the specimens are immersed in the LBE under different temperatures. Hence, the microstructure and phases of the multiphase ceramic coatings are not affected by the LBE temperatures. Hence, it is indicated that the LBE temperatures have no obvious effect on the microstructure and phases of the multiphase ceramic coatings.

The micro-hardness of the specimens after LBE corrosion at different temperatures for 300 h is shown in Figure 12, where one can see that they behave distinctly. The hardness of the coating without immersion in the LBE is 1852.4 HV$_{0.2}$, which is far higher than that of the substrate (230–277 HV$_{0.2}$). The hardness of the specimens after immersion in the LBE at 723 and 823 K for 300 h has minimum values of 1813 HV$_{0.2}$ and 1594.8 HV$_{0.2}$ separately.

To directly determine the chemical composition of the coating, Figure 11 presents the X-ray diffraction spectrum, where no new secondary phases predominantly detected in the layer are labelled. However, the intensity of some peaks is different, and it is noted that the phases of the coating is stable while the specimens are immersed in the LBE under different temperatures. Hence, it is indicated that the LBE temperatures have no obvious effect on the microstructure and phases of the multiphase ceramic coatings.

![Figure 8](image1.png)  
**Figure 8.** Melting process of LBE at different heating time points on the surface of CLAM steel.

![Figure 9](image2.png)  
**Figure 9.** Melting process of LBE at different heating time points on the surface of coating.

![Figure 10](image3.png)  
**Figure 10.** Infiltration of LBE sample: (a) CLAM steel; (b) coating.
labelled. However, the intensity of some peaks is different, and it is noted that the phases of the coating is stable while the specimens are immersed in the LBE under different temperatures. Hence, it is indicated that the LBE temperatures have no obvious effect on the microstructure and phases of the multiphase ceramic coatings.

Figure 11. XRD spectrum of the samples without and after LBE corrosion: (a) without corrosion; (b) 723 K; (c) 823 K.

The micro-hardness of the specimens after LBE corrosion at different temperatures for 300 h is shown in Figure 12, where one can see that they behave distinctly. The hardness of the coating without immersion in the LBE is 1852.4 HV0.2, which is far higher than that of the substrate (230–277 HV0.2). The hardness of the specimens after immersion in the LBE at 723 and 823 K for 300 h has minimum values of 1813 HV0.2 and 1594.8 HV0.2 separately.

Figure 12. Micro-hardness of the samples without and after immersion in LBE at different temperatures for 300 h.

Figure 13 gives the mechanical properties of the specimens both after LBE corrosion at different temperatures. The shear strength of the sample without LBE corrosion is determined to be in between 280 and 290 MPa, whereas it decreases by 0.35% after corrosion at 723 K and by 0.61% after corrosion at 823 K. The coatings on the CLAM steel surface can inhibit LBE corrosion and present a high shear strength, due to the greater number of generated compounds (e.g., Al2O3, TiO2, FeCr, and so on) formed in the metallurgical reaction during the laser in-situ reaction process. The corrosion of the LBE has no effect on the shear strength of the coating.
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Figure 13. Shear strength of the samples.

From Figure 14a, one can see that there are two typical fracture morphologies of the tear samples. The fracture of the upper part of the coating is brittle fracture; moreover, ductile fractures are also detected near the substrate. The EDS analysis of the cleavage fracture 002 region shows that the contents of Ti and Al are high, confirming that the coating can be embrittled by increasing the content of the ceramic phase (Table 3). In addition, the Fe content is higher in the 001 and 003 regions where dimples appear, indicating that the increase in the iron content of the substrate can increase the toughness of the coating.

Figure 14. Morphology of samples: (a) shear fracture of coatings; (b) points for EDS analysis.

Table 3. Chemical composition of each point in Figure 14.

| Point | C   | O   | Al  | Cr  | Fe  | V  |
|-------|-----|-----|-----|-----|-----|----|
| 1     | 3.24| 3.00| 43.60| -   | 4.73| 1.59|
| 2     | -   | 7.09| 51.72| 1.87| 1.65|-   |
| 3     | 3.47| 6.28| 36.68| 2.95| 15.12| -  |

4. Conclusions

The crystalline and amorphous multiphase ceramic coatings on the surface of CLAM steel were fabricated by oxygen acetylene flame spraying and laser in-situ reaction technology, aiming to protect the substrate from LBE corrosion. The LBE corrosion temperature was found to exert no observable effect on the microstructure and phase of the coatings. The micro-hardness of the specimens after immersion in LBE at 723 and 823 K for 300 h had minimum values of 1813 HV₀.₂ and 1594.8 HV₀.₂.
respectively. Moreover, the shear strength of the $\text{Al}_2\text{O}_3$–$\text{TiO}_2$ multiphase coatings decreased slightly as the temperature of the LBE increased, and the shear strength of the coatings decreased by 0.35% and 0.61% after corrosion at 723 and 823 K, respectively. Such crystalline and amorphous multiphase ceramic coatings exhibit very strong LBE corrosion resistance, which can be used as candidate structural materials for accelerator-driven subcritical systems.

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