Synergistic effect of in-situ proton irradiation and lead-bismuth eutectic corrosion on amorphous FeCrAlTiMo coatings

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Abstract

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Full Text

Preamble

In-situ proton irradiation/lead-bismuth eutectic corrosion synergistic effect on amorphous FeCrAlTiMo coatings

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Abstract

The synergistic effect of simultaneous proton irradiation and lead-bismuth eutectic (LBE) corrosion on amorphous FeCrAlTiMo coatings was investigated. Results demonstrated that proton irradiation had a positive effect on the LBE corrosion resistance of the coating. Both experimental and simulation results revealed that irradiation-induced annihilation of free volume reduced diffusion kinetics and led to a slower corrosion rate at 600 °C, challenging the traditional wisdom that irradiation damage negatively affects materials corrosion. Finally, a novel mechanism of irradiation-decelerated corrosion was proposed, providing a new approach for designing nuclear structural materials with enhanced corrosion and irradiation resistance.

Keywords: Synergistic effect; Proton irradiation; Lead-bismuth eutectic; Decelerated corrosion

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1. Introduction

Generation IV advanced nuclear energy systems impose extremely challenging operating environments characterized by higher temperatures, more intense irradiation, and more corrosive coolants [1]. Consequently, the compatibility between in-reactor structural materials and corrosive coolants has become one of the key bottlenecks restricting their development [2]. In recent years, coolant corrosion and irradiation-assisted corrosion have remained focal concerns for structural materials in advanced nuclear reactors [3, 4]. To accurately evaluate the service life of in-reactor components, a thorough understanding of the synergistic effects of simultaneous coolant corrosion and irradiation is critical.

To date, numerous post-irradiation corrosion and in-situ irradiation corrosion studies have been conducted to elucidate irradiation effects on corrosion in various coolants, including liquid lead-bismuth eutectic (LBE) [5], molten salts [6], water-based environments [7], supercritical CO_2 [8], and others. A growing consensus has emerged that irradiation accelerates the corrosion rate of structural materials in current reactors. This irradiation-accelerated corrosion mechanism can generally be attributed to two main aspects. First, displacement damage effects introduce considerable numbers of vacancies and interstitials, which evolve into dislocation lines, loops, walls, voids, bubbles, and other defects through migration and aggregation. These defects provide fast pathways for elemental movement, increasing diffusion rates and thus accelerating overall corrosion [9]. Second, irradiation-enhanced diffusion can induce spatial changes in chemical composition distribution, manifesting as phase transformations, precipitation, and segregation, which particularly promote localized corrosion [10].

However, some experiments have also confirmed the existence of irradiation-decelerated corrosion, such as proton-irradiated Ni-Cr alloys in high-temperature molten salts [11] and Au^{2+}-irradiated FeCrAlTiMo coatings in high-temperature liquid LBE [12], indicating that multiple competitive mechanisms introduced by irradiation co-affect corrosion. Nevertheless, explanations for this potential irradiation-decelerated corrosion mechanism have remained elusive. Clearly, from the perspective of long-term safe service of in-reactor structural materials, irradiation-accelerated corrosion is undesirable. If an irradiation-decelerated corrosion mechanism can be revealed and adapted material systems can be designed and developed, it would greatly advance the development of advanced nuclear reactors.

Herein, based on the irradiation-accelerated corrosion mechanisms mentioned above, an amorphous FeCrAlTiMo high-entropy alloy (HEA) coating was specially designed and prepared. Subsequently, the synergistic effects of simultaneous proton irradiation and LBE corrosion were investigated. The results show that proton irradiation can decelerate corrosion of the FeCrAlTiMo coating in LBE, contradicting the commonly reported irradiation-accelerated corrosion. This deceleration was determined and quantified through cross-sectional TEM analysis of the corrosion-induced oxide film thickness. Combining experimental and molecular dynamics (MD) simulation results, we propose an irradiation-decelerated corrosion mechanism resulting from irradiation-induced annihilation of free volume in amorphous alloys, which weakens elemental diffusion. Moreover, this work provides a new strategy for designing innovative structural materials for future advanced nuclear reactors.

2.1 Corrosion/irradiation sample fabrication

The pristine FeCrAlTiMo coating microstructure was identified by TEM before corrosion/irradiation tests, as shown in Fig. 1 [Figure 1: see original paper]. Both bright-field and dark-field TEM images were featureless, with no significant local contrast observed (Fig. 1b-c). EDS mapping results showed all coating components were uniformly distributed without distinguishable element segregation or aggregation (Fig. 1d). Moreover, selected-area electron diffraction (SAED) patterns at different coating depths presented similar diffraction halos corresponding to amorphous characteristics (Fig. 1e-g). These results collectively indicated that the pristine FeCrAlTiMo coating possessed a completely amorphous phase structure.

Magnetron sputtering was employed for fabrication. A 12Cr2W1Mn ferritic/martensitic (F/M) steel, designed specifically for lead-cooled fast reactors (LFR), was selected as the substrate with a final machined thickness of approximately 80 m and dimensions of 10\$$\times 10mm^{\{2\}.AFeCrAlTiMoalloytarget(99.99wt.\times 10}${-6}\$ mbar. During deposition, the Ar working pressure was 5\$×10^{-4}\$ mbar, the working temperature was 350 °C, the sputtering power density was approximately 6.6 W/cm^2, the substrate bias voltage was -100 V, the target-substrate distance was 15 cm, and the deposition rate was 20 nm/min. The coating’ s nominal composition was determined by EDS as Fe-22.2 at%, Cr-21.6 at%, Al-20.2 at%, Ti-16.1 at%, and Mo-19.9 at%, with a coating thickness of approximately 2.0 m. This thickness was sufficiently thin to allow most energetic protons to penetrate the entire substrate/coating sample foil (~82 m), demonstrating that the corrosion/irradiation experiment was feasible and could be successfully achieved, as shown in Fig. 2 [Figure 2: see original paper] and Fig. 3 [Figure 3: see original paper].

2.2 Corrosion/irradiation experiments

Simultaneous proton irradiation and LBE corrosion experiments were conducted using the 3 MV tandem accelerator (HVEE, High Voltage Engineering Europa) at the Institute of Nuclear Science and Technology, Sichuan University, Chengdu, China. Facility details can also be found in our previous research [13]. The substrate/coating sample foil was sandwiched between two graphite washers, with the coated side directly exposed to liquid LBE and the proton beam injected from the opposite side. As long as the protons could pass through the entire sample foil, the coating was irradiated and corroded simultaneously.

Notably, the window allowing the proton beam to reach the sample foil was 3 mm in diameter, while the hollow inner diameter of the graphite washer was 5 mm. Consequently, two distinct areas were obtained on the coated side: one "corrosion only" area and another "corrosion plus irradiation" area. The synergistic effect of irradiation and corrosion was elucidated by comparing coating behavior between these two areas. A schematic of this corrosion/irradiation experiment facility is shown in Fig. 3a [Figure 3: see original paper].

After the accelerator vacuum chamber pressure reached 1$\times10^{-7}$ mbar, the heater was activated to raise the corrosion cell temperature to the target value (600 °C) over approximately 1 hour. The proton beam with particle energy of 5.5 MeV was then introduced, and the experiment officially began. During irradiation/corrosion, the proton beam flux was 5.6$\times10^{12}$ ions/(cm$^2\cdot$s) and the average beam current was 3.48 A/cm^2. The total irradiation/corrosion time was 12 hours, yielding a total proton fluence of 2.0$\times10^{17}$ ions/cm^2. The oxygen concentration in LBE was 3.6$\times10^{-3}$ wt.%. Once the target duration and proton fluence were achieved, the proton beam was cut off and the heater was stopped, concluding the experiment.

2.3 Sample characterization

Coating microstructure and formed oxide film were characterized using a field-emission scanning electron microscope (FESEM, INSPECT F50, FEI) and a field-emission transmission electron microscope (TEM, FEI Talos F200s Super-X) equipped with an energy dispersive spectrometer (EDS). TEM thin foils were prepared by dual-beam focused ion beam (FIB, Thermo Scientific Helios 5 CX) lift-out from both “corrosion only” and “corrosion plus irradiation” areas. Before FIB milling, a platinum (Pt) layer approximately 0.5 m thick was deposited to protect the surface from subsequent Ga ion irradiation damage. These TEM foils were thinned gradually using 30 kV, 16 kV, 8 kV, 5 kV, and 2 kV Ga ions to thicknesses of ~1000 nm, ~500 nm, ~200 nm, ~100 nm, and ~50 nm, respectively. The final milling voltage and current were 2 kV and 20 pA, respectively, sufficiently low to minimize FIB damage. After the irradiation/corrosion experiment, the sample foil was immersed in a mixed solution of H_2O_2 (hydrogen peroxide), CH_3COOH (acetic acid), and CH_3CH_2OH (ethanol) with a volume ratio of 1:1:1 at room temperature to remove residual LBE from the surface.

2.4 Molecular dynamics (MD) simulations

MD simulations were performed using the Large-scale Atomic/Molecular Massively Parallel Simulator (LAMMPS) software package [14] to calculate free volume evolution and diffusion coefficient variations of coating components before and after irradiation. First, a simulation box labeled FCATM (110) containing 75,600 atoms with a BCC crystal structure was constructed, in which Fe, Cr, Al, Ti, and Mo atoms were randomly distributed to occupy lattice sites, as shown in Fig. 8j [Figure 8: see original paper]. The simulation employed an NPT ensemble starting from the crystalline FCATM (110) structure at 873.5 K (600 °C). The system was heated to 5500 K over 400 ps, then isothermally equilibrated in an NVE ensemble for 400 ps, and finally cooled from 5500 K back to 873.5 K within 400 ps and isothermally equilibrated for another 400 ps. The completely amorphous FeCrAlTiMo coating, labeled FCATM’ (110), was obtained through structural analysis using OVITO software (Fig. 8k) [Figure 8: see original paper].

Diffusion rates of each element before and after 5.5 MeV proton irradiation at a dose of 2.0\$×10^{17}\$ ions/cm^2 on the FCATM’ (110) surface were also calculated. Interatomic interactions between Fe, Cr, Al, Ti, and Mo atoms were described by embedded-atom method (EAM) potentials [15], which were validated by comparing defect formation energies, equilibrium volumes, elastic moduli, and heats of formation for several binary compounds with ab initio simulations and experiments. Due to the high proton energy, interactions between protons and other atoms were described using the Ziegler-Biersack-Littmark (ZBL) potential [16] during irradiation simulations. This ZBL potential includes an additional switching function that connects the ZBL and EAM potentials and smoothly reduces curvature to zero between inner (0.7 Å) and outer (1.5 Å) cutoffs.

In diffusion simulations, periodic boundary conditions were applied in three directions. For cascade simulations, periodic boundary conditions were applied in x and y directions, while the z direction used fixed boundary conditions.

The simulated system' s stress relaxation geometry was first optimized using the conjugate gradient descent method. The Number-Pressure-Temperature (NPT) ensemble (at standard atmospheric pressure) at 600 °C was then employed within the simulation box. The system was simulated for approximately 400 ps, sufficient to ensure temperature distribution stability. Finally, mean square displacements (MSDs) of all atoms were recorded during the subsequent 600 ps simulation. The timestep was set to 1 fs in all simulations. Cascade simulation was conducted following NPT equilibration for 400 ps, using the Number-Volume-Temperature (NVT) ensemble at 600 °C with a timestep of 0.001 fs. All atomic configurations were visualized using the Open Visualization Tool (OVITO) software package [17]. Volume analysis during simulation was completed using the Voro++ program [18]. Diffusion rates of all atoms and each atom type were calculated by fitting MSD versus time data using a linear function and least-squares method with reference to Einstein' s equation (1) [19]:

$$D = \lim_{t\to\infty} \frac{\langle \Delta r^2(t) \rangle}{6t}$$

where $\langle \Delta r^2(t) \rangle$ is the mean square displacement as a function of time t. The calculation method for $\langle \Delta r^2(t) \rangle$ is as follows:

$$\langle \Delta r^2(t) \rangle = \langle [r(t) - r(0)]^2 \rangle$$

3.1 Evidence of irradiation-decelerated corrosion in FeCrAlTiMo coatings

The thin sample foil (substrate + coating) with a total thickness of approximately 82 m (Fig. 2) [Figure 2: see original paper] was simultaneously exposed to 600 °C liquid LBE (Fig. 3d) [Figure 3: see original paper] and proton beams in a previously constructed corrosion/irradiation experiment facility [13]. Fig. 3a [Figure 3: see original paper] presents the core diagram of this facility, where two contrasting areas were flexibly obtained: a central area subjected to both liquid LBE corrosion and proton irradiation, and an outer area exposed only to liquid LBE (Fig. 3c) [Figure 3: see original paper]. According to SRIM simulation (Fig. 3b) [Figure 3: see original paper], the sample foil thickness (82 m) was far from the Bragg peak position (98 m) where most protons would be deposited, indicating that proton irradiation damage was introduced into the coating without significant hydrogen implantation. Therefore, the effect of hydrogen injection on coating corrosion behavior was negligible.

Scanning electron microscope (SEM) images at various resolutions revealed remarkable differences in LBE corrosion behavior between irradiated and unirradiated areas (Fig. 3e-j) [Figure 3: see original paper]. The unirradiated area was covered by continuous, dense oxide particles (Fig. 3g) [Figure 3: see original paper], while the average size and number density of oxide particles in the irradiated area were significantly reduced (Fig. 3j) [Figure 3: see original paper], implying irradiation-decelerated corrosion. Since proton irradiation was the only difference between the two areas, it was clearly responsible for slowing the LBE corrosion rate of the coating.

3.2 Quantification of decelerated corrosion through oxide film analysis

To further confirm irradiation-decelerated corrosion, Fig. 4 [Figure 4: see original paper] presents cross-sectional TEM analysis of oxide films formed in irradiated and unirradiated areas for quantitative comparison of LBE corrosion behavior. High-angle annular dark-field (HAADF) images (Fig. 4a-d and f-i) [Figure 4: see original paper] showed that both irradiated and unirradiated coating surfaces were covered by thin, continuous oxide films characterized by a double-layer structure: an outer oxide layer (OOL) with gray contrast and an inner oxide layer (IOL) with dark contrast. EDS mapping results (Fig. 4e) [Figure 4: see original paper] revealed that the oxide film in unirradiated areas showed Cr and O enrichment in the OOL, while Al, Ti, and O were enriched in the IOL, eventually identified as hexagonal Cr_2O_3 OOL and mixed amorphous Al_2O_3/rutile TiO_2 IOL, as shown in Fig. 5 [Figure 5: see original paper]. In contrast, the irradiated area showed Cr and Ti signals in the OOL, identifying hexagonal Cr_2O_3 and rutile TiO_2, and Al signals in the IOL, identifying amorphous Al_2O_3 (Fig. 5) [Figure 5: see original paper]. These results indicated that irradiation promoted separation of Al_2O_3 and TiO_2.

A transitional area between the oxide film and alloy coating, characterized by depletion of oxide-forming elements (Al, Ti, and Cr), formed after corrosion and is commonly called the transitional layer (TL). Al depletion was more severe than that of Cr and Ti in both irradiated and unirradiated areas (Fig. 4e and j) [Figure 4: see original paper]. Additionally, Al, Ti, and Cr depletion in the irradiated area was significantly weaker than in the unirradiated area, also implying that irradiation slowed corrosion.

To quantitatively compare corrosion behavior between irradiated and unirradiated areas, oxide layer and transitional layer thicknesses were summarized from twenty independent HRTEM and HAADF images, as shown in Fig. 6 [Figure 6: see original paper]. Comparing minimum, maximum, and average thicknesses of the outer oxide layer, inner oxide layer, total oxide layer, and transitional layer under identical corrosion conditions (time and temperature), corrosion was distinctly weaker in the irradiated area. As shown in Fig. 6c [Figure 6: see original paper], based on total oxide layer thickness, the unirradiated area corroded approximately 1.4–1.5 times more than the irradiated area. In summary, our experimental results confirmed that in-situ proton irradiation decelerated the LBE corrosion rate of the FeCrAlTiMo coating.

4.1 Mechanism analysis of proton-decelerated corrosion

Under saturated oxygen conditions, LBE corrosion on the coating manifested as oxidation (Fig. 4) [Figure 4: see original paper]. Irradiation had little effect on the double-layer structure and phase composition (Cr_2O_3, TiO_2, and Al_2O_3) of the oxide film formed on the exposed coating (Fig. 5 and Fig. 6) [Figure 5: see original paper][Figure 6: see original paper], meaning that irradiation retarded corrosion (Fig. 6) [Figure 6: see original paper] primarily by affecting corrosion kinetics rather than thermodynamics. This suggests a mechanism by which irradiation reduced coating element diffusion kinetics.

However, irradiation effects on corrosion typically appear to be accelerated [4, 20-22], attributed to irradiation-induced structural defects (dislocation loops, voids, bubbles, etc.) and spatial redistribution of chemical components (precipitation and segregation). Energetic incident particles displace target atoms in cascades, inevitably creating vacancies, interstitials, and other irradiation defects that provide fast pathways for atomic movement and increase elemental diffusion rates, thus accelerating corrosion [9]. As shown in Fig. 7 [Figure 7: see original paper], the FeCrAlTiMo coating maintained its amorphous structure after exposure to the high-temperature/LBE/irradiation multi-field environment, with no discernible precipitation or segregation detected, indicating good structural stability. Amorphous metallic glasses differ from traditional crystalline materials in their unique structural feature of long-range disorder and short-range order, making them free of crystalline defects such as dislocation lines, loops, and walls [23]. Owing to this stable amorphous nature and absence of crystalline defects, the contribution of typical irradiation-induced defects to enhanced corrosion was greatly weakened in this study.

Building on previous work, metallic glasses are generally perceived to consist of tightly bonded atomic clusters that overlap and form a percolating "skeleton" of the glassy structure, with loosely bound free-volume regions embedded within it [24]. Analogous to crystalline materials, these free-volume regions are treated as typical structural defects inside metallic glasses, and it has been widely demonstrated that increased free volume fraction leads to significantly increased diffusion coefficients [25, 26]. In fact, subtle differences could still be observed in SAED patterns of pristine, irradiated, and unirradiated coatings (Fig. 8b, e, and h) [Figure 8: see original paper]. The diffraction halo diameters for pristine, unirradiated, and irradiated coatings were 9.007 1/nm, 9.031 1/nm, and 9.097 1/nm, respectively (Fig. 8c, f, and i) [Figure 8: see original paper], indicating that average interatomic distances became smaller and atomic arrangements became tighter. In other words, equivalent high-temperature annealing and irradiation promoted free volume annihilation, thus reducing free volume fraction, as reported in other studies [27, 28].

Since unirradiated and irradiated coatings underwent identical thermal aging, thermal effects are not discussed here and only irradiation effects are considered. To address potential inaccuracies in experimental measurements, the coating' s volumetric response to proton irradiation and accompanying variations in elemental diffusion coefficients were further calculated using molecular dynamics simulation. Fig. 8l and m [Figure 8: see original paper] show the evolution of average volume per atom (V) over time for unirradiated and irradiated coatings, respectively, over 600 ps. Averaging V over recorded time yielded V values of 30.136 Å^3/atom for irradiated coating and 30.763 Å^3/atom for unirradiated coating, indicating reduced free volume after irradiation, consistent with SAED pattern results (Fig. 8c, f, and i) [Figure 8: see original paper].

Fig. 8n [Figure 8: see original paper] shows mean square displacement (MSD) changes for each atom in irradiated (dotted line) and unirradiated (solid line) coatings over recorded time. The slope of the MSD versus time curve for irradiated coating was significantly lower than for unirradiated coating, implying a smaller diffusion rate. According to Einstein' s equation [19], calculated diffusion coefficients for unirradiated and irradiated coatings are plotted in Fig. 8o [Figure 8: see original paper]. Diffusion coefficients for both total and individual atoms decreased after irradiation. Lower diffusion coefficients mean slower diffusion kinetics, which reasonably explains the decelerated LBE corrosion rate in irradiated coatings—i.e., thinner oxide and transitional layers (Fig. 3) [Figure 3: see original paper].

Based on this analysis, a mechanism considering irradiation-reduced diffusion is proposed to explain how irradiation enhanced corrosion resistance. In this study, the FeCrAlTiMo coating retained its amorphous structure during simultaneous irradiation and corrosion. Interstitial-like and vacancy-like point defects generated by irradiation damage cascades were transient, unstable, and completely annihilated, unable to evolve into large-scale structural defects such as dislocations [29]. Conversely, irradiation-induced thermal spikes and viscous flow resulted in annihilation and redistribution of free volume [27, 30], reducing coating diffusion kinetics and leading to decelerated corrosion. This challenges the traditional wisdom that irradiation accelerates corrosion. This abnormal irradiation-decelerated corrosion mechanism is hypothesized to be generally effective in amorphous alloys, independent of corrosive medium type. This effect can be exploited to design new amorphous nuclear structural materials with high phase structural stability to achieve better synergistic corrosion and irradiation resistance, which has important implications for structural material design and the rapid development of future advanced nuclear energy systems.

Conclusions

The synergistic effect of simultaneous proton irradiation and LBE corrosion on FeCrAlTiMo coating was investigated. Surface morphology, elemental composition, microstructure, and crystal structure of the oxide scale were systematically analyzed. The following conclusions can be drawn:

1. The composition and crystal structure of the oxide scale formed on the FeCrAlTiMo coating after exposure to 600 °C molten LBE were nearly unaffected by proton irradiation. The oxide scale in both irradiated and unirradiated areas consisted of a Cr_2O_3 outer layer and a mixed TiO_2/Al_2O_3 inner layer. Notably, irradiation promoted separation of Al_2O_3 and TiO_2.
2. Proton irradiation decelerated LBE corrosion of the FeCrAlTiMo coating. The thicknesses of the inner and total oxide layers decreased significantly, while the outer layer increased slightly after irradiation.
3. Proton irradiation-decelerated corrosion can be attributed to irradiation-induced annihilation and redistribution of free volume, which reduced coating element diffusion kinetics and led to slower corrosion.

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Data availability

All data generated or analyzed during this study are included in the published article and its supplementary information files.

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