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

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Date: 2025-02-13T00:00:00+00:00

Abstract

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This study investigates the synergistic effect of proton irradiation and lead-bismuth eutectic (LBE) corrosion on amorphous FeCrAlTiMo coatings. The results demonstrate that proton irradiation exerts a positive influence on the LBE corrosion resistance of the coating. Experimental and simulation results confirm that irradiation-induced free volume annihilation reduces diffusion kinetics, leading to a decreased corrosion rate at 600°C, which challenges the conventional perception that irradiation damage negatively impacts material corrosion. Finally, a novel mechanism of irradiation-decelerated corrosion is proposed, offering new insights for designing nuclear structural materials with superior corrosion resistance and irradiation tolerance.

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 showed that irradiation-induced annihilation of free volume reduced diffusion kinetics and led to a slower corrosion rate at 600 °C, which challenges the traditional wisdom that irradiation damage negatively affects materials corrosion. Finally, a novel mechanism of irradiation-decelerated corrosion is 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

Introduction

Generation IV advanced nuclear energy systems impose extremely challenging operating environments, characterized by higher temperatures, stronger irradiation fields, 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 material 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 the effects of irradiation 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. Gradually, a consensus has emerged that irradiation accelerates the corrosion rate of structural materials in today' s reactors. The mechanism of irradiation-accelerated corrosion can generally be attributed to two main aspects. First, it is well established that displacement damage effects caused by irradiation introduce a considerable number of vacancies and interstitials, which further evolve into dislocation lines, loops, walls, voids, bubbles, and other defects through migration and aggregation. These defects can provide fast pathways for elemental movement in materials, increasing diffusion rates and thus accelerating overall corrosion [9]. Second, irradiation-enhanced diffusion can induce spatial changes in the distribution of chemical composition within materials, manifesting as phase transformations, precipitation, segregation, etc., which particularly promotes 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. Obviously, from the perspective of long-term safe service of in-reactor structural materials, irradiation-accelerated corrosion is undesirable. If one of the irradiation-decelerated corrosion mechanisms can be revealed, and material systems adapted to these mechanisms can be designed and developed, it will greatly promote 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. The deceleration was determined and quantified by cross-sectional TEM analysis, which measured the thickness of the corrosion-induced oxide film. Combining experimental and simulation (molecular dynamics, MD) results, we propose an irradiation-decelerated corrosion mechanism resulting from irradiation-induced annihilation of free volume in amorphous alloys, which weakens elemental diffusion. Moreover, our work provides a new strategy for designing innovative structural materials for future advanced nuclear reactors.

2.1 Corrosion/irradiation sample fabrication

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

Magnetron sputtering was used as the fabrication method. A 12Cr2W1Mn ferritic/martensitic (F/M) steel, specifically designed for lead-cooled fast reactors (LFR), was selected as the substrate. The substrate was machined to a final thickness of approximately 80 m and dimensions of $10\$\times10mm^{\{2\}}.AFeCrAlTiMoalloytarget(99.99wt.\times10\{-6\}\$$ 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 nominal composition of the coating 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%. The coating thickness was approximately 2.0 m, which was thin enough to allow most energetic protons to penetrate the entire substrate/coating sample foil (~82 m), proving 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

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

Notably, the window that allowed the proton beam to irradiate 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 was "corrosion only" and the other was "corrosion plus irradiation." The synergistic effect of irradiation and corrosion was elucidated by comparing the corrosion behavior of the coating in these two areas. A schematic of this corrosion/irradiation experiment facility is shown in Fig. 3a [Figure 3: see original paper].

After the pressure of the accelerator vacuum chamber reached 1$×10^{-7}$ mbar, the heater was started to raise the corrosion cell temperature to the target temperature (600 °C) over a period of approximately 1 hour. Then, the proton beam with a particle energy of 5.5 MeV was introduced to the sample, and the experiment officially began. During irradiation/corrosion, the proton beam flux was 5.6$×10^{12}$ ions/($cm^2 \cdot s$) and the average beam current was 3.48 A/cm^2. The total irradiation/corrosion time was 12 hours, the total proton fluence was 2.0$×10^{17}$ ions/cm^2, and the oxygen concentration in LBE was 3.6$×10^{-3}$ wt.%. Once the targeted duration and proton fluence were reached, the proton beam was cut off and the heater was stopped, concluding the experiment.

2.3 Sample characterization

The microstructure of the coating and formed oxide film was characterized by 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). The TEM thin foils were prepared by a dual-beam focused ion beam (FIB, Thermo Scientific Helios 5 CX) instrument, with lift-out from both the "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, which were 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 dynamic (MD) simulations

MD simulations were performed using the Large-scale Atomic/Molecular Massively Parallel Simulator (LAMMPS) software package [14] to calculate the evolution of free volume in the coating and the variation of diffusion coefficients 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 used an NPT ensemble and started from the crystalline structure FCATM (110) 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 down to 873.5 K within 400 ps and isothermally equilibrated for another 400 ps. The completely amorphous FeCrAlTiMo coating, labeled FCATM' (110), was obtained by structural analysis using OVITO software (Fig. 8k) [Figure 8: see original paper].

The diffusion rates of each element before and after 5.5 MeV proton irradiation with a dose of 2.0$×10^{17}$ ions/cm^2 on the FCATM' (110) surface were also calculated. The interatomic interactions between Fe, Cr, Al, Ti, and Mo atoms were described by embedded-atom method (EAM) potentials [15]. These EAM potentials were validated by comparing formation energies of defects, equilibrium volumes, elastic moduli, and heats of formation for several binary compounds with ab initio simulations and experiments. Due to the high energy of the protons, the interaction between protons and other atoms was described using the Ziegler-Biersack-Littmark (ZBL) potential [16] during the irradiation simulation process. This ZBL potential includes an additional switching function that connects the ZBL and EAM potentials and smoothly reduces the curvature to zero between an inner (0.7 Å) and outer (1.5 Å) cutoff. In diffusion simulations, periodic boundary conditions were applied in three directions. In cascade simulations, periodic boundary conditions were applied in the x and y directions, while the z direction used fixed boundary conditions. The stress relaxation geometry of the simulated system was first optimized using the conjugate gradient descent method. Then, a Number-Pressure-Temperature (NPT) ensemble (at standard atmospheric pressure) at 600 °C was employed inside the simulation box. The system was simulated for approximately 400 ps,

which was sufficient to ensure temperature distribution stability. Finally, the MSDs of all atoms were recorded during the subsequent 600 ps simulation. The timestep was set to 1 fs in all simulations. The cascade simulation was conducted following equilibration in the NPT ensemble for 400 ps, using a Number-Volume-Temperature (NVT) ensemble at 600 °C with a timestep of 0.001 fs. All atomic configurations were displayed using the open visualization tool (OVITO) software package [17]. Volume analysis in the simulation process was completed using the Voro++ program [18]. The diffusion rates of all atoms and each atom type were calculated by fitting the MSD versus time data using a linear function and the least-squares method with reference to Einstein' s equation.

3.1 Evidence of irradiation-decelerated corrosion of FeCrAl-TiMo 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]. Figure 3a [Figure 3: see original paper] presents the core diagram of the corrosion/irradiation experiment facility, where two distinct areas were 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 were deposited, indicating that proton irradiation damage was introduced into the coating without significant hydrogen implantation. Therefore, the effect of hydrogen injection on the coating' s corrosion behavior was negligible.

Scanning electron microscope (SEM) images at different resolutions of the irradiated and unirradiated areas revealed remarkable differences in LBE corrosion behavior between them (Fig. 3e-j) [Figure 3: see original paper]. The unirradiated area was covered by continuous and dense oxide particles (Fig. 3g) [Figure 3: see original paper], while the average size and number density of oxide particles formed 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 certainly the sole factor responsible for slowing the LBE corrosion rate of the coating.

3.2 Quantification of decelerated corrosion by analyzing oxide film

To further confirm the effect of irradiation-decelerated corrosion, Fig. 4 [Figure 4: see original paper] presents cross-sectional TEM analysis of the oxide film formed in irradiated and unirradiated areas for quantitative comparison of LBE corrosion behavior. From the HAADF images (Fig. 4a-d and f-i) [Figure 4: see original paper], both the irradiated and unirradiated coating surfaces were

covered by a thin, continuous oxide film characterized by a double-layer structure with an outer oxide layer (OOL) in gray contrast and an inner oxide layer (IOL) in dark contrast. According to the EDS mapping results (Fig. 4e) [Figure 4: see original paper], the oxide film formed in unirradiated areas showed enrichment of Cr and O in the OOL, while Al, Ti, and O were enriched in the IOL, which was 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 to the unirradiated area, where a mixed Al_2O_3/TiO_2 IOL was observed, the irradiated area showed signals from Cr and Ti in the OOL, identifying hexagonal Cr_2O_3 and rutile TiO_2, and signals from Al 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.

Additionally, a transitional area between the oxide film and alloy coating, characterized by depletion of oxide-forming elements (Al, Ti, and Cr), formed after corrosion, 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]. Furthermore, the depletion of Al, Ti, and Cr 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, we summarized the thicknesses of the oxide layer and transitional layer from twenty independent HRTEM and HAADF images, as shown in Fig. 6 [Figure 6: see original paper]. Comparing the minimum, maximum, and average thicknesses of the outer oxide layer, inner oxide layer, total oxide layer, and transitional layer of the irradiated and unirradiated areas 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 roughly 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 the diffusion kinetics of coating elements in this study.

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 (precipitates and segregation). Energetic incident particles displace target atoms in cascades, inevitably resulting in vacancies, interstitials, and other irradiation defects. These defects provide fast pathways for atomic movement and increase diffusion rates of certain elements, thus accelerating corrosion [9]. As shown in Fig. 7 [Figure 7: see original paper], the amorphous structure of the FeCrAlTiMo coating was maintained after exposure to the high-temperature/LBE/irradiation multi-field environment, and no discernible precipitation or segregation was detected, indicating good structural stability. It is well established that amorphous metallic glasses differ from traditional crystalline materials in their unique structural feature of long-range disorder and short-range order, which makes them free of crystalline defects such as dislocation lines, loops, and walls [23]. Owing to the 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 mainly 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 an increase in free volume fraction leads to a significant increase in diffusion coefficient [25, 26]. In fact, from the SAED patterns (Fig. 8b, e, and h) [Figure 8: see original paper], some subtle differences could still be observed in the pristine, irradiated, and unirradiated coatings. The diameters of diffraction halos of the 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 the average interatomic distance became smaller and atomic arrangement 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 the unirradiated and irradiated coatings underwent the same thermal aging, the thermal effect will not be discussed here and only irradiation effects will be considered. Given possible inaccuracies in experimental measurements, the coating' s volumetric response to proton irradiation and the accompanying variation in element diffusion coefficient were further calculated by molecular dynamics (MD) simulation. Fig. 8l and m [Figure 8: see original paper] show the evolution of average volume per atom (Vp) over time for the unirradiated and irradiated coatings, respectively, over 600 ps. Averaging Vp over the recorded time yielded Vap, with the irradiated coating' s Vap (30.136 $Å^3$/atom) being less than that of the unirradiated coating (30.763 $Å^3$/atom), indicating that free volume in the coating was reduced after irradiation. This was consistent with the SAED pattern results (Fig. 8c, f, and i) [Figure 8: see original paper].

Fig. 8n [Figure 8: see original paper] shows the change in mean square dis-

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

Based on the above analysis, a mechanism involving irradiation-reduced diffusion is proposed to explain how irradiation enhanced corrosion resistance. In this study, the FeCrAlTiMo coating retained its amorphous structure after simultaneous irradiation and corrosion. The interstitial-like and vacancy-like point defects generated by irradiation damage cascades were transient, unstable, and completely annihilated, and could not 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 mechanism of irradiation-decelerated corrosion is hypothesized to be generally effective in amorphous alloys, independent of corrosive medium type. Therefore, this effect can be adopted 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 the design and rapid development of structural materials for future advanced nuclear energy systems.

Conclusions

The synergistic effect of simultaneous proton irradiation and LBE corrosion on FeCrAlTiMo coating was investigated. The 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 could promote 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 diffusion kinetics of coating elements and led to slower corrosion.

Acknowledgements

This work was supported by the National Natural Science Foundation of China (Grant No. U2241235) and the Nuclear Power Institute of China.

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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