# 5 - 9 Microstructure of TiAlN Coating after LBE Corrosion with Pre-irradiation

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Lead-bismuth eutectic (LBE) has great potential advantages as coolant material for lead-cooled fast reactor (LFR) and accelerator driven sub-critical system (ADS)[1]. On this account, the corrosion of structural materials by LBE is an urgent problem needed to be solved. TiAlN coating exhibits excellent properties, such as high hardness and melting point, which leads it to be one of the promising candidates as protective coating on fuel cladding of LFR[2]. In order to study the performance of TiAlN coating under harsh service conditions of LFR, considerable works, LBE corrosion tests of TiAlN coating after ion irradiation have been carried out.

In this work, TiAlN coatings were prepared on WFeNi substrates by cathodic arc ion plating method, with Cr layer as a buffer. Different damage levels have been pre-created in the TiAlN coating by 1.4 MeV $N^{5+}$ self-ion irradiation with the dose of $4\times10^{15}$ and $2\times10^{16}$ ions/cm$^2$ at 320 kV Multi-discipline Research Platform for Highly Charged Ions (IMP, Lanzhou). Then the coatings were exposed to 450 ℃ LBE for 3 000 h. By transmission electron microscope (TEM), the microstructure of the coating was characterized before and after experiments.

The results show that TiAlN coating exhibits excellent LBE corrosion resistance. Figure 1 shows the TEM images of the microstructure near the coating surface before and after corrosion. There is a thin nano-scale layer covered on the surfaces of original coating. With energy dispersive spectroscopy (EDS) analysis shown in Fig. 1(e), it's confirmed that the layer is composite oxides of Al and Ti, which acts as the first barrier against to LBE. After corrosion, these layers still remain intact. Average thicknesses of the oxide layers of the coatings are are shown in Fig. 2. The thickness of the oxide layer has obviously increased after corrosion, and pre-irradiation seems to have no significant effect on oxide thickness. During the corrosion test, oxygen atoms in LBE have the opportunity to diffuse through the initially formed thin oxide layer and continue to oxidize at the interface with Ti and Al elements in the coating[3], which thickens oxide layer on the surface of the coating.

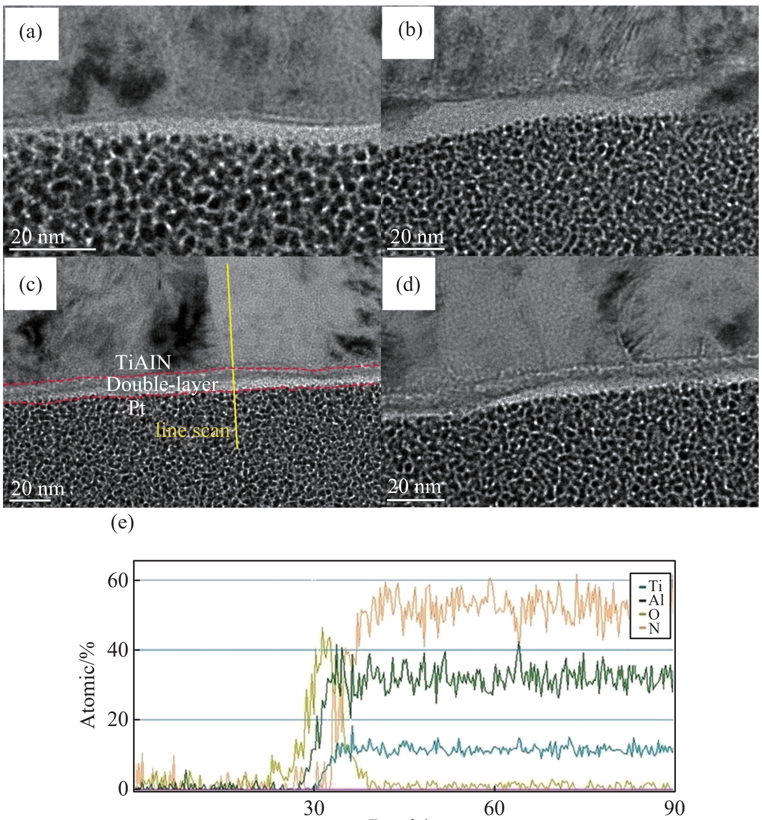


Fig. 1 (color online) TEM images of TiAlN coating (a) original sample, and samples with different pre-irradiation fluences (b)un-irradiated, (c) $4\times10^{15}$ and (d) $2\times10^{16}$ ions/cm$^2$, (e) EDS line scan on (c) after corrosion for 3 000 h.

It is worth noting that for pre-irradiation as shown in Fig. 1(c) and (d), the oxide layers present a structure of double-layer. In Fig. 1(e), along the depth direction the content of Al begins to increase before that of Ti, which confirms that the oxide layer on the surface of the coating is composed of the outer layer of $Al_2O_3$ and the inner layer of $Al_2O_3/TiO_2$. The double-layered structure indicates that pre-irradiation created a significant effect on the formation of oxide layer. We suggest that after the irradiation-modification, initial surface oxide layer and the near-surface of the coating may allow significant differences in the diffusion rate of the elements Al, Ti and O. Such differences finally lead to the new formed oxide layer with a double-layer structure during the following corrosion process. The mechanism of double-layer oxide is shown in Fig. 3. Note that, although the irradiation significantly changes the structure of the oxide layer, it still does not essentially influence the corrosion resistance of the coating.

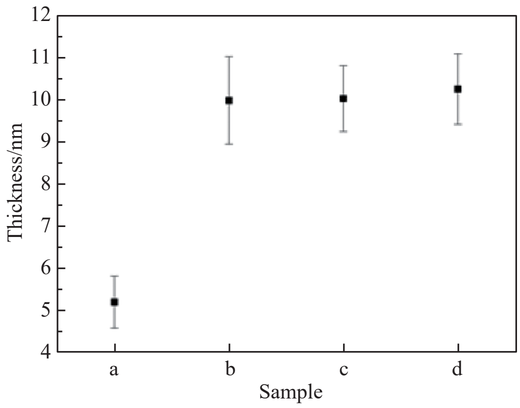


Fig. 2 Average thickness of oxide layers on the surface of TiAlN coatings in Fig. 1(a)∼(d).

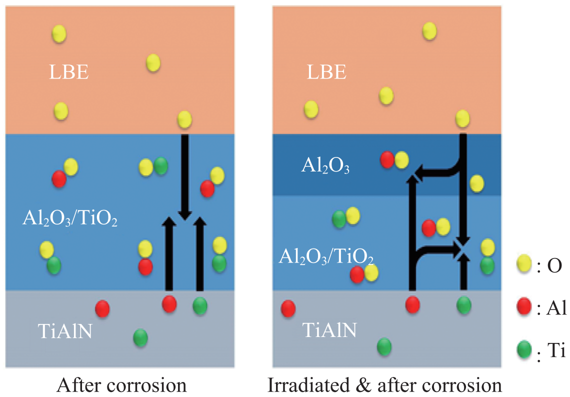


Fig. 3 (color online) Double-layer oxide mechanism on the surface of the TiAlN coating.

## References

[1] Handbook on Lead-bismuth Eutectic Alloy and Lead Properties, Materials Compatibility, Thermal-hydraulics and Technologies: 2015 Edition, OECD Nuclear Science, (2015).

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[3] H. Ichimura, A. Kawana, Journal of Materials Research, 8(1993)1093.