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Alumina-Forming Coating on Metal Baskets for U-Recovery in Pyroprocessing

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1. Introduction

Electrorefining is a key unit process in pyroprocessing technology for the treatment of spent nuclear fuel, in which uranium is recovered as a metallic dendrite that grows on the cathode, positioned within a basket structure; the cathode and basket are immersed together in a LiCl-KCl molten salt electrolyte. This U-recovery basket has conventionally been fabricated from Al_2O_3 ceramic to provide electrical insulation between the basket and the growing U dendrite. However, because Al_2O_3 ceramics are brittle and highly susceptible to thermal shock, repeated heating and cooling cycles during electrorefining operation induce cracking and eventual fracture of the basket, limiting its operational lifetime.

To overcome this thermal-shock limitation, a mechanically robust tungsten (W) basket was considered as an alternative. While W offers superior thermal-shock resistance, its high electrical conductivity allows U dendrites to grow directly along the basket surface, causing unintended electrical shorting and compromising the electrorefining process. Conventional ceramic coatings such as Y_2O_3 were also evaluated as an electrically insulating layer on W, but exhibited poor physical adhesion to the substrate and were therefore unsuitable for practical application.

These considerations indicate that an ideal material for the U-recovery basket must simultaneously satisfy three requirements: chemical inertness toward the LiCl-KCl-U-UCl_3 molten salt system, electrical insulation to prevent dendrite-induced shorting, and resistance to thermal-shock-induced degradation. In this study, a pack cementation aluminizing approach was investigated as a means of forming a robust, Al_2O_3-forming aluminide layer on a W substrate, thereby combining the thermal-shock resistance of W with the electrical insulation required for stable long-term electrorefining basket operation.

2. Methods and Results

In this section, the specimen preparation procedure and pack cementation aluminizing process are described, followed by the surface and cross-sectional characterization results used to identify the phase and morphology of the resulting coating. Characterization includes SEM-EDS analysis of the surface and cross-section, and XRD phase identification of the coated specimen.

2.1 Methods

2.1 Experimental Methods

W plate specimens (10 mm × 10 mm × 1 T) were used as the coating substrate. Prior to coating, the specimen surfaces were polished sequentially using 320-grit SiC paper followed by 1 μm diamond suspension, and then ultrasonically cleaned in deionized water. Pack cementation aluminizing was carried out using a pack mixture consisting of 15 wt.% Al powder (donor), 3 wt.% NH_4Cl (activator), and 82 wt.% Al_2O_3 powder (inert filler). The specimen was buried in the pack mixture inside an alumina crucible and placed in a quartz reactor under flowing Ar atmosphere at 1000 ℃ for 5 h. Under these conditions, NH_4Cl decomposes to generate HCl gas, which reacts with the Al powder to form volatile $AlCl_x$ species; these species subsequently transport Al to the specimen surface, where they react to form an aluminide layer.

2.2 Results

2.2.1 Surface Microstructure and Phase Identification

The surface morphology of the aluminized specimen was examined by scanning electron microscopy (SEM) at X500 and X3,000 magnifications (Fig. 1 and Fig. 2, respectively), and the elemental composition was analyzed by energy-dispersive X-ray spectroscopy (EDS). The coated surface exhibited a dense layer composed of well-developed polyhedral grains (Fig. 1, Fig. 2). EDS point analysis at three representative locations, indicated in Fig. 2, consistently yielded Al:W atomic ratios of approximately 80:20 at.%, close to the stoichiometry of the Al_4W intermetallic phase. EDS elemental mapping of the surface (Fig. 3) further confirmed a uniform distribution of Al and W across the coated layer, consistent with the point analysis results. X-ray diffraction (XRD) analysis of the coated surface (Fig. 4) was performed over a 2θ range of 20°–80°, and the measured pattern showed good agreement with the reference stick pattern of Al_4W (PDF #65-1241) at the major diffraction peaks (22.6°, 26.5°, 40.5°), confirming

that the coating consists predominantly of a single-phase Al_4W layer.

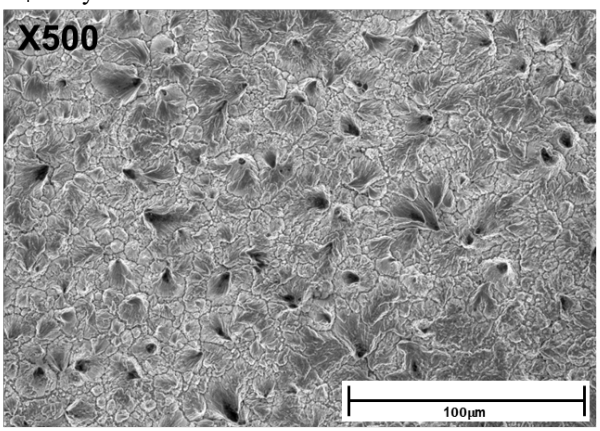

Fig. 1. SEM image of the aluminized W surface at X500 magnification

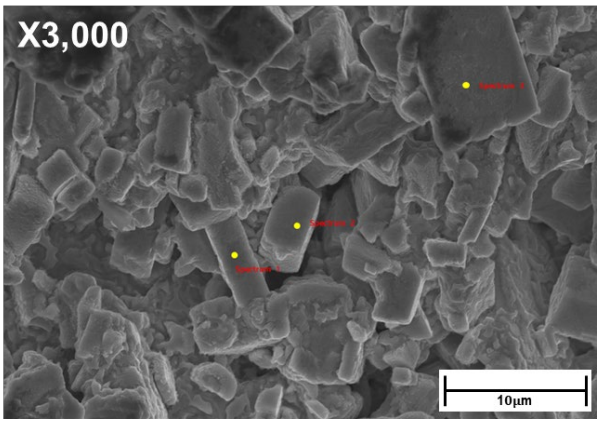

Fig. 2. SEM image of the aluminized W surface at X3,000 magnification, with EDS point analysis locations indicated.

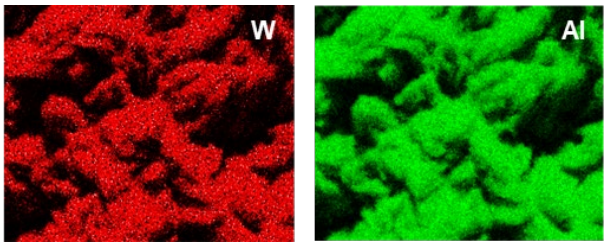

Fig. 3. EDS elemental mapping images of the aluminized W surface showing the distribution of W (left) and Al (right).

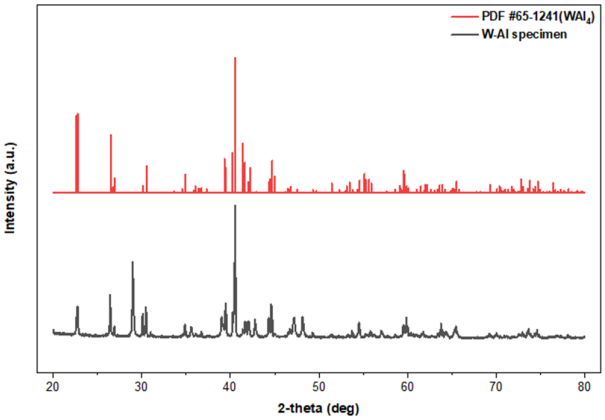

Fig. 4. XRD pattern of the aluminized W-Al specimen surface, overlaid with the reference stick pattern of Al_4W (PDF #65-1241).

2.2.2 Cross-Sectional Analysis

To characterize the coating thickness and its distribution across the coating/substrate interface, cross-sectional specimens were prepared and examined by SEM-EDS (Fig. 5). The aluminide coating layer was continuous and uniform, with a thickness of approximately 130 μm. EDS elemental mapping across the cross-section (Fig. 6) showed a distinct boundary between the Al-rich coating and the W substrate, consistent with the line-profile results. An EDS line profile (Fig. 7) acquired across the cross-section, from the coating surface toward the substrate, showed a stable composition of approximately 80 at.% Al and 20 at.% W throughout the coating layer, transitioning sharply to approximately 100 at.% W within a narrow interfacial region at the coating/substrate boundary. No broad interdiffusion zone with a gradual compositional gradient was observed, indicating that a single stable intermetallic phase, rather than a sequence of multiple Al-W intermetallic layers, formed at the interface under the present processing conditions.

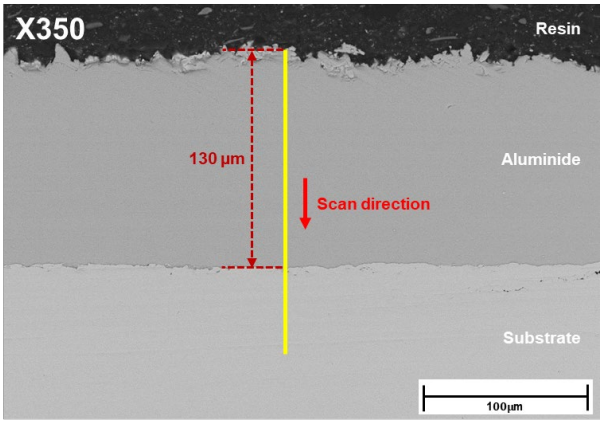

Fig. 5. Cross-sectional SEM image of the aluminized W specimen showing the resin, aluminide coating, and W substrate layers.

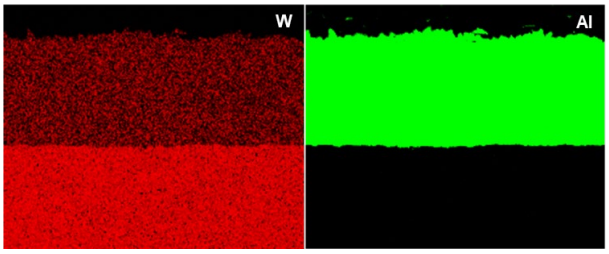

Fig. 6. Cross-sectional EDS elemental mapping images showing the distribution of W (left) and Al (right) across the aluminide coating and W substrate.

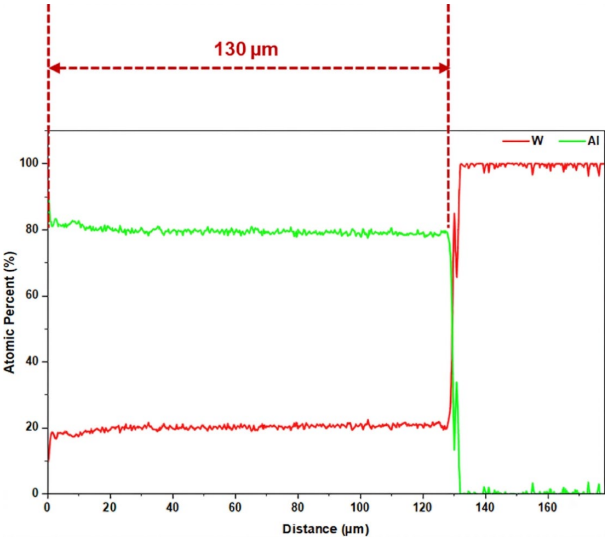

Fig. 7 . EDS line profile of W and Al across the coating/ substrate interface, showing an aluminide coating thickness of approximately 130 µm.

3. Conclusions

A pack cementation aluminizing process was applied to W plate specimens (10 mm × 10 mm × 1 T) using a pack mixture of 15 wt.% Al, 3 wt.% NH_4Cl, and 82 wt.% Al_2O_3 at 1000 ℃ for 5 h, with the aim of forming a robust, electrically insulating aluminide coating for use as a U-recovery basket, or crucible, material in electrorefining. Surface SEM observation revealed a dense, well-developed polyhedral grain morphology, and EDS point analysis consistently showed Al:W atomic ratios of approximately 80:20 at.%, close to the Al_4W stoichiometry. This phase assignment was corroborated by XRD, which showed good agreement between the measured pattern and the Al4W reference stick pattern (PDF #65-1241) at the major diffraction peaks. Cross-sectional SEM-EDS confirmed a uniform coating thickness of approximately 130 µm, with the EDS line profile transitioning sharply from the Al-rich coating (~80 at.% Al) to the W substrate (~100 at.% W) within a narrow interfacial region, without evidence of a broad interdiffusion zone. These results collectively demonstrate that the selected pack cementation conditions reliably produce a uniform, single-phase Al_4W coating on W, satisfying the electrical insulation requirement for the intended U-recovery basket, or crucible, application. Building on this baseline coating, future work will extend the process to Mo substrates, evaluate the effect of Y_2O_3 doping on coating microstructure and oxidation resistance, and investigate process modifications—such as reduced cooling rate and improved specimen preparation—to address surface cracking and coating delamination observed during cross-sectional sample preparation.

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