Preparation of Nickel-Chromium Composite Coating on Tungsten via Molten Salt Thermal Diffusion

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Abstract

Tungsten is a commonly used shielding material in Sr-90 radioisotope thermoelectric generators, but its high-temperature oxidation resistance is poor. To improve its oxidation resistance under high-temperature operating conditions, this study prepared a nickel-chromium composite coating on tungsten using the molten salt thermal diffusion method, and conducted analysis and testing on coatings prepared at different molten salt temperatures. The results show that when the molten salt temperature is 950 °C, the impurity phosphorus element in the coating gradually disappears, the prepared nickel-chromium coating becomes purer, and it exhibits good oxidation resistance and spalling resistance. The study reveals that dense Cr_2O_3 forms on the surface of the oxidized sample; additionally, this sample possesses the strongest adhesion strength, and the coating exhibits a fine-grained structure. Therefore, 950 °C is a suitable temperature for preparing nickel-chromium coatings by the molten salt thermal diffusion method.

Full Text

Preamble

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Preparation of Ni-Cr Composite Coatings on Tungsten by Molten Salt Thermal Diffusion Method

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Abstract

[**Background**] Tungsten is a commonly used shielding material in Sr-90 radioisotope thermoelectric generators; however, its resistance to high-temperature oxidation is relatively poor.
[**Purpose**] This study aims to enhance the oxidation resistance of tungsten under high-temperature conditions.
[**Methods**] A nickel-chromium composite coating was prepared on tungsten using the molten salt thermal diffusion method. Coatings produced at different molten salt temperatures were characterized using scanning electron microscopy, energy dispersive spectroscopy, and X-ray diffraction. Additionally, cyclic oxidation tests and isothermal oxidation tests were conducted to evaluate the oxidation resistance and spallation resistance of the coatings. The adhesion strength between the coatings and substrate was assessed using the indentation method.
[**Results**] The results indicate that the primary phase composition of the coatings is γ-(Ni,Cr). When the molten salt temperature is 950 ℃, the coating surface exhibits a blocky morphology, the impurity phosphorus element gradually disappears, and the prepared nickel-chromium coating is more pure with good oxidation resistance and anti-spallation properties. The study reveals that a dense Cr_2O_3 layer formed on the surface of the oxidized sample. Furthermore, this sample demonstrated the highest adhesion strength, and the coating exhibited a fine crystalline structure.
[**Conclusions**] Therefore, 950 ℃ is the optimal temperature for preparing nickel-chromium coatings using the molten salt diffusion technique.

Keywords: Molten salt diffusion, Nickel-chromium coating, High-temperature oxidation resistance, Adhesion

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Introduction

Radioisotope thermoelectric generators (RTGs) are power generation devices that utilize the Seebeck effect to convert decay heat from radioactive isotopes into electrical energy [1]. They are commonly employed in extreme environments such as polar regions, deep space, and deep sea to power detectors, sensors, and coastal warning lights [2-4]. Sr-90 is one of the suitable nuclides for these

batteries. During its decay, it produces a large number of electrons that generate bremsstrahlung X-rays, requiring high-density materials for shielding. Tungsten is a high-melting-point, high-density material with excellent radiation resistance [5] and is frequently used as an X-ray shielding material, making it ideal for RTG applications. However, tungsten exhibits poor oxidation resistance at high temperatures, with significant oxidation occurring above 600 ℃ [6].

The SENTINEL 100F is a 100-watt-class RTG that uses Sr-90 as nuclear fuel, and its shielding layer temperature serves as a specific parameter reference for this study. During normal operation, the shielding layer temperature of the SENTINEL 100F typically ranges from 593 to 643 ℃, while under accident conditions, the temperature can reach up to 852 ℃ [7]. Although the RTG system undergoes vacuum treatment, complete vacuum cannot be achieved, and vacuum stability may not be maintained during accident conditions or long-term use. Consequently, implementing oxidation prevention measures is necessary.

Research has shown that one of the most effective methods is depositing an anti-oxidation coating on the tungsten surface. Chromium coatings are typical anti-oxidation coatings with high melting points; during oxidation, they form a dense Cr_2O_3 layer on the surface that prevents oxygen diffusion into the substrate. The SENTINEL 100F prevents tungsten oxidation through hard chromium plating [7]; however, hard chromium coatings exhibit poor adhesion to the substrate and are brittle, potentially leading to cracking and spallation under stress at high temperatures. In contrast, nickel-chromium coatings demonstrate better mechanical properties and adhesion [8,9]. Therefore, Ni-Cr binary alloy coatings can be considered as high-temperature anti-oxidation coatings for tungsten.

During the initial oxidation stage of Ni-Cr alloy coatings, Cr_2O_3, NiO, and $NiCr_2O_4$ form on the surface. Whether nickel oxides are completely replaced by chromium oxides depends on the chromium content. When the chromium content is sufficiently high, selective oxidation occurs, generating only Cr_2O_3 and providing oxidation protection. In the Ni-Cr system, the critical chromium concentration for selective oxidation is approximately 20 wt.% [10].

Various methods exist for preparing Ni-Cr alloy coatings [11-14], with co-electrodeposition in aqueous solutions being the most widely studied. Zhang et al. [14] prepared a Ni-9.6 wt.% Cr nanocomposite film with a grain size of 39 nm using co-electrodeposition; this nanocrystalline structure facilitates selective oxidation of chromium, and good oxidation resistance was demonstrated at 800 ℃. Liu et al. [8] successfully prepared a nanocrystalline Ni-Cr coating with excellent mechanical properties on a 30CrNiMo steel substrate using pulsed current co-electrodeposition, significantly improving the substrate' s microhardness and wear resistance. Firouzi-Nerbin et al. [15] deposited a Ni-Cr coating on a copper substrate using a sulfate-chloride electroplating solution combined with co-electrodeposition and investigated the effects of direct and pulsed current on cathode efficiency, alloy composition, grain size, microhardness, morphology, and corrosion properties.

However, the co-electrodeposition process faces several challenges, primarily due to the different deposition potentials required for Cr and Ni co-deposition [16,17]. Additionally, electroplating suffers from poor coating-substrate adhesion. To address these issues, this paper proposes a novel approach for preparing Ni-Cr coatings. First, a chemical nickel coating is deposited on the tungsten substrate, followed by chromium diffusion into the nickel coating using the molten salt thermal diffusion method. This two-step preparation avoids the complex operation of adjusting Cr and Ni to the same potential. Molten salt thermal diffusion is a technique where samples are placed in inorganic molten salt, and active elements in the salt diffuse into the sample surface to form controllable coatings. This technique is essentially a form of chemical plating that, compared to electroplating, produces dense and uniform coatings while further enhancing coating-substrate adhesion at high temperatures.

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1. Methodology

1.1 Materials

In this study, fluoride salts were selected as the base salt due to their good fluidity, high thermal stability, and high solubility for active elements [18]. To avoid the toxicity of hexavalent chromium, trivalent chromium salts were chosen as the solute [19]. The molten salt formulation used in this study consisted of FLiNaK (a eutectic mixture of LiF, NaF, and KF), CrF_3, and chromium powder. Tungsten samples were circular specimens measuring 10$×10×$3 mm. All chemicals were reagent-grade: sodium fluoride, chromium powder, and ethanol were provided by Sinopharm Chemical Reagent Co., Ltd.; lithium fluoride and chromium fluoride were provided by Shanghai China Lithium Industrial Co., Ltd. and Shanghai Macklin Biochemical Co., Ltd., respectively; potassium fluoride was provided by Xinxiang Yellow River Fine Chemical Industry Co., Ltd.; tungsten specimens (purity >99.99%) were provided by Qinghe County Tengfeng Metal Materials Co., Ltd. and subjected to wire cutting.

1.2 Preparation of Ni-Cr Composite Coatings

Before the experiment, sample surfaces were polished with sandpaper to remove oxide scale and achieve a mirror finish. The samples were then ultrasonically cleaned in distilled water and ethanol, after which they were sent to Shanghai Parker Company for electroless nickel plating. The resulting electroless nickel coating thickness was approximately 10 μm. During the electroless nickel plating process, a small amount of phosphorus element was inevitably introduced; the phosphorus content in the prepared electroless nickel coating was about 1-3 wt.%.

The entire molten salt chromizing process was conducted in a glove box under an argon atmosphere with O_2 and H_2O contents below 1 ppm, as moisture

and oxygen can exacerbate corrosion of fluoride salts [20,21]. In this study, the molten salt consisted of a mixture of FLiNaK, CrF_3, and chromium powder, with a FLiNaK-to-CrF_3 ratio of 95 wt.%-5 wt.%; chromium powder was added in appropriate amounts. During the experimental procedure, the molten salt mixture was first placed in a stainless steel crucible and transferred to a resistance furnace. The furnace temperature was then adjusted to 750-950 ℃, and the electroless nickel-plated tungsten samples were immersed in the molten salt for 4 h using stainless steel wire. Finally, after the molten salt treatment, the samples were removed and cleaned with distilled water to remove residual salt from the surface.

1.3 Testing and Characterization

This study used a D8 Advance X-ray diffractometer (XRD) to detect the phase composition of the coatings and post-cyclic-oxidation coatings. The diffractometer operated at a scanning rate of 8°/min with a scanning range of 10-90°, using Cu Kα radiation ($\lambda = 1.5418$ Å). A LEO 1530vp scanning electron microscope (SEM) was used to observe the surface and cross-sectional morphologies of the samples. Energy dispersive spectroscopy (EDS) attached to the SEM was used for local elemental analysis. High-temperature cyclic oxidation tests and isothermal oxidation tests were conducted on the samples using an SXL-1200 box furnace in air atmosphere. The cyclic oxidation temperature was 850 ℃, with each cycle consisting of heating at 850 ℃ for 1 h followed by cooling to room temperature, for a total of 6 cycles. Before the cyclic oxidation experiments, alumina crucibles were preheated to constant weight to hold the samples, and each sample (crucible + sample) was weighed using an electronic balance with an accuracy of 10^{-4} g.

After the experiment began, weighing was performed once per cycle. Following each weighing, a curve plotting the weight gain per unit area versus time was constructed to evaluate the high-temperature oxidation resistance and spallation resistance of the composite coatings [10]. The isothermal oxidation test temperature was 650 ℃ for 300 h, with samples weighed at oxidation times of 50 h, 100 h, 150 h, and 300 h to plot the oxidation weight gain per unit area. Coating adhesion was evaluated using an 8150 LK Rockwell hardness tester with a diamond cone indenter with a 120° apex angle under loads of 30 kg, 45 kg, 60 kg, 100 kg, and 150 kg. Finally, the minimum load causing coating cracking or spallation was determined through SEM observation.

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2. Results and Discussion

2.1 Coating Microstructure

Figure 1 [Figure 1: see original paper] shows SEM images of the electroless nickel coating and Ni-Cr coatings prepared in molten salt at 750-950 ℃ for 4 h. The

untreated electroless nickel coating (Figure 1(a)) exhibits a dense and uniform surface composed of many blocky particles. The Ni-Cr coatings prepared at 750 ℃ (Figure 1(b)) and 850 ℃ (Figure 1(c)) show similar surface morphologies, appearing granular with some pores. After treatment at 950 ℃ (Figure 1(d)), the coating surface morphology differs significantly from those treated at 750 ℃ and 850 ℃, presenting a blocky structure. This morphology resembles a molten state, suggesting that the coating surface may have undergone elemental dissolution at high temperature.

The XRD patterns of Ni-Cr coatings prepared in molten salt at 750-950 ℃ for 4 h are shown in Figure 2 [Figure 2: see original paper]. Four phases are identified: Ni_3P, Cr_3P, γ-(Ni,Cr), and the substrate W, with the γ-phase being the predominant component. The presence of phosphorus-containing phases is due to the small amount of P element in the electroless nickel coating. At a molten salt temperature of 750 ℃, some Ni_3P diffraction peaks are present in the coating. As the temperature increases to 850 ℃ and 950 ℃, Ni_3P gradually transforms into Cr_3P because Cr has a lower standard electrode potential than Ni and more readily reacts with P to form stable compounds. Simultaneously, as the temperature increases, the intensity of W diffraction peaks gradually decreases, indicating that more chromium diffuses into the electroless nickel coating and the coating density increases, making it difficult for X-rays to penetrate the coating to reach the substrate.

Figure 3 [Figure 3: see original paper] presents the cross-sectional SEM morphology and EDS elemental analysis of the electroless nickel coating and Ni-Cr coatings prepared in molten salt at 750-950 ℃ for 4 h. The coating thickness is approximately 10 μm. The cross-section of the electroless nickel coating (Figure 3(a)) is uniform and dense, with relatively uniform distribution of Ni and P elements. At a molten salt temperature of 750 ℃ (Figure 3(b)), a crack is observed in the middle of the coating filled with many granular materials. The EDS elemental map reveals that these granular filling materials are phosphorus. This phenomenon occurs because phosphorus on the coating surface reacts with diffusing chromium to form Cr_3P, creating a driving force for phosphorus diffusion from the coating interior to the outer surface. At this stage, phosphorus has diffused to the middle of the coating. At 850 ℃ (Figure 3(c)) and 950 ℃ (Figure 3(d)), the coatings are dense and uniform without through-thickness cracks or spallation. Chromium completely diffuses into the Ni coating, while phosphorus diffuses to the coating surface, with partial phosphorus loss observed at 950 ℃. This corresponds to the morphology in Figure 1(d), where elemental dissolution occurs on the coating surface.

The mechanism for this phenomenon is analyzed as follows: The electroless nickel coating is considered an amorphous structure at room temperature, which is inherently unstable. When the temperature reaches 300 ℃, it transforms into a crystalline mixture of Ni and Ni_3P. When the phosphorus content reaches 11 wt.%, the melting point of this mixture is 880 ℃ [22]. Therefore, the elemental dissolution on the coating surface at 950 ℃ may occur because, before

chromium completely forms stable compounds with the electroless nickel, the phosphorus content at the coating surface reaches 11 wt.%, causing the Ni and Ni_3P crystalline mixture on the coating surface to melt. This phenomenon is beneficial for oxidation resistance because it reduces phosphorus content, which is advantageous since related studies have shown that Ni-P coatings oxidize 100 times faster than pure nickel in the 800-1000 ℃ temperature range [23].

2.2 High-Temperature Oxidation Testing and Characterization

The appearance of samples after cyclic oxidation testing in air is shown in Figure 4 [Figure 4: see original paper], where the yellow regions on sample surfaces represent tungsten oxide. It can be observed that the pure tungsten sample surface is completely oxidized, while the electroless nickel coating and Ni-Cr coatings prepared at 750 ℃ and 850 ℃ show varying degrees of spallation. The Ni-Cr coating prepared after 950 ℃ molten salt treatment remains relatively intact, demonstrating the best anti-spallation performance. The strong anti-spallation property may be attributed to high temperature increasing the coating-substrate adhesion, which was systematically tested subsequently.

The weight gain curves after cyclic oxidation in air are presented in Figure 5 [Figure 5: see original paper]. Faster weight gain indicates more rapid oxidation. The figure shows that the electroless nickel coating provides some oxidation protection compared to pure tungsten, but the effect is relatively weak. After molten salt chromizing, the oxidation resistance improves significantly. As the molten salt temperature increases from 750 ℃ to 950 ℃, the oxidation resistance improves progressively, with the best performance at 950 ℃. This improvement is attributed to two main factors: first, at 950 ℃, the reduced phosphorus content on the coating surface enhances oxidation resistance; second, the samples prepared at 750 ℃ and 850 ℃ exhibit relatively poor anti-spallation performance, and once spallation occurs, oxidation resistance naturally decreases. As shown in Figure 5, during the initial oxidation stage before spallation occurs, the weight gain rates for all three temperatures are relatively slow. After some time, spallation begins on samples prepared at 750 ℃ and 850 ℃, leading to rapid weight gain.

Since the coating prepared at 950 ℃ demonstrates the best performance, detailed characterization of the oxidized sample was conducted. Figure 6 [Figure 6: see original paper] shows the SEM images, XRD pattern, and EDS line scan results of the Ni-Cr coating prepared in molten salt at 950 ℃ for 4 h after cyclic oxidation. Figure 6(a) shows the surface morphology of the oxide layer, which appears granular. EDS elemental analysis of selected regions reveals atomic percentages of 54.43% for O and 45.57% for Cr, with no Ni or P detected, suggesting the formation of Cr_2O_3 on the coating surface. Figure 6(b) shows the XRD pattern after cyclic oxidation, revealing γ-(Ni,Cr) and Cr_2O_3 phases, which confirms Cr_2O_3 formation. The absence of NiO and $NiCr_2O_4$ in the oxide layer is primarily related to selective oxidation of chromium. According to Wagner' s theory, when a binary alloy A-B is exposed to oxygen, where A is

the more noble element and B is the less noble element, only the oxide of B (BO) forms if the concentration of B in the alloy is sufficiently high, while A diffuses from the alloy/oxide layer interface into the alloy [24]. Therefore, in the Ni-Cr alloy system, when the chromium concentration is high enough, selective oxidation occurs. The critical chromium concentration for selective oxidation is approximately 20 wt.%. Figure 6(d) shows the cross-sectional EDS line scan analysis in weight percentage, where the chromium content in the coating clearly exceeds 20 wt.%. Additionally, the Cr and O contents increase significantly near the coating surface, again confirming Cr_2O_3 formation. The region containing only Ni and Cr corresponds to the γ-phase in the XRD pattern in Figure 6(b). Meanwhile, oxygen exists only on the coating surface without diffusing into the substrate, indicating excellent oxidation resistance of the coating. Figure 6(c) shows the cross-sectional morphology after cyclic oxidation, revealing no through-thickness cracks in the coating.

Since nuclear batteries are designed for long-term operation, isothermal oxidation tests were also conducted for extended durations on the Ni-Cr coating prepared at 950 ℃ for 4 h. Figure 7 [Figure 7: see original paper] shows the appearance of samples after 300 h of isothermal oxidation at 650 ℃ in air. The pure tungsten sample surface is completely oxidized (Figure 7(a)), while the coated sample surface appears black with no tungsten oxide formation (Figure 7(b)), indicating the coating remains intact. Figure 8 [Figure 8: see original paper] shows the weight gain of samples after isothermal oxidation at 650 ℃ in air. At oxidation times of 50 h, 100 h, 150 h, and 300 h, the oxidation weight gain of the Ni-Cr coated sample is far lower than that of pure tungsten, with a relatively slow increase rate. This demonstrates that the coating maintains good oxidation resistance during long-term isothermal oxidation testing.

2.3 Coating Adhesion Testing

To verify the effect of high temperature on Ni-Cr coating adhesion, the indentation method was used to evaluate adhesion strength. Adhesion can be assessed through the critical load, defined as the minimum load causing coating cracking or spallation, where a higher critical load indicates stronger coating-substrate bonding [25]. Figure 9 [Figure 9: see original paper] shows the indentation morphologies of the electroless nickel coating and Ni-Cr coatings prepared in molten salt at 750-950 ℃ for 4 h. At a load of 60 kg, the electroless nickel coating developed obvious cracks. Since the applied loads were discrete, the critical load for electroless nickel coating is between 45-60 kg. Similarly, the critical loads for all samples were determined and are listed in Table 1 . Sample A has a critical load below 30 kg, Sample B between 100-150 kg, and Sample C above 150 kg. This demonstrates that adhesion between the coating and substrate improves significantly after high-temperature molten salt chromizing of the electroless nickel coating. The low adhesion of Sample A is primarily due to phosphorus not diffusing to the sample surface, forming a crack filled with granular material in the middle of the coating (Figure 3(b)). Sample C, prepared at the highest

molten salt temperature, shows the strongest coating-substrate adhesion, which explains its excellent anti-spallation performance.

Additionally, anti-spallation performance is related to coating grain size. Based on the XRD patterns in Figure 2, the Debye-Scherrer formula was used to estimate the average grain size of Sample C' s coating to be approximately 29 nm, indicating a fine-grained structure:

$$D = \frac{K\lambda}{\beta \cos\theta}$$

where D represents the coating grain size, K is the Scherrer constant, λ is the X-ray wavelength, β is the full width at half maximum of the diffraction peak, and is the Bragg diffraction angle. Fine-grained Ni-Cr alloy coatings can produce oxide layers with finer grain structures, which exhibit better high-temperature plasticity and creep properties [26,27]. Stress generated at high temperatures can be relieved through creep rather than cracking or spallation; therefore, the fine-grained structure also contributes to the coating' s strong anti-spallation performance.

Table 1 Critical loads of electroless nickel coating and Ni-Cr coatings prepared in molten salt at 750-950 ℃ for 4 h

Sample	Type	Critical Load (kg)
Electroless Ni	-	45-60
A (750 ℃)	Ni-Cr	<30
B (850 ℃)	Ni-Cr	100-150
C (950 ℃)	Ni-Cr	>150

Conclusion

This study successfully prepared Ni-Cr composite coatings on tungsten surfaces using the molten salt thermal diffusion method. The results show that Ni-Cr coatings prepared at 750 ℃ and 850 ℃ exhibit granular surface morphologies, while the coating prepared at 950 ℃ shows a molten blocky morphology. Four phases were identified in the coatings: Ni_3P, Cr_3P, γ-(Ni,Cr), and the substrate W, with the solid solution γ-phase as the main component. As the temperature increased from 750 ℃ to 950 ℃, Ni_3P gradually transformed into Cr_3P, while phosphorus diffused to the coating surface and gradually disappeared at 950 ℃. The coating prepared at 950 ℃ demonstrated the best oxidation resistance and anti-spallation performance. Detailed investigation revealed that a dense Cr_2O_3 layer formed on the surface of the oxidized sample, which exhibited the strongest adhesion and a fine-grained structure. Therefore, 950 ℃ is the optimal temperature for preparing Ni-Cr coatings using the molten salt thermal diffusion method.

Author Contributions

CAO Xueshan: Data curation, manuscript writing
DAI Jianxing: Review and editing
ZHANG Wei: Investigation
YU Guojun: Conceptualization, methodology

All authors have read and approved the final manuscript.

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Note: Figure translations are in progress. See original paper for figures.