Effects of the different interlayer deposition processes on the microstructure of Cr/TiN coating

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Abstract

Cr/TiN coatings were deposited on the surface of Zircaloy-4 alloy by physical vapor deposition. The Cr interlayers were prepared by magnetron sputtering (MS) and multi-arc ion plating (MAIP), respectively. To elucidate the effects of the different interlayer processes on the microstructure of Cr/TiN coating, the morphological features, components of surface particles, surface roughness, bonding strength, phase constituents, and high-temperature oxidation resistance were carefully investigated. In comparison to the Cr/TiN coating with the Cr interlayer prepared by the MS, the Cr/TiN coating with the Cr interlayer prepared by the MAIP exhibits a blurry bonding interface and higher coating roughness. Interestingly, the latter one has lower surface porosity, together with better adhesion and higher oxidation resistance.

1. Introduction

Zirconium alloys have been generally used as fuel cladding materials in nuclear reactors owing to the low thermal neutron absorption cross sections, excellent stability of irradiation, good compatibility with coolants and fuels, and favorable mechanical properties [1, 2]. However, they are prone to fail due to the high temperature oxidation [3, 4]. In addition, the high surface roughness also shortens the service life of the fuel cladding materials. In order to slow down the oxidation failure, it is effective to prepare the coating with high oxidation resistance on the surface of zirconium alloy.

Physical vapor deposition (PVD) is an advanced technology to obtain various high-quality coatings under the low-temperature condition [5, 6]. The coatings prepared by PVD show the merits such as high purity and compact texture [5–7]. As well known, MS and MAIP are the two most common methods of the PVD technology. The MS method has the superiorities of low deposition temperature and low surface roughness, but the MS coating has a relatively weak bonding strength [8–10]. The MAIP technology has the superiorities of high deposition rate and high ionization rate, but the surface roughness of the MAIP coating become relatively high due to the effect of macroparticles [11–14]. In the past decades, various high-performance coatings including composite coating and multi-layer coating have been prepared by the MS and MAIP methods [15]. In terms of the multi-layer coatings, the strengthening effect of the interface between layers can greatly reduce the internal stress of coatings, decrease the occurrence and expansion of the cracks, increase the oxidation resistance, and obtain better adhesion [15–20]. Among the ceramic coating, the TiN coating is effective in protecting the Zircaloy-4 (Zr-4) alloy tubes from being oxidized in the pressurized water reactor environments [21, 22]. For the metallic coating in the accident tolerant fuel cladding, the Cr coating has been widely studied due to the high-temperature oxidation resistance, along with its ascendant binding ability [23–25]. Therefore, a Cr layer often serves as a transition layer to reduce the residual stress between the coatings and the substrates, thereby...
enhancing the bonding strength of them \[24, 25\]. Based on the above consideration, we aimed to deposit the multi-layer coating of Cr/TiN by the PVD method for the sake of prolonging the service life of zirconium alloy.

The effects of different interlayer preparation processes on the microstructure and properties of the Cr/TiN coating have been carefully investigated. For the convenience of the following description, MS/MS represents a method to prepare the Cr interlayer by MS and the TiN layer by MS. Similarly, MAIP/MS represents a method to prepare the Cr interlayer by MAIP and the TiN layer by MS.

2. Experimental

The Cr/TiN coatings were deposited on the surface of Zr-4 alloy by the multi-function sputtering system (TSU-650). The dimension of samples is 20 mm × 20 mm × 5 mm. The Cr interlayers were prepared by mid-frequency magnetron sputtering and multi-arc ion plating, respectively. The corresponding targets are Cr-rectangular-target (500 mm × 80 mm × 4 mm) and the Cr-circular-target (Φ100 mm × 18 mm), and the purity of targets is 99.99%. The TiN layers were prepared by direct-current magnetron sputtering, and the target is TiN-circular-target (Φ100 mm × 18 mm) with 99.99% purity. Before the deposition of Cr/TiN coatings, the samples were treated with the following steps: First, the samples were mechanically ground and polished; Second, the samples were cleaned ultrasonically in the alcohol solution for 45 min, and then were dried; Third, the samples were placed in the vacuum chamber, and then were pre-sputtered (160 sccm argon flow, −800 V bias, 50% bias duty ratio) for 10 min. Based on our previous work \[25, 26\], the optimized process parameters of different Cr/TiN coatings are shown in tables 1 and 2, respectively.

The scanning electron microscopy (SEM, CARL ZEISS EVO 18) was used to characterize the surface and cross-section morphology of Cr/TiN coatings. The energy dispersive spectroscopy (EDS, OXFORD X-MAX) was used to analyze the element composition of the surface particles. The X-Ray Diffraction (XRD, D/max-2550 VL P^{-1}C^{-1}) was applied for the analysis of phase constituents. For the tests of the surface roughness and the bonding strength, the mechanical roughness tester (RC50H) and the scratch tester (WS-2005) were employed, respectively. The high-temperature oxidation resistance was tested in the furnace (SX2-2.5-10) at 800 °C, and the oxidized sample was weighed and recorded three times every 30 min by the electronic balance (BS210S) with an accuracy of 0.1 mg. In this way, the average oxidation weight gain of the sample was determined.

3. Results and discussion

3.1. Coating morphology

Figure 1 is the original surface morphologies of the two types of Cr/TiN coatings. The Cr/TiN coating prepared by MS/MS exhibits a relatively flat surface (figure 1(a)), while many macroparticles are visible on the surface of the Cr/TiN coating prepared by MAIP/MS (figure 1(b)). The point EDS analyses were performed and the results are shown in table 3. It can be seen that the elemental mass fractions of each point are similar. Given that the TiN layers were prepared under the same process parameters, the macroparticles particles were formed in the processes of preparing Cr interlayer. The macroparticles are the inherent features of MAIP due to the following reasons \[27–29\]. When the arc-current density is excessively high or the duration of the electric arc on the same position is too long, the target will be melted and transferred directly to the surface of substrate in the form of

| Table 1. Deposition parameters for Cr interlayers prepared by different methods. |
|-------------------------------|-------------------------------|
| Parameters for magnetron sputtering | Parameters for multi-arc ion plating |
| Working temperature | Working temperature |
| 360 °C | 400 °C |
| Working current | Working current |
| 5 A | 65 A |
| Substrate bias | Substrate bias |
| −200 V | −200 V |
| Bias duty ratio | Bias duty ratio |
| 50% | 50% |
| Deposition time | Deposition time |
| 8 h | 3.5 h |
| Nitrogen flow | Nitrogen flow |
| 0 sccm | 0 sccm |
| Argon flow | Argon flow |
| 40 sccm | 110 sccm |
| Distance from target to substrate | Distance from target to substrate |
| 10 cm | 16 cm |
| Working pressure | Working pressure |
| 1.5 Pa | 1.7 Pa |
micro droplets, so as to form the macroparticles [27]. Moreover, under the conditions of high temperature and high current density, the ionized plasma can easily be piled up on the surface of Zr-4 alloy, so as to form the macroparticles [28]. In addition, the trace of Cr was detected on the surface of coatings due to the residual Cr in the vacuum chamber.

Noticeably, the irregular holes can be observed for both the Cr/TiN coatings, as the dark spots in figure 1. This can be explained by two possibilities, one is that the micro-particles would peel off owing to the compressive stresses effect [29], and the other is that the subsequent atoms would be occluded by the biggish adjacent atoms due to the shadow effect [30]. By using the Image J software, the surface morphology and the statistics of holes in Cr/TiN coatings is shown in figure 2 and table 4, respectively. In table 4, the area fraction of holes in the MAIP/MS coating is 0.17%, which is 1/2 of that of the MS/MS coating. During the deposition process, the incident particles with the higher energy will have a greater migration probability. The migration can transfer the kinetic energy to other particles and cause re-sputtering phenomenon, thus causing the effects of
filling holes and reducing the hole numbers [31]. In MS, the energy of particles is only $1 \sim 10$ eV, which is much lower than the energy of particles in MAIP ($100 \sim 1000$ eV) [31]. Furthermore, the particles can diffract better in MAIP, which is beneficial to the particle diffusion on the surface of coatings [30]. Thus, the Cr/TiN coating prepared by the MAIP/MS method has smaller porosity than the Cr/TiN coating prepared by the MS/MS method.

Figure 3 presents the cross-sectional morphologies of the Cr/TiN coatings. The interface between the coating and the substrate is clear in figure 3(a), in contrast to the relatively blurry interface in figure 3(b). During the deposition of Cr interlayer, the plasma formed by MAIP affects the surface of substrate with higher energy, which brings about the increase of the substrate temperature. Consequently, the plasma penetrates into the substrate and thus results in a blurry interface [29].

### 3.2. Surface roughness

Figure 4 shows the surface roughness of the Cr/TiN coatings, with a testing length of 1.6 mm. The surface roughness of the MS/MS coating and the MAIP/MS coating is 0.064 $\mu$m (figure 4(a)) and 0.092 $\mu$m (figure 4(b)), respectively. In figure 4(b), the relatively large fluctuation degree is related to the macroparticles, which are formed in the process of preparing Cr interlayer by MAIP. The macroparticles can enhance the shadow effect, and make the low valley of the film surface be shaded. Therefore, the Cr/TiN coating prepared by MAIP/MS method has a higher roughness.

| The methods of Cr interlayers | Count | Total area ($\mu$m$^2$) | Average size ($\mu$m$^2$) | Area fraction(%) |
|-------------------------------|-------|------------------------|--------------------------|-----------------|
| Magnetron sputtering          | 186   | 8.377                  | 0.045                    | 0.34            |
| Multi-arc ion plating         | 81    | 4.197                  | 0.052                    | 0.17            |
3.3. Bonding strength

The bonding strength of coatings was tested by the scratch method. In this method, the critical load of the sample is determined by combining the acoustic emission signal, the friction signal and the scratch morphology [32–34]. That is also the normal force when the coating failed [34]. During the experiment, the length of the scratch was 4 mm, the normal force increased from 0 N to 50 N, and the loading speed of force was 50 N min\(^{-1}\). In figure 5, the frictional force increased approximately linearly and mutated when the coating began to fail. At the same time, the acoustic emission signal appeared, together with the inside and the edge of the scratch started to desquamate [35].

Figure 5(a) indicates that the critical load of the Cr/TiN coating prepared by MS/MS is 27.5 N. Similarly, figure 5(b) indicates that the Cr/TiN coating prepared by MAIP/MS has a critical load of 32.3 N. In figure 5(b), as the load increases, the acoustic signal is disturbed. This is because the indenter hits the macroparticles during the loading process [36]. Figure 6 shows the morphology of the Cr/TiN coating failure in the scratch test. The peeling area of the coating in figure 6(a) is larger than that in figure 6(b). It is further shown that the Cr/TiN coating prepared by MAIP/MS method has a better bonding strength. The zirconium matrix metal deformed greatly under the action of pressure, so that the load concentrated in the front half of the indenter during the sliding process. Therefore, the coating in the front part of the indenter bulged due to the extrusion deformation, thus resulting in the failure of the bonding area between the substrate and the coating.

The higher bonding strength of the Cr/TiN coating prepared by MAIP/MS can be ascribed to the following two main reasons: on one hand, the higher ionization rate of MAIP (80% ∼ 90%) can make the plasmas with higher energy and higher temperature, thus causing the increased diffusivity of sputtered atoms, which is beneficial to the enhancement of bonding strength [27, 37]; On the other hand, compared with the MS, the
directional acceleration of the magnetic field and the electric field in the MAIP can make the plasmas with higher mass velocity, thus causing the stronger bombardment intensity, which can make the coatings have the higher bonding strength with the Zr-4 alloy [38].

3.4. High-temperature oxidation resistance

Figure 7 shows the phase constituents of the substrate and Cr/TiN coatings after 6 h oxidation in the atmospheric environment. It can be seen that the phase ZrO₂ was formed by the reaction of substrate and oxygen in the air for the un-coated substrate. In contrast, the oxidized coatings are consisted of Cr, TiO₂ and Cr₂O₃, without the presence of TiN and ZrO₂ phases. This indicates that the outer TiN layer has been completely oxidized, the Cr interlayers were partially oxidized, and the Cr/TiN coatings served as the barrier to protect the substrate in the high-temperature environment.
**Table 5.** EDS analysis for substrate after 6 h oxidation.

| Number of the spectra | C   | O   | Zr   |
|-----------------------|-----|-----|------|
| Spectra A1            | 3.75| 13.29| 82.96|
| Spectra A2            | 4.80| 16.25| 78.94|
| Spectra A3            | 3.35| 16.83| 79.81|
| Spectra A4            | 3.69| 13.48| 82.83|
| Average               | 3.90| 14.96| 81.14|

**Table 6.** EDS analysis for Cr/TiN coatings after 6 h oxidation.

| Number of the spectra | C   | O   | Ti  | Cr  |
|-----------------------|-----|-----|-----|-----|
| Spectra 1             | 1.22| 17.03| 59.45| 22.29|
| Spectra 2             | 0.88| 17.15| 59.81| 22.16|
| Average               | 1.05| 17.09| 59.63| 22.23|
| Spectra 3             | 3.6 | 25.30| 56.20| 14.90|
| Spectra 4             | 1.35| 25.03| 57.13| 16.49|
| Spectra 5             | 0.95| 20.96| 55.11| 22.98|
| Average               | 1.97| 23.76| 56.15| 18.12|

**Figure 8.** The surface morphology (a) and cross-sectional morphology (b) of the substrate after 6 h oxidation.

**Figure 9.** Surface morphologies of the Cr/TiN coatings with the Cr interlayers prepared by magnetron sputtering (a) and multi-arc ion plating (b) after 6 h oxidation.
Figure 8 is the surface morphology and the cross-sectional morphology of the substrate after 6 h oxidation. To further examine the composition of the oxide layer, the EDS point analyses were carried out and the results are listed in Table 5. Obviously, the oxide layer is mainly ZrO₂. In Figure 8(b), the thickness of the oxide layer is about 20 μm. As well known, the holes in the oxide layer as seen in Figure 8(a) provide channels for the oxygen diffusion, so that the oxide film of the substrate is thicker than that of the Cr/TiN coatings. Figure 9 is the surface morphologies of the Cr/TiN coatings after 6 h oxidation. Compared with the oxidized Cr/TiN coating prepared by MS/MS, the oxidized Cr/TiN coating prepared by MAIP/MS exhibits less loose granular structures without visible holes. It indicates that a denser oxide film was formed after the oxidation of the MAIP/MS coating. To further determine the composition of the oxide, EDS point analyses were performed and the results are listed in Table 6, the oxides of the MAIP/MS-coating contain a higher oxygen (O) content and a lower chromium (Cr) content. This indicates that the diffusion of oxygen can be more effectively prevented by the dense oxide film formed after the oxidation of MAIP/MS-coating, thereby reducing the oxidation rate of the Cr interlayer.

Figure 10 shows the cross-sectional morphologies and EDS line-scan analyses of the Cr/TiN coatings with the Cr interlayers prepared by magnetron sputtering (a) and multi-arc ion plating (b) after 6 h oxidation.

Figure 10. Cross-sectional morphologies and EDS line-scan analyses of the Cr/TiN coatings with the Cr interlayers prepared by magnetron sputtering (a) and multi-arc ion plating (b) after 6 h oxidation.
oxidation. It can be seen that both types of the Cr/TiN coatings have not fallen off, and can prevent the substrate from being oxidized. From the EDS line scanning results, the element diffusion occurs at the TiN/Cr interface and Cr/substrate interface after 6 h oxidation. In addition, the existence of holes in the coating provides channels for the diffusion of the element Zr and causes the element segregation phenomenon. Moreover, a trace Zr in the oxidized TiN-coating area was detected due to the higher porosity of the Cr/TiN coating prepared by MS/MS. Furthermore, the descending rate of the element O in the oxidized Cr/TiN coating prepared by MAIP/MS is faster than that in the oxidized Cr/TiN coating prepared by MS/MS. This indicates that the Cr/TiN coating prepared by MAIP/MS has higher high-temperature oxidation resistance.

Figure 11 is the oxidation weight gain curves of Cr/TiN-coated samples and substrate at 800 °C. The oxidation processes of the Cr/TiN-coated samples can be described as follows: (i) The first stage is the oxidation of TiN layer, which can form a dense TiO₂ oxide film, and lead to the slow oxidation weight gain process; (ii) The second stage is the oxidation of Cr interlayer after the complete oxidation of TiN layer, and the dense oxide (Cr₂O₃) can fill the pores in coatings, thereby further blocking the oxidation of samples [25]. During the high-temperature oxidation experiment, the monoclinic phase (ZrO₂) formed by the oxidation of substrate is porous and has no protective effect [39]. Consequently, compared with the dense oxide film formed by the oxidation of Cr/TiN coatings, the substrate possesses the much bigger weight gain. In addition, in comparison with the Cr/TiN-coated sample prepared by MS/MS, the Cr/TiN-coated sample prepared by MAIP/MS has the smallest weight gain, which is about 1/4 of that of the substrate. It indicates that this method can make the Cr/TiN coating possess better high-temperature oxidation resistance, which is related to its lower surface porosity (table 4). The holes attenuate the structural stability of coatings, provide channels for oxygen to diffuse into the interior of coatings, and make it easier to form an oxide film [38, 40, 41]. Therefore, the oxidation of coatings is intensified.

4. Conclusion

(1) The Cr/TiN coating prepared by MS/MS exhibits a flat surface with higher porosity. The surface roughness of the Cr/TiN coating prepared by MAIP/MS become high due to the existence of macroparticles.

(2) The bonding strength of the Cr/TiN coating prepared by MAIP/MS is enhanced owing to the high energy for MAIP, and the interface between coating and substrate become blurry.

(3) After 6 h oxidation, the dense Cr₂O₃ and TiO₂ significantly prevent the further diffusion of the element oxygen. The holes and granular structures appear on the surface of the Cr/TiN coating prepared by MS/MS, exhibiting higher oxidation weight gain than the Cr/TiN coating prepared by MAIP/MS.
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