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ARTICLE

Characteristics of Transition Layer at Soft Metal-Substrate Interface for Metal Seal

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Abstract: The pressure-actuated metal seal with soft metal coating has been widely used in complex working conditions such as high temperature, low temperature and high pressure. The investigation of the characteristics and binding strength of the transition layer between the soft metal coating and the superalloy substrate is important to improve the sealing performance and to model and simplify the working through-process of metal sealing. The distribution characteristics of elements at soft metal-substrate interface and the binding strength between coating and substrate under different thicknesses and material combinations of coating layer were studied by experimental methods. The results indicate that the thickness of soft metal coating has little influence on the interface morphology of GH4169-Cu, GH4169-Ag and Cu-Ag, but has an influence on the thickness of transition layer between different metals, while this influence is weakened with increasing the coating thickness, and the thickness of transition layer is about 2 μm when the coating thickness is more than 30 μm. The cross-cut test shows that the Cu, Ag and Cu-Ag coatings are all well combined with nickel-based superalloy GH4169 substrate. The materials of soft metal, i.e. the coating materials, have significant influence on the characteristic of transition layer and the surface characteristics of coating after cross-cut test.

Key words: pressure-actuated metal seal; nickel-based superalloy; copper coating; silver coating; element transition layer

The pressure-actuated metal seal is widely used in the complex working conditions, such as high temperature, low temperature and high pressure, for mechanical devices such as liquid rocket engine[1–3]. The nickel-based superalloy GH4169 (i. e., Inconel 718 alloy) has a wide service temperature range, and has a good comprehensive performance in −253–650 °C[4–6], which is a common material used in the metal seal of liquid rocket engine[3,7]. In general, the surface of metal seals is coated with soft metal that has good plasticity[2,8]. The plastic deformation is able to change the surface topography characteristics[9–13], and the micro-plastic deformation by soft metal coating can ensure good meshing between sealing surfaces[1–2].

Up to now, most of the research on superalloy surface coating is focused on surface modification to improve the characteristics of lubrication, wear resistance and corrosion resistance at high temperature. The distribution and content of elements in the coating have an important influence on these properties. Cai et al[14] experimentally studied the influence of particle size of CrAlY powder on CrAlY content in electrodeposited Ni-CrAlY coating of GH4169. Kong et al[15] analyzed the distribution characteristics of Al, Ti, Si and N elements in TiAlSiN coating on surface of GH4169 alloy by cathodic arc ion plating.

The high-temperature protective coating on the surface of GH4169 alloy is also widely used, and there are various coating materials. Dong et al[16] studied the microstructure and oxidation characteristics at 800 °C of Al_2O_3/Cr composite coating on the surface of Inconel 718 alloy. Han et al[17] studied the performance of CoCrAl coating on the surface of

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GH4169 alloy by plasma thermal spraying, and found that the thermal shock resistance of the modified coating after cold and hot cycling process is significantly improved. Zheng et al[18] studied the transformation behavior of YSZ@Ni core-shell anoparticles in YSZ@Ni coating on the surface of GH4169 alloy by laser cladding.

There are many studies on the improvement in lubrication and wear resistance of coating on the surface of GH4169 alloy. Zhou et al[19] studied the property of plasma-sprayed cermet coating on the surface of Inconel 718 alloy, and the tribological properties of two Cr_3C_2/NiCr coatings at the used temperature from room temperature to 800 °C were studied. Mu et al[20] studied the lubrication behavior of multilayer VN-VN/Ag composite coating on surface of Inconel 718 alloy at 25–700 °C. Fang et al[21] studied the diffusion and dissipation behavior of Ag in NiCrAlY/Ag coating on surface of Inconel 718 alloy under sliding wear from room temperature to 800 °C.

In addition, soft metal coating with pure silver or pure copper has seldom been found in the abovementioned studies. The silver was used as a bonding layer to connect Ni superalloy HAYNES 214 and silicon carbide (SiC) ceramic by Hattali et al[22]. The results show that reaction between SiC and Ni is significantly avoided by the silver layer, but the thin silver layer (2 μm with obvious uneven coating) has limited effect, and coating with more than 50 μm in thickness will be more effective.

However, for pressure-actuated metal seal, the surface of nickel-based superalloy GH4169 is coated with a soft metal such as copper or silver that has good plasticity, and the function of the coating in this case is completely different from the abovementioned studies. Additionally, depending on the coating material and its service function, the coating is either too thin, such as only 2 μm for TiAlSiN coating thickness[15], or too thick, such as CoCrAl coating with a thickness of 160 μm[17] or Ag foil layer with a thickness of 200 μm[22], in abovementioned studies. It is significantly different from the thickness of soft metal coating on surface of metal sealing structures. In addition, there is no post-plating heat treatment for the soft metal coating, the element diffusion behavior between the soft metal coating and the substrate is weak, and element transition phenomenon is more obvious. The function of the soft metal coating in pressure-actuated metal seal is completely different from the coatings in the abovementioned studies. During the working process of metal seal, the soft metal undergoes deformation and element migration, where soft metal elements migrate to the surface of the mating flange[2].

Furthermore, the characteristic of transition layer at interfaces between soft metal and substrate determines constraint mode between coating layer and substrate in finite element analysis for pressure-actuated metal seal. This study focused on characterizing transition layer from soft metal to substrate, and provided a basis for determining the thickness of soft metal coatings on metal seals and modeling the soft metal layers in finite element analysis. Therefore, in this study, the distribution characteristics of elements at soft metal-substrate interface under Cu plating, Ag plating and Cu-Ag composite plating cases were analyzed. The binding strength between coatings and substrate was evaluated by cross-cut test, and the influence of thickness of coating layer on transition layer characteristics at interface between soft metal and substrate was studied.

1 Experiment

In order to improve the sealing performance, the pressure-actuated metal seal used in extreme working conditions such as aerospace was coated with soft metals[1–2], such as copper, silver, gold and other soft metals, as shown in Fig. 1. According to the characteristics of the coating and the substrate, the substrate samples with 15 mm in length, 6 mm in width and 5 mm in thickness were extracted and designed, and a hole with 2.5 mm in diameter was formed on one side of the substrate sample to facilitate the lifting during plating. Soft metal material was electroplated on the sample by a mature electroplating process. According to the practical case of metal seal, electroplating process was the last process, so there is no heat treatment process after electroplating process.

The material of substrate used in this experiment was nickel-based superalloy GH4169, which was manufactured by Gaona Aero Material Co., Ltd according to standard GJB713-89. The material of soft metal coating was copper or silver. Based on the experience for metal seals, the thickness of coating was generally 10–40 μm. In order to enhance the surface activity of the substrate, a nickel layer with 5 μm in thickness was electroplated on GH4169 before the soft metal plating.

The main processing parameters of silver plating are as follows: silver concentration in the plating solution was about 30 g/L; pH=12–12.5 for the plating solution; current density was about 1 A/dm²; plating time was calculated according to thickness of plating layer with plating speed of 100 s/μm. The main processing parameters of copper plating were as follows: copper concentration in the plating solution was about 25 g/L; pH=10–10.5 for the plating solution; current density was 1–1.8 A/dm²; plating time was calculated according to thickness of plating layer with plating speed of 270 s/μm.

Single metal coating and multi-metal composite coating were both used in industrial processes, so three combinations of substrate and coating materials were adopted in this study, including the single metal coatings of Cu layer and Ag layer,

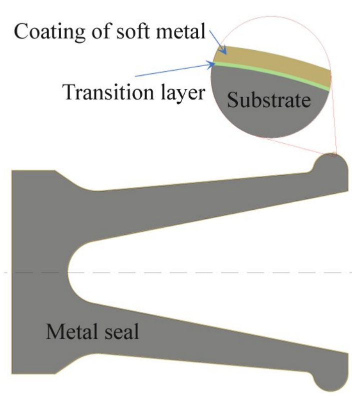

Fig.1 Sketch of coating of soft metal for metal seal

and the multi-metal composite coating such as pre-plated Cu followed by Ag plating, as listed in Table 1. When composite coating was adopted, the pre-plated layer has little influence on the service performance of metal seal. Thus, the thickness of pre-plated layer remained unchanged at 10 μm in this study. The experimental plans for the match between substrate and coating material and the thickness of coating layer are listed in Table 1. The substrate samples were activated on the surface and then electroplated according to the planned order and thickness of soft metal coatings.

After surface activation of the substrate samples, the test samples were individually electroplated according to the order and thickness of the soft metal coatings listed in Table 1. The morphology and elemental distribution characteristics at interface between coating and substrate were observed by scanning electron microscope (SEM) and energy disperse spectrometer (EDS). According to standard GB/T 5270-2005, the binding situation between coating and substrate was analyzed by cross-cut test.

2 Results and Discussion

2.1 Morphology of interface between soft metal coating and substrate

Fig.2–Fig.4 illustrate the interface morphology and elemental distribution characteristics on cross section under various combinations of soft metals and coating thicknesses, in which element Ni represents the GH4169 substrate material.

Table 1 Coating thicknesses of single metal and multi-metal composite (μm)

Thickness	Cu	Ag	Cu-Ag
Pre-plated Cu	-	-	10
Soft metal coating	10	10	-
	20	20	20
	30	30	30
	40	40	40

For the copper plating, the boundary between GH4169 and Cu cannot be observed from the cross section morphology, as shown in Fig.2a_1–2d_1. The reasons are that colors of GH4169 alloy and copper are similar in the image and they are well combined without any obvious defects. The element distribution shows that elements Ni (i.e., GH4169 alloy) and Cu are uniformly distributed in both the substrate and the coating, and there is a clear boundary between them. When the thickness of copper coating varies from 10 μm to 40 μm, the interface characteristics between GH4169 alloy and Cu remain consistent and relatively smooth. However, when the thickness of Cu coating reaches 40 μm, the degree of unevenness on the outer face of Cu coating is significant, as shown in Fig. 2d_1 and 2d_3.

The cross section morphology of GH4169 substrate/Ag coating is different from that of GH4169 substrate/Cu coating. The boundary between GH4169 alloy and Ag is distinct, as shown in Fig. 3a_1 – 3d_1. The darker color in SEM image represents the GH4169 substrate, and the lighter color represents the Ag coating. The cross section morphology and the element distribution both show that the combination between GH4169 alloy and Ag is relatively dense, and there is no defect. The element distribution shows that elements Ni (i.e., GH4169 alloy) and Ag are uniformly distributed in both the substrate and the coating, and there is a clear boundary between the two elements. When the thickness of silver coating varies from 10 μm to 40 μm, the interface characteristics between GH4169 alloy and Ag remain consistent and relatively smooth, and the outer face of Ag coating is also relatively smooth.

For the composite coating of pre-plated Cu with 10 μm in

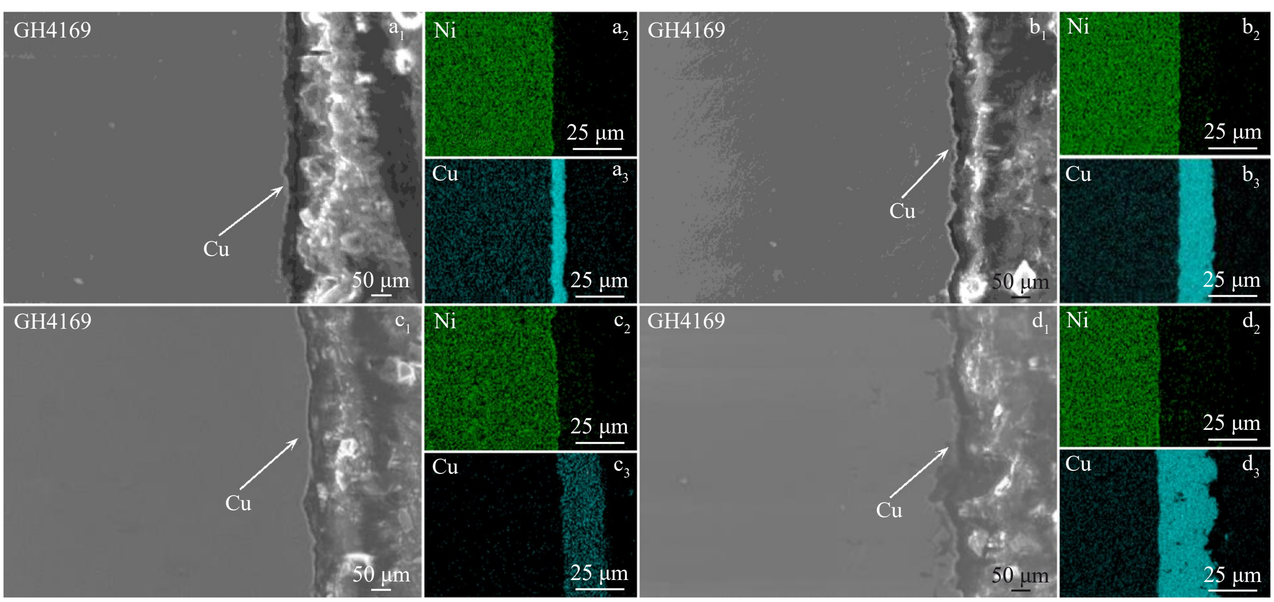

Fig.2 SEM interface morphologies (a_1 – d_1) and EDS element mappings (a_2 – d_2, a_3 – d_3) of GH4169 with different thicknesses of Cu coating: (a_1–a_3) 10 μm, (b_1–b_3) 20 μm, (c_1–c_3) 30 μm, and (d_1–d_3) 40 μm

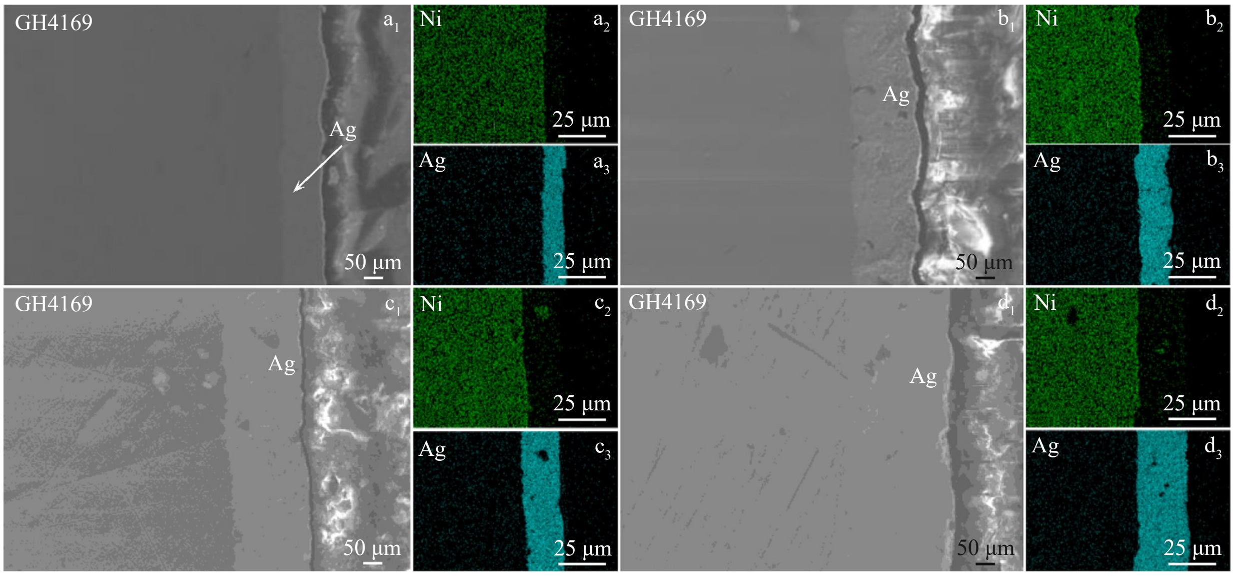

Fig.3 SEM interface morphologies (a_1–d_1) and EDS element mappings (a_2–d_2, a_3–d_3) of GH4169 with different thicknesses of Ag coating: (a_1–a_3) 10 µm, (b_1–b_3) 20 µm, (c_1–c_3) 30 µm, and (d_1–d_3) 40 µm

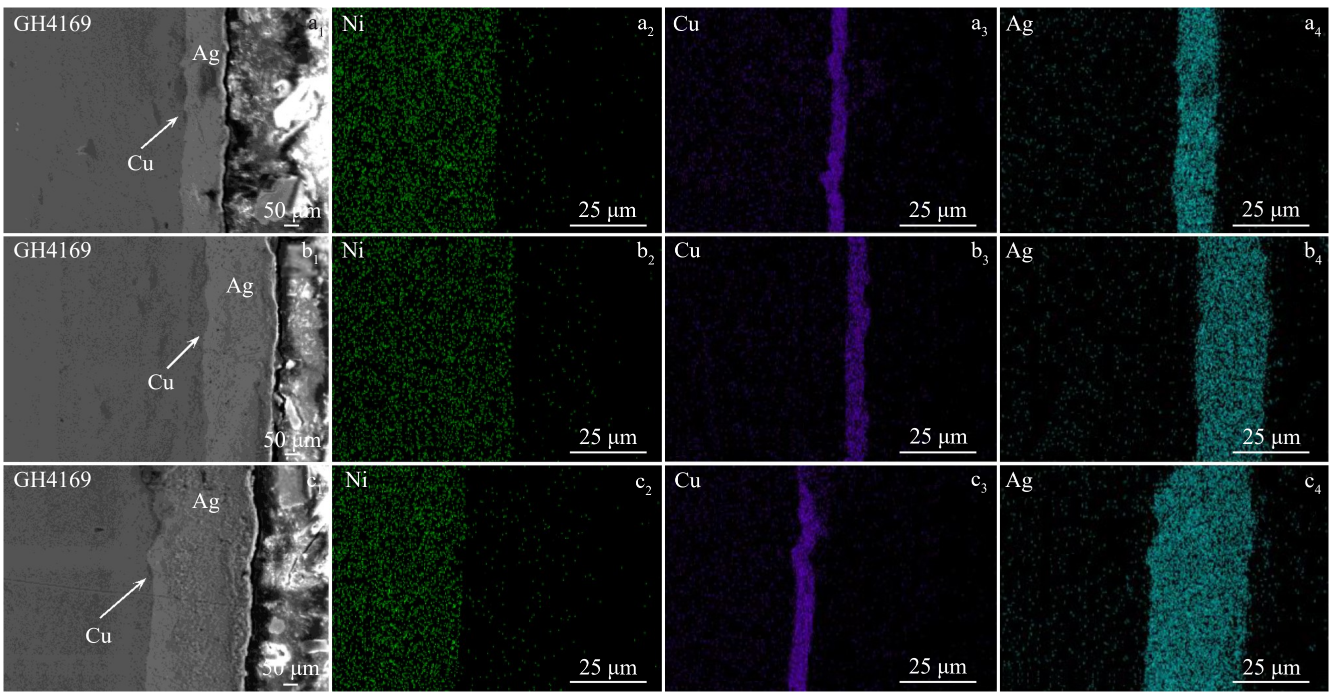

Fig.4 SEM interface morphologies (a_1–c_1) and EDS element mappings (a_2–c_2, a_3–c_3, a_4–c_4) of GH4169-Cu with different thicknesses of Ag coating: (a_1–a_4) 20 µm, (b_1–b_4) 30 µm, and (c_1–c_4) 40 µm

thickness and Ag coating with 20–40 µm in thickness, the interface morphology of the copper coating and silver coating on the cross section is consistent with the interface morphologies of the single coating in Fig. 2 and Fig. 3. It is difficult to distinguish the boundary between GH4169 substrate and copper coating, but the silver coating can be clearly seen, and then the boundary between Cu coating and Ag coating can be distinguished, as shown in Fig. 4a_1–4c_1. It can be seen from EDS mapping that the Cu coating is located between the GH4169 substrate and the Ag coating, each element is evenly distributed in the corresponding zones, the boundaries between each zone are complete and clear, and the interface between different elements is relatively smooth. The thickness of the silver coating has little influence on the Cu-Ag interface and the morphology of the outer face of silver coating.

2.2 Transition phenomenon at interface and parameter influence

The characteristics of element change and concentration distribution along scanning direction can be characterized by

EDS line scan. The variation of element concentration can be used to study the elements transition or elements diffusion between substrate and coating, and the abrupt-change region of element concentration can be regarded as the transition layer or diffusion layer. The EDS line scan was performed on the test samples from inside to outside (substrate to coating) under different combinations of soft metals and coating thicknesses (Table 1). Fig. 5–Fig. 7 illustrate the distributions of element concentration, in which GH4169 substrate is still represented by element Ni.

It can be seen from the EDS line scan results that there is an abrupt-change region for element concentration between GH4169 alloy and Cu coating, as shown in Fig. 5, which can be regarded as the mutual transition/diffusion region of elements Ni and Cu. The thickness of transition region is defined as L. In this region, as the scanning distance increases from inside to outside, the peak intensity of element Ni gradually decreases and finally tends to 0. However, the intensity of element Cu gradually increases and finally is basically equal to the intensity of element Cu in the coating.

Similarly, there is an abrupt-change region (L in thickness) for element concentration between GH4169 alloy and Ag coating, as shown in Fig. 6. In this region, as the scanning distance increases from the inside to the outside, the peak intensity of element Ni gradually decreases and finally tends to 0, while the intensity of element Ag gradually increases and finally is basically equal to the intensity of element Ag in the coating.

Similar to the single soft metal coating, there is an abrupt-change region (L_1 in thickness) for element concentration between GH4169 alloy and Cu coating, and an abrupt-change region (L_2 in thickness) for element concentration between Cu coating and Ag coating for the copper-silver composite coating, as shown in Fig. 7. As the scanning distance increases from the inside to the outside, the peak intensity of element Ni gradually decreases and finally tends to 0. The intensity of element Cu gradually increases, reaches a stable level, then decreases and tends to 0. The peak intensity of element Ag gradually increases and reaches a stable level that is the same as the intensity of element Ag in Ag coating.

Fig. 8 illustrates the variation of thickness of transition layers with increasing the thickness of soft metal coating. For the single metal coating, with increasing the thickness, the thickness of the transition layer between GH4169 alloy and Cu coating (L_{Cu}) decreases rapidly at first, and then increases slowly. However, the thickness of the transition layer between GH4169 alloy and Ag coating (L_{Ag}) is basically unchanged at first and then increases. When the thickness of soft metal coating increases from 10 μm to 20 μm, the L_{Cu} decreases by nearly 0.7 μm, and the changing rate approaches −30%. However, L_{Ag} is almost unchanged and slightly smaller than L_{Cu}. When the thickness of soft metal coating increases from 20 μm to 40 μm, the L_{Cu} increases by nearly 0.3 μm, and the changing rate is nearly 20%. L_{Ag} increases by about 0.4 μm, the changing rate is nearly 25%, and L_{Ag} is generally larger than L_{Cu}. The thickness (L) of the transition layer is basically about 2 μm. With the increase in coating thickness, the influence of the coating thickness on the thickness of transition layer is weakened and negligible.

Compared with the single soft metal coating, the composite coating has some influence on the transition/diffusion behavior of copper and silver. The expected thickness of pre-

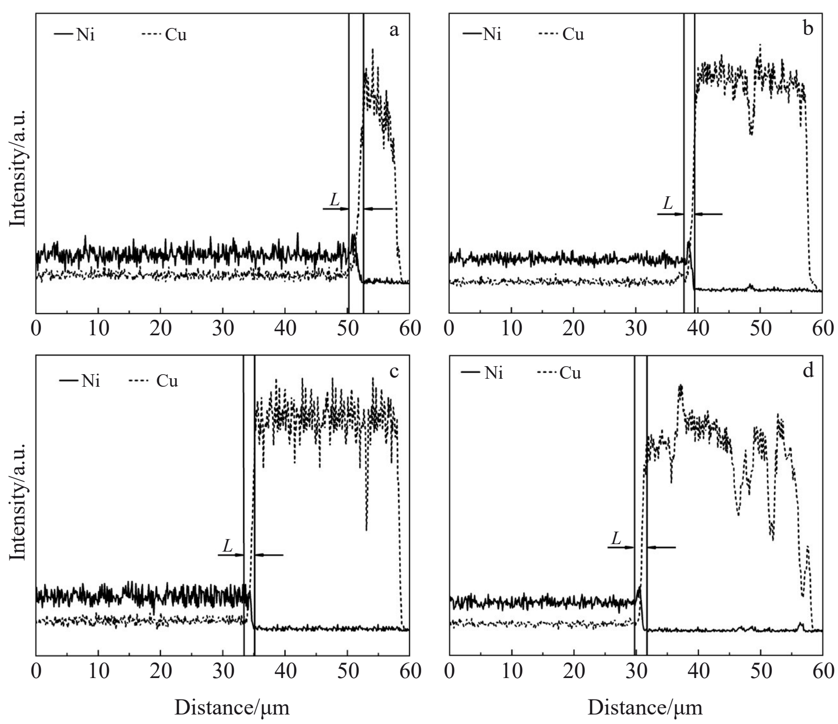

Fig.5 EDS line scan results for GH4169 with different thicknesses of Cu coating: (a) 10 μm, (b) 20 μm, (c) 30 μm, and (d) 40 μm

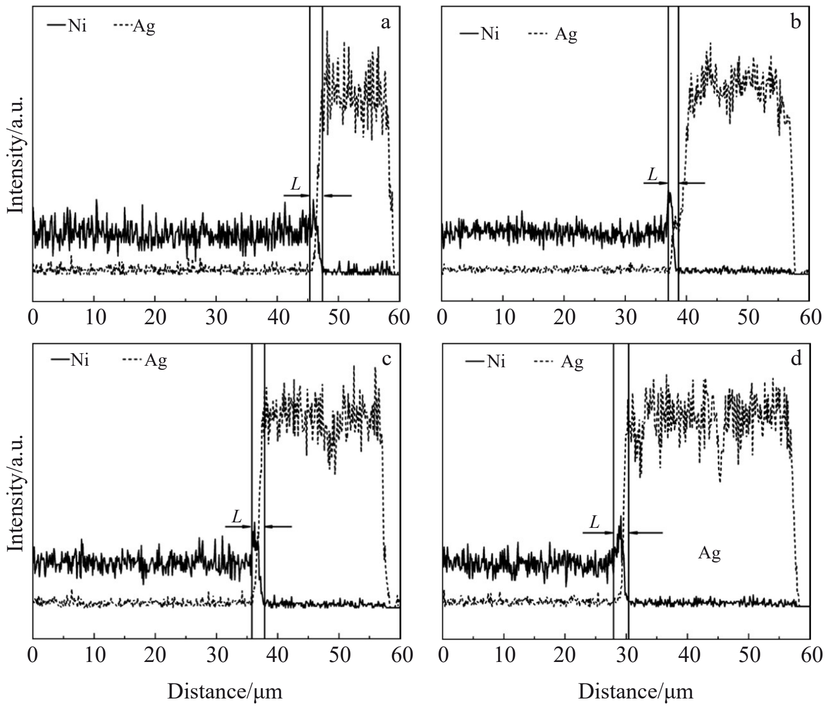

Fig.6 EDS line scan results for GH4169 with different thicknesses of Ag coating: (a) 10 μm, (b) 20 μm, (c) 30 μm, and (d) 40 μm

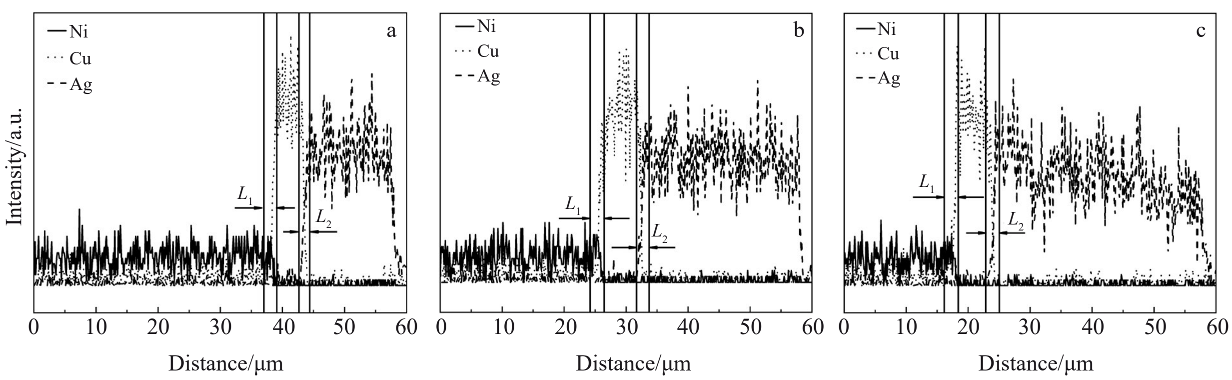

Fig.7 EDS line scan results for GH4169-Cu with different thicknesses of Ag coating: (a) 20 μm, (b) 30 μm, and (c) 40 μm

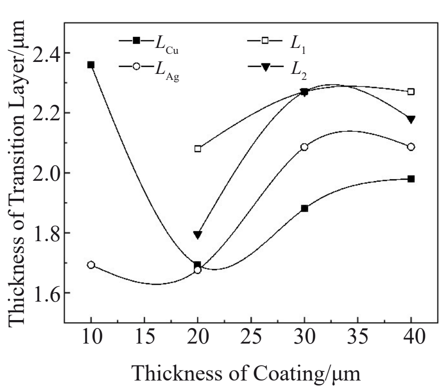

Fig.8 Influence of thickness of coatings on transition layer

plated Cu is constant. The thickness (L_1) of the transition layer between GH4169 alloy and Cu coating is about 2.2 μm with a variation of ±5%, which is less than L_{Cu} with the same coating thickness. With the increase in the thickness of Ag coating, the variation trend of thickness of the transition layer (L_2) between Cu coating and Ag coating for the multi-metal composite coating is similar to that of the single Ag coating, but L_2 is greater than L_{Ag}. Although there is a decrease in L_2 when the thickness of Ag coating increases from 30 μm to 40 μm, the decrement is very small, which is about −4%.

2.3 Binding strength between coatings and substrate

Fig. 9 – Fig. 11 illustrate the results of cross-cut test under different combinations of soft metals and coating thicknesses. The straight line in the figures is the grid area by the knife. It is bright, indicating that the knife penetrates the substrate. However, the other area is darker, which is the coating areas. After the cross-cut test, there will be a material accumulation on the edge grid area of Cu coating, and the accumulation of coating material will become more obvious with increasing the thickness of the coating, as shown in Fig.9. However, the

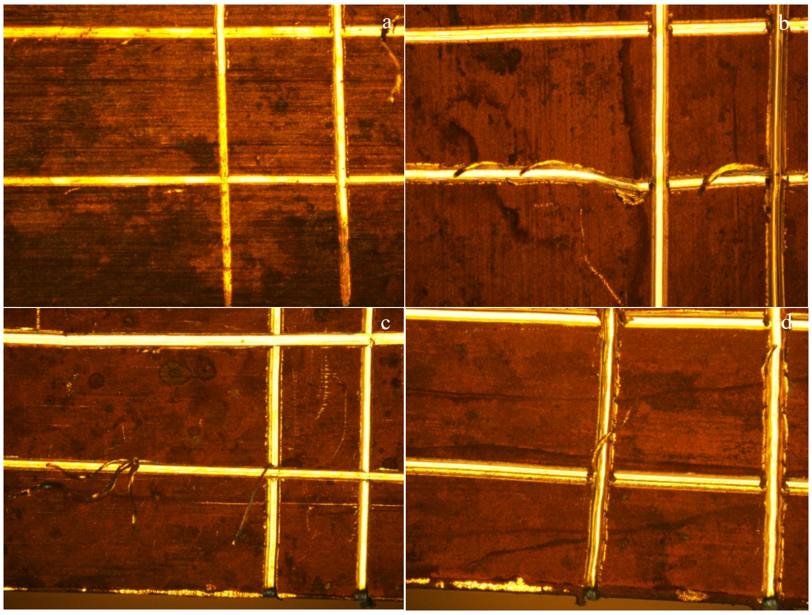

Fig.9 Results of cross-cut test for GH4169 with different thicknesses of Cu coating: (a) 10 μm, (b) 20 μm, (c) 30 μm, and (d) 40 μm

middle area among the grids is relatively flat and the phenomenon of coating peeling off the substrate is not observed. It indicates that Cu coatings with different thicknesses and the substrate are well combined.

Similar to the Cu coating, the phenomenon of material accumulation on the edge grid area after the cross-cut test is also presented for Ag coating. The accumulated phenomenon also becomes more obvious with increasing the thickness of the coating, as shown in Fig.10. However, the degree of material accumulation for Ag coating is less than that for the Cu coating, and the smoothness of the grid area is better. The middle area within the grids is flat for different thicknesses of Ag coating, indicating that the coating is well combined with the substrate. The phenomenon of coating peeling off the substrate is not observed for Ag coatings with different thicknesses.

The similar characteristics are also presented for the Cu-Ag composite coating. Although Cu coating and Ag coating cannot be distinguished visually, the phenomenon of material accumulation on the edge grid area after the cross-cut test is also presented. With the increase in the thickness of the composite coating, the material accumulation becomes more obvious, and the middle area within the grids is flat and the phenomenon of coating peeling off the substrate is not observed, as shown in Fig. 11. The degree of material

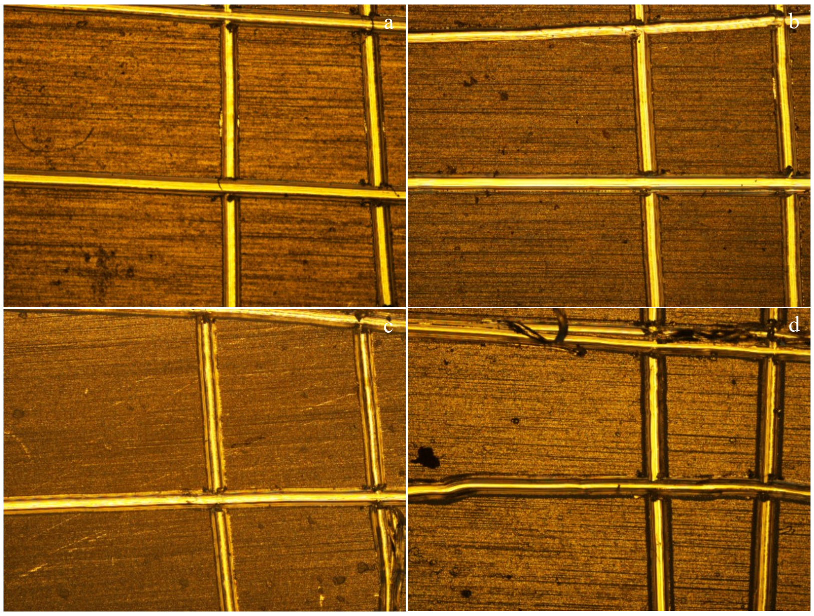

Fig.10 Results of cross-cut test for GH4169 with different thicknesses of Ag coating: (a) 10 μm, (b) 20 μm, (c) 30 μm, and (d) 40 μm

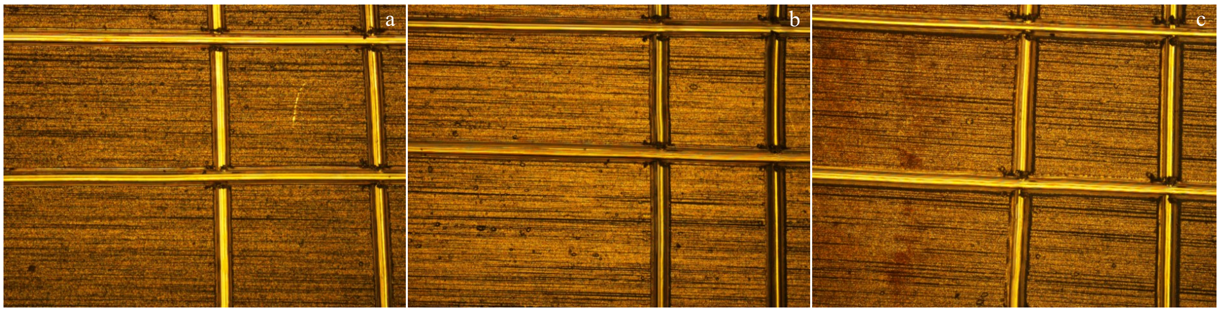

Fig.11 Results of cross-cut test for GH4169-Cu with different thicknesses of Ag coating: (a) 20 μm, (b) 30 μm, and (c) 40 μm

accumulation for composite coating is less than that for the silver coating, and the smoothness of the grid area is also better.

3 Conclusions

1) The thickness of soft metal coating has little influence on the morphology of the interface of GH4169-Cu, GH4169-Ag and Cu-Ag, and two metal layers are well combined and have a smooth interface. When the thickness of the copper coating is large (such as 40 μm), the degree of unevenness on the outer face of the coating is significant. However, the thickness of silver coating has little influence on morphology of the outer face of the coating.

2) For the single metal coatings, the influence of thickness of coating on the thickness of transition layer depends on the coating material. When the thickness of coating is less than 20 μm, the thickness of copper transition layer (L_{Cu}) is much larger than the thickness of silver transition layer (L_{Ag}). When the thickness of coating is greater than 20 μm, L_{Cu} and L_{Ag} both increase with the increase in coating thickness, but $L_{Cu} < L_{Ag}$. Compared with the single soft metal coating, the multi-metal composite coating has a certain influence on the transition/diffusion behavior of copper and silver, which reduces the transition depth of copper and increases the transition depth of silver. When the thickness of coating is greater than 30 μm, the thickness of transition layer between different metal layers basically stabilizes at about 2 μm.

3) The cross-cut test indicates that the substrate and the coating are well combined. No peeling phenomenon of the coating from the substrate is observed for all three kinds of coatings, and the coating can play the expected role. However, material accumulation occurs on the edge grid area, and the accumulation degree increases with increasing the thickness of the coating. The accumulation degree of coating material for the Cu-Ag composite coating is less than that for the silver coating, and the accumulation degree of coating material for the silver coating is less obvious than that for the copper coating.

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金属密封软金属和基体界面过渡层特征

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摘　要：带有软金属镀层的自紧式金属密封广泛应用于高温、低温、高压等复杂工况。研究软金属镀层和高温合金基体界面过渡层特征及结合强度对提升密封性能和金属密封工作全过程建模与简化具有重要意义。采用试验方法研究了不同软金属镀层材料及厚度组合下软金属和基体界面元素分布特征和镀层-基体结合强度。结果表明：软金属镀层厚度对GH4169-Cu、GH4169-Ag和Cu-Ag界面形貌几乎没有影响，但对不同金属之间的过渡层厚度有影响；随着镀层厚度增加，该影响减弱，镀层厚度大于30 μm后，过渡层厚度稳定在2 μm左右。划格试验表明Cu、Ag和Cu-Ag复合镀层与基体GH4169均结合较好。软金属材料对过渡层特征和镀层划格试验后表面特征有显著影响。

关键词：自紧式金属密封；镍基高温合金；铜镀层；银镀层；元素过渡层

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