HIGH-TEMPERATURE OXIDATION BEHAVIOR OF AN N5 NANOCRYSTALLINE COATING DEPOSITED WITH ESD ON A Ni-BASED SINGLE-CRYSTAL SUPERALLOY

VISOKOTEMPERATURNA OKSIDACIJA NANO KRISTALINIČNE PREVLEKE VRSTE N5, NANEŠENE Z ESD POSTOPKOM NA POVRŠINO MONOKRISTALA NI SUPERZLITINE

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An N5 nanocrystalline coating was prepared on a Ni-based single-crystal superalloy with electrospark deposition. The morphology, chemical composition and phase constitution of the coating were analyzed with scanning electron microscopy (SEM), energy-dispersive spectrometry (EDS) and X-ray diffraction (XRD), respectively. The high-temperature oxidation resistance of the substrate and coating at $1100\,^{\circ}$ C was tested during a static isothermal oxidation experiment. The results show that the electrospark-deposited coating with a columnar crystal structure is nanocrystalline; there is no elemental interdiffusion between the substrate and the coating, effectively preventing a decrease in the mechanical properties of the substrate alloy due to interdiffusion. Ta has a different oxide morphology in the single-crystal superalloy and its nanocrystalline coating, and it improves the adhesion of the oxide film due to the pinning effect.

Keywords: high-temperature oxidation, nanocrystalline coating, electrospark deposition

V članku je opisana priprava in analiza obnašanja nanokristalinične prevleke vrste N5, nanešene na površino monokristala Ni superzlitine s postopkom električnega iskrenja (ESD; angl.: electrospark deposition). Morfologija prevleke, njena kemijska in fazna sestava, je bila analizirana s pomočjo vrstičnega elektronskega mikroskopa (SEM), energijske disperzijske spektroskopije (EDS) in rentgenske difrakcije (XRD). Odpornost prevleke in podlage proti visokotemperaturni oksidaciji je bila določena pri 1100 °C s pomočjo statičnega izotermičnega preizkusa. Rezultati analiz so pokazali, da je bila z ESD postopkom izdelana prevleka s stebričasto kristalno strukturo sestavljena iz nanokristalov. Pri tem ni prišlo do interdifuzije kemijskih elementov med podlago in prevleko, kar učinkovito prepreči poslabšanje mehanskih lastnosti podlage zaradi interdifuzije. Tantal (Ta) ima drugačno morfologijo oksida (TaO_x) v monokristalni superzlitini kot v nankokristalinični prevleki, kar izboljša adhezijo oksidnega filma zaradi učinka pripenjanja.

Ključne besede: visoko temperaturna oksidacija, nanokristalinična prevleka, postopek elektro-iskrilne depozicije

1 INTRODUCTION

Ni-based single-crystal superalloys are often used in turbine blades of aero-engines or jet engines because of their excellent mechanical properties and high-temperature oxidation resistance. The high-temperature oxidation resistance of an alloy is as important as its mechanical properties when used for blades in service under harsh conditions above 1100 °C. To avoid a loss of a turbine blade caused by high-temperature oxidation, it is common to apply a protective coating on its surface. However, due to the uneven temperature distribution in the parts of a blade, the loss of a blade surface caused by high-temperature oxidation is not uniform, which directly leads to a premature failure of the coating in specific areas, and the coating needs to be repaired. If all the coating is peeled off and the blade is recoated, then the

blade becomes thinner and its service life is shortened. Under normal circumstances, after a blade coating is peeled off 2–3 times, the blade cannot be used due to the loss of thickness. To avoid the above problems, a protective coating should be applied to localized areas of the blade, which can prolong the service life and avoid an accelerated loss of the blade.

At present, arc spraying and plasma spraying are used commonly to repair coatings, but these methods produce coatings that tend to form holes and cracks^{1,2} and easily peel off,³ resulting in a significant decrease in the oxidation resistance of a coating. To improve the resistance of the coating to defects, we chose the electrospark-deposition method. The characteristics of the electrospark-deposition method are as follows: the coating layer and the substrate are generally metallurgically bonded, the adhesion between the coating and the substrate is strong, high-temperature oxidation peeling does not occur easily,⁴ the size of the coated object is unlim-

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ited, which facilitates local repairs of workpieces,5 and the core of a workpiece hardly heats up, which prevents annealing or deformation of the workpiece. In general, the method is simple and convenient to perform, which makes it easy to repair the turbine blades of aero-engines and prolongs their service life. In 1999, a nanocrystalline coating with excellent adhesion and high-temperature oxidation resistance was proposed by Wang et al.6 At present, nanocrystalline coatings are widely used in the repair of aero-engine turbine blades. In the current experiment, an N5 nanocrystalline coating was deposited on a Ni-based single-crystal superalloy substrate with the electrospark-deposition method as a repair coating for aero-engine turbine blades. The advantages of electrospark deposition of an N5 nanocrystalline coating on a Ni-base single-crystal superalloy are as follows:

- (i) the deposited coating is tightly combined with the substrate, which can minimize the peeling of the coating;
- (ii) the substrate and coating have the same composition, which can effectively reduce element interdiffusion and prevent a decrease in the mechanical properties of the substrate;⁷
- (iii) the nanocrystalline coating has good adhesion, which can effectively improve the peeling resistance of the oxide film; and
- (iv) the nanocrystalline coating has rich grain boundaries, which can accelerate the diffusion rate of elemental Al, reduce the critical content of elemental Al in the surface film, and improve the oxidation resistance of the oxide film.⁶

Recently, Wang et al.7 investigated the oxidation behavior of a nanocrystalline coating sputtered on a nickel-based single-crystal superalloy, showing that the sputtered nanocrystalline coating improved the oxidation and scale-spallation resistance of the single-crystal superalloy. In contrast to the sputtering technology, the ESD technology has obvious advantages, such as strong adhesion, possibility of local repairs, no need for a large vacuum chamber, etc. However, to the best of the authors' knowledge, there is still no report about the oxidation resistance of an ESD nanocrystalline coating on a single-crystal superalloy of the same composition. Thus, in this paper, an N5 nanocrystalline coating was deposited on the surface of a Ni-based single-crystal superalloy substrate using the electrospark deposition method. The high-temperature oxidation resistance of the substrate and coating was tested during a high-temperature oxidation experiment. The micromorphologies, chemical composition and phase constitution of the coating were analyzed with SEM, XRD and EDS. The oxidation resistance of the N5 nanocrystalline coating and the effect of Ta on the oxidation resistance of the coating were discussed.

2 EXPERIMENT

The coating was prepared with the electrospark-deposition method. Both the substrate and deposition materials are Ni-based single-crystal superalloys. The main components of the materials are shown in Table 1. After quenching and tempering the Ni-based single-crystal superalloy, wire cutting was used to obtain a substrate with a size of 20 mm \times 10 mm \times 3 mm and a cylindrical electrode of ϕ 5 mm × 80 mm. Before the deposition, the samples were polished with sandpaper to ensure that the oxide film was completely removed and then they were cleaned with acetone to remove oily contaminants. The coating was deposited with SD-D5A electrospark-deposition equipment. The process was performed manually under argon for protection. Deposition parameters were as follows: a power of 4 gears, frequency of 6 gears, electrode speed of 5 gears, argon flow rate of 20 L/min, and alloy electrode specific deposition time of 1.5 min/cm².

The patterned substrate and the deposited material were polished with SiC sandpaper and placed in an oxidation crucible. Isothermal-oxidation experiments were carried out at 1100 °C in a muffle furnace. The weight change of the samples was measured with an electronic balance. The sensitivity of the balance was 0.1 mg, and the samples were weighed every 20 h to observe the oxidation rate. Before weighing, the samples were removed and cooled to room temperature. X-ray diffraction (XRD) was used to analyze the phase constitution of the samples; the morphology of the samples and elemental distribution in the cross-sections of the samples were observed with scanning electron microscopy (SEM).

Table 1: Composition of the Ni-based single-crystal superalloy N5 (w/%)

Elements	Ni	Co	Cr	Mo	W	Ta	Al	Re
Composition	Bal.	8.3	7.3	1.5	5.0	6.4	5.9	2.4

3 RESULTS AND DISCUSSION

According to the morphology of the N5 coating surface from Figure 1a, the coating surface is orange peel-like and relatively flat with many features that resemble melting craters of different sizes. When the electrode material and the substrate are in contact, creating a short circuit, a plasma arc with an extremely high temperature is instantaneously generated between the electrode and the substrate (the temperature can reach 5000-25000 K). The electrode material melts to form metal droplets at high temperatures. The plasma accelerates the splashing of the metal droplets on the substrate, where they cool down and solidify, forming the craterlike shapes shown in Figure 1a. The presence of many craters is the main reason for the extensive roughness of the coating deposited by ESD. Figure 1b shows the backscattering morphology of a cross-section where the

Table 2: Oxidation kinetics of the Ni-based single-crystal superalloy and N5 nanocrystalline coating at 1100 °C within specified time ranges

Temperature (°C)	Sample	Oxidation duration (h)	k (mg ² cm ⁻⁴ s ⁻¹)	Oxidation duration (h)	k (mg ² cm ⁻⁴ s ⁻¹)	
1100	Substrate	0-20	2.13×10^{-6}	20-100	4.05×10^{-7}	
	Coating	0-20	5.05×10^{-6}	20-100	6.75×10^{-7}	

thickness of the coating is approximately 40 μ m, and the interface between the coating and the substrate is clearly defined, resembling a metallurgical bond without cracks. As shown in **Figure 1c**, there are no microholes at the interface between the coating and substrate; the substrate has a typical γ/γ' structure, and the disordered cubic γ' phase is uniformly distributed in the matrix phase of the γ solid solution.

The morphology of the N5 coating etched with a CuSO₄ solution is shown in **Figure 1d**. After etching, the grains of the N5 coating are uniform ultrafine columnar grains (marked as arrows)⁸ of a nanoscale size. The N5 coating deposited with ESD is a nanocrystalline coating. The formation of a nanocrystalline coating shows that grain refinement occurs during the process of ESD deposition. The electrode material is heated to the melting point in a very short time and then it rapidly cools. The cooling rate of the coating may be reduced to

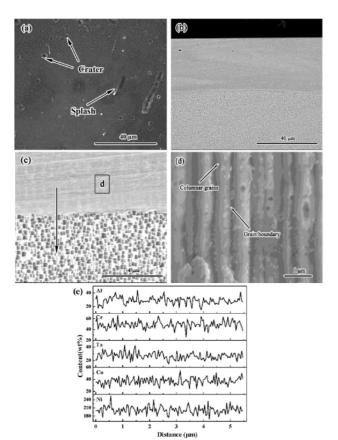


Figure 1: Morphology of the ESD N5 nanocrystalline coating: a) surface morphology, b) and c) back-scattering cross-sectional morphology, d) etched morphology, e) EDS line-scanning results of the N5 nanocrystalline coating

10⁵–10⁶ K/s,⁹ and the cooling rate is so fast that it is difficult for the crystal nuclei to grow. Therefore, the grain size of the coating decreases, showing a fine grain structure and forming a nanocrystalline coating.

Figure 1e shows the EDS line-scanning results for the N5 nanocrystalline coating. As shown in Figure 1e, there is no obvious change in the content of elements along the longitudinal direction of the coating through the interface, indicating that there is a chemical-potential difference between the coating and substrate, suppressing the interdiffusion in the high-temperature environment.

The kinetics curves for the high-temperature oxidation of the Ni-based single-crystal superalloy substrate and N5 nanocrystalline coating are shown in **Figure 2**. Both kinetics curves follow the parabolic law in accordance with the following formula:

$$y^2 = kt \tag{1}$$

where y is the oxidation gain and t is the oxidation time. **Table 2** includes the parabolic-rate constant calculated in accordance with Equation (1). The calculation results show that the parabolic constant of the N5 nanocrystalline coating is larger than that of the Ni-based single-crystal superalloy substrate.

During the initial stage of oxidation (0–20 h), an oxide film is formed quickly on the surface of the coating, and the weight gain is rapid. During the stable oxidation stage (20–100 h), the oxide film on the surface of the coating protects the internal alloy, the rate of the oxidation weight gain becomes slower and the curve is gentle. According to the oxidation-kinetics curves, the oxidation

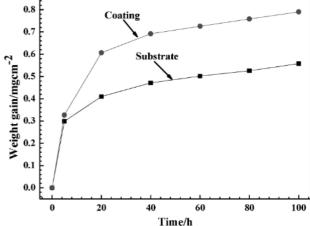


Figure 2: Oxidation kinetics curve for the Ni-based single-crystal superalloy substrate and N5 nanocrystalline coating

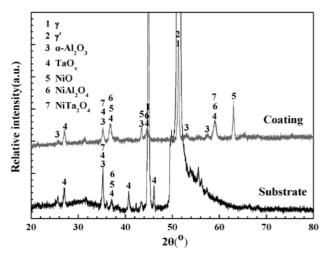


Figure 3: X-ray diffraction of the Ni-based single-crystal superalloy substrate and N5 nanocrystalline coating

weight gain of the coating is higher than that of the substrate. The result shows that after 100 h of high-temperature oxidation, the weight gain per unit area of the Nibased single-crystal superalloy substrate is 0.56 mg/cm², and the weight gain per unit area of the N5 nanocrystalline coating is 0.8 mg/cm². The oxidation weight gain per unit area of the coating is 42.85 % higher than that of the substrate.

Figure 3 shows the XRD patterns of the Ni-based single-crystal superalloy substrate and N5 nanocrystalline coating after the oxidation at 1100 °C for 100 h. The strongest peaks are assigned to γ/γ' , indicating that neither the substrate nor the coating undergoes a phase change after the high-temperature oxidation. After the oxidation, the phase constitutions of the substrate and the coating are almost the same. The oxide film on the surface contains α -Al₂O₃, NiO, TaO_x, NiTa₂O₄ and NiAl₂O₄. The peaks corresponding to TaO₂ and Ta₂O₅ appear in the spectrum, so we choose to use TaO_x. The contents of α-Al₂O₃ and NiAl₂O₄ in the substrate are lower than those in the coating. The XRD results show that the phase compositions of the substrate and the coating are basically the same after the high-temperature oxidation experiment.

The surface morphology of the Ni-based single-crystal superalloy substrate and N5 nanocrystalline coating after the oxidation at 1100 °C for 100 h is shown in Figure 4. After oxidation, the surface of the Ni-based single-crystal superalloy is smooth, without cracks, but localized areas of the oxide film are wrinkled and peeled off (Figure 4a). The surface morphology of the N5 nanocrystalline coating after oxidation is shown in Figure 4c. A layer with a fine and dense spinel structure is formed on the surface of the N5 nanocrystalline coating

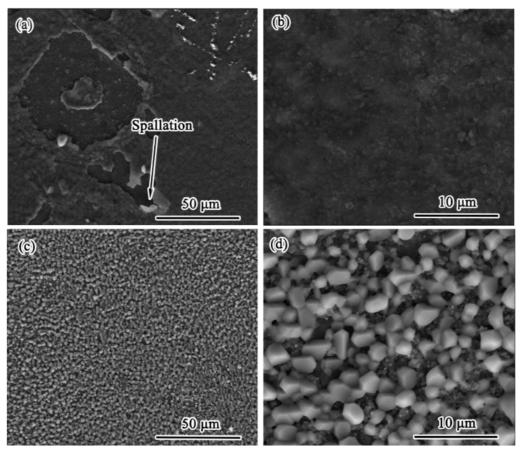


Figure 4: Morphology after oxidation: a) and b) Ni-based single-crystal superalloy substrate, c) and d) N5 nanocrystalline coating

after oxidation. Combined with the XRD spectrum analysis, the results show that this layer is a NiAl₂O₄ spinel. There was no oxide-film shedding or wrinkling on the surface of the coating after oxidation, indicating that the adhesion of the N5 nanocrystalline coating was strong. The main reasons for the strong adhesion of the nanocrystalline coating are as follows:

- the oxidation film formed by the nanocrystalline coating has a smaller grain size, which is conducive to releasing growth-related stress and thermal stress through plastic deformation,
- (2) the nanocrystalline coating has good capacity for deformation, which is conducive to releasing stress generated by the film during the thermal cycle, 10
- (3) the nanocrystalline coating has a columnar crystal structure and the oxide is continuously oxidized along the columnar crystals of the coating, exhibiting a mechanical pinning effect similar to that of active elements,
- (4) fine and compact oxide grains can prevent metal interdiffusion and reduce the formation of vacancies. Therefore, even after 100 h of oxidation at a high temperature, there was no cracking or peeling of the oxide film.

The cross-sectional morphology of the Ni-based single-crystal superalloy substrate and N5 nanocrystalline

coating after the oxidation at 1100 °C for 100 h is shown in Figure 5. The morphology of the cross-section (Figure 5b) shows that the boundary between the coating and the substrate is clearly defined, and there is no secondary reaction zone (SRZ) or element-rich topologically close packing (TLP) through the interface between the coating and the substrate, indicating that no elemental interdiffusion occurred during the process of high-temperature oxidation. Figure 5c shows that after the oxidation of the Ni-based single-crystal superalloy, the oxide film has a layered structure, the upper oxide film is loose, the lower oxide layer is dense and the density distribution is uniform. Table 3 shows the EDS point results of the oxide films of the N5 single-crystal superalloy and its nanocrystalline coating. According to the EDS and XRD results, the upper oxide film is mainly NiAl₂O₄ (point 1) and the lower oxide films is Al₂O₃ (point 3). There are a few white dots (point 2) in the lower layer of the Al₂O₃ film. The EDS results confirmed that the white dots in the Al₂O₃ film are TaO_x. The morphology of the cross-section of the N5 nanocrystalline coating after oxidation is shown in Figure 5d. The oxide film also shows a layered structure. However, the upper oxide film is dense and tightly bound to the lower oxide film. Combining the EDS and XRD results, we find that the upper oxide film has a spinel structure formed by

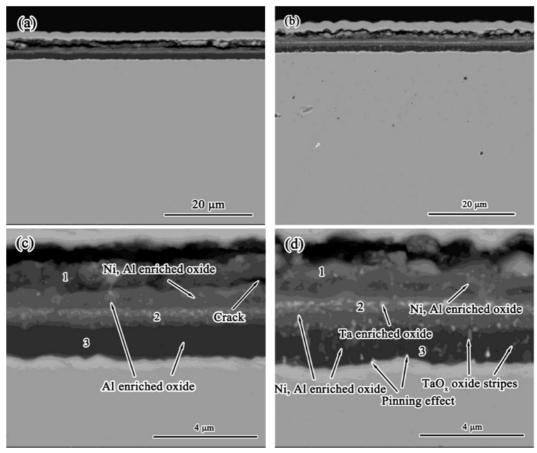


Figure 5: Cross-sectional morphology of: a) and c) Ni-based single-crystal superalloy substrate; b) and d) N5 nanocrystalline coating

NiAl $_2O_4$ (point 4), and the composition of the lower oxide film still includes TaO_x (point 5) and Al_2O_3 (point 6). However, there is an obvious difference in the oxide film between the N5 single-crystal superalloy and its nanocrystalline coating. The nanocrystalline coating includes many TaO_x oxide stripes, distributed in the lowest layer of the oxide film and, moreover, at the interface of the oxide film and coating. TaO_x oxides in the Al_2O_3 film improve the adhesion of the oxide film due to the pnning effect.

Table 3: EDS point results for oxide films of N5 single-crystal superalloy and its nanocrystalline coating

Atomic per- centage of el- ements (x/%)	О	Al	Cr	Co	Ni	Ta
1	41.79	31.06	2.24	2.15	20.84	0.54
2	52.94	39.83	1.75	1.44	12.54	0.96
3	59.78	33.65	0.93	0.76	4.59	0.30
4	42.20	26.70	2.96	2.12	19.31	1.87
5	39.71	24.64	4.13	3.96	7.31	3.86
6	56.36	31.01	1.52	1.45	6.32	0.69

According to the previous studies, a nanocrystalline coating can significantly improve the oxidation resistance of a substrate.11,12 The main reason is the fact that the formation of a nanocrystalline coating reduces the grain size of the coating. Compared with the alloy substrate, the nanocrystalline coating has more abundant grain boundaries, which can effectively improve the diffusion coefficient of Al, reduce the critical Al content required for the transformation of Al from internal oxidation to external oxidation, shorten the time for the formation of the Al₂O₃ film, and form a protective Al₂O₃ film in a short time to improve the oxidation resistance of the coating. In this study, the opposite behavior occurred. The oxidation rate of the substrate alloy is found to be slower than that of the N5 nanocrystalline coating. Although the weight gain of the coating due to oxidation is higher, the adhesion of the coating is significantly better than that of the substrate. As shown in Figures 4a and 5c, many oxide-film spalling points (Figure 4a) on the substrate surface and cracks in the oxide film (Figure 5c) are observed. However, the surface of the oxide film on the N5 nanocrystalline coating is smooth, without spalls and wrinkles (Figure 4c). In the cross-sectional morphology (Figure 5d), the bond between the oxide film and the substrate is tight, without holes and cracks, indicating that the N5 nanocrystalline coating significantly improves the adhesion to the substrate. The strong adhesion of the N5 nanocrystalline coating mainly comes from two sources. On the one hand, the addition of reactive elements can improve the adhesion of the coating and slow down the growth rate of the oxide film.¹³ An active element, Ta, is added to the N5 nanocrystalline coating to improve the resistance of the coating to peeling. On the other hand, the nanocrystalline

coating has good adhesion because of its good plastic-deformation properties.

In general, pores are generated due to the formation of oxide films. When the amount of porosity is small, the effect on the resistance of the film to stripping is weak. However, once the amount of porosity is large enough, the adhesion between the oxide film and the coating decreases, and the coating is prone to oxidative spalling. It is generally believed that an addition of reactive elements to an alloy can inhibit the formation of pores in the oxide film, thereby improving the spalling resistance of the oxide film. Ta, as a reactive element, plays an important role in the high-temperature oxidation resistance of the substrate and coating. Research on the effect of Ta on the oxidation resistance of alloys is convincing.14-18 Park et al. 15 showed that the effect of Ta on the oxidation resistance of coatings depends on the amount of Ta. Wang et al.⁷ reported that the presence of Ta in sputtered nanocrystalline coatings improved the adhesion and oxidation resistance of oxide films. Han et al.16 thought that when the content of Ta was less than 0.05 w/%, the presence of Ta had little effect on the high-temperature oxidation performance of the coating. However, in the present paper, the amount of Ta in the Ni-based single-crystal superalloy is as high as 6.4 w/%. At the end of the static isothermal oxidation experiment, elemental Ta accumulates at the grain boundaries of the Al₂O₃ columnar crystals (Figure 5). Ta is an active element, enhancing the resistance to stripping of the nanocrystalline coating. In contrast, an uneven distribution and peroxidation of Ta on a substrate results in the growth of pores in the oxide film, accelerating the oxide spalling. Chen et al. 16 reported that when the valence state of impurity ions is greater than Al3+, the high-valence metal ions occupy O2holes and react with oxygen to form many oxides, resulting in cavities in the oxide film, and then, the oxide film easily peels off.

According to the high-temperature oxidation-kinetics curve (Figure 2 and Table 2), the weight gain of the coating is always higher than that of the substrate during a constant-temperature static-oxidation experiment. The nanocrystalline coating is formed by columnar crystals with rich grain boundaries so that the migration rate of Ta is faster than that in the substrate. This situation leads to a larger Ta accumulation in the coating. The presence of a large amount of Ta makes the coating absorb oxygen faster, resulting in a greater weight gain due to oxidation. The EDS test results from Tables 4 and 5 prove this point. The above point is the main reason why the weight gain of the coating is greater than that of the substrate in this high-temperature oxidation experiment. Although the weight gain of the coating is faster, the stripping resistance of the oxide film of the nanocrystalline coating is substantially improved.

Ta, an insoluble element, is often added to superalloys to improve their mechanical properties.^{19,20} In this study, a Ni-based single-crystal superalloy is used as a protective coating, and Ta is found in the substrate-andcoating aggregate, in an Al₂O₃ film, after high-temperature oxidation. According to Wang et al.7, the Gibbs free energy of Ta and Al oxides follows the trend of TaO₂ > $Al_2O_3 > Ta_2O_5$. It is speculated that Ta may be in the high-valent cation state (Ta5+) at the grain boundaries of the Al₂O₃ film. Elemental Ta moves along the grain boundaries in accordance with the concentration gradient. High-valent Ta⁵⁺ preferentially occupies O²⁻ holes and is finally partially oxidized to Ta₂O₅ on the surface, or in the middle, of the oxide film. In the N5 nanocrystalline coating, the dispersion of Ta is relatively uniform and the nanocrystalline coating has rich grain boundaries, so the diffusion rate of Ta is faster in the nanocrystalline coating. Studies showed that the diffusion coefficients of ions along the grain boundaries are several orders of magnitude higher than the lattice diffusion coefficient.21,22 The rapid diffusion of Ta makes the aggregation of Ta at the grain boundaries of the coating more significant, which is one of the reasons why the oxidation weight gain of the nanocrystalline coating is greater than that of the substrate. In Figure 5d, the white dots in the alumina oxide film show the flow dynamics, where the flow moves from the Ni-based single-crystal superalloy substrate to the alumina oxide film in the N5 nanocrystalline coating, and the alumina oxide film forms ribbons. This experimental phenomenon further proves the theoretical speculation. The above experimental demonstration further clarified the influence of Ta on the oxidation performance of the coating, providing a theoretical basis for the design and preparation of Ni-based single-crystal superalloy coatings with ESD.

4 CONCLUSIONS

- 1. An N5 nanocrystalline coating with a columnar grain structure was successfully prepared on an N5 single-crystal superalloy substrate using the ESD technology; it exhibits a strong metallurgical bonding with its substrate and there are no microholes at the interface.
- 2. During oxidation no secondary reaction zone or topologically close-packed phases formed as there was no interdiffusion due to the chemical-potential balance between the substrate and coating.
- Ta has a different oxide morphology in the nanocrystalline coating and single-crystal superalloy, and it improves the adhesion of the oxide film due to the pinning effect.

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