



Article Interdiffusion Behaviour of NiCoCrAlY Coating and N5 Single Crystal Superalloy

Jiaxu Cao^{1,2}, Yingkun Liu^{1,2}, Chunming Deng^{2,*}, Kun Yang² and Feng Li¹

- ¹ School of Materials and Energy, Guangdong University of Technology, Guangzhou 510006, China; vikkycool767158226@163.com (J.C.); lingling_014@163.com (Y.L.); fengli@gdut.edu.cn (F.L.)
- ² The Key Lab of Guangdong for Modern Surface Engineering Technology, National Engineering Laboratory for Modern Materials Surface Engineering Technology, Guangdong Institute of New Materials, Guangzhou 510651, China; yangkun@gdinm.com
- * Correspondence: dengchunming@gdinm.com

Received: 24 February 2020; Accepted: 13 April 2020; Published: 16 April 2020



Abstract: This paper aims at investigating the microstructure and phases evolution of single crystal superalloy/high temperature protective coating during high temperature static oxidation, and exploring the influence of element interdiffusion behaviour on microstructure and phase evolution of the single crystal superalloy substrate. A NiCoCrAlY high-temperature protective coating was deposited on the Ni-based single-crystal superalloy by low-pressure plasma spraying technology. The coated samples were subjected to static oxidation for 200 h at a constant temperature of 1100 °C. Scanning electron microscope, energy dispersive spectrometer and X-ray diffraction were used to characterise the microstructure and phase after interdiffusion between the coating and the substrate at high temperature. The results showed that a dense thermally grown oxide layer was formed on the surface of the NiCoCrAlY coating after oxidation for over 100 h. The only interdiffusion zone was formed after oxidation for 50 h, while both interdiffusion zone and secondary reaction zone could be observed after oxidation for over 100 h. The thickness of interdiffusion zone and secondary reaction zone is increased with the extension of oxidation time, and the grain growth of topological close-packed phase in the secondary reaction zone is found. Al, Cr and Co in the coating diffuse from the coating to the substrate, while Ni and refractory materials like Ta, Mo, Re and W diffuse from the coating to the substrate. The interdiffusion of coating and substrate leads to the instability of γ/γ' phase in the substrate, which finally results in the formation of W, Re and Cr-rich needle-like topological close-packed phase in the substrate.

Keywords: single crystal superalloy; NiCoCrAlY coating; static oxidation; interdiffusion

1. Introduction

As the main materials used for advanced aero-engine blades, Ni-based single crystal superalloys possess excellent creep strength and fatigue resistance performance at high service temperature [1–5]. In contrast, the oxidation resistance of Ni-based single crystal superalloys is poor for the increased service temperature in high thrust-ratio turbine engines, and one of the most effective ways is to apply with protective coatings like MCrAlY, PtAl coating [6–10]. MCrAlY coatings (M is generally Ni, Co or Ni and Co) have good compatibility with single crystal superalloys at high temperature. Simultaneously, the sufficient content of Cr and Al in the coating ensure its oxidation and hot corrosion resistance [11–14].

However, the huge differences in composition between coating and underlying substrate can lead to element interdiffusion during long-term high-temperature service, resulting in the segregation of insoluble elements such as Ta in the substrate and the precipitation of harmful topological close-packed

phase [15–17]. The main components of topological close-packed phases are W, Mo and Re, which are added in the superalloy for strengthening the comprehensive performance of superalloys at high temperature [18–20]. There is no doubt that the precipitation of harmful topological close-packed phases will not only deplete the refractory reinforcing elements in the substrate but also be the source of rack propagation, resulting in the system failure [21,22]. Up to now, few reports are available on the precipitation of topological close-packed phases during high temperature oxidation process. In order to obtain a higher quality coated surface, many researchers have carried out related research, for example, Fernande-Abia proposed a mechanistic model for cutting forces in turning with nose radius tools [23–26]. In addition, generally MCrAIY coatings were widely deposited on superalloy by thermal spray technology including low-pressure plasma spraying, air plasma spraying and high velocity oxy/air fuel, physical vapor deposition and electron beam-physical vapor deposition, depending on the coating technical requirements. Low-pressure plasma spraying is the most widely used and successful process for the high coating performance especially for blade of aero-engine because of the shrouded Ar environment and relatively low working pressure. However, few studies focusing on interdiffusion between low-pressure plasma-sprayed MCrAlY coatings and single crystal superalloy was found, which is still insufficient to understand the mechanism of interdiffusion between Ni-based superalloy (especially the single crystal superalloys) and protective MCrAIY coatings [27–30].

In this paper, the interdiffusion behaviour of N5 single crystal superalloy/NiCoCrAlY coating are investigated during high-temperature static oxidation process. The microstructure evolution, interdiffusion of elements and precipitation of characteristic phase are studied after static oxidation respectively. The research is helpful for the understanding of interdiffusion behaviour between LPPS sprayed protective MCrAlY coatings and single crystal superalloy, the precipitation process of the precipitated phase as well, which will provide a certain guideline for the estimation of service life of superalloy.

2. Experimental Procedure

N5 Ni-based single crystal superalloy and NiCoCrAlY is used as substrate and protective coating respectively, and their chemical compositions are shown in Table 1. The substrate was cut into sheet-shaped blocks with a dimension of 100 mm × 15 mm × 2.5 mm by electric discharge machining. Prior to thermal spraying, the substrate was grit-blasted at a pressure of 0.3–0.5 MPa using 120# white fused alumina. A low-pressure plasma spraying device (Oerlikon Metco, Freienbach, Switzerland) was used to deposit NiCoCrAlY coating on the substrate. The deposition parameters are shown in Table 2. The coated sample was cut into a dimension of 10 mm × 15 mm × 2.5 mm with a Secotom-15 precision cutting machine for static oxidation test.

Component (wt %)	N5 Single Crystal	NiCoCrAlY Coating
Ni	Balance	31.41
Со	7–9	38.40
Cr	6–8	19.73
Al	6–8	10.44
Мо	2–4	-
Та	6–8	-
Re	3–4	-
Y	-	0.01
W	4–6	-

Table 1. Nominal compositions of N5 single crystal superalloy and NiCoCrAlY coating.

Parameters	LPPS
Pressure (Pa)	40
Current (A)	1800
Power (kW)	126
Plasma gas (Ar) (slpm) ^a	110
Plasma gas (He) (slpm) ^a	-
Plasma gas (H ₂) (slpm) ^a	6
Powder feed rate (g min ⁻¹)	30
Spraying distance (mm)	450
Torch speed (mm/s)	300

Table 2. Deposition parameters of NiCoCrAlY coating prepared by low pressure plasma spraying.

^a SLPM: standard litres per minute.

Then, the constant temperature static oxidation test was performed in a muffle furnace (KSF1400, kilns, Shanghai, China). The NiCoCrAlY coated/N5 single crystal superalloy samples and the uncoated samples were placed in an Alumina crucible at 1100 °C (Air, 1 atm). After a predetermined oxidation time (0, 50, 100, 200 h), the sample is taken out for the characterizations.

The X-ray diffractometer (D8-Advance, Bruker, Karlsruhe, Germany) was used to analyse the phase of the coating using Cu K α -ray as the diffraction source with a scanning step of 0.02°/s and a scanning range of 2 θ from 10° to 90°. Field emitting-scanning electron microscope (FE-SEM, Nava-Nano-430, FEI, Hillsboro, OR, USA) equipped with energy spectrum (EDS, Oxford INCAx-sight 6427, Oxford, UK) were used to analyse the morphology and chemical compositions of coating and substrate.

3. Results and Discussions

3.1. LPPS Sprayed NiCoCrAlY Coating on N5 Single Crystal Superalloy

Figure 1 presents the morphology and XRD pattern of the as-deposited coating prepared by low-pressure plasma spraying technology (LPPS). The cross-sectional image of the coating shows a laminar microstructure, typically for thermal spray coating. The coating had an average thickness of 60 μ m and a density of over 99%. The coating was well bonded with the grit-blasted substrate, and a distinct interface of coating/substrate was observed. No significant oxide region was observed in the LPPS coating. According to the XRD pattern, the main phase of the as-deposited coating consists of γ -Ni/ γ '-Ni₃Al [31].



Figure 1. Cross-sectional back-scattered electron (BSE) image and X-ray diffraction (XRD) diagram of as-deposited coating: (a) cross-sectional BSE-images, (b) XRD diagram.

3.2. Static Oxidation of NiCoCrAlY Coated N5 Single Crystal Superalloy

Figure 2 shows the enlarged BSE images of the thermally grown oxide (TGO) layer after static oxidation of coating at 1100 $^{\circ}$ C for different time. After static oxidation for 50 h, a 2.20 μ m TGO layer

appeared on the coating, and the TGO is Cr_2O_3 and $NiCr_2O_4$ according to the XRD result. With the extension of the oxidation time to 100 h and 200 h, the thickness of TGO layer is gradually increased to be 3.75 and 4.34 µm. However, the TGO layer is composed of 2 layers, including the spinel Ni(Cr,Al)₂O₄ top layer and α -Al₂O₃ bottom layer after oxidation for 100 and 200 h. During the oxidation process, the Al in the coating diffuses outward and the O from the surrounding atmospheric environment diffuses inward, and θ -Al₂O₃ is firstly formed on the coating surface except the former spinel oxide, afterwards θ -Al₂O₃ undergoes phase transformation into stable and dense equiaxed crystal α -Al₂O₃ at 1100 °C. The surface morphologies for bare and coated substrate oxidized at 1100 °C for 200 h are shown in Figure 3. For bare substrate, some relatively loose oxides with spinel structure are observed, while very dense oxide is formed on the coated substrate. Dense α -Al₂O₃ at the bottom of TGO can effectively prevent the diffusion of external gases into the interior of the coating/substrate, therefore increasing the service life of the coating/single crystal superalloy significantly [32].



Figure 2. BSE images and XRD patterns of NiCoCrAlY coating after static oxidation at 1100 °C for different time: (**a**) 0 h; (**b**) 50 h; (**c**) 100 h; (**d**) 200 h; (**e**) XRD patterns.



Figure 3. SEM images of sample surface after static oxidation at 1100 °C for 200 h: (**a**) bare substrate; (**b**) coated substrate.

3.3. Interdiffusion Behaviour between NiCoCrAlY Coating and N5 Single Crystal Superalloy

Figure 4 presents the cross-sectional morphology of NiCoCrAlY coating/N5 single crystal superalloy after static oxidation at 1100 °C for 0, 50, 100 and 200 h. Generally the Al in the coating will diffuse into the substrate because of the high concentration of Al in the coating, resulting in the following phase transformation in the substrate [33]:

$$3\gamma$$
-Ni + [Al] $\rightarrow \gamma'$ -Ni₃Al, (1)

$$\gamma'$$
-Ni₃Al + 2 [Al] \rightarrow 3 β -NiAl. (2)



Figure 4. Cross-section of NiCoCrAlY coating/N5 single crystal superalloy after static oxidation at 1100 °C for different time: (**a**) 0 h; (**b**) 50 h; (**c**) 100 h; (**d**) 200 h.

According to the above-mentioned phase transformation process, after static oxidation for 50 h, an interdiffusion zone (IDZ) with a thickness of 17 µm was first formed at the coating/substrate interface, and the dark phase in the IDZ was the γ' -Ni₃Al phase, while the secondary reaction zone (SRZ) was not found. However, as the oxidation continued to be 100 h, the content of γ' -Ni₃Al phase in IDZ was decreased, and a SRZ with a large amount of granular TCP phases also began to appear in the raft γ 'substrate. The diffusion after 200 h oxidation is similar to that after 100 h oxidation, and the difference lies in more TCP content and larger TCP crystal in the SRZ with the extension of oxidation time. The thickness of IDZ and SRZ is also significantly affected by the oxidation time, as shown in Figure 5. It can be seen that the thickness of IDZ and SRZ is sharply increased during the oxidation

process from 0 h to 100 h, but the growth rate for both zones is obviously slowed down in the next 100 h [34,35].



Figure 5. Variation of interdiffusion zone (IDZ) and secondary reaction zone (SRZ) for NiCoCrAlY coating/N5 single crystal superalloy with static oxidation time at 1100 °C.

In order to analyse the interdiffusion behaviour, the distribution of elements was characterised using EDS every 5 μ m from coating sub-surface (20 μ m away to coating surface) to the interior substrate after oxidation for different time, as presented in Figure 6. In the as-deposited NiCoCrAlY coating on N5 single crystal superalloy, the content of Al, Cr, Co, and Ni is 10.44 wt %, 19.73 wt %, 38.40 wt %, and 31.41 wt % in the coating and 7.13 wt %, 7.31 wt %, 7.93 wt %, and 60.27 wt % in the substrate, respectively. From Figure 6a, the concentration of elements ranged between coating and substrate, indicating the interdiffusion had occurred after oxidation for 50 h. In addition to being selectively oxidized, Al, Cr and Co in the coating diffused into the substrate at the same time, leading to the gradual decrease of Al, Cr and Co in the coating with the lapse of oxidation time, while Ni diffuses from the substrate to the coating according to the concentration profiles after oxidation for different time. Moreover, Mo, W, Ta and Re elements were also detected in the coating according to Figure 6b–d, indicating that these elements diffuse into the coating. It is worth noting that the concentration fluctuation of Ni, Cr and Co at the IDZ is connected with the precipitation phase.



Figure 6. Energy dispersive spectrometer (EDS) elemental concentration profiles of NiCoCrAlY coating/N5 single crystal superalloy with depth from coating to substrate after static oxidation at 1100 °C: (**a**) 0 h; (**b**) 50 h; (**c**) 100 h; (**d**) 200 h.

3.4. Precipitation of TCP

Formation of needle-like fragile TCP phase has significantly negative effect on the high temperature performance, such as high temperature strength, creep, etc. As mentioned before, with the extension of oxidation time, more TCP phases were precipitated and growth, which is verified from Figure 7. The size of the TCP phase is nearly doubled after oxidation from 100 to 200 h, and the bright core region and the grey shell region can be clearly observed in Figure 7b. The needle-like TCP phase was oriented at 45° or 135° with the substrate. EDS revealed that TCP phase was rich in Cr and Re, as shown in Figure 7c,d. This is consistent with σ -TCP phase in nickel-based superalloy [36,37].



Figure 7. Cross-sectional BSE images and EDS element maps of precipitation phase: (**a**) 100 h, (**b**) 200 h, (**c**) EDS Cr distribution in Figure **b**, (**d**) EDS Re distribution in Figure (**b**).

The chemical compositions for the core region and the shell region of σ -TCP phase were measured by EDS. It is found that the content of Ta, W and Re was 3.14 wt %, 16.14 wt % and 20.53 wt % in the bright core region, and the content of Cr, Mo and Re was 40.15 wt %, 4.56 wt %, 28.92 wt % in the grey shell region. Therefore the precipitation of the TCP phase consumes a large amount of solid solution strengthening elements such as Ta, Mo, W, Re; this is the reason for the decrease of high temperature creep properties of the single crystal superalloys after applying protective coatings. Both Al and Cr in the coating diffuse into the substrate during high temperature oxidation process, while the solid solution of both elements varies greatly. For Al, it forms γ' -Ni₃Al and β -NiAl with Ni in the substrate depending on the concentration of Al during the diffusion. However the solid solubility of Cr in the Ni-based superalloy is much lower than that of Al, α -Cr is easily precipitated in the substrate during the diffusion process. While the refractory elements such as W and Re have much higher solid solubility in α -Cr than Al, Re will hasten the precipitation of α -Cr. As a whole, the interdiffusion of coating and substrate leads to the instability of γ/γ' phase in the substrate, which finally results in the formation of W, Re and Cr-rich needle-like TCP phase [38,39].

4. Conclusions

The NiCoCrAlY high-temperature protective coating was deposited on the Ni-based single-crystal superalloy by low-pressure plasma spraying technology. The microstructure and phase evolution of single crystal superalloy/high temperature protective coating during high temperature static oxidation, and the influence of element interdiffusion behaviour on microstructure and phase evolution of single crystal superalloy substrate were investigated systematically. Some important conclusions can be drawn as follows:

- The N5 Ni-based superalloy/NiCoCrAlY coating forms a dense α -Al₂O₃ TGO, which can significantly improve the oxidation resistance performance of substrate.
- IDZ appears after static oxidation for 50 h, then SRZ enriched with TCP phase will be observed at 100 h, afterwards the TCP phase will gradually grow with the extension of the oxidation time.
- Al, Cr and Co in the coating diffuses from the coating to the substrate, while Ni and refractory materials like Ta, Mo, Re and W diffuse from the coating to the substrate.
- The interdiffusion of coating and substrate leads to the instability of γ/γ' phase in the substrate, which finally results in the formation of W, Re and Cr-rich needle-like TCP phase. The concentration change law and the precipitation process of precipitation phase are of great significance to improve the reliability of the coating/single crystal superalloy system and reasonably predict its life.

Author Contributions: Conceptualization, J.C., C.D., K.Y. and F.L.; methodology, J.C. and C.D.; software, J.C., C.D. and Y.L.; validation, J.C. and C.D.; formal analysis, J.C. and C.D.; investigation, J.C. and C.D.; resources, J.C., C.D. and K.Y.; data curation, J.C., C.D. and Y.L.; writing—original draft preparation, J.C. and C.D.; writing—review and editing, J.C. and C.D.; funding acquisition, C.D., K.Y. and F.L. All authors have read and agreed to the published version of the manuscript.

Funding: This work was supported by the Guangdong Provincial Key Area Research and Development Program (No. 2019B010936001), the Guangdong Provincial Natural Science Foundation Project (No. 2016030312015, No. 2020A1515010948), and the Guangdong Academy of Sciences Project (No. 2020GDASYL-20200104028).

Conflicts of Interest: The authors declare no conflict of interest.

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