Fabrication of a PtAl2 Single-Phase Coating and Its High-Temperature Oxidation Resistance Postprint

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Abstract

A Pt coating was deposited on nickel-based cast superalloy K38G by pulse electroplating, and a platinum-aluminum coating comprising a single-phase PtAl outer layer and a -NiAl inner layer was obtained through step-heating pack cementation aluminizing treatment. Isothermal and cyclic oxidation tests were performed on the single-phase PtAl coating at 1100 °C, and the oxidation behavior and failure mechanism under both conditions were analyzed. The results indicate that the single-phase PtAl coating demonstrates favorable isothermal oxidation resistance; the initial rapid weight gain primarily originates from -Al O formation, which rapidly transforms to -Al O with the weight gain subsequently plateauing. However, the cyclic oxidation resistance of the single-phase PtAl coating is inferior; thermal stresses generated during cyclic oxidation induce localized spalling or detachment of the PtAl layer, thereby triggering premature coating failure. Consequently, the single-phase PtAl coating is unsuitable for high-temperature load-bearing service environments, with its failure and degradation during cyclic oxidation primarily arising from PtAl layer spallation and rapid depletion of Al element in the -NiAl layer adjacent to spalled regions.

Full Text

Preamble

Title: OXIDATION PROPERTY AND FAILURE MECHANISM OF A SINGLE PHASE PtAl2 COATING

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Abstract: Pt-modified aluminide coating has attracted great attention due to its advantage of the integrated property in resisting both high temperature oxidation and hot corrosion. By the presence of Pt, the spallation trend of the grown oxide scale and the detrimental effect of S can be restrained at a very low level. Besides, Pt could promote -Al O formation and stabilize -NiAl phase. Thus Pt-modified aluminide (Pt-Al) coating has been widely used in some crucial applications requiring reliability and extended service life. There are mainly PtAl , -(Ni, Pt)Al and / -NiPtAl phases existing inside Pt-Al coating. In this work, a single phase PtAl coating was prepared on a Ni-base K38G superalloy through pulse-electroplating of Pt and pack aluminization under stepped heating mode. At 1100 °C, the isothermal oxidation behavior of the single phase PtAl coating was evaluated by thermogravimetric analysis (TGA). Cyclic oxidation test of the PtAl coating was performed within a vertical muffle furnace at the same temperature. The results indicate that the singular PtAl coating possesses quite good isothermal oxidation resistance. However, its resistance against cyclic oxidation is very poor. The cyclic stress induced by repeated heating and cooling has caused visible detachment of PtAl coating layer, which the spallation of PtAl in further would lead to a premature failure of the whole coating system. Partial spallation of PtAl layer, including undesirable consumption of Al inside -NiAl nearby the spallation act the main reason responsible for the final failure. Accordingly, it is not appropriate to apply single phase PtAl coating in the high temperature services involving stress and load. The degradation mechanism of the singular PtAl coating is investigated by discussing the stress generated from cyclic heating and cooling.

Keywords: Ni-base superalloy, Pt-Al coating, thermogravimetric analysis (TGA), isothermal oxidation, cyclic oxidation

1. Introduction

Nickel-based superalloys exhibit excellent mechanical properties at elevated temperatures, such as creep resistance, stress rupture strength, and fatigue resistance, due to their unique coherent / phase structure, making them widely used as high-temperature structural materials in aerospace and energy industries. In practical applications, nickel-based superalloys must also withstand attack from corrosive media such as O , S, and Cl in service environments. Above 1000 °C, damage from high-temperature oxidation far exceeds that from corrosive salts. Considering mechanical performance requirements, the content of major oxidation/corrosion-resistant elements such as Al and Cr in superalloys is quite limited. Therefore, high-temperature components are typically protected by applying high-temperature protective coatings to prevent rapid failure of the substrate alloy from oxidation attack.

Aluminide (Al-diffusion) coatings have been widely used on superalloy hot-section components since the 1950s due to their simple process and low cost[1].

The concept of Pt-modified aluminide coatings was first proposed by Cape[2], and Boone et al.[3~5] conducted a series of studies on Pt-modified aluminide coatings. Their results demonstrated that the presence of Pt significantly improves the coating' s resistance to oxide scale spallation, and the coating' s hot corrosion and cyclic oxidation performance are markedly improved compared with conventional aluminide coatings. Domestic research on Pt-Al modified coatings also began early[6~8]. To date, researchers worldwide have maintained continuous interest in Pt-Al coatings[9~14].

Common constituent phases in Pt-modified aluminide coatings include PtAl and Pt-dissolved -NiAl or / phases. Based on preparation processes and coating phase compositions, these coatings can be classified as single-phase PtAl coatings, PtAl +(Ni,Pt)Al dual-phase coatings, (Ni,Pt)Al single-phase coatings, and / -NiPtAl coatings. Existing reports on Pt-Al coatings have primarily focused on the high-temperature oxidation behavior and performance of single-phase (Ni,Pt)Al or PtAl +(Ni,Pt)Al dual-phase coatings, with few studies on the preparation and high-temperature oxidation performance of single-phase PtAl coatings. This work investigates the high-temperature oxidation performance and failure modes of single-phase PtAl coatings through isothermal and cyclic oxidation tests.

2. Experimental Procedures

The nickel-based cast superalloy K38G was used as the substrate material, with main chemical composition (mass fraction, %) of: Cr 16.34, Co 8.38, Al 4, W 2.66, Mo 1.77, Ta 1.75, Nb 0.76, C 0.16, B 0.01, and Ni balance. Prior to experiments, the K38G alloy substrate was cut into plate specimens measuring 15 mm × 10 mm × 2 mm, ground with 1000-grit sandpaper, dry-blasted with corundum grit, and then ultrasonically cleaned in acetone and alcohol before being dried for use.

Before electroplating, the alloy specimens were given a conventional Ni strike treatment to obtain a Ni layer approximately 5 m thick. The platinum plating solution was prepared using K PtCl (Pt content about 40%, mass fraction) as raw material, with appropriate KNO added, boiled and cooled to obtain orange-yellow crystals. The crystals were dissolved by heating with HCl or H SO , and the pH was adjusted to 1~2 for use. During Pt plating, a Pt wire mesh (diameter 0.5 mm) was used as the anode, the water bath temperature was set to 40~60 °C, the current density was 10~20 mA/cm^2, the deposition rate was controlled at 6 m/h, and the plating time was 1.5 h, yielding a Pt coating approximately 9 m thick.

The K38G samples with Pt deposits were embedded in an Al O crucible containing the pack cementation mixture, which consisted of FeAl powder (Al content 49%, mass fraction) plus 2% NH Cl (mass fraction), and placed in a stainless steel tube furnace. Before heating, the tube furnace was evacuated to ~10 Pa and backfilled with high-purity Ar (99.99%), repeated twice, and then maintained under Ar atmosphere to create a sealed inert environment. To remove hydrogen and relieve stresses, the samples were held at 200 °C for 1 h, 600 °C for 2 h, and 1000 °C for 3 h, with heating rates controlled within 10 °C/min.

Oxidation tests were conducted at 1100 °C in air. Isothermal (static) oxidation was performed in a VersaTherm Analyzer 771-0596 thermogravimetric analyzer (TGA) for 20 h, with mass data recorded every 30 s. Cyclic oxidation tests were carried out in a conventional vertical muffle furnace, with each cycle consisting of 60 min holding in the furnace plus 10 min cooling outside the furnace, for a total of 200 cycles. Mass changes of specimens were measured using a Sartorius BP-211D electronic analytical balance with an accuracy of 10   g.

The phase structures of coatings and oxide scales were analyzed using an X' Pert PRO X-ray diffractometer (XRD). The microstructure of coatings and the chemical composition of oxidation products were examined using an FEI Inspect F 50 field-emission scanning electron microscope equipped with an Oxford INCA X-MAX energy-dispersive X-ray analyzer (EDAX). To prevent spallation of the oxide scale during metallographic polishing, the cross-sectional samples with oxide scales were electroless Ni-plated before preparation.

3. Experimental Results and Discussion

3.1 Preparation of Single-Phase PtAl Coating

In practice, because the Pt electroplating process window is relatively narrow and the superalloy substrate contains active elements such as Al and Cr, a pre-plating Ni (Ni-strike) treatment is generally required to obtain high-quality Pt coatings[15]. Figure 1a [Figure 1: see original paper] shows the cross-sectional morphology of the K38G substrate after 90 min of Pt plating, where the Ni layer thickness is 5 m and the Pt layer thickness is 9 m. Due to the sandblasting treatment, visible gaps exist between the pre-plated Ni layer and the K38G substrate, while the Pt coating bonds well with the pre-plated Ni layer, and the Pt layer itself is continuous and dense. Figure 1b shows the cross-sectional morphology of the Pt-Al coating after pack aluminization for 3 h using the stepped heating method. It can be seen that the obtained single-phase PtAl layer has a thickness of about 60 m, beneath which is a -NiAl layer twice as thick as the PtAl coating. A band of white particles is distributed between the PtAl layer and the -NiAl layer; these white particles are PtAl particles. No visible interdiffusion zone (IDZ) appears between the -NiAl layer and the K38G substrate. Semi-quantitative EDAX analysis results indicate that the PtAl layer mainly contains Al, Pt, and Ni, with Al content of 64.3 at%, Pt content of 25.5 at%, and Ni content of 10.2 at%. Compared with conventional aluminide coatings, the total coating thickness after the same aluminization process is generally much greater for Pt-Al coatings because the presence of the Pt coating can accelerate Al diffusion[16-18], with the Pt layer serving as an "Al source transfer station" during aluminization.

Figure 2 [Figure 2: see original paper] shows the XRD spectra of the K38G superalloy after Pt deposition and subsequent aluminization treatment. Due to the use of conventional diffraction with penetration depth exceeding 10 m, the diffraction peaks of the Pt-plated sample include both the Pt layer and the pre-plated Ni layer. The figure also shows that the diffraction peaks of the pre-plated Ni layer are relatively narrow, indicating good crystallinity, while the Pt layer peaks are significantly broader, which is caused by large stresses within the plated Pt layer. Therefore, stepped heating is required before aluminization to eliminate stresses and precipitated H , preventing cracking of the Pt coating due to rapid heating. After pack aluminization for 3 h at 1000 °C in a protective atmosphere, the diffraction peaks of Pt and Ni disappear, becoming a single PtAl phase. The PtAl layer is thick enough to exceed the XRD detection depth, consistent with the results in Figure 1b.

3.2 Isothermal Oxidation Performance

Figure 3 [Figure 3: see original paper] presents the phase analysis results of the oxide scale on the single-phase PtAl coating after isothermal oxidation at 1100 °C for 20 h in the TGA. After 20 h of static oxidation, a single -Al O scale grew on the coating surface. Due to the short oxidation time, the underlying phase remains single-phase PtAl , with no observed phase transformation or degradation of PtAl .

Figure 4 [Figure 4: see original paper] shows the kinetic curve of the single-phase PtAl coating during isothermal oxidation at 1100 °C for 20 h, with the inset illustrating the relationship between the square of mass gain (ΔW) and oxidation time (t). As shown in the figure, the mass gain ΔW of the PtAl coating after 20 h is about 0.9 mg/cm^2, while the mass gain reached 0.7 mg/cm^2 within the initial 1 h (indicated by the dashed line in the figure). After 5 h, the mass change of the coating sample tended to level off. When ΔW is squared, the initial segment of the curve does not follow a linear relationship and does not satisfy the condition $\Delta W^2 = k \cdot t$ (where k is the kinetic constant). After 5 h, the curve follows a linear relationship, with the fitted k value being 6.31×10^{3} $mg^2/(cm \cdot h)$.

Since the Al content in the PtAl coating exceeds 60 at%, sufficient Al source is available for the rapid initial oxidation to form -Al O . It is speculated that the transient -Al O forms in the initial stage. Meanwhile, the fast-growing -Al O quickly transforms to the slower-growing -Al O at 1100 °C, after which the oxide scale mass gain becomes slow and gradually follows parabolic kinetics.

Figure 5 [Figure 5: see original paper] shows the cross-sectional morphology of the PtAl coating after isothermal oxidation at 1100 °C for 20 h. After 20 h of static oxidation, a continuous Al O scale formed on the PtAl coating surface, and the numerous white particles distributed between the PtAl coating and the -NiAl layer disappeared. Their contrast is intermediate between PtAl and -NiAl, suggesting that a phase transformation from PtAl to -(Ni,Pt)Al occurred in this region. When part of the PtAl layer is magnified, needle-like

Al O is observed on the outermost surface of the Al O scale, with a morphology characteristic of -Al O scales, indicating that fast-growing -Al O indeed formed in the early oxidation stage, consistent with the kinetic mass gain curve results.

Due to the short oxidation time and limited Al consumption, no PtAl phase transformation or degradation is observed between the surface Al O scale and the PtAl coating. However, significant elemental interdiffusion occurred between the PtAl coating and the -NiAl layer, causing the disappearance of the numerous distributed white particles. Meanwhile, because the static oxidation temperature is higher than the aluminization temperature, the interdiffusion effect between -NiAl and the K38G substrate is significantly stronger than during aluminization, resulting in a visible interdiffusion zone (IDZ) between -NiAl and the K38G substrate.

Based on the static oxidation results, it can be inferred that during subsequent static oxidation, the PtAl layer and the underlying -NiAl can continuously supply Al to support the growth and healing of the surface -Al O scale, demonstrating good isothermal oxidation resistance.

3.3 Cyclic Oxidation Performance and Failure Mechanism

Figure 6 [Figure 6: see original paper] shows the mass change curves of the K38G alloy and single-phase PtAl coating during cyclic oxidation at 1100 °C for 200 cycles. For the K38G alloy, the sample mass continuously increased up to 10 cycles, after which it rapidly decreased, reaching a mass change of -17.2 mg/cm^2 at 200 cycles.

Due to the low Al content in the K38G superalloy, a protective single Al O scale cannot be formed during high-temperature oxidation[19]. It is speculated that within 10 cycles, non-protective mixed oxides first formed (Cr O , TiO , and NiAlO spinel, etc.[20]), after which spallation of the mixed oxides caused the large mass loss. For the single-phase PtAl coating, unlike the rapid mass gain to 5 h followed by slow mass gain in static oxidation, obvious mass loss (~1 mg/cm^2) was observed after just 2 cycles of cyclic oxidation. The coating sample only began to show mass gain behavior after 20 cycles. With increasing cyclic oxidation cycles, the coating sample mass started to increase, reaching a plateau after 120 cycles, demonstrating some oxidation resistance. Although the overall mass loss of the single-phase PtAl coating is significantly better than that of the K38G alloy, the initial mass loss during cyclic oxidation indicates that the oxidation resistance of the PtAl coating is noticeably reduced compared with its static oxidation performance, failing to achieve complete oxidation resistance.

Figure 7 [Figure 7: see original paper] shows the cross-sectional morphologies of the K38G alloy and PtAl coating after 200 cycles of cyclic oxidation at 1100 °C. The K38G alloy surface formed mixed oxides with localized nodular protrusions, and typical internal oxidation occurred beneath the surface layer. After 200 cycles of cyclic oxidation, the K38G alloy surface exhibited continuous undulations, related to oxide scale spallation. After 200 cycles, the PtAl coating sample maintained an intact surface Al O scale, but partial detachment of the PtAl coating from the -NiAl layer occurred with void formation. Compared with the as-received single-phase PtAl coating (Figure 1b) and after static oxidation for 20 h (Figure 5), the PtAl layer thickness decreased significantly, while the interdiffusion zone (IDZ) became noticeably thicker and the gray-white precipitates (mainly Cr, W, Mo, and Ta, etc.) became coarser. Since PtAl is more brittle and has a lower thermal expansion coefficient than -NiAl[21], the thermal cycling process causes detachment along the coating direction and forms wrinkles to release stress, similar to the failure behavior of thermal barrier coatings due to thermally grown oxide (TGO)[22].

The detached portion of the PtAl layer in Figure 7b was magnified and subjected to EDAX line scanning, with results shown in Figure 8 [Figure 8: see original paper]. In Figure 8b, the Al and O peaks coincide, corresponding to the PtAl layer and -NiAl layer surface in the line scan region of Figure 8a, indicating that an Al O scale formed on both the PtAl layer and -NiAl layer surfaces (Figure 8b). The formation of Al O on the -NiAl surface demonstrates that the voids penetrated through and were directly exposed to air. EDAX point analysis of the spalled PtAl coating revealed a Pt content of 9.7 at% in the coating, consistent with the slightly higher Pt content near the PtAl layer in the EDAX line scan profile. The -NiAl beneath it also formed Al O , consuming some Al, causing precipitation of a small amount of -Ni Al, which mainly distributed along -phase grain boundaries (Figure 8a). The precipitation of -Ni Al primarily results from Al consumption in the -phase, including outward Al O scale formation and inward elemental interdiffusion, occurring through the following reaction: 3NiAl → Ni Al + 2Al. Meanwhile, -Ni Al precipitation at grain boundaries occurs mainly because: (1) grain boundary diffusion coefficients are several orders of magnitude higher than bulk diffusion coefficients at high temperatures[23]; (2) grain boundaries are defects where new phases preferentially nucleate at high-energy defect sites[24].

For high-temperature oxidation, especially cyclic oxidation, sample mass changes mainly result from mass gain due to oxide scale formation and mass loss due to coating/scale spallation. Thermal cycling generates thermal stresses, causing the coating and oxide scale to expand during heating and contract during cooling, producing corresponding thermal stresses. Generally, the thermal expansion coefficient of ceramics is significantly lower than that of metallic alloys. Therefore, during cyclic oxidation of conventional high-temperature protective coatings (such as MCrAlY or aluminide coatings), the oxide scale partially or completely spalls, after which Al from the coating diffuses to the surface to continue repairing and forming a new Al O scale. When the amount of oxide scale growth exceeds spallation, the sample mass continues to increase; when spallation exceeds growth, the coating mass decreases until the Al content in the coating falls below that required to repair the Al O scale, leading to coating failure.

Based on the static and cyclic oxidation results, the PtAl coating fully possesses the ability to form an -Al O scale. Its oxidation resistance is not poor when subjected to no or limited external forces, and literature[25] also reports that Pt-Al coatings with similar structures have good isothermal oxidation resistance. However, due to its inherent brittleness, the PtAl phase has a lower coefficient of thermal expansion (CTE) than both the -NiAl layer and the K38G alloy substrate. During heating (cooling), its expansion (contraction) is significantly less than that of the underlying layers, meaning the PtAl layer experiences tensile stress during heating and compressive stress during cooling. Therefore, during cyclic oxidation, the PtAl layer undergoes repeated alternating tensile (compressive) stresses, and its failure mode and process can be represented by Figure 9 [Figure 9: see original paper]. Figure 9a shows the as-received single-phase PtAl coating on the K38G superalloy substrate with a -NiAl layer, with no internal stresses present. After heating, both the substrate and coating expand, but the PtAl expands less than -NiAl, so the PtAl side at the PtAl / -NiAl interface experiences tensile stress (Figure 9b). After cooling, the K38G substrate and -NiAl contract to their original state, while the expanded PtAl contracts more slowly and to a lesser extent, thus experiencing compressive stress applied by the -NiAl layer (Figure 9c). When impurities or other defects exist at the PtAl / -NiAl interface and the compressive stress is sufficiently large, the compressive stress direction will be deflected by the defects or impurities, generating oblique compressive stress as shown in Figure 9d. When the resultant oblique compressive stress exceeds the bonding strength between PtAl and -NiAl, the PtAl layer spalls, manifesting as wrinkling and void formation. Through wrinkling and spallation, the stresses within the PtAl layer are finally released.

It should be noted that after PtAl layer spallation, on one hand, it will completely spall as oxidation continues; on the other hand, its spallation exposes the underlying -NiAl layer to air, leading to rapid consumption of the Al source in the coating' s -NiAl layer. Abnormal rapid consumption of Al is generally considered one of the main causes of high-temperature protective coating failure[26,27]. Therefore, coating spallation and rapid Al consumption during cyclic oxidation constitute the primary failure mechanisms of the single-phase PtAl coating.

Although PtAl is a brittle phase, its Pt and Al contents are both significantly higher than those in -(Ni,Pt)Al. Moretto et al.[28] reported that single-phase PtAl layers eventually evolve into -(Ni,Pt)Al after long-term high-temperature oxidation. If the distribution of PtAl phase in the coating is controlled to obtain a PtAl + -NiAl composite structure similar to that between the PtAl and -NiAl layers in Figure 1b, the brittleness drawback of the PtAl phase can be compensated by its dispersion distribution. Moreover, it can store more Pt and Al elements and transform into -(Ni,Pt)Al during high-temperature service (Figure 5), resulting in excellent comprehensive performance.

4. Conclusions

(1) A single-phase PtAl coating with a -NiAl layer was obtained on the K38G superalloy through pulse electroplating deposition and stepped heating pack aluminization.

(2) The single-phase PtAl coating possesses good isothermal oxidation resistance. The rapid initial mass increase is caused by the formation of fast-growing -Al O . As -Al O rapidly transforms to -Al O , the coating mass gain slows down.

(3) The single-phase PtAl coating exhibits poor cyclic oxidation resistance. Thermal cycling stresses cause partial spallation of the PtAl layer and create through-thickness voids that directly expose the underlying -NiAl layer to air, accelerating rapid consumption of Al in the internal -NiAl layer and resulting in premature failure.

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