High temperature characteristics of Ir–Ta coated and aluminized Ni-base single crystal superalloys

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Abstract

High temperature oxidation and hot corrosion properties of Ir–Ta coated and aluminized Ni-base superalloys are presented. An Ir–Ta binary alloy, proposed as a novel metallic bond coat material, was coated on a Ni-base single crystal superalloy TMS-75 using electron beam physical vapor deposition, followed by a conventional low activity Al pack cementation process. Cyclic oxidation tests and hot corrosion tests revealed that these Ir–Ta coated and aluminized specimens showed reasonably good oxidation and hot corrosion resistance. In addition, it was found that the formation of TCP phases is suppressed by the presence of the Ir–Ta enriched layer. These results indicated that the Ir–Ta alloy system is promising as a new metallic bond coat material for high temperature structural materials.

Keywords: EB-PVD; Ir–Ta coating; Low activity Al pack cementation process; Diffusion barrier; Ni-base single-crystal superalloy

1. Introduction

To fabricate internal combustion engines with higher heat efficiency is one of the most effective means to reducing CO2 emission. Continuous efforts have thus been made for increasing operating temperatures of jet engines and gas turbines. In practice, the inlet gas temperature in the state-of-the-art gas turbines has already reached 1773 K [1], which is well above the melting points of Ni-base superalloys used for their turbine blades and vanes. Therefore, the improvement of thermal barrier coatings (TBCs) for Ni-base superalloys is becoming increasingly important.

Since 1960s, TBCs have widely been used for blades and vanes of gas turbines [2–5]. TBCs generally consist of two coating layers; a metallic bond-coat layer and a ceramic top-coat layer. Typically, simple Al-pack cementation, PtAl [6] or MCrAlY is used as a bond-coat material for preventing the internal oxidation of substrates, and yttria stabilized zirconia (YSZ) as a top-coat material for insulating heat conduction, respectively. However, the increase in operating temperature for current turbine systems has brought about serious problems, such as the acceleration of inter-diffusion between substrates and bond-coats, which may promote the precipitation of detrimental phases in the substrates, and the rapid growth of a thermally grown oxide (TGO) on the bond-coat surface [4,5]. This leads to further enhancement of the top-coat spallation [2,3] and accordingly shortens the service life of TBCs. Development of novel bond coat materials will thus be essential in realizing next-generation gas turbines or jet engines.

In order to overcome these problems, Narita et al. proposed Re-modified aluminate coatings [7]. They reported that in this coating system, Re and Cr from the substrate form a σ phase layer which works as a diffusion barrier to suppress the outer-diffusion of Ni from the substrate. In this coating system, however, the σ phase formation takes place only when the Cr amount in the substrates is sufficiently high.

Above these as backgrounds, we have proposed an Ir–Ta binary alloy system as a candidate for the novel bond-coat material because Ir has a high melting temperature (2716 K) [8], excellent chemical stability and low oxygen permeability, while Ta also has high melting temperature (3269 K), chemical resistance to aggressive propellant gases.
and low diffusion rates in Ni-base superalloys [9,10]. Our preliminary investigation has shown that aluminized Ir–Ta-coated specimens have both good resistance against oxidation and ability to work as a diffusion retarder [11,12].

In the present study, the high temperature properties of aluminized Ir–Ta coatings have been assessed in a more detailed manner so as to evaluate the applicability of the coatings to heat-resistant turbine blades or vanes. Cyclic hot corrosion tests [13–15] and longer-term cyclic oxidation tests were conducted for the coated and uncoated specimens, and the results are discussed in relation to their microstructural characteristics.

2. Experimental details

In this paper, all specimens were prepared from a heat-treated Ni-base single crystal superalloy TMS-75 (Ni: 63.1, Co: 12.6, Cr: 3.5, Mo: 1.29, Al: 13.7, Ta: 2.05, Hf: 0.04, Re: 1.66 in at.%) [16,17]. They were cut into \(17 \times 17 \times 2 \text{ mm}^2\) plates, polished with emery paper up to \#600, and buffed with 0.25 \(\mu\text{m}\) diamond paste, and subsequently cleaned with acetone in an ultrasonic bath. An Ir–20 at.% Ta alloy, fabricated by the vacuum arc melting method, was selected as a source material, which was coated on substrates using an Electron Beam Physical Vapor Deposition (EB-PVD) method. The typical coating conditions were as follows, chamber pressure: \(1.2–3.5 \times 10^{-3} \text{ Pa}\), electron beam acceleration voltage: \(10 \text{ kV}\). Under these conditions, the typical deposition rate of approximately \(10 \mu\text{m/h}\) was obtained for a single-sided coating. After that, the specimens were turned over and the other side of the specimens was coated. Entire surfaces including the edges were coated in this manner.

The conventional Al-pack cementation was then conducted for a part of the Ir–Ta-coated and uncoated specimens. In this process, specimens were embedded in the mixture powders of Al, Fe, NH\(_4\)Cl and Al\(_2\)O\(_3\), and were heated at 1373 K for 5 h under flowing Ar atmosphere. In this way, four kinds of specimens were prepared namely: uncoated TMS-75, Ir–Ta-coated TMS-75, uncoated and Al-pack cemented TMS-75 and Ir–Ta-coated and Al-pack cemented TMS-75. Hereafter, these specimens are denoted as ‘TMS-75’, ‘Ir–Ta’, ‘TMS-75 + Al’ and ‘Ir–Ta + Al’, respectively.

In order to evaluate the high temperature properties, cyclic oxidation tests and cyclic hot corrosion tests were carried out for all kinds of specimens. As for the cyclic oxidation tests, one heating cycle was defined as heating a sample at 1373 K for 20 h in air, followed by air-cooling. The heating temperature was selected based on the estimation that the hottest section of substrate materials for blades and vanes will reach 1373 K for the next-generation gas turbines [18]. The tests were conducted for 30 cycles. The oxidation resistance of the specimens was evaluated by measuring the weight change of the sample in the passage of oxidation cycles.

Hot corrosion tests were conducted at 1173 K, because the reduction of turbine lifetime caused by the hot corrosion is reported to become most severe at temperatures ranging from 1100 to 1200 K [19]. Specimens dipped in Na\(_2\)SO\(_4\) molten salts were heated at 1173 K for 20 h in air, followed by air-cooling, and this procedure was defined as one heating cycle. This procedure was carried out for 10 heating cycles and the corrosion resistance of the specimens was evaluated by measuring the weight change of the samples in the passage of the tests.

After the hot corrosion tests, the defect morphologies of the ‘TMS-75 + Al’ and ‘Ir–Ta + Al’ specimens were investigated using a polarization measurement. It is known that chemical [20], electrical [21,22] and electrochemical techniques [23,24] are useful in evaluating the protective quality of coatings. In this study, the electrochemical technique, i.e. the polarization measurement, was applied because it was believed to be a more reliable nondestructive technique than any other methods. The two samples were soaked in a \(0.5\text{M-}\text{H}_2\text{SO}_4 + 0.05\text{M-KSCN}\) solution purged using Ar. Potentials, which were measured using the saturated calomel electrode (SCE) as the reference electrode, were automatically increased from \(-0.45\) to \(+0.45\) V at 0.38 V/s. The area exposed to the electrolyte was approximately \(0.5 \text{ cm}^2\). The test temperature was 298 K.

The microstructural evolution of the specimens was investigated by scanning electron microscopy equipped with energy dispersive spectroscopy (SEM-EDS), electron-probe microanalysis (EPMA) and X-ray diffractometry (XRD).

![Fig. 1. Weight change of specimens as a function of oxidation cycle. Specimens were kept at 1373 K for 20 h and air-cooled for each cycle.](image-url)
3. Results and discussions

3.1. Oxidation resistance

Fig. 1 shows the changes in weight of the ‘TMS-75’, ‘Ir–Ta’, ‘TMS-75 + Al’ and ‘Ir–Ta + Al’ as a function of oxidation cycles. The sample weight gain is mainly attributed to the oxidation of the surface, while weight loss is attributed to the exfoliation of volatile oxide scales from the specimen surface. Therefore, a sample demonstrating low weight change, especially low weight loss during this oxidation test, can be regarded as having a good oxidation resistance. As for unaluminized specimens, sample weight linearly decreased with a rate of approximately 2.25 (mg/cm² per oxidation cycle) after several oxidation cycles, suggesting that these specimens had completely lost protective properties against oxidation. As for Ta-coated and aluminized specimens, sudden decrease of weight after a few oxidation cycles is due to the spallation of surface layers. These results indicated that the aluminized ‘TMS-75 + Al’ and ‘Ir–Ta + Al’ have better oxidation resistance than other specimens investigated in this study. In ‘TMS-75 + Al’, the sample weight increased after a few oxidation cycles and then became stable until the 10th cycle. After that, the sample weight gradually decreased, suggesting that the spallation or exfoliation of surface oxidation layer started after 10 oxidation cycles. On the other hand, the weight change of ‘Ir–Ta + Al’ is much smaller than that of ‘TMS-75 + Al’, especially between the 10th and 30th oxidation cycles. For instance, the decrease in sample weight in ‘Ir–Ta + Al’ between the 10th and 30th cycles (2.38 mg/cm²) is about four times smaller than that in ‘TMS-75 + Al’ (13.0 mg/cm²), indicating that the former has better oxidation resistance at 1373 K.

Fig. 2. (a) Cross-sectional microstructure and (b) corresponding chemical composition profile of ‘TMS-75 + Al’ [11].
Oxidation at high temperatures can involve microstructural evolutions of substrate materials. Cross-sections of the ‘TMS-75 + Al’ and ‘Ir–Ta + Al’, which demonstrated smaller weight change in the cyclic oxidation test in this study, are thus observed to investigate the difference in microstructural evolution of the two specimens. Figs. 2 and 3 [11] show the (a) cross-sectional microstructure and (b) corresponding concentration profiles of solute elements in ‘TMS-75 + Al’, before the oxidation test (Fig. 2) and after 30 oxidation cycles (Fig. 3), respectively. As shown in Figs. 2 and 3, cross-sectional microstructure of ‘TMS-75 + Al’ had a drastic change. It originally had a duplex Ni–Al-based outer layer and an inner Al-diffusion layer structure while after the tests, ‘TMS-75 + Al’ consisted of three layers, an Al2O3-based outer oxide layer of less than 10 μm in thickness, a Ni–Al-based intermediate layer of approximately 160 μm in thickness and an Al-diffusion inner layer of 100 μm in thickness. In addition, precipitates of topologically close packed (TCP) phases, which are brightly contrasted in Fig. 3(a), spread all over the observed region.

The cross-sectional microstructure of ‘Ir–Ta + Al’ also changed after the oxidation tests as shown in Figs. 4 and 5 [11]. While as-prepared ‘Ir–Ta + Al’ before the cyclic oxidation tests consisted of three layers: the Ir-, Ta-, Ni- and Al-enriched layer, the Ni–Al-based layer and the Al-diffusion layer from the surface, after cyclic oxidation tests, the cross section of ‘Ir–Ta + Al’ can be categorized into four layers: the Al2O3-based oxide layer with a thickness of ~20 μm, the Ir-, Ta-, Ni- and Al-enriched layer with a thickness of 50–60 μm, the Ni–Al-based layer with a thickness of ~30 μm and the Al-diffusion layer with a thickness of 20–30 μm stacking in this order from the surface. Note that the Ni-content in the oxide layer is much smaller in ‘Ir–Ta + Al’ than in ‘TMS-75 + Al’. Also, in
'Ir–Ta + Al', precipitates TCP phases were observed only within 300 \( \mu \)m in depth from the surface.

These microstructural changes during oxidation tests can be explained in the following manner. When the aluminized specimens are exposed under hot air, the \( \text{Al}_2\text{O}_3 \) layer forms on the surface, which prevents the internal oxidation of the specimens. During the oxidation tests, \( \text{Al} \) is continuously consumed in order to compensate for the spallation or evaporation of surface oxides. Simultaneously, interdiffusion between the \( \text{Al} \)-enriched layer and substrate occurs for the uniform distribution of solute elements. Consequently, inward diffusion of \( \text{Al} \) in the \( \text{Al} \)-enriched surface layer to the substrate and alternative outward diffusion of solute elements in the substrate to the surface, such as \( \text{Ni} \), \( \text{Cr} \), and \( \text{Co} \), occur during the oxidation tests. In addition, the concentration of \( \text{Al} \) in the surface area decreases with increasing oxidation cycles. These phenomena will lead to two detrimental effects, (i) formation of brittle spinel oxides such as \( \text{NiAl}_2\text{O}_4 \) or volatile \( \text{NiO} \) and (ii) precipitation of detrimental TCP phases in the substrate.

Thermodynamic investigation confirmed that \( \text{NiAl}_2\text{O}_4 \) or \( \text{NiO} \) forms when the aluminum activity is low and those of oxygen and nickel are high [4]. Once an uniform and dense \( \text{Al}_2\text{O}_3 \) layer forms on the surface, such secondary oxides would never form because the permeability of nickel into the \( \text{Al}_2\text{O}_3 \) scale is very low. However, cyclic thermal stress during the oxidation test may initiate cracks in the oxide scale. If \( \text{Ni} \) is present right beneath the cracked oxide layer, it can react with oxygen in the gas and/or \( \text{Al} \) to form \( \text{NiAl}_2\text{O}_4 \) and/or \( \text{NiO} \) due to the low \( \text{Al} \) activity (\( \text{Al} \) concentration) at the reaction zone. The \( \text{NiAl}_2\text{O}_4 \) and/or \( \text{NiO} \) can then continue to grow by the transport (of oxygen gas) through the cracked \( \text{Al}_2\text{O}_3 \) layer. The slight decrease in weight for ‘TMS-75 + Al’ after 10 oxidation cycles can thus be explained by the spallation of brittle \( \text{NiAl}_2\text{O}_4 \), or exfoliation of volatile \( \text{NiO} \). Indeed, in our previous

![Cross-sectional microstructure and corresponding chemical composition profile of 'Ir–Ta + Al'](image)

Fig. 4. (a) Cross-sectional microstructure and (b) corresponding chemical composition profile of ‘Ir–Ta + Al’ [11].
investigation, X-ray analysis demonstrated the presence of NiAl₂O₄ and NiO in the ‘TMS-75 + Al’ after 10 oxidation cycles [11]. In addition, Ni concentration in the oxide layer in ‘TMS-75 + Al’ (typically 30 at.%) is much higher than that in ‘Ir-Ta + Al’ (less than 5 at.%) after 30 oxidation cycles, as shown in Figs. 3(b) and 5(b), respectively. These results support the above discussion. On the other hand, it is shown that ‘Ir-Ta + Al’ maintains stable Al₂O₃-based surface layer even after 30 oxidation cycles, resulting in the smaller mass change (2.38 mg/cm²) than that of ‘TMS-75 + Al’ (13.0 mg/cm²) from 10 to 30 oxidation cycles. Although further investigation would be required to understand the oxidation kinetics, it is speculated that the Ir-Ta-Al enriched oxide layer just beneath the surface, as shown in Fig. 5(b), may hinder the outward diffusion of Ni, thus retard the formation of NiAl₂O₄ and NiO.

It is also important to retard the microstructural evolution of substrate materials. Precipitation of TCP phases is of particular concern because it drastically impairs the creep property of substrate materials [25,26]. The TCP phase generally form by the segregation of refractory elements such as Mo, W and Re and a certain amount of Al [25,26]. During oxidation tests, while Ni, Cr and Co diffuse outward to the surface, these refractory elements tend to retain due to the lower diffusibility. The retained refractory elements and inward diffusing Al give rise to the precipitation of TCP phases. Therefore, the smaller the interdiffusion of solute elements is, the less precipitation of TCP phases occurs. From Figs. 3(a) and 5(a), it is clear that the precipitation of TCP phases is less in ‘Ir-Ta + Al’ than ‘TMS-75 + Al’, indicating that interdiffusion of alloying elements were suppressed by the presence of the Ir-Ta-enriched layer. It is
also expected ‘Ir–Ta + Al’ will not deteriorate creep properties of the substrates after cyclic oxidation tests.

The comparative study of oxidation kinetics in ‘Ir–Ta + Al’ and ‘TMS-75 + Al’ at 1373 K showed that ‘Ir–Ta + Al’ (i) demonstrates a lower weight change during the cyclic oxidation tests, (ii) forms a more stable oxide layer and (iii) has less amount of TCP phase precipitation. All results indicated the beneficial effects of the Ir–Ta coatings, which may be again attributed to the role of the Ir–Ta-enriched layer in retarding the interdiffusion of alloying elements. In the previous paper [11], where cyclic oxidation tests were conducted up to 10 cycles, the authors discussed the effect of the ‘diffusion retarder’ of ‘Ir–Ta’ on oxidation resistance and phase stabilities of substrates. Current study agreed with the discussions in the previous report, and also proved that the ‘diffusion retarder’ layer remains effective even for much longer oxidation tests (up to 30 oxidation cycles).

3.2. Hot corrosion resistance

Having a good hot corrosion resistance is another important requirement for coatings of high temperature structural materials. Comparisons of hot corrosion properties have been carried out in this section.

Fig. 6 shows the sample weight change as a function of hot corrosion cycles. Similar to the cyclic oxidation tests, the sample weight gain is mainly attributed to the formation of brittle and corrosive products such as sulfides or oxides while weight loss is mainly attributed to the evaporation of volatile oxides or spallation of such brittle and corrosive products as mentioned previously. Therefore, the significant variation in sample weight of the specimens except for ‘Ir–Ta + Al’ and ‘TMS-75 + Al’, indicates the repeated formation and spallation of brittle or corrosive products during corrosion tests, implying the poor corrosion resistance. On the other hand, sample weight changes of both ‘Ir–Ta + Al’ and ‘TMS-75 + Al’ are similar and smallest, suggesting that they have better corrosion resistance than the others. However, microstructural investigations revealed that they have different morphologies. Backscattered electron (BSE) images with concentration maps of these two specimens, derived from EPMA are shown in Fig. 7. In ‘TMS-75 + Al’ three kinds of layers are observed; an outer oxide layer with a thickness of about 5 μm (in Fig. 7(a)), an intermediate Ni-Al based layer with a thickness of about 60 μm (in Fig. 7(a)) and an inner Al-diffusion layer with a thickness of about 30 μm (in Fig. 7(a)) from the surface. Note that in the BSE image, there are a number of dark spots corresponding to cracks or voids. The distribution of these dark spots agrees very well with that of the oxygen enriched region. In addition, in the Ni–Al based layer, co-segregation of Cr and S is observed as red spots, which propagated to approximately 70 μm in depth from the surface. These results indicate that, internal oxidation and sulfuration occurred in ‘TMS-75 + Al’. In the case of ‘Ir–Ta + Al’, on the other hand, an Ir–Ta–Al enriched layer lies in between the oxide layer and the Ni–Al enriched layer. The number of cracks and vacancies are much smaller than those in ‘TMS-75 + Al’. Neither the enrichment of O, nor the co-enrichment of Cr and S are observed in the ‘Ir–Ta + Al’ specimens.

Phase identification of the surfaces of the (a) ‘TMS-75 + Al’ and (b) ‘Ir–Ta + Al’, after the hot corrosion tests was conducted by X-ray diffraction analysis as in Fig. 8. Na2SO4, which was used as the molten salt for the corrosion test, was not identified for either specimen, confirming that the molten salt was thoroughly removed from the surface when the samples were cleaned. NiAl and Al2O3 phases were identified on both surfaces of the specimens. In addition, the CrS phase was identified in ‘TMS-75 + Al’ whilst not sulfides but Ta2O5 and NaTaO3 were identified in ‘Ir–Ta + Al’. These results are consistent with the EPMA analysis described above.

In addition to the microstructural investigation, the polarization measurement enables us to understand the difference in defect morphologies of coated specimens. The results of the polarization measurement after the hot corrosion tests are shown in Fig. 9. ‘TMS-75 + Al’ gave almost two times higher current density than ‘Ir–Ta + Al’ when the electron potential was between −0.1 and +0.2 V. Since the increase of current density can be attributed to the anodic active dissolution of the base metal through some defects, this investigation also indicates that the number density of defects generating in the specimen is smaller in ‘Ir–Ta + Al’ than in ‘TMS-75 + Al’. It should also be noted that the large variation in current density of ‘TMS-75 + Al’ at the potential of 0.2 V or above suggests the occurrence of pitting corrosion in the substrate.

From these investigations, it is revealed that ‘Ir–Ta + Al’ has the higher corrosion resistance than any other specimens at 1173 K. There may be two major roles of the Ir–Ta layer for improving hot corrosion resistance: (i) a diffusion barrier of solute elements as described in Section 2
and (ii) formation of tantalum oxides to protect the corrosive attacks by S.

It is well established that S and O in the molten salts diffuse very quickly at intergranular sites [27]. Especially, S diffuses extremely quickly and attacks grain boundaries, which gives rise to the formation of cracks. Also, when small voids are formed in a coating layer, O and S segregate at these small voids to form oxides and sulfides, which may become crack propagation sites. In ‘TMS-75 + Al’ as explained in the previous section, Ni, Co and Cr easily diffuse outwards. The outward diffused Cr and S thus formed CrS as shown in Fig. 7, which may have enhanced the initiation and propagation of cracks. Although further investigation is required to understand the detailed mechanism of CrS formation and the reason why only CrS is detected, it is concluded that many cracks and voids generated in ‘TMS-75 + Al’ are attributed to the formation of CrS which would degrade the mechanical properties of the coatings.

In ‘Ir–Ta + Al’, on the other hand, $\alpha$-Al$_2$O$_3$, NiAl, Ta$_2$O$_5$ and NaTaO$_3$ peaks were identified from X-ray analysis. Lee et al. [28] reported that NaTaO$_3$ is formed from Na$_2$SO$_4$ and Ta$_2$O$_5$ according to the following reactions:

$$\text{Na}_2\text{SO}_4 = \text{Na}_2\text{O} + \text{SO}_3 \quad (1)$$

$$\text{Ta}_2\text{O}_5 + \text{Na}_2\text{O} = 2\text{NaTaO}_3 \quad (2)$$

Since the melting point of NaTaO$_3$ is 2083 K, NaTaO$_3$ will not be melted during the hot-corrosion tests and thus will not work as the corrosive agents. In addition, Ta$_2$O$_5$, a melting point of 2163 K, is known as the stable oxide. In ‘Ir–Ta + Al’, the presence of both $\alpha$-Al$_2$O$_3$ and Ta$_2$O$_5$ on the specimen surface may have protected the surface from so-called ‘sulfur and oxygen attack’ and prevented

Fig. 7. Concentration map of (a) ‘TMS-75 + Al’ and (b) ‘Ir–Ta + Al’ after hot-corrosion tests; (1) oxide layer, (2) Ni–Al based layer, (3) inner Al-diffusion layer, (4) substrate, (5) Ir–Ta–Al enriched layer.
the formation of sulfides such as CrS and voids. It is also suggested that the Ir–Ta–Al enriched layer hinders the outward diffusion of Cr to the surface, thus avoiding the reaction between Cr and S. These results can explain the better corrosion resistance of ‘Ir–Ta+Al’.

This study confirmed that the aluminized Ir–Ta coating has both good oxidation and hot corrosion properties, thus, it is promising as a novel bond-coat material. As a next step, optimization of the coating parameters, such as Al-pack cementation condition, Ir–Ta composition, and further investigation of oxidation and hot corrosion properties at various temperatures will be required in order for this alloy system to be practically feasible.

4. Conclusions

Ir–Ta coated and aluminized coatings are obtained by an EB-PVD of Ir–Ta on Ni-base single crystal superalloy TMS-75, followed by an Al-pack cementation process. Cyclic oxidation tests at 1373 K and hot-corrosion tests with dipped molten salts (Na₂SO₄) at 1173 K were carried out to characterize the high temperature properties of the coating and the following results were obtained.

- The Ir–Ta coated superalloy with Al-pack cementation showed good oxidation resistance. Moreover, the precipitation of the third phase was suppressed.
- The hot corrosion tests and the following investigations revealed that in the Ir–Ta coated superalloy with Al-pack cementation, Ir–Ta–Ni–Al enriched layer prevented the infiltration of oxygen and sulfur, and retarded the formation of voids and cracks in the inner layer, confirming that the Ir–Ta coated superalloys with Al-pack cementation also has better corrosion resistance.

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