EFFECT OF Re AND Ta ON HOT CORROSION RESISTANCE OF NICKEL-BASE SINGLE CRYSTAL SUPERALLOYS

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Abstract

Hot corrosion behavior of several Ni-base single crystal (SX) superalloys with different Re, Ta and Cr contents at 750 °C and 900 °C in static air has been systematically investigated using the “deposit recoat” method. It was shown that, both Re and Ta can improve the hot corrosion resistance of SX superalloys. Re may alter the activities of elements and therefore delay the deformation and cracking of oxide scale, and may enable the self-healing of the oxide scale. The advantage to hot corrosion properties of adding Ta depends on Cr content in the superalloys. The beneficial effect of Ta is more pronounced in alloys with a low Cr content. Ta was found to exhibit a beneficial effect by promoting the formation of NaTaO_3 and TaS_2 , and therefore inhibiting the formation of liquid Na_2MoO_4 and Ni-sulfides. It seems that Re and Ta are promising elements that allow us to design hot corrosion resistant superalloys with superior high temperature properties.

Introduction

Hot corrosion resistant superalloys are extensively used in industrial gas turbines (IGTs) because of their outstanding mechanical strength and hot corrosion resistance. In order to improve the inlet temperature of IGTs, much effort has been made to improve the high temperature strength of the hot corrosion resistant superalloys. Generally, around 12-16 wt.% Cr along with at least 3 wt.% Ti is required for acceptable hot corrosion resistance [1-4]. However, a high Cr content limits the potential to use more refractory elements (Re, Mo, W and Ta) to further improve the mechanical strength of the alloys, i.e., one needs to balance the hot corrosion resistance, high temperature strength and microstructural stability carefully during alloy design.

As revealed by previous researchers, W and Mo show detrimental effects on hot corrosion resistance, because their acid oxides can accelerate the corrosion rate dramatically [5-7]. Re and Ta may be the choices for stronger alloys, but very limited information is available regarding their effects on the hot corrosion resistance and mechanism, especially in alloys with low Cr content.

Matsugi and coworkers [8-10] have investigated the effect of Re on hot corrosion behavior and clearly showed that the hot corrosion resistance was improved remarkably by small Re additions, though the sample analysis and mechanism were not given. Caron and Czech also reported the same effect [11, 12]. They regarded the effect of Re as being a diffusion barrier of the reactive elements. But, there is no corresponding experimental evidence. Gurrappa [13] has observed Re-containing products in hot-corroded samples of CMSX-4. They explained the hot corrosion behavior of the alloy as electrochemical in nature. However, role of Re was not mentioned. Therefore, further investigation was needed to better understand the mechanism of Re on hot corrosion resistance.

Fryburg [14-16] has studied the hot corrosion behavior of several Ni-base superalloys and concluded that Ta improved the hot corrosion resistance by facilitating the formation of solid NaTaO_3 and suppressing the formation of liquid $\text{Na}_2\text{Mo(W)O}_4$. It has been reported that a better hot corrosion resistance would be obtained if the Ta/(Mo+W) compositional ratio is close to unity in the alloy [10, 17]. In our previous work [18], beneficial effect of Ta was also observed. It seems that Ta promoted the formation of NaTaO_3 and (Cr, Ti) TaO_4 , with the latter spinel phase acting as a diffusion barrier of ions. However, results acquired by Zhang [19] and Cui [20] demonstrated that Ta deteriorated the hot corrosion resistance, for reasons unstated. Currently, most work studying the effect of elements on hot corrosion resistance focuses on the formation of oxygen-containing oxides or spinels, such as Cr_2O_3 [21, 22], NaTaO_3 [14, 15] and (Cr, Ti) TaO_4 [18], while the formation of sulfides that may dramatically affect the corrosion mechanisms [23] has not been monitored.

The aim of our present paper is to study the effect of Re and Ta on hot corrosion behavior of SX Ni-base superalloys with different Cr contents, trying to analyze the corrosion mechanism and provide new perspective in alloy design for high strength hot corrosion resistant SX superalloys.

Experimental

Nominal chemical compositions of the experimental alloys are listed in table 1. The experimental alloys were divided into three groups to conduct hot corrosion test: (I) E1 (12Cr-0Re) and E7 (12Cr-2Re); (II) E7 (12Cr-2Re-4Ta) and E71 (12Cr-2Re-6Ta); (III): E74 (5Cr-6Ta) and E76 (5Cr-9Ta), with contents of other elements identical in each group.

SX rods of the experimental alloys measuring 16 mm in diameter and 220 mm in length were fabricated by high rate solidification (HRS) method. All rods received proper heat treatment. Hot corrosion specimens were cut from as heat-treated rods. The surfaces of the samples were prepared by grinding on SiC paper to #800 and all the samples were chamfered before test. Ultrasonic clean with acetone and alcohol was performed after all the preparation processes.

Hot corrosion test was carried out at 750 °C and 900 °C. The salt-recoating method, which has been described in detail in our previous paper [24], was employed in the experiments. Specimens were placed on a hot Ni plate and sprayed with saturated aqueous solution of Na_2SO_4 until their surfaces were coated with 0.3~0.5 mg/cm^2 salt. The sprayed specimens were put into separated corundum crucibles and placed in static air at 1 atm at test temperature to perform the hot corrosion test. The test was periodically interrupted every 20 h and the samples were removed from the furnace for weight monitoring and salt-recoating. The time interval between being taken out and weighted was 1 h in

every cycle. Each data point of the mass change was the average of three samples.

Scanning electron microscopy (SEM) equipped with an energy dispersive spectroscopy (EDS), electro-probe microanalyser

(EPMA) and X-ray diffraction (XRD) were used to characterize the microstructure and to identify the corrosion products. To prevent dissolving the water soluble corrosion products into water, all the cross-sectioned samples were ground and polished in kerosene [14].

Table 1 Nominal chemical compositions of the experimental alloys in wt.%.

Alloy	Cr	Co	Mo	W	Ti	Al	Ta	Re	Ni
E1	12.00	9.00	1.90	4.00	3.90	3.40	4.00	-	Bal.
E7	12.00	9.00	1.90	4.00	3.90	3.40	4.00	2.00	Bal.
E71	12.00	9.00	1.90	4.00	3.90	3.40	6.00	2.00	Bal.
E74	5.00	10.00	2.00	6.00	-	5.50	6.00	3.00	Bal.
E76	5.00	10.00	2.00	6.00	-	5.50	9.00	3.00	Bal.

Results

Hot Corrosion Resistance and Products at 750 °C

The three groups of samples were put together to conduct the hot corrosion test at 750 °C. Here we only show a representative macroscopic appearance of the samples corroded for 200 h in Fig. 1, due to the observation that the morphologies of all the samples were similar during the whole test. The sprayed salt still can be seen and the sample was still complete.

Figure 1 Macroscopic appearance of the samples corroded for 200h at 750 °C.

Figure 2 (a) Weight gain versus time curves for the experimental alloys with a surface coating of Na_2SO_4 at 750 °C. (b) and (c): Representative surface and cross-section morphologies of the samples corroded for 200 h at 750 °C. (d) EDS result of point A and B in (b) and (c).

Weight gain versus time curves for the experimental alloys are shown in Fig. 2a. The mass change of all alloys followed the linear law. Representative surface and cross-section morphologies of the samples corroded for 200 h are illustrated in Fig. 2b and 2c.

The surfaces of the samples were covered by pure condensed Na_2SO_4 , as was confirmed by EDS (Fig. 2d). The interface of the substrate and the salt coating was smooth and flat (Fig. 2c).

Hot Corrosion Resistance and Products at 900 °C

Re Effect Hot corrosion kinetics of group I alloys are shown in Fig. 3. It can be seen that, the mass change of the two alloys were similar in the initial 100 h. Both curves followed parabolic law. After 100 h, corrosion kinetics of the Re-free alloy changed. E1 experienced rapid mass change in the last 100 h. The overall corrosion kinetics of the Re-containing alloy followed multi-stage parabolic law and the inflection points were 320 h and 400 h. The mass of E7 increased slowly during the whole test.

Figure 3 Weight gain versus time curves for group I alloys with a surface coating of Na_2SO_4 at 900 °C.

The macroscopic morphologies of the group I alloys corroded at 900 °C is given in Fig. 4. After 20 h corrosion, the surfaces of both alloys became black, but they were still flat and complete (Fig. 4a and 4d). As of 100 h, the surfaces of E1 samples were covered with small protrusions (Fig. 4b), while the time that protrusions appeared on E7 was 160 h (Fig. 4e and 4f). After protrusions occurred, the oxide scale on E1 became extremely fragile and started to crack and spall off. As of 200 h, the E1 sample was severely damaged. Cracking and spallation was remarkable (Fig. 4c). However, the E7 samples were nearly unchanged after the protrusions appeared. Until 440 h, the E7 samples were still compact and complete.

Figure 4 Macroscopic morphologies of the group I alloys corroded at 900 °C with a surface coating of Na_2SO_4 . (a) E1 for 20 h. (b) E1 for 100 h. (c) E1 for 200 h. (d) E7 for 20 h. (e) E7 for 100 h. (f) E7 for 160 h. (g) E7 for 440 h.

The cross-section morphologies of E1 are illustrated in Fig. 5, in which the phases were identified according to the EDS and XRD analyses (When comparing the cross-section morphologies of

protrusions on E1 and E7, we removed the residual salt on the samples.). The morphology of the protrusion on E1 at 100 h is given in Fig. 5a. The outer layer of the protrusion was (Ni,Cr,Ti)-O oxides. At the tip of the protrusion, the outer oxide layer spalled off. The sub-surface layer was mixed oxides of Al, Cr, Ni and Ta. Cracks parallel to and perpendicular to the surface of the protrusion can be seen (Fig. 5a and 5b). The inner sulfide layer was almost continuous. Molten salt penetrated the outer oxide layer at 120 h (Fig. 5c). A continuous and compact salt layer can be seen at the middle of the oxide layer. As of 200 h, both the salt layers and the NiO layers became loose and porous (Fig. 5d). Dissolved NiO grains dispersed in the honeycomb salt layer. (Al,Ni,Cr)-O oxide network extended into the substrate. CrS_x ($1.000 < x < 1.500$) particles can be seen in the front of the corrosion layer (The stable phases in Cr-S system can be denoted as CrS_x , with $1.000 < x < 1.500$ [25]).

The cross-section morphologies of E7 are presented in Fig. 6. The outer (Cr,Ni,Ti)-O oxide layer was still complete and compact at 200 h. A continuous residual salt layer can be seen at the outermost of the sample surface. The inner Al_2O_3 layer was almost continuous. The cross-section morphology of the protrusion formed on E7 at 440 h is given in Fig. 6b (The sample was washed by boiling water for comparison with E1.). The (Cr,Ni,Ti)-O oxide layer on protrusions was still integrate after 440 h. The amount of the inner sulfides was much less than that in E1 at 100 h. Cracks and holes can be seen.

Figure 5 (a) Cross-section morphology of protrusion on E1 at 100 h. (b) Higher magnification of the rectangle area in (a). (c) Typical cross-section morphology of flat region on E1 at 120 h. (d) Typical cross-section morphology of E1 at 200 h.

Figure 6 (a) Typical cross-section morphology of E7 at 200 h. (b) Cross-section morphology of protrusion on E7 at 440 h.

Ta Effect in High Cr Alloys Hot corrosion kinetics of group II alloys at 900 °C are given in Fig. 7. The corrosion kinetics of E7 (12Cr-2Re-4Ta) and E71 (12Cr-2Re-6Ta) were similar, both of the two alloys followed multi-stage parabolic law (The group I and group II were conducted as two individual tests. Differences found in Fig. 3 and Fig. 7 for E7 were probably due to the differences in the salt amount or its uniformity in the two tests.).

The macroscopic appearances of the group II alloys corroded for 240 h at 900 °C are shown in Fig. 8. After 240 h corrosion, the two samples were still compact and complete. Yellow products can be seen on their surfaces.

Figure 7 Weight gain versus time curves for group II alloys with a surface coating of Na₂SO₄ at 900 °C.

Figure 8 Macroscopic appearances of the group II alloys corroded for 240 h at 900 °C with a surface coating of Na₂SO₄. (a) E7. (b) E71.

XRD results of the samples corroded for 20 h and 240 h are illustrated in Fig. 9. After 20 h corrosion, the main products on E7 were Cr₂O₃, TiO₂ and spinels. While for E71, Ta-containing products TaO₂ and TiTaO₄ were detected besides Cr₂O₃, TiO₂ and spinels. As of 240 h, the phases detected on E7 and E71 were similar. The main constituent of their corrosion scales were Cr₂O₃, Na₂Cr₂Ti₆O₁₆ and residual salt.

Figure 9 XRD results of the group II samples with different corrosion time. (a) E7 for 20 h. (b) E71 for 20 h. (c) E7 for 240 h. (d) E71 for 240 h.

Surface morphologies and EDS analyses of the corrosion scales formed after 240 h exposure at 900 °C are presented in Fig. 10. The surface morphologies of E7 and E71 were similar. Their surfaces were covered by a condensed salt film (mark as “A” in Fig. 10). Cracks were produced because of cooling and surface tension. EDS analysis of the salt film (Fig. 10c) indicated that the salt wasn’t the initially sprayed pure sodium sulfate. Chromate and titanate, which were probably introduced by the dissolution of the corresponding oxides, were also a part of the salt film. Besides the salt film, a white needle-like phase (marked as “B” in Fig. 10) containing Na, Cr, Ti and O (Fig. 10d) was observed, with its amount on E71 more than on E7.

Cross-section morphologies of the corrosion scales are given in Fig. 11. After 20 h exposure, the structures of the corrosion scales formed on the two alloys were similar. They consisted of an outer oxide layer, an inner Al₂O₃ layer and a sulfide layer. The dominant constituents of the outer layer were Cr- and

Ti-containing oxides and spinels. White phases enriching in Ta can be observed in E71. As of 240 h, the structures of the corrosion scales on E7 and E71 were almost unvaried, excepting

the outermost thick salt layer. Cr_2O_3 was the main products on E7 and E71.

Figure 10 Surface morphologies of the corrosion scales formed on the samples of group II alloys after 240 h exposure at 900 °C. (a) E7. (b) E71. (c) and (d): EDS results of A and B in (a) and (b).

Figure 11 Cross-section morphologies of group II alloys corroded for different time at 900 °C. (a) E7 for 20 h. (b) E71 for 20 h. (c) E7 for 240 h. (d) E71 for 240 h.

Ta Effect in Low Cr Alloys Typical hot corrosion curves of group III alloys are shown in Fig. 12. In the initial 60 h, the mass changes of the two alloys were similar. After that, E74 experienced rapid mass gain, while the mass change of E76 was still slow. The abrupt mass change of E76 occurred at 140 h. The final mass gain of E74 was almost three-fold that of E76.

Figure 12 Weight gain versus time curves for group III alloys with a surface coating of Na_2SO_4 at 900 °C.

The macroscopic morphologies of the group III alloys are presented in Fig. 13. The corrosion products on E74 started to peel off at 60 h, while the surfaces of E76 were still compact and complete at that time. After 200 h corrosion, both of the surfaces of the two alloys were loose. Cracking and spallation were remarkable.

Figure 13 Macroscopic morphologies of the group III alloys corroded at 900 °C with a surface coating of Na_2SO_4 . (a) E74 for 60 h. (b) E76 for 60 h. (c) E74 for 200 h. (d) E76 for 200 h.

XRD patterns of the corrosion products formed on E74 are given in Fig. 14. When corrosion time was less than 60 h, the corrosion products did not change significantly. They consist of Al_2O_3 , NaTaO_3 and NiO . After 60 h, peaks of Al_2O_3 and NaTaO_3 disappeared, only NiO can be detected. The products formed on E76 can be found in our previous paper [24]. Compared with E74, Al_2O_3 and NaTaO_3 were still detectable on E76 after 120 h exposure. NaTaO_3 still existed even at 200 h.

Cross section morphologies of E74 are shown in Fig. 15. A three layer structure including an outer oxide layer, an inner oxide layer and an inner sulfide layer was observed. After 20 h corrosion, a thin (Co, Cr, Al, Ta)-doped NiO layer was formed at the surface of the sample and some CrS_x particles dispersed on the inner

depleted zone. With corrosion time increased, both the NiO layer and the sulfide layer became thicker and the inner oxides appeared. As of 80 h, the microstructure and composition of the corrosion scale changed substantially. The outer layer became thicker and porous. Cracks can be seen between the outer layer and the inner layer. Besides the previously existing CrS_x particles, liquid NiS_x network was observed in the inner layer (Fig. 15d and 15g). As of 200 h, the entire substrate was corroded (Fig. 15e). Cracks extended from the interface of the porous outer layer and the inner network to the interior of the substrate. A white layer enriched in Ni and W can be observed between the outer layer and the inner network (Fig. 15f).

Figure 14 XRD patterns of E74 corroded for different time.

The initial corrosion scale formed on E76 was also composed of an outer doped NiO layer, an inner Al_2O_3 layer and an inner CrS_x layer. With corrosion time increased, the outer NiO layer thickened. The Al_2O_3 layer developed to an Al_2O_3 -dominant multi-oxides network layer. However, the constituent of the sulfide layer was different from that of E74. TaS_2 was formed instead of NiS_x . The constituent of the sulfide layer changed in this order: $\text{CrS}_x \rightarrow \text{CrS}_x + \text{TaS}_2 \rightarrow \text{TaS}_2 \rightarrow \text{CrS}_x + \text{TaS}_2$ ($1.000 < x < 1.5000$). The detailed evolutionary process of corrosion scales on E76 was reported in our previous paper [24].

Discussion

Two temperature regimes of molten sulfate-induced hot corrosion have been studied for nickel and their alloys by previous researchers. At temperatures around 900 °C, pure Na_2SO_4 is liquid. Hot corrosion occurred around this temperature is termed as Type-I hot corrosion. However, hot corrosion can also occur below the Na_2SO_4 melting point of 884 °C. Rapid attack at temperatures of around 700 °C is called Type-II hot corrosion. Gas containing SO_3 is necessary to develop the liquid NiSO_4 - Na_2SO_4 which is the corrosion medium of the Type-II hot corrosion of Ni-base alloys [26]. In the present test, SO_3 was not introduced in the test system, and the evaporation of SO_3 from the Na_2SO_4 salt at 750 °C was not enough for NiSO_4 - Na_2SO_4 formation. Thus, no significant reaction between the solid salt deposit and the alloys was observed at 750 °C in the present experiment. The test method should be modified for better investigation of the low temperature hot corrosion behavior of the alloys. As the inlet temperature of IGTs increases, the operating

temperature of the blades increases. Type-I hot corrosion is thus of high importance. The discussion will focus on hot corrosion at

900 °C.

Figure 15 Cross-section morphologies of E74 with different corrosion time. (a), (b), (c) and (e) correspond to the cross-section of 20 h, 40 h, 80 h and 200 h. (f) Higher magnification of the corrosion layers in (e). (g) Higher magnification of the rectangle area in (c). (d) EDS result of the region “B” in (g). (h) EDS result of the region “A” in (f).

In the molten Na_2SO_4 , the following reaction occurs [23]:

The hot corrosion of all alloys can be divided into two stages: an initiation stage and a propagation stage. As soon as the coated sample was placed into the furnace, it was oxidized by the molten salt (initiation stage). With corrosion proceeding, the basicity of the salt increases, the dissolution of protective oxides occurred. The degradation process stepped into the propagation stage.

Re Effect

According to the above hot corrosion test results, Re improved hot corrosion resistance of the alloy remarkably. This was probably because Re addition increased the activity of Cr in the alloys. As a result, the Cr_2O_3 -dominant scale on Re-containing alloy was more pure and compact than that on Re-free alloy. The integrated Cr_2O_3 layer was more resistant to molten salt and more effective in inhibiting the inward diffusion of oxygen and sulfur. Thus, the formation of protrusions and penetration of salt on Re-containing alloys was postponed, and hot corrosion performance was improved.

For Re-free alloy, the protective oxide scale was easily destroyed. Thus, the released oxygen and sulfur traverse through the scale and caused inner oxidation and sulfidation. Due to the big PBR (Pilling-Bed-Worth Ratio) value of the inner corrosion productions ($\text{PBR}_{\text{Al}_2\text{O}_3} = 1.28$, $\text{PBR}_{\text{CrS}_x} = 2.8 \sim 3.87$), compressive stress was generated in the substrate. Simultaneous deformation of the scale and the substrate then occurred to accommodate the stress. As a result, protrusions were produced and tensile stress was generated in the corrosion scale. Consequently, cracks were produced at tip of the protrusion.

After protrusions and cracks were produced in E1 at 100 h, the alloy experienced rapid corrosion because more corrodents were

introduced in the substrate (Fig. 5c). Penetration of salt and fluxing of oxides were remarkable on E1 (Fig. 5d). For E7, protrusions appeared at 160 h, but the mass change of E7 did not increase correspondingly. The inflection points of the curve were 320 h and 400 h. The overall corrosion kinetics of Re-containing alloy followed multi-stage parabolic law, which was probably because cracks in the outer scale open and close periodically. This phenomenon has also been observed in previous literatures studying the oxidation behavior of Cr [27, 28]. This observation may also be attributed to the increased activity of Cr in Re-containing alloy, which allows more Cr to diffuse outward to heal the cracks in the protective scale.

Ta Effect

As was well known, Cr_2O_3 was the most effective scale to resist Type-I hot corrosion attack [2, 17, 21-23, 29-31]. Thus, the different hot corrosion behavior of alloys containing different Ta can be related to the evolution of Cr_2O_3 scale in the alloys.

In E7 and E71 which have high Cr content, Cr_2O_3 was formed as soon as the samples were put into the furnace. During the whole test, Cr was sufficient to keep the completeness of the Cr_2O_3 scale. The Cr_2O_3 scale can well protect the substrate from hot corrosion. Although small amount of Ta-containing products TaO_2 and TiTaO_4 were formed in alloys with higher Ta content (E71), they did not affect the corrosion kinetics of alloy.

On the contrary, in E74 and E76 which have medium Cr content, Cr was insufficient to promote the formation of a continuous Cr_2O_3 scale. Instead, sufficient Ni enabled the formation of a NiO layer which was susceptible to Na_2SO_4 -induced hot corrosion [23]. For E76 which has higher Ta content, NaTaO_3 , which inhibited the formation of $\text{Na}_2\text{Mo(W)O}_4$ and resulted in postponed acidic fluxing [14]. This was formed as soon as the test started and remained throughout the test. The compact oxide layer can act as a diffusion barrier of oxygen. A low oxygen partial pressure

which permitted the formation of TaS₂ was thus established. The detailed discussion of formation of sulfides can be found in our previous paper [24]. Formation of TaS₂ led to the release of Cr due to the decomposition of CrS_x, and therefore promoted the formation of Cr₂O₃, which can lower the diffusion rate of ions and the oxygen partial pressure in turn. However, for E74, the catastrophic hot corrosion caused by Na₂Mo(W)O₄ made the outer NiO layer loose and porous. As a result, the inner oxygen partial pressure was increased. According to Ellingham diagram for sulfides [24, 32] and the M-O-S diagrams [15, 24, 33] produced by HSC chemistry® version 6.0, TaS₂ formation was not favorable in E74. Because of the large amount of sulfur introduced into the substrate through the porous oxide scale, Cr was insufficient for sulfide formation. Liquid NiS_x was formed which further increased the corrosion by accelerating the diffusion of ions. After NiS_x was formed, the corrosion rate of E74 increased dramatically. The sample was severely damaged at 200 h.

Summary

The following conclusions can be drawn based upon analyses of the results obtained in this work:

- (1) No significant reaction between the solid salt deposit and the alloys was observed using the present test method without introducing SO₃. The hot corrosion behavior of the alloys at 750 °C is similar.
- (2) The overall corrosion kinetics of the Re-containing alloy follows multi-stage parabolic law. Re postpones the deformation and cracking of oxide scale and enables the self-healing of cracks in oxide scale. It is probably because Re alters the activities of elements.
- (3) Ta promotes the formation of TaS₂ besides Ta-containing oxides and spinels, and inhibits the formation of Na₂Mo(W)O₄ and NiS_x. The beneficial effect of Ta is more pronounced in alloys with lower Cr content.

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