Hot Corrosion Behavior of a Powder Metallurgy Superalloy Under Gas Containing Chloride Salts

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Abstract
Hot-corrosion behavior of a powder metallurgy superalloy (Alloy 1) under gas containing chloride salts at 700 °C, 750 °C and 800 °C were investigated via weight-gain measurements, X-ray diffraction (XRD), scanning electron microscopy (SEM), and electron probe micro analysis (EPMA). The hot-corrosion behavior of a similar alloy (Alloy 2) at 800 °C and the same conditions were also carried out for comparison. The experimental results showed that the average mass gain of Alloy 1 increases as the temperature elevates. The corrosion kinetics followed linear power law at 700 °C, 750 °C and 800 °C. The corrosion layers obtained after 100 h of hot corrosion were mainly composed of Cr2O3, TiO2, Al2O3, NiO and NiCr2O4. The cross-sectional morphologies and corresponding elemental maps indicated that the chloride salts penetrated into the corrosion product and caused it to produce many cavities and cracks. According to these results, the hot corrosion of Alloy 1 under gas containing chloride salts is confirmed to be an accelerated oxidation process due to the damage in integrity of the oxide film caused by continuous corrosion with chloride salts. Compared to Alloy 2, the increased Co and Al content in Alloy 1 with better hot corrosion resistance at 800 °C promoted the rapid formation of continuous Cr2O3 and Al2O3 protective films on the alloy surface in which Co inhibited internal oxidation of Al through the third element effect.

Keywords Powder metallurgy superalloy · Gas hot corrosion · Chloride salts · Accelerated oxidation · Third element effect

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Introduction

Nickel-based superalloys consisting of a continuously disordered $\gamma$ matrix and ordered $\gamma'$ precipitates are widely used in aircraft turbine engines and subjected to a highly aggressive environment, in which both good mechanical properties and high oxidation resistance are required [1–3]. Powder metallurgy superalloys have quickly become the preferred material for advanced aero-engine disks with the advantages of uniform structure, no macro-segregation, high yield strength and decent fatigue performance. At the same time, among current turbine manufacturing technologies, powder metallurgy is regarded as a mature and reliable method for high-performance turbine disks. In order to improve the service temperature and comprehensive performance of powder metallurgy superalloys, the third generation of powder metallurgy superalloys, e.g., Alloy 1 (its composition is listed in Table 1), was invented by Beijing Institute of Aeronautical Materials on basis of the second generation of powder metallurgy superalloys, e.g., Alloy 2 (Table 1). The main difference between the two alloys lies in the composition of Co, Al, Cr et al. Alloy 1 is the key material to ensure the high performance and reliability of the most advanced aero-engines.

Normally, the oxidation/corrosion resistances of Ni-based superalloys are primarily determined by the content of Cr/Al due to the thermally-induced growth of continuous protective $\mathrm{Cr}_2\mathrm{O}_3/\mathrm{Al}_2\mathrm{O}_3$ scales [4–7]. Compared to $\mathrm{Cr}_2\mathrm{O}_3$ scales, $\mathrm{Al}_2\mathrm{O}_3$ scales provide superior oxidation resistance at temperatures above 871 °C [8, 9]. However, $\mathrm{Cr}_2\mathrm{O}_3$ exhibits better hot corrosion resistance to sulfate other than $\mathrm{Al}_2\mathrm{O}_3$ because Cr-rich phases (gamma) tend to be more resistant to sulfate induced corrosion because $\mathrm{Cr}_2\mathrm{O}_3$ is able to establish protective behavior faster than $\mathrm{Al}_2\mathrm{O}_3$ [10–12]. Co improves mechanical properties at high temperature, but the effects of Co on oxidation behavior are unclear as different researchers have concluded differing results. For example, Choi et al. [13] indicated that the addition of Co could slightly increase the isothermal oxidation resistance of $\mathrm{Ni}_3\mathrm{Al}$ base alloy but decreased the cyclic oxidation resistance at 1000–1200 °C. Weiser et al. [14] reported that the replacement of Ni by Co in Ni–9Al–8W–8Cr (at.%) alloy enhanced the oxidation resistance at low temperature ($\leq$850 °C). Ismail et al. [15] indicated that an increase in Co concentration decreased the oxidation performance of Co–Ni base superalloys at 800 °C. Therefore, the effect of Co on oxidation resistance is complex, and may be affected by other elements, environments, and temperatures. However, the addition of Co can improve the hot corrosion resistance of Ni-based superalloys [13, 16, 17].

In marine environment, gas hot corrosion caused by chloride salts could significantly reduce the service life of powder turbine disks [18]. Hot corrosion became a topic of important and popular interest in the late 1960s as gas turbine engines of military aircraft suffered from corrosion during operation over seawater [19–26]. For wrought and

| Table 1 | Chemical composition of two powder metallurgy superalloys (wt%) |
|---------|-------------------------------------------------------------|
| Alloy | Cr | Al | Co | Ti | W | Mo | Nb | Ta | Ni |
|-------|----|----|----|----|---|----|----|----|----|
| Alloy 1 | 13.0 | 3.6 | 20.0 | 3.5 | 4.3 | 2.9 | 1.5 | 1.5 | Balance |
| Alloy 2 | 15.8 | 2.2 | 13.0 | 3.8 | 4.1 | 4.3 | 0.8 | – | Balance |

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cast superalloys, many attentions have been paid on their hot corrosion behaviors [15, 27–33]. However, there are few studies on high temperature corrosion of powder metallurgy superalloys in which the mechanism is still not clear. At present, investigations of Alloy 1 are mainly concentrated on microstructure, preparation process and mechanical properties, while resistance and mechanism of gas hot corrosion of Alloy 1 remain unclear.

In this study, gas hot corrosion tests at different temperatures were performed to obtain corrosion kinetics of Alloy 1 to evaluate corrosion resistance. Phase constitutions of corrosion layers were detected by X-ray diffraction (XRD). Surface and cross-sectional morphologies as well as elemental distributions in corrosion layers were observed by scanning electron microscopy (SEM) and electron probe micro analysis (EPMA), respectively. Since the service temperature of the turbine disk developed from Alloy 1 will reach 800 °C. In order to compare the hot corrosion resistance at this temperature, the gas hot corrosion tests of Alloy 2 at 800 °C were also carried out. The experimental results were analyzed to uncover the hot corrosion mechanism that would promote a better understanding of corrosion resistance in Alloy 1.

Experimental Procedures

Specimens Preparation

The chemical compositions of Alloy 1 and Alloy 2 were analyzed and listed in Table 1. Specimens with dimensions of 30 mm in length, 10 mm in width and 2 mm in thickness were used. Before gas hot corrosion tests, specimens were ultrasonically cleaned in acetone and ethanol, then dried at 120 °C for 1 h.

Gas Hot Corrosion and Oxidation Tests

The gas hot corrosion tests were carried out with reference to China Aviation Standard HB 7740–2004 which is the most-widely used by China Aviation Industry to evaluate corrosion resistance of superalloys and high temperature protective coatings. The principle of gas hot corrosion test is to use a special test equipment (Fig. 1) to form corrosive gas through continuous mixed combustion of compressed air, atomized aviation fuel and artificial seawater, then the gas is sprayed on the surface of specimen to cause corrosion. The equipment is an atmospheric low-speed gas corrosion test device, which has the characteristics of accurate control of test parameters and wide adjustment range. It can better simulate the temperature, corrosion medium and cold/hot alternation of the working environment of turbine components. According to the standard HB 7740-2004, it is a cycle of continuous corrosion for 55 min at the test temperature and cooling out for 5 min. The chloride salts in artificial seawater mainly containing NaCl and KCl were used in the present paper since powder metallurgy superalloy suffers severe hot corrosion from high temperature gas containing chloride salts during operation over seawater.
The original artificial seawater containing chloride salts was prepared according to the composition in Table 2. Specimens were inserted in the gas hot corrosion test equipment for corrosion of 25 h, 50 h, 75 h, 100 h at 700 °C, 750 °C and 800 °C. Afterward, specimens were air cooled to room temperature and weighed using an electronic balance with an accuracy of 0.0001 g. Oxidation of 100 h in air at 700 °C, 750 °C and 800 °C was also carried out for comparison.

To calculate the corrosion rate at each temperature, at least three specimens tested for 100 h need to be washed using alkali with 40% NaOH + 60% Na2CO3 at 500 °C, removing corrosion products until the surface of the specimen was metallic. The weight of the corrosion products was calculated by subtracting the weight after alkali washing from the original weight of the specimen in which the average corrosion rate can be obtained.

### Characterization

Phase constitutions of corrosion layers were determined by XRD. After that cross-sectional specimens were embedded in an epoxy, then polished with water-based sandpaper of different particle sizes, and finally the coating samples were polished to a mirror-like effect with a polishing agent. SEM and EPMA were employed to

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**Table 2** Composition of original artificial seawater solution (wt%)

| Composition | Content (g L⁻¹) |
|-------------|----------------|
| NaCl        | 27             |
| MgCl₂       | 12.8           |
| KCl         | 1              |
| CaCl₂       | 1              |
| Deionized water | Balance        |
investigate the cross-sectional morphologies and elemental distributions in corrosion layers.

**Results and Discussion**

**Corrosion Kinetics and Characterization**

Figure 2 illustrates corrosion rate of Alloy 1 at 700 °C, 750 °C and 800 °C. It is clear that mass gain increases gradually as the time extends and the temperature elevates. It is suggested that the mass gain of Ni-based superalloy during isothermal oxidation process follows a relationship of the form [34–36]:

\[
(\Delta W)^n = kt,
\]

where \( \Delta W \) is the mass gain per unit area, \( n \) is the rate exponent, \( t \) is the exposure time at a particular temperature, and \( K \) is the isothermal rate constant. Mohanty and Shores [37] confirmed that corrosion kinetics of high alloy stainless steels can also be described by Eq. (1). Moreover, they found that corrosion kinetic follows a square power law at low temperature while a linear one at high temperatures. According to Fig. 1, it is clear that corrosion kinetics follow linear power law \((n=1)\) at 700 °C, 750 °C and 800 °C. Thus, isothermal rate constants \( K \) can be obtained by use of regression analysis (see Table 3). In addition, the correlation coefficients \( R \) of regression analyses nearly equals to 1, indicating that linear power law is applicable to Alloy 1.

Figure 3 illustrates the oxidation/corrosion rates of Alloy 1 at different temperatures in air and in gas containing salts. It is evident that the oxidation rate of Alloy 1 during oxidation is much lower than that during gas hot corrosion, the former is only about one tenth of the latter, which implies that the corrosion layer is not protective.
Figure 4 illustrates the corrosion rates of Alloy 1 and Alloy 2 at different temperatures. It is clear that the corrosion rates of Alloy 1 are significantly lower than that of Alloy 2 at 800 °C, indicating that Alloy 1 has more superior hot corrosion resistance at this temperature.

Surface Morphologies and Phase Constitutions of Corrosion Layer

Figure 5a illustrates the surface morphology of Alloy 1 after gas hot corrosion of 100 h at 700 °C. It can be seen that fine particles of corrosion products were almost all over the surface of the specimen, indicating that the corrosion products have yet formed films on the surface of Alloy 1. Figure 5b is the XRD spectra of Alloy 1 after hot corrosion of 25 h and 100 h at 700 °C. It shows that corrosion products are Cr$_2$O$_3$ after hot corrosion of 25 h, while NiO, TiO$_2$ and NiCr$_2$O$_4$ appear after 100 h. This confirms that the types of corrosion products will change during hot corrosion of 100 h. Furthermore, the corrosion products demonstrate that the process of hot corrosion is mainly oxidation at this temperature.

Figure 6a depicts the surface morphology of Alloy 1 after hot corrosion of 100 h at 750 °C. It is seen that corrosion products have formed a dense film on the surface. Figure 6b is the XRD spectra of Alloy 1 after hot corrosion of 25 h and 100 h at 750 °C. It reveals that corrosion products are Cr$_2$O$_3$, NiO, TiO$_2$ and NiCr$_2$O$_4$ after hot corrosion of 25 h, which differs from that of at 700 °C. The corrosion products after hot corrosion of 100 h are the same as those after 25 h. At the same time, Fig. 6b also validates that the process of hot corrosion is mainly oxidation.

Figure 7a depicts the surface morphology of Alloy 1 after hot corrosion of 100 h at 800 °C. The corrosion film becomes loose and porous. Figure 7b is the XRD spectra of Alloy 1 after hot corrosion of 25 h and 100 h at 800 °C. It presents that the corrosion products after hot corrosion of 25 h and 100 h are the same as those at 750 °C. This confirms again that the process of hot corrosion is mainly oxidation.

It is known that NiCr$_2$O$_4$ can be formed by the following reaction:

\[
\text{NiO} + \text{Cr}_2\text{O}_3 = \text{NiCr}_2\text{O}_4,
\]

\[\Delta G^\theta_T = -1378.26 + 0.3677T/K \text{ (kJ mol}^{-1} \text{).} \tag{2}\]

This equation suggests that free energies, $\Delta G^\theta$, of NiCr$_2$O$_4$ are negative at 700, 750 and 800 °C. The negative $\Delta G^\theta$ values prove that NiCr$_2$O$_4$ is formed from NiO and Cr$_2$O$_3$ spontaneously, thus providing theoretical bases to understand the appearance of NiCr$_2$O$_4$.
Cross-Sectional Morphologies

Together with measurements of mass gain and phase constitution, severity of hot corrosion attack was studied by cross-sectional morphologies of corrosion layers. Figure 8a displays cross-sectional morphology of Alloy 1 after 100 h of gas hot corrosion at 700 °C. It is clear that the corrosion layer is not complete and dense, and there are voids in it. There is a clear gap between the corrosion layer and the substrate, which means the adhesion of the corrosion layer is poor. The surface of the substrate under the corrosion layer has an area with a different color contrast from the substrate, and its depth is about 2.2 μm. EDS analysis shows that this area is a Cr-poor zone. Figure 9 illustrates electron probe elemental maps of cross section. It shows that the corrosion layer is composed of Cr₂O₃, TiO₂, Al₂O₃, and
Fig. 5  
(a) SEM image after hot corrosion of 100 h and  
(b) XRD analyses after hot corrosion of 25 h and  
100 h of Alloy 1 at 700 °C

Fig. 6  
(a) SEM image after hot corrosion of 100 h and  
(b) XRD analyses after hot corrosion of 25 h and  
100 h of Alloy 1 at 750 °C

Fig. 7  
(a) SEM image after hot corrosion of 100 h and  
(b) XRD analyses after hot corrosion of 25 h and  
100 h of Alloy 1 at 800 °C
NiO, without sulfide. Moreover, there is no obvious sulfurization inside the substrate. Figure 9 also validates that Na in the corrosion layer appear, which means the salts have yet penetrated into the corrosion layer.

Figure 8b and c displays cross-sectional morphology of Alloy 1 after 100 h of gas hot corrosion at 750 °C and 800 °C, respectively. It is clear that the integrity of the corrosion layer deteriorates, which is divided into many pieces by cracks and tends to spall along cracks. There are a large amount of cavities between the corrosion layer and the substrate, which means the adhesion of the corrosion layer
is poor. The depth of Cr-poor zone is about 3.4 μm and 5.4 μm at 750 °C and 800 °C, respectively. Figures 10 and 11 illustrate electron probe elemental maps of cross section. It shows that the corrosion layer is composed of Cr₂O₃, TiO₂, Al₂O₃, and NiO at 750 °C and 800 °C. As the same as 700 °C, Element Na in the corrosion layer appear and there is no obvious sulfurization inside the substrate.

Figure 12 displays the cross-sectional elemental maps after hot corrosion of 25 h of Alloy 1 and Alloy 2 at 800 °C. In the initial stage of corrosion, a continuous corrosion layer consists of Cr₂O₃ and Al₂O₃ appears on the surface of Alloy 1, without obvious internal oxidation. The thickness of oxide layer is about 4 μm. In contrast, the Cr₂O₃ layer of Alloy 2 is thinner, about 2.5 μm. Furthermore, no continuous Al₂O₃ layer is formed on the surface, but the internal oxidation of Al is serious, and Al₂O₃ penetrates into the substrate like fingers.

**Corrosion Mechanism of Alloy 1**

It is evident from the comparison of oxidation in air and gas hot corrosion that salts accelerate the failure of Alloy 1. Normally, oxidation of alloys is selective, which depends on the content of a certain element and its oxidation free energy [38]. The products during oxidation in air of Alloy 1 are mainly oxides of Cr₂O₃, TiO₂ and...
NiCr$_2$O$_4$, et al. Under the gas hot corrosion condition in this test, the corrosion layer of Alloy 1 is mainly composed of Cr$_2$O$_3$, TiO$_2$, Al$_2$O$_3$, NiO and NiCr$_2$O$_4$, without sulfide. Moreover, it is evident that the corrosion rate of Alloy 1 during oxidation is much lower than that during gas hot corrosion (Fig. 3). Therefore, the hot corrosion behavior under gas containing chloride salts is actually accelerated oxidation, which is obviously different from the hot corrosion mechanism of the cooperating mechanism of oxidation and sulfurization of powder superalloy in molten NaCl–Na$_2$SO$_4$ salts [39]. This is because the salt in this study mainly chlorine and only a small amount of sulfur in aviation fuel.

The presence of chloride salts such as NaCl will cause great damage to the protective oxide film. NaCl can reacts with the oxide film as follows [40]:

$$4\text{NaCl} + 2\text{Cr}_2\text{O}_3 + 3\text{O}_2 = 2\text{Na}_2\text{CrO}_4 + 2\text{CrO}_2\text{Cl}_2,$$

$$4\text{NaCl} + \text{Cr}_2\text{O}_3 + 3/2\text{O}_2 = 2\text{Na}_2\text{SO}_4 + 2\text{CrO}_2\text{Cl}_2,$$

$$2\text{NaCl} + \text{Al}_2\text{O}_3 + 1/2\text{O}_2 = 2\text{NaAlO}_2 + \text{Cl}_2,$$

CrO$_2$Cl$_2$ may evaporate in a gaseous form or become chromate:
It can be seen from the cross-sectional element distribution maps that the corrosion layer contain Na but without Cl (Figs. 9, 10, 11), this infers that $\text{CrO}_2\text{Cl}_2$ was likely to evaporate in a gaseous form. Due to these reactions, the oxide films became loose and porous, further promoting salts penetration.

At the beginning of the hot corrosion, Alloy 1 is corroded slightly by the selective oxidation and oxides of Cr, Al, and Ti appear on the surface. With the extension of time, chloride salts penetrated the oxide film and reacted, destroying the integrity of the oxide film and making it less protective. Moreover, the oxide film formed during gas hot corrosion easily spalled from the substrate. Normally, a higher oxidation rate always develops more interfacial cavities [41]. From this reaction:

$$\text{CrO}_2\text{Cl}_4 + 2\text{NaCl} + 2\text{H}_2\text{O}(g) = \text{Na}_2\text{CrO}_4 + \text{HCl}.$$  \hspace{1cm} (6)

![Fig. 12 Cross-sectional elemental maps after hot corrosion of 25 h at 800 °C: a Alloy 1, and b Alloy 2](image_url)
work, it was found that a higher oxidation rate during gas hot corrosion than that during oxidation in air (Fig. 3). Therefore, compared with oxidation in air, more interfacial cavities occurred in the oxidation layers formed by gas hot corrosion (Fig. 8), causing poorer adhesion and a higher driving force for spallation. Once the oxide layer spalls, the substrate corrosion will accelerate.

Compared to Alloy 2, Alloy 1 has better gas hot corrosion resistance at 800 °C (Fig. 4). In the initial stage of corrosion, a continuous corrosion layer composed of Cr$_2$O$_3$ and Al$_2$O$_3$ appeared on the surface of Alloy 1, without obvious internal oxidation (Fig. 12a). In contrast, for Alloy 2, beneath the Cr$_2$O$_3$ layer, significant internal oxidation of Al occurred (Fig. 12b). This is mainly due to the difference in the content of Co and Al. Firstly, Alloy 1’s Co content is significantly higher than that of Alloy 2 (Table 1). Previous studies indicated that the diffusion coefficient of Cr in Co exceeds the same value for Ni at lower temperatures (≤850 °C); therefore, the increase of Co content promotes the outward diffusion of Cr to the oxidation front and enhances the oxidation resistance at low temperature (≤850 °C) [14]. Furthermore, increased Co increases the Cr content in γ matrix as a result of the increased γ’ fraction [41]. Moreover, the initial fast outward diffusion of Co due to oxidation also increased the Cr content below scale. All of them are believed to favor the growth of a continuous Cr$_2$O$_3$ scale on Alloy 1 in a shorter transient stage (Fig. 12a). With the growth of Cr$_2$O$_3$ scale, the oxygen partial pressure at the Cr$_2$O$_3$ scale/underlying substrate interface further decreased. The selective oxidation of Al started, therefore, a continuous Al$_2$O$_3$ layer formed on Alloy 1 (Fig. 12a). These imply that the increase of Co also slightly promoted the growth of Al$_2$O$_3$ through the third elemental effect. The mechanism of the third element effect is not yet clear, but some ideas include: inhibition of internal oxidation of Al, complete solubility between Al$_2$O$_3$ and Cr, inhibition of the growth of external oxides [42, 43]. These ideas were basically confirmed by comparing the hot corrosion of above two alloys. Secondly, there is the significant difference of Al content between Alloy 1 (3.6 wt%) and Alloy 2 (2.2 wt%), extra Al in Alloy 1 might be enough for the continuous alumina layer to form and protect the alloy [44].

According to preceding analyses, the hot corrosion of Alloy 1 under gas containing chloride salts was confirmed to be an accelerated oxidation process due to the damage in integrity of the oxide film caused by continuous corrosion with chloride salts. Compared to Alloy 2, Alloy 1 has better hot corrosion resistance at 800 °C, which is attributed to the increase of Co and Al content. The increased Co and Al content can promote the rapid formation of continuous Cr$_2$O$_3$ and Al$_2$O$_3$ protective films on the alloy surface with better hot resistance in which Co inhibits internal oxidation of Al through the third element effect.
Conclusions

In this study, hot corrosion tests under gas containing chloride salts at different temperatures were performed. Phase constitutions, surface and cross-sectional morphologies, elemental distributions of corrosion layers had been studied. The conclusions can be summarized as follows:

- The average mass gain of Alloy 1 increased as the temperature elevates. The corrosion kinetics followed linear power law at 700 °C, 750 °C and 800 °C. The corrosion rates during oxidation in air were much lower than that of gas hot corrosion.
- The corrosion layers obtained after 100 h of hot corrosion were mainly composed of Cr$_2$O$_3$, TiO$_2$, Al$_2$O$_3$, NiO and NiCr$_2$O$_4$. The cross-sectional morphologies and elemental maps indicated that the chloride salts penetrated into the corrosion layers and caused lots of cavities and cracks.
- The hot corrosion of Alloy 1 under gas containing chloride salts was confirmed to be an accelerated oxidation process due to the damage in integrity of the oxide film caused by continuous corrosion with chloride salts.
- Compared to Alloy 2, Alloy 1 has better hot corrosion resistance at 800 °C, which is attributed to the increase of Co and Al content. The increased Co and Al content can promote the rapid formation of continuous Cr$_2$O$_3$ and Al$_2$O$_3$ protective films on the alloy surface with better hot resistance in which Co inhibits internal oxidation of Al through the third element effect.

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Data Availability The datasets generated during and/or analyzed during the current study are available from the corresponding author on reasonable request.

Declarations

Conflict of interest The authors have no relevant financial or non-financial interests to disclose.

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