

A Study of the Hot Salt Corrosion Behavior of Three Nickel-Based Single-Crystal Superalloys at 900 °C

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Abstract: A study of the hot salt corrosion behavior of three nickel-based single-crystal superalloys at 900 °C was conducted. We discovered that the corrosion layer on each alloy was distinctly enriched with Mo, Ni, S, and O, primarily comprising sulfides and oxides. Notably, variations in oxygen distribution across the alloys revealed that the elemental composition plays a pivotal role in their corrosion resistance. These insights not only advance our understanding of the mechanisms driving thermal corrosion in nickel-based single-crystal superalloys but also lay the groundwork for designing alloys with enhanced durability tailored to high-temperature applications. This research marks a significant step toward the optimal design and utilization of superalloys in sectors demanding exceptional material stability under thermal stress.

Keywords: high-temperature corrosion; Ni-based superalloy; molten salt corrosion; corrosion in marine environments



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1. Introduction

Nickel-based single-crystal superalloys exhibit excellent mechanical properties and creep resistance at elevated temperatures, making them vital for aerospace and power generation applications [1]. However, the demanding service environment characterized by extremely high temperatures and humidity can lead to thermal corrosion on the alloy blade surface, posing a significant challenge. Consequently, understanding the thermal corrosion behavior of superalloy materials and enhancing their corrosion resistance is an urgent issue [2]. Zhang et al. [3] investigated that the corrosion products of Inconel 718 consisted mainly of an outer layer composed of Ni_2CrO_4 , Cr_2O_3 , and Fe_3O_4 and an inner layer composed of Ni_3S_2 . Yang's team [4] investigated the hot corrosion behavior of nickel-based single crystals at 900 °C in three different salt mixtures. It was revealed that, during the thermal corrosion process, in the γ' depleted layer of nickel-based single-crystal superalloys, the corrosion products were mainly linear TaS_2 and pitted CrS . Additionally, the high-temperature performance of single-crystal superalloys has been enhanced by including high-melting-point alloying elements such as Re, W, Ta, and Mo [5–8]. Song's team investigated the thermal corrosion behavior of nickel-based single-crystal superalloys in molten Na_2SO_4 salt at 900 °C and demonstrated the effect of the Al/Ti ratio and Re on the corrosion of nickel-based single-crystal superalloys through first-principle calculations [9]. The thermal corrosion of nickel-based single-crystal superalloys at 900 °C was investigated by the introduction of a salt-coating method by Gong et al. Elemental analyses and microstructural results revealed the corrosion mechanism of nickel-based single-crystal superalloys, and undulating surfaces, multilayered external oxide layers, and internal CrS

layers were observed during the hot corrosion process [10]. Nevertheless, the mechanism of the behavior of superalloys composed of complex salts and the interaction between the corrosive salts are still unclear.

To solve these issues, this paper aims to investigate the corrosion behavior and mechanisms of three nickel-based single-crystal superalloys with distinct compositions in a hot salt environment at 900 °C. The effect of the presence of Co, Mo, Al, Cr, W, and Ti elements on the corrosion resistance of nickel-based superalloys was revealed.

2. Materials and Methods

The specific alloys chosen for this study, which are commonly used in practical applications, represent different generations of nickel-based single-crystal superalloys, each with its unique composition and properties. The compositions of the three nickel-based single-crystal superalloys utilized in this study are summarized in Table 1. The three single-crystal superalloys were obtained through directional solidification to ensure the crystals grow uniformly and continuously, minimizing the formation of grain boundaries and defects and eliminating the complexities introduced by grain boundaries and other microstructural features. Three alloy bars underwent standard heat treatment to prepare the specimens for analysis, followed by machining into a $\Phi 10 \times 170$ mm substrate. Subsequently, they were cut into $\Phi 9 \times 3$ mm cylinders using electrical discharge machining (EDM). The specimens were then polished using SiC sandpaper, with grit sizes ranging from 80# to 3000#. To quantify the corrosion rate, weight loss measurements were conducted with utmost precision. In this study, a balance with an accuracy of 0.001 was utilized for weight measurements. Each sample was measured 3 times to ensure the accuracy of the results. Additionally, it was ensured that the balance was operated under identical temperature and humidity conditions before and after the measurements to eliminate any potential influence of these factors on the measurement outcomes. In addition, before measurement, the pre-corrosion samples were ultrasonically cleaned to remove any surface contaminants, while the post-corrosion samples were cleaned with acetone immersion, allowed to dry at room temperature, and handled with minimal disturbance to preserve their corrosion state. Using the crucible method, we conducted thermal corrosion tests on specimens immersed in a 3:1 mass ratio mixture of Na_2SO_4 and NaCl salts. The specimens were placed in a corundum container and heated to 900 °C in a resistance furnace. Mass loss was precisely measured using an electronic balance before and after corrosion. Microstructural characterization was performed using a field-emission SEM (Tescan Mira 4) and EDS (Oxford Xplore30 Aztec one) under controlled environmental conditions.

Table 1. The elemental composition of 3 Ni-based single-crystal superalloys (wt.%).

	Cr	Re	Co	Mo	Ta	Ti	W	Al	Hf	Ni
Alloy 1	6.25	2.82	9.51	0.58	6.51	0.94	6.37	5.72	0.09	Bal.
Alloy 2	7	3.08	7.83	1.52	6.50	-	5.01	6.01	0.11	Bal.
Alloy 3	3.5	5	6	0.4	8	0.15	6.5	5.8	0.03	Bal.

3. Results and Discussion

Figure 1 exhibits the kinetic profiles and macroscopic morphology of the three nickel-based single-crystal superalloys during corrosion. The profiles reveal the alloys' mass gain rates over time, while morphology provides insights into surface features and structural evolution. Table 2 summarizes the fitted parameters and outcomes. After 40 h, weight gain curves flatten as protective corrosion layers initially form but become porous and peel off, leading to salt infiltration and accelerated corrosion. Mass increases significantly after 40 h, indicating rapid corrosion rates, with darker, scaly, and peeling/skinning corrosion products observed. When comparing the kinetic curves of the three alloys, it is clear that the magnitude and trend of the rate of change in weight for Alloy 3 is relatively small.

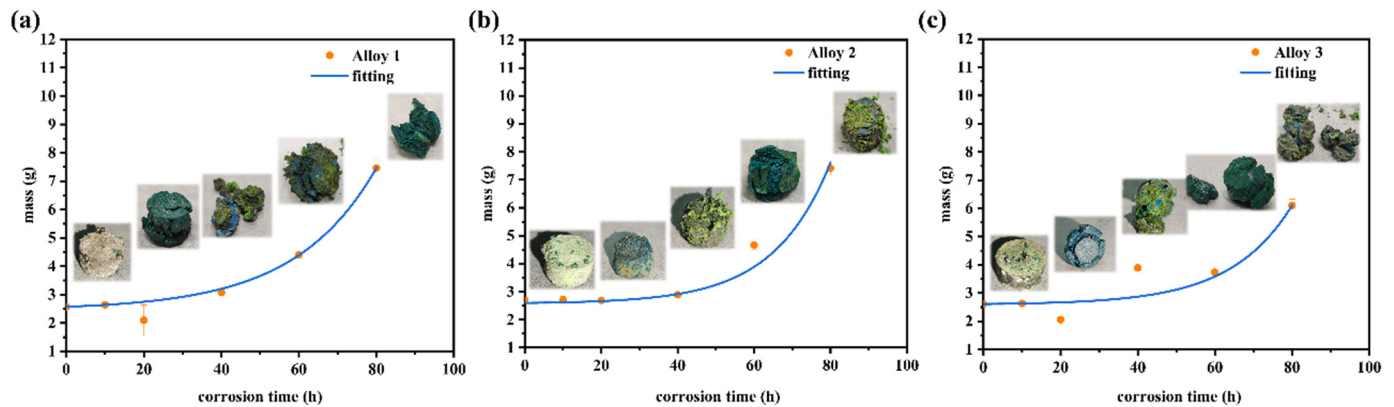


Figure 1. Kinetic profiles and macroscopic morphology of three nickel-based single-crystal superalloys (a) Alloy 1; (b) Alloy 2; (c) Alloy 3.

Table 2. Parameters of kinetic curve fitting for hot salt corrosion of three nickel-based single-crystal superalloys.

	Alloy 1	Alloy 2	Alloy 3
Modelling	ExpDec1	ExpDec1	ExpDec1
Equation	$y = A_1 \cdot \exp(-x/t_1) + y_0$	$y = A_1 \cdot \exp(-x/t_1) + y_0$	$y = A_1 \cdot \exp(-x/t_1) + y_0$
y_0	2.46 ± 0.03	2.57 ± 0.13	2.59 ± 1.44
A_1	0.11 ± 0.022	0.02 ± 0.02	0.02 ± 0.25
t_1	-20.97 ± 1.26	-14.88 ± 2.56	-15.87 ± 36.43
Reduced Chi-Sqr	0.58	8.64	919.05
R^2 (COD)	0.99	0.95	-0.69
Adjust R^2	0.99	0.91	-1.81

Samples with a corrosion duration of 20 h were selected for morphological characterization. As shown in Figure 2a, the upper layer of the surface of Alloy 1 after corrosion formed a white irregular 1–3 μm length of massive corrosion products, labeled with number 1. At the same time, a large number of round granular products, continuous and uniform in size, with smooth contours and small sizes and diameters below 1 μm , were also present on the surface, labeled with number 2. Needle-like products, 4–6 μm in length, were also observed, which were long and interconnected, labeled with number 3. By observing Figure 2b, it is evident that the surface morphology of Alloy 2 following 20 h of hot salt corrosion is dominated by the presence of needle-like substances. At the location marked with 1, relatively thick, trunk-like, and needle-like substances are distinctly visible, while at the location marked with 2, fine, dense, continuous, and compact needle-like substances are observed. The results presented in Figure 2c indicate three distinct types of surface corrosion product morphology for Alloy 3. Region 1 is characterized by a significant number of irregular lumps, measuring 4–7 μm in width. Region 2 exhibits elongated needle-like materials, while Region 3 is populated by granular material with a diameter ranging from 1 to 2 μm .

Figure 3 shows the surface XRD results of three alloys (Alloy 1, Alloy 2, and Alloy 3) after hot salt corrosion at 900 °C. It can be observed that when the three samples underwent hot corrosion for 20 h, the main product peak on the surface of the alloys was that of NiO, and a small amount of Al₂O₃ could also be detected. Due to the exfoliation on the surface of the alloys, a large number of NiMoO₄ phases could be detected in the alloys, and the peaks of the compounds of NiO, Al₂O₃, and Ta with smaller intensities were detected in Alloy 3. CrReO₂ compounds were also detected in Alloy 3, which proved that the elements Re and Cr reacted with O atoms together.

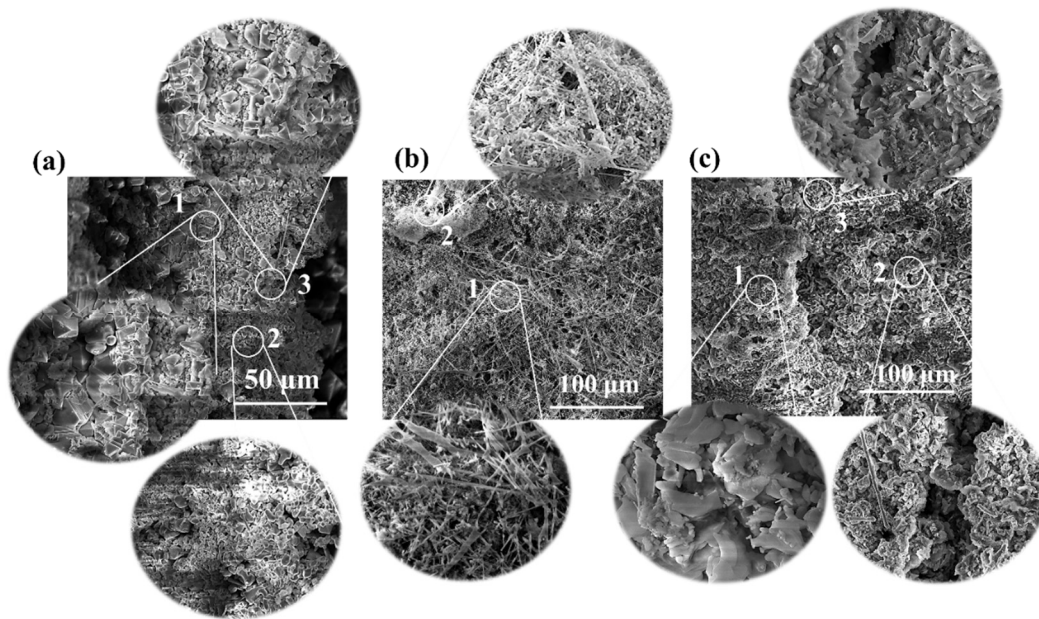


Figure 2. SEM images of the bottom surfaces of corrosion samples of three alloys: (a) Alloy 1; (b) Alloy 2; (c) Alloy 3.

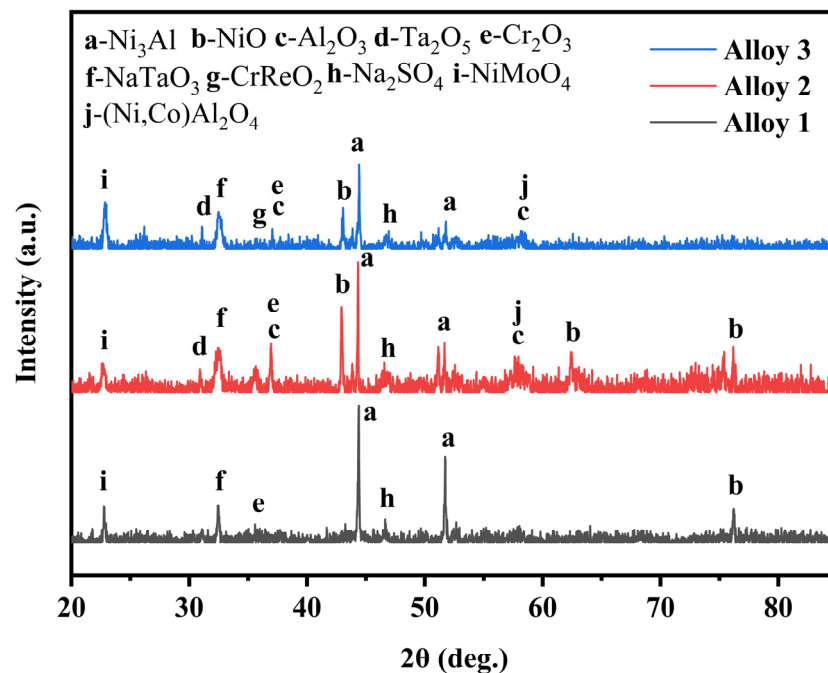


Figure 3. Surface XRD results of three nickel-based single-crystal superalloys after thermal corrosion at 900 °C for 20 h.

Figure 4 shows the SEM images depicting corrosion cross-sections of the three nickel-based single-crystal superalloys. Each alloy's corrosion layer is roughly segmented into two distinct layers. Alloy 1 exhibits an outermost loose, dark grey layer of uneven thickness, followed by a 10–15 µm granular inner layer. Alloy 2 has a similar but thinner inner layer, lacking the outermost one. The outermost layer of Alloy 3 is the thickest (30–40 µm), lumpy, and uneven, with corrosion channels and voids indicating potential internal diffusion and corrosion by S, O, and Cl elements from the molten salt. In addition, comparing the corrosion sections of the three alloys, we can clearly observe that the thickness of the inner layer of Alloy 3 is much lower than that of the other two alloys, which, combined with

the corrosion mechanism proposed in this paper, proves that the corrosion performance of Alloy 3 is much better than that of the other two alloys.

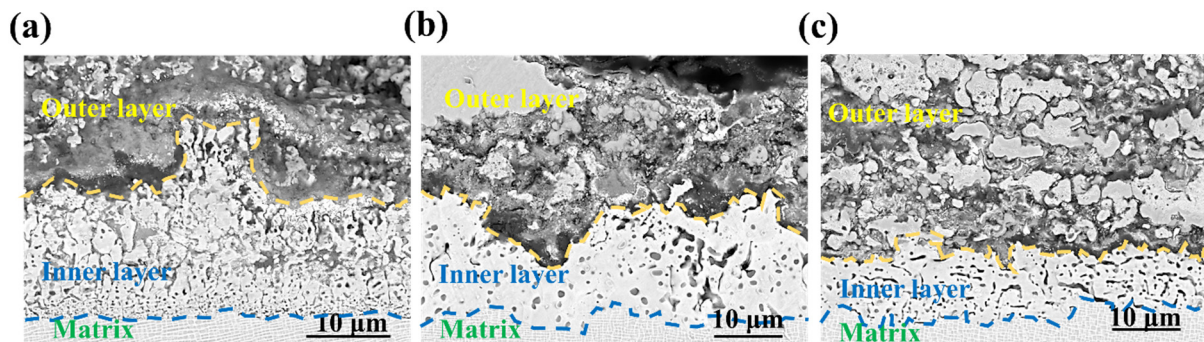


Figure 4. Cross-sectional morphology of three nickel-based single-crystal superalloys after hot salt corrosion: (a) Alloy 1; (b) Alloy 2; (c) Alloy 3.

Figures 5–7 present the EDS analysis results of the three nickel-based single-crystal superalloys in the cross-section after corrosion. As previously mentioned in Table 1, these alloys contain the main constituent elements. Combined with observations from Figure 4, it is evident that the outermost corrosion layer in Figure 5 is enriched with Mo, Ni, Ti, S, and O elements, indicating that the main constituents in this region are sulfides and oxides of Mo and Ni. Additionally, a large number of O and Al elements are concentrated at the transition position between the outer layer and inner layer, suggesting that a significant number of aluminum oxide products are primarily present here. In the inner layer region of Alloy 1's corrosion product layer, particles are enriched with O and Cr elements and also contain a certain number of Ta, Ti, W, and O elements. It is presumed that these particles are primarily composed of the oxides of Ta, Ti, and W. The layer that is closer to the substrate contains a large number of O and Al elements. The inner part closest to the substrate is more complex, with the enrichment of oxides and sulfides of various alloy elements. The distribution is highly uneven, with W, Ni, and Ti being more aggregated. There is also a portion enriched with Mo, Hf, and a small amount of S.

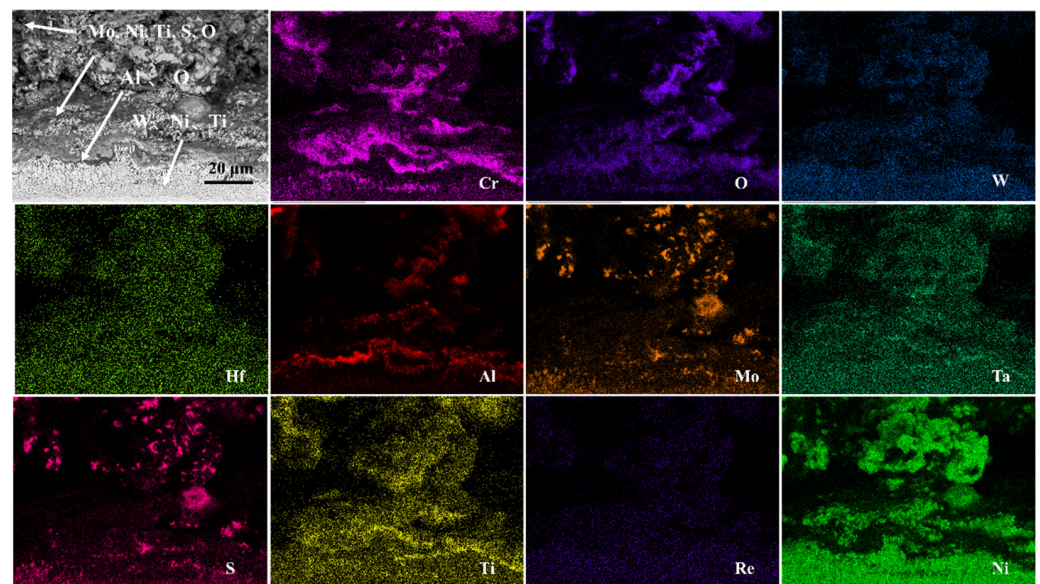


Figure 5. Cross-sectional morphology and corresponding elemental distribution of Alloy 1 after corrosion at 900 °C for 20 h.

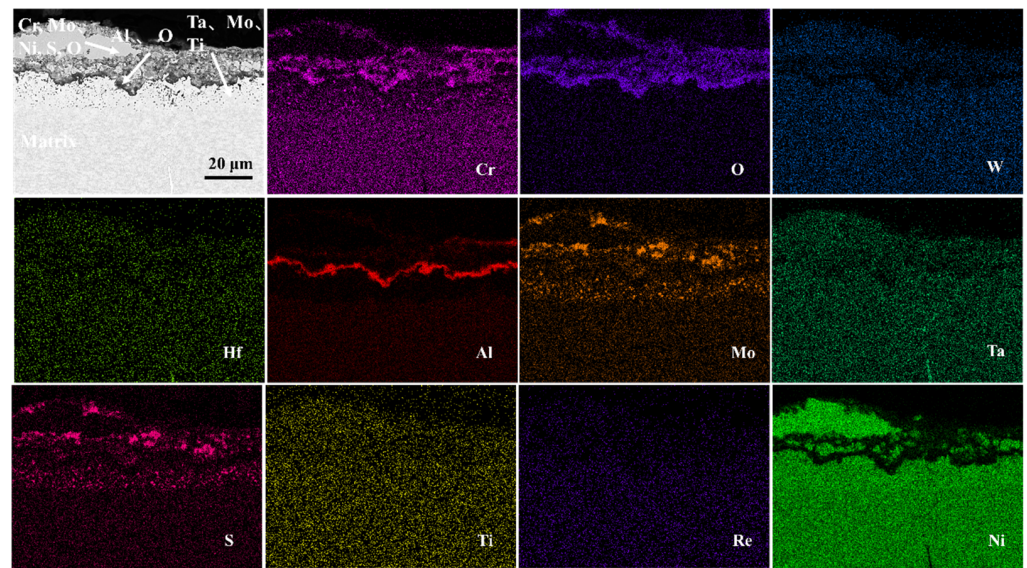


Figure 6. Cross-sectional morphology and corresponding elemental distribution of Alloy 2 after corrosion at 900 °C for 20 h.

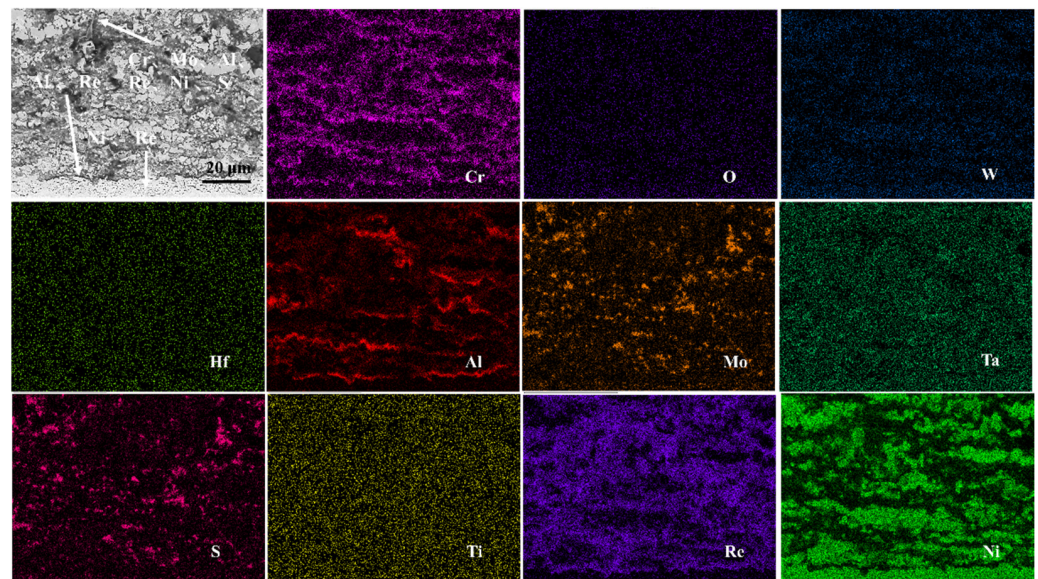


Figure 7. Cross-sectional morphology and corresponding elemental distribution of Alloy 3 after corrosion at 900 °C for 20 h.

By observing Figure 6, it is evident that the outermost corrosion layer is enriched with Cr, Mo, Ni, S, and O elements. This suggests that the main components of this region are sulfides and oxides of Cr, Mo, and Ni, with small amounts of Hf and Ta also present. Notably, a significant amount of Al and O, along with a certain amount of Cr and W, accumulate at the transition position between the outer and inner layers. This suggests a high concentration of alumina products and a smaller amount of Cr_2O_3 in this region. The distribution of elements in the inner layer becomes evident after passing through the Al_2O_3 -enriched area. Firstly, the presence of O cannot be observed, indicating that it is blocked by the external corrosion products. However, S can still be detected, indicating that it penetrated through the porous and sparse corrosion products of the outer layer to reach the alloy's substrate. The particles in the inner corrosion product layer are enriched with Ta, Ti, and Mo and contain smaller amounts of Hf, W, and O. This suggests that there is a significant number of alumina products and a smaller amount of Cr_2O_3 in this region.

The elemental distribution within the corrosion pattern of Alloy 3 appears more muted than the patterns observed in Alloys 1 and 2. Examining Figure 7, it becomes evident that the outer layer of Alloy 3 contains substantial amounts of Cr, Mo, Al, Re, Ni, and a notable quantity of S. Notably, the presence of O and W elements is minimal, rendering them almost imperceptible. This suggests that the outermost corrosion products of Alloy 3 are primarily composed of sulfides derived from these five elements. At the interface between the outer and inner layers, large accumulations of Al and Re are observed, accompanied by a defined quantity of S. Closer to the substrate, within the inner layer, elements such as Ti and Ta, along with a substantial amount of Ni, are found in a particulate form. This suggests that the corrosion products in this region possess reduced quantities of S and O.

According to Wagner's theory, the corrosion mechanism of the three alloys at 900 °C essentially involves the oxidation process of metals, characterized by the migration of metal cations and cation vacancies [11]. Thermal corrosion, however, accelerates the failure of oxides through the presence of S, Cl, and O atoms in molten salts. Hence, we illustrate the corrosion mechanism using Alloy 1 as an example in Figure 8.

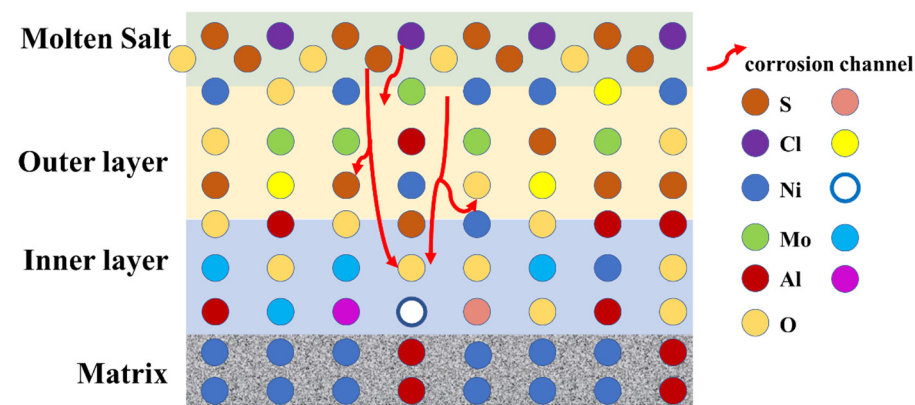


Figure 8. Diagram of the corrosion mechanism of nickel-based single-crystal superalloys, using Alloy 1 as an example.

Given that the melting points of Na_2SO_4 and NaCl molten salt media are below 900 °C, they decompose into SO_4^{2-} and Cl^- ions. During the initial stages of thermal corrosion, the surface of the specimen covered with Na_2SO_4 and NaCl exhibits only a limited amount of O^{2-} . These O^{2-} molecules preferentially react with Al, Mo, and Cr to form oxides, followed by reactions with Ni and Ti [12]. While the reaction of Ni with the molten salt may facilitate the formation of an Al_2O_3 layer, the loose and easily exfoliating nature of NiO reaction products can compromise their protective role, potentially accelerating the degree of melted salt corrosion [13].

As the substrate undergoes further corrosion, a significant number of void channels emerge. S and O atoms continue to penetrate along these channels, corroding deeper into the material. During this process, Ta preferentially forms Ta_2O_5 oxides during oxidation, which subsequently react with the corrosive media to ultimately exist as stable solid NaTaO_3 spinel [12,14]. The ongoing oxidation reactions enhance the activity of O^{2-} ions. Once the O atoms concentration reaches a level sufficient for oxide dissolution, Cr_2O_3 oxides preferentially dissolve, followed by Al_2O_3 oxides. This explains the decreasing abundance of these oxides as the corrosion layer moves away from the substrate [15]. Concurrently, sulfides become abundantly present in the outer corrosion layer.

Combined with previous findings [16], Figure 9 shows Spearman's correlation coefficients between the changes in the weights of the major elements and the weights of the metals of a variety of single-crystal superalloys subjected to a 20 h thermal corrosion test at 900 °C. An r-value of less than 0 indicates a negative correlation, while an r-value of more than 0 indicates a positive correlation. Additionally, p-values of less than 0.05/0.01 are considered to be statistically significant/highly significant and indicate that the probability of

the differences in the sample being caused by a sampling error is low. Based on the graphs, we can observe that the elements Re, Cr, and Co are negatively correlated with the amount of metal weight change in most of the single-crystal superalloys, which indicates that an increase in the concentration of these three elements leads to a decrease in the amount of metal change. This finding emphasizes the key role of these elements in enhancing the corrosion resistance of the alloys, with the p -value for Cr being less than 0.001 for one group of alloys, suggesting that the correlation was calculated with particularly high accuracy. In contrast, increases in the elements Mo and W have a detrimental effect, which is consistent with the results of the experimental analyses presented in this paper.

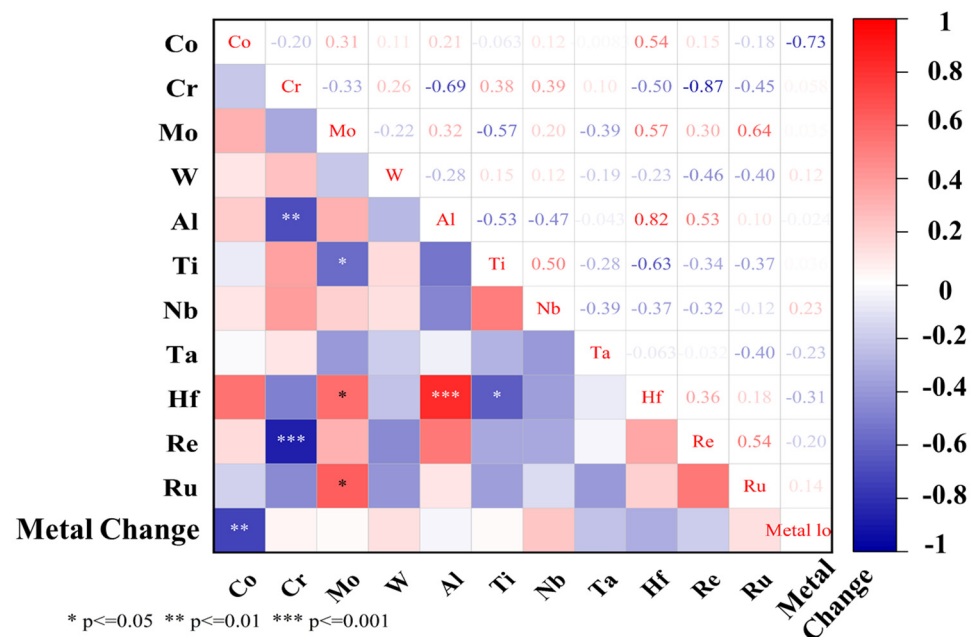


Figure 9. Spearman's correlation coefficients between changes in the weights of the major elements and the weights of the metals of a variety of single-crystal superalloys subjected at 900 °C for 20 h.

4. Conclusions

In this paper, the hot salt corrosion of three typical nickel-based single-crystal superalloys was investigated at 900 °C in a mixture of NaSO₄ and NaCl molten salts. The following conclusions were drawn:

1. The outer corrosion layers of all three alloys were enriched in Mo, Ni, S, and O. However, Alloy 1 showed O enrichment in all corrosion processes, whereas Alloy 2 showed O enrichment only in the outer layer. On the other hand, Alloy 3 showed very little O enrichment in the corrosion products.
2. The corrosion mechanism of nickel-based single-crystal superalloys at high temperatures was briefly analyzed. In the early stage of corrosion, S and O first reacted with Al, Mo, and Cr to form oxides, and then with Ni and Ti. With the oxidation reaction and the presence of corrosive substances, the substrate from the void channel underwent further corrosion, during which some oxides were dissolved, and thus the protective oxide film was reduced, and the substrate was further corroded.
3. It was also found that the presence of the elements Re, Cr, and Co enhanced the corrosion resistance of the alloy. By contrast, the presence of elements Mo and W exerted the opposite effect.

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and funding acquisition, L.H.; supervision, Y.L. (Yong Liu). All authors have read and agreed to the published version of the manuscript.

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

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