

Article

High-Temperature Oxidation Behaviors of 321 Steel with Y or Nb Micro-Alloying

Ximing Yang^{1,2}, Zhijie Zeng¹, Xu Wang¹, Xing Li¹, Chengjun Guo^{1,2,*}, Xiangpeng Xiao^{1,2,*}  and Bin Yang¹

¹ Faculty of Materials Metallurgy and Chemistry, Jiangxi University of Science and Technology, Ganzhou 341000, China; 1450668389@126.com (X.Y.)

² Jiangxi Advanced Copper Industry Research Institute, Yintan 335000, China

* Correspondence: guochengjun1128@126.com (C.G.); xiao_xiangpeng@126.com (X.X.)

Abstract: The effects of Y or Nb addition on the oxidation behavior of 321 steel at high temperatures were investigated by scanning electron microscopy (SEM), energy spectroscopy (EDS) and X-ray diffractometer (XRD). At the same time, the oxidation mechanism and oxidation kinetics of rare earth Y or Nb addition are explored. The results show that temperature greatly influences the high-temperature oxidation resistance of the alloys, and the oxidation phenomenon of the alloy becomes more obvious as the temperature increases. Adding 0.5 wt.% Nb or 0.045 wt.% Y elements can effectively improve the oxidation resistance of 321 stainless steel at high temperatures. The addition of rare earth Y can promote the diffusion of Cr in the matrix, leading to increased Cr content in the oxide film and the eventual formation of a dense Cr₂O₃ film, which effectively hinders the continuation of the oxidation reaction. As a result of the Nb addition, the outward diffusion of Cr elements can be effectively inhibited, Cr and O ion bond can be strengthened, the oxidation rate can be reduced, the adhesion rate of oxide film can be increased, and the oxidation resistance of 321 stainless steel can be improved.

Keywords: 321 stainless steel; high-temperature oxidation; oxidation kinetics; micro-alloying



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1. Introduction

A typical austenitic stainless steel is 321 stainless steel. The alloy element titanium is added to the material, improving the stability of 321 stainless steel and preventing carbide precipitation at 400–900 °C. In addition, it can improve the high-temperature characteristics of the material, and 321 stainless steel performs much better than 316 L in high-temperature environments. Moreover, 321 stainless steel has good oxidation resistance and creep strength. In large boiler superheaters, reheaters, steam pipeline, petrochemical heat exchangers, and polysulphuric acid production equipment are still widely used [1–3]. As the heat-resistant steel in the boiler tube is under high temperature and high pressure for a long time, at the same time, the pipe wall temperature is dozens of degrees higher than the steam temperature. The organization and performance of the steel will seriously deteriorate in such an environment for a long time and then damage the normal operation of the equipment. There is a big security risk. Therefore, it is very important to improve further the oxidation resistance of 321 alloys at high temperatures [4,5].

Relevant research shows that [6,7], the high-temperature oxidation resistance of heat-resistant steel is closely related to the formation of dense oxide film on the alloy surface. Using plasma technology to metalize or carburize the alloy surface can form a compact and stable coating with excellent high-temperature oxidation resistance. In contrast, directly adding alloying elements to austenitic steels is a simple process. It has also been shown to promote the formation of a dense, stable, high-temperature resistant oxide layer on the steel surface [8,9]. In the early stages of oxidation, the metal is easily oxidized. Relevant research shows that adding rare earth elements can improve the oxidation resistance of

the alloy [10–13]. Since the active element effect was proposed in 1937, numerous pieces of research have been conducted on the addition of rare earths and other active elements in heat-resistant alloys and steels [14–16]. Based on Liu X's [17] findings, rare earths are easily separated at the oxide grain boundary, and because rare earth ions have a large radius, metal ions cannot diffuse outward, so that the outward diffusion of chromium ions becomes the control step of the process. The process has a low oxidation rate and the oxide layer from growing deeper. Yan [18] discussed the effect of Y on the cyclic oxidation behavior of HP40 heat-resistant steel at 1100 °C. Studies also analyzed the continuous oxidation performance of heat-resistant steel at a high temperature [19]. These theories show that metals are easily oxidized in the early stages of oxidation. As rare earth elements are added, a continuous outer oxide layer can be formed, thereby reducing the oxidation rate, increasing the adhesion of oxides during the oxidation process, and hindering the expansion of the metal substrate during oxidation [11,12,20].

On the other hand, Nb, as a micro-alloying element, is usually added to the alloy to improve high-temperature strength and creep resistance [21]. In addition, Nb, as a strong carbide-forming element can replace Cr and C to form stable NbC, and refine the grain and improve the overall performance of steel. When Nb and oxygen are heated to high temperatures, stable oxides are formed [22,23]. Hyung [24], in a small amount of Nb elements in ferritic stainless steel, found that Nb in the oxide film and metal matrix interface is easy to bias the formation of Nb₂O₅ and laves phase, which can promote the combination of Cr and O ions to improve oxidation resistance.

The effect of micro-alloying on the oxidation behavior of steel has been reported, but the effect of Y or Nb on the high-temperature oxidation of 321 heat-resistant steel and the formation of oxides on the surface remain to be determined. Therefore, in this study, Y and Nb were added to the SUS321 alloy to influence oxidation behavior. The oxidation kinetics and surface oxide formation characteristics of the alloy at 600 °C, 650 °C, and 700 °C, and the formation of oxide film were systematically analyzed using the weight increment method.

2. Materials and Methods

A heat-resistant steel sus321 is used in this experiment. Four samples with different compositions were prepared by doping 0.015 wt.% Y, 0.045 wt.% Y, and 0.5 wt.% Nb, respectively, and they are numbered according to their ingredients as shown in Table 1. The sample is cut into 30 mm × 10 mm × 8 mm rectangular blocks, sanded with 2000 grit sandpaper, polished, degreased in an acetone bath, and then ultrasonically cleaned in an ethanol bath. In the experiment, samples were taken and weighed every 20 h. According to the national standard "Method for the determination of oxidation resistance of steel", the sample is subjected to a constant temperature test in a box-type resistance furnace in the air medium, and the temperature control accuracy of the resistance furnace is ±5 °C. The samples were subjected to constant temperature oxidation experiments at 600 °C, 650 °C, and 700 °C, with an accumulated oxidation time of 200 h. During the alloy isothermal oxidation process, weigh the samples were oxidized for 10 h, 20 h, 40 h, 60 h, 80 h, 100 h, 120 h, 140 h, 160 h, 180 h, and 200 h, and photos were taken to observe the alloys. The sample weight increase is measured by an electronic balance (Model Number: FA200 Manufacturer: Shanghai Hengping Scientific Instrument Co., Ltd., Shanghai, China) with 0.1 mg accuracy. Using an X-ray diffractometer (Model Number: Empyrean Manufacturer: PANalytical, Malvern, UK), the phase composition of the oxide film of the alloy was analyzed. An electron microscope (Model Number: MLA650F Manufacturer: FEI, Hillsboro, OR, USA) equipped with an energy spectrometer was used to observe the oxide film's morphology and composition.

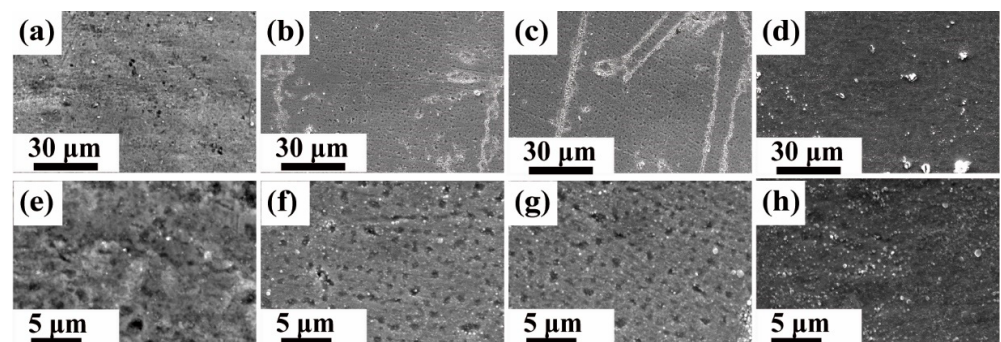
Table 1. Chemical composition of samples.

Number	C	Si	Mn	P	S	Cr	Ni	Ti	Y	Nb	Fe
Y ₀	0.06	0.55	1.0	0.02	0.001	18	10.5	0.45	0	0	Bal
Y _{0.015}	0.06	0.55	1.0	0.02	0.001	18	10.5	0.45	0.015	0	Bal
Y _{0.045}	0.06	0.55	1.0	0.02	0.001	18	10.5	0.45	0.045	0	Bal
Nb _{0.5}	0.06	0.55	1.0	0.02	0.001	18	10.5	0.45	0	0.5	Bal

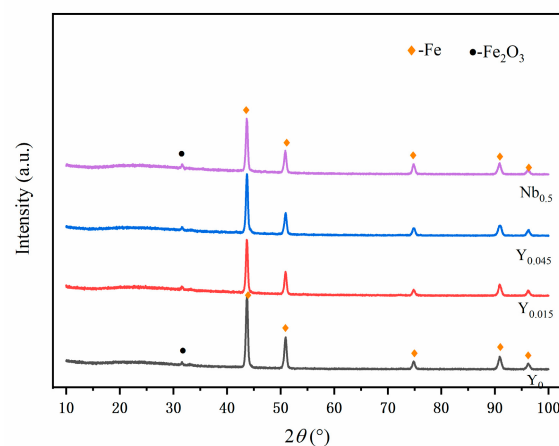
3. Results

3.1. Oxidation Result at 600 °C

Figure 1 shows the SEM image of the alloy oxidized at 600 °C for 200 h. As shown in Figure 1, no peeling phenomenon was observed in the four groups of samples. After 200 h oxidation at 600 °C, the results showed that the oxidation degree of the alloy surface was lower than before. In addition, by comparing Figure 1e–h, the composition significantly affects the morphology of the oxide film. The micro-addition of Y made the oxide film on the alloy surface more compact, and the appearance of oxidation pores could be observed (Figure 1f,g). Silver clusters appeared on the surface of the Nb_{0.5} alloy, and many oxide particles appeared on the surface.

**Figure 1.** SEM image of sample oxidation at 600 °C. (a,e) Y₀; (b,f) Y_{0.015}; (c,g) Y_{0.045}; (d,h) Nb_{0.5}.

Tests and analysis were performed using XRD, on an alloy that has been oxidized at 600 °C for 200 h to determine the oxide film phase composition. As shown in Figure 2, the main diffraction peak of the four alloy groups is the Fe matrix. The presence of Fe₂O₃ was also observed. The results show that the main composition of the oxide film is Fe₂O₃ at 600 °C, and the addition of Y or Nb elements had little effect on the oxidation resistance of the alloys at this temperature.

**Figure 2.** XRD diagram of sample oxidation at 600 °C.

3.2. Oxidation Result at 650 °C

Figure 3 shows the SEM image of the alloy oxidized at 650 °C for 200 h. As seen from Figure 3a, the oxide film on the surface of Y_0 alloy is distributed in a “network chain” shape, and a small amount of oxide clusters are observed. The addition of 0.015 wt.% did not significantly improve the oxidation resistance of the alloy. As seen from Figure 3b,f, many oxide particles with a gully distribution are formed. When continuing to increase rare earth content, as shown in Figure 3c,g, the oxide particles on the surface of the $Y_{0.045}$ alloy are more evenly distributed, the oxide layer is denser, and the oxide crystal nucleus is denser. Additionally, dense oxide films were also observed on the surface of $Nb_{0.5}$ alloy, which could effectively prevent oxygen diffusion into the matrix. The oxidation resistance of 321 stainless steel can be improved effectively by adding the proper amount of Y or Nb.

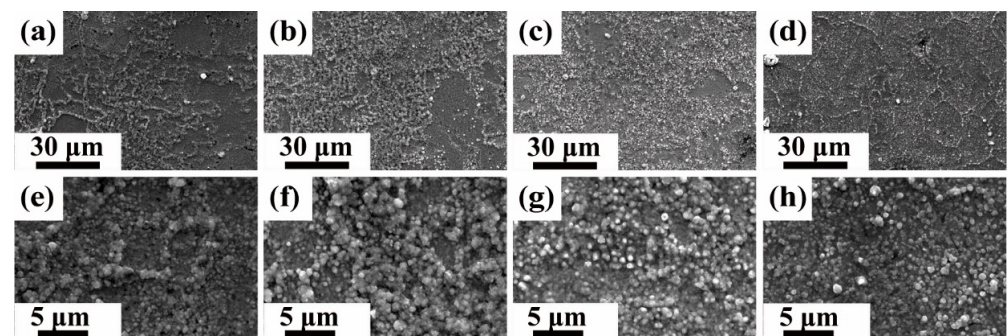


Figure 3. SEM image of sample oxidation at 650 °C. (a,e) Y_0 ; (b,f) $Y_{0.015}$; (c,g) $Y_{0.045}$; (d,h) $Nb_{0.5}$.

Figure 4 shows the XRD patterns of the four groups of alloys oxidized at 650 °C for 200 h. $Y_{0.015}$ alloys only detected an obvious matrix diffraction peak. This also suggests that the specimen under 650 °C temperature occurs only during mild oxidation. The $Y_{0.015}$ alloy spectrum shows a weak surface oxide diffraction peak, and the main components are Fe_2O_3 and Cr_2O_3 . This further indicates that the addition of 0.015 wt.% Y will deteriorate the oxidation resistance of 321 stainless steel.

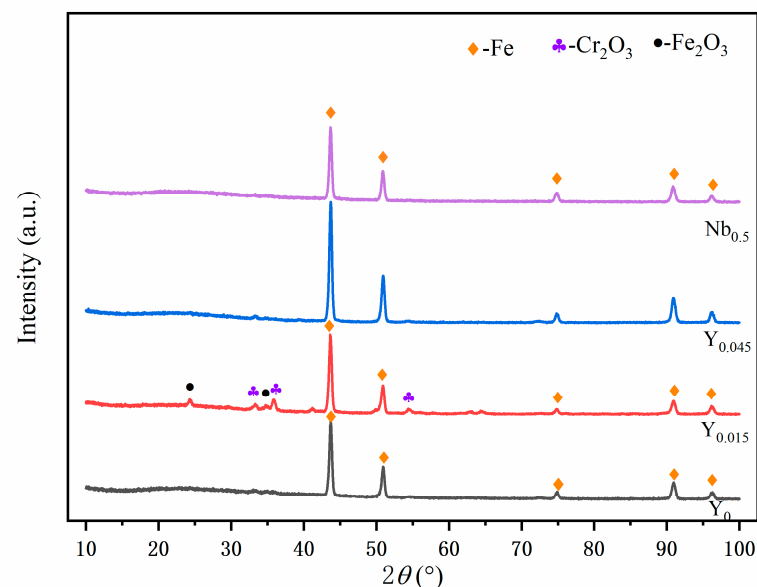


Figure 4. XRD diagram of sample oxidation at 650 °C.

3.3. Oxidation Results at 700 °C

Further detection by SEM imaging was found (Figure 5). The addition of components has a significant effect on the oxidation resistance of the alloy. The temperature increase also

greatly affects the alloy (Figures 1 and 3). The distribution of the oxide layer on the surface of the four groups of alloys is different. As shown in Figure 5a,e, it is observed that the oxide layer on Y_0 alloy surface is thicker and fluffier, with honeycomb distribution. Oxide layer adhesion is poor, and the loose and porous oxide layer cannot effectively inhibit the diffusion of oxygen, which does not provide effective protection against high-temperature oxidation. In contrast, as shown in Figure 5b,f, the surface of $Y_{0.015}$ alloy has slight cracks and clumps. When the Y content of rare earths is increased, as shown in Figure 5c,g, it is obvious that the oxide layer on the surface is more evenly distributed, and no cracking occurs on the surface. At high temperatures, rare earth Y can enhance the alloy's oxidation resistance by moderate amounts. The surface oxide layer of $Nb_{0.5}$ alloy is granular and more uniform and compact.

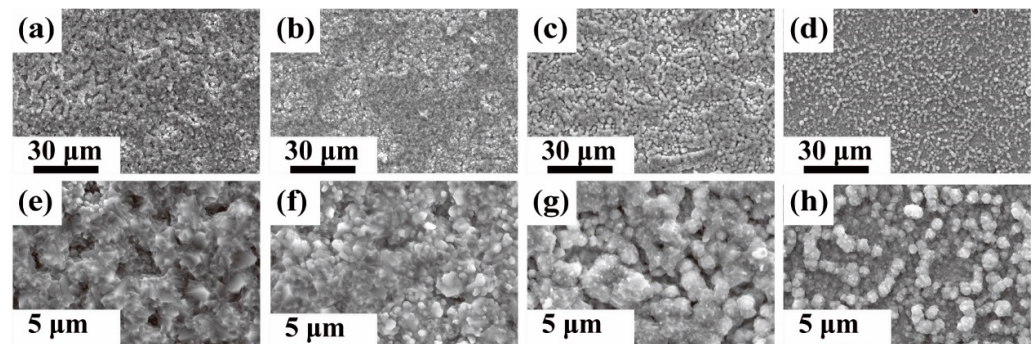


Figure 5. SEM image of sample oxidation at 700 °C. (a,e) Y_0 ; (b,f) $Y_{0.015}$; (c,g) $Y_{0.045}$; (d,h) $Nb_{0.5}$.

Figure 6 shows the EDS diagram of the four groups of alloys oxidized at 700 °C for 200 h. The main distribution of Cr, Ni, Fe, Y, and Nb elements can be observed. Only a few Fe-rich pores were observed in Y_0 alloy, despite very different Fe enrichments in the four sample groups (Figure 6b). A single addition of a small amount of rare earth element $Y_{0.045}$ alloy makes the Fe-rich concentration more widely distributed, but compared with $Nb_{0.5}$ alloy, a single addition of rare earth element has little effect on the alloy's high-temperature oxidation resistance. The $Nb_{0.5}$ Fe-rich alloy is bright, which is consistent with the XRD analysis (Figure 7). An alloy's oxidation resistance depends primarily on whether Cr can generate Cr_2O_3 to prevent oxygen from entering the matrix. In contrast, the Ni and Cr concentration and distribution are more uniform in $Nb_{0.5}$ alloy. It can be seen that the addition of Nb can accelerate the diffusion rate of Cr and Ni, and the addition of Y and Nb can keep the content of Cr stable in the oxide layer and promote the formation of the Cr_2O_3 oxide film, as shown in Figure 8d,f,h.

Figure 7 shows the XRD diagram of a after alloy oxidation at 700 °C for 200 h. In the four groups of samples, the main diffraction peak is the matrix. In addition, also observed oxide of the diffraction peak significantly. The main oxides are Cr_2O_3 and Fe_2O_3 . Additionally, a weak Fe_3O_4 diffraction peak was also detected in the $Y_{0.045}$ alloy.

Figure 8 shows the alloy's cross-section morphology and EDS analysis in backscattered electron image (BSE image) after the sample was oxidized at 700 °C for 200 h. Y_0 alloy did not form an oxide film to protect the matrix effectively. The thickness of the dense oxide layer of the alloy is about 0.6 μm. The Fe content in the surface layer is very small (Figure 8b). The corrosion of the oxide layer on the matrix was observed. The thickness of the oxide film of the matrix steel was uneven and there were cracks (Figure 8a). As shown in Figure 8c,d, the $Y_{0.045}$ alloy oxide layer thickness is about 0.6 μm, but Cr content is stable and the alloy oxide layer is denser, with fewer cracks and holes. The alloy thickness of $Nb_{0.5}$ in the oxide layer is thinner, about 0.4 μm (Figure 8f). The interface between the oxide layer and the alloy matrix is straight, indicating that the oxide layer has good adhesion.

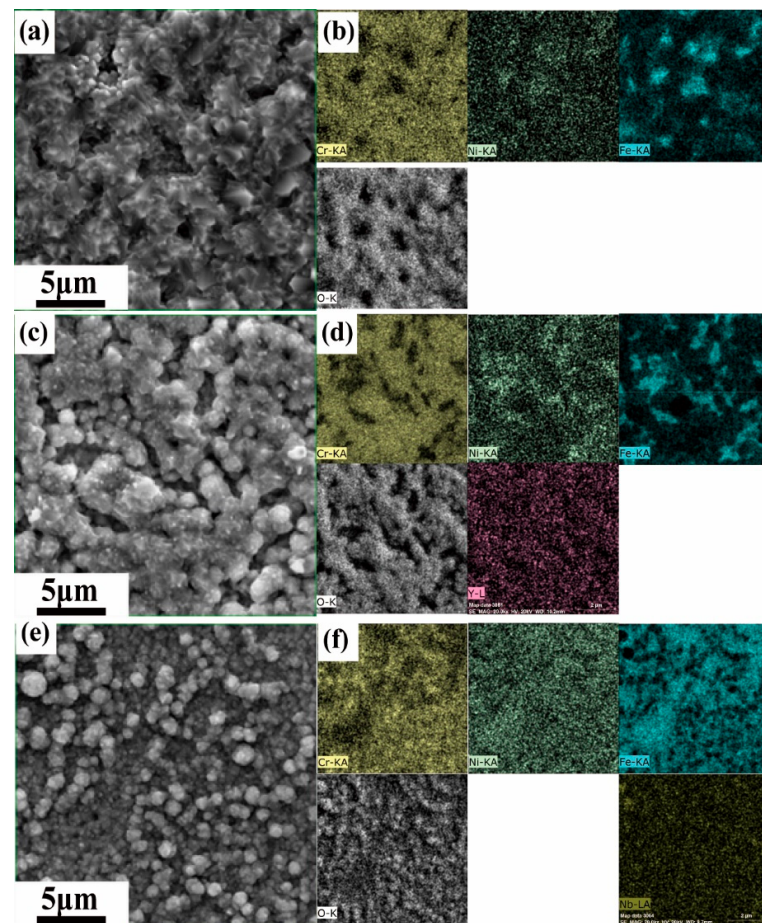


Figure 6. EDS diagram of sample oxidation at 700 °C. (a,b) Y_0 ; (c,d) $Y_{0.045}$; (e,f) $N_{0.5}$.

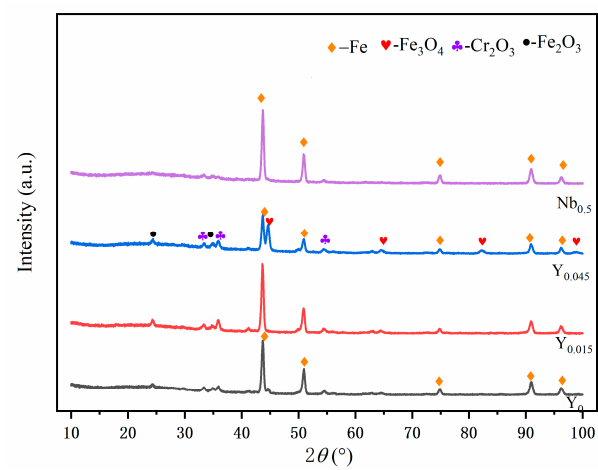


Figure 7. XRD diagram of sample oxidation at 700 °C.

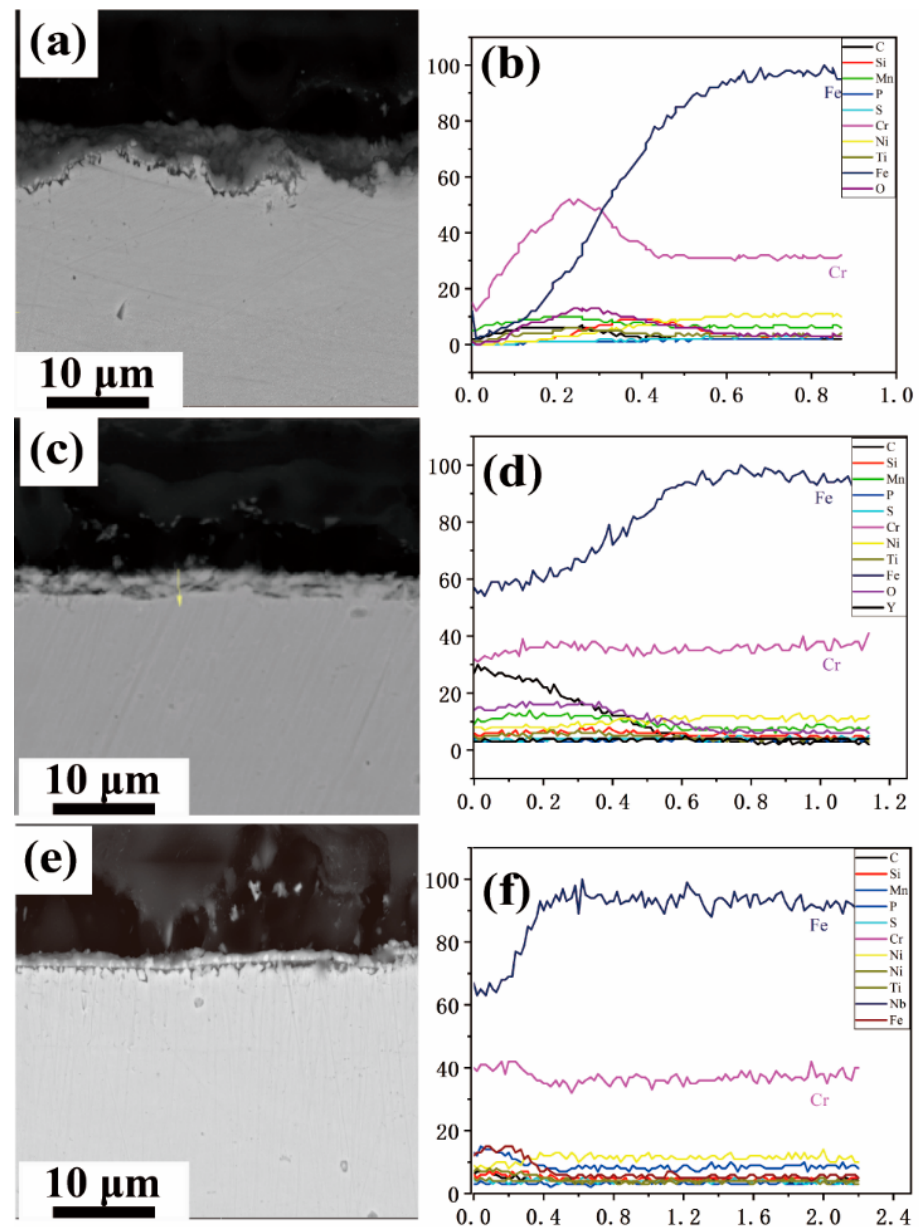


Figure 8. BSE diagram of sample oxidation at 700 °C. (a,b) Y_0 ; (c,d) $Y_{0.045}$; (e,f) $N_{0.5}$.

4. Discussion

4.1. Effect of Component Addition on Oxidation Properties

The main elements are heat-resistant Fe and Cr, while other common elements include Ni and Si. Whether a continuous and dense Cr_2O_3 layer is formed in the inner layer of the oxide film is a key factor affecting the oxidation resistance of heat-resistant steel. There are several oxidation reactions of Fe and Cr in heat-resistant steel.





It can be determined whether oxidation is feasible and also the order of oxide formation by using the Richardson–Ellingham diagram in thermodynamics of oxidation. Therefore, the possible sequence of oxidation products of S321 steel is $\text{SiO}_2 > \text{MnO} > \text{Cr}_2\text{O}_3 > \text{FeO} > \text{Fe}_3\text{O}_4 > \text{NiO} > \text{Fe}_2\text{O}_3$. In the oxidation process, due to the slow nucleation rate and a small content of Si and Mn, O on the substrate surface immediately reacts with Cr, and the surface is oxidized rapidly to form Cr_2O_3 . As the reaction progresses, FeO oxides preferentially form on the steel surface and further promote oxidation inside the steel. The oxide layer containing FeO on the surface is usually loose, porous, and easy to shed, which greatly reduces the oxidation resistance of the steel. The further oxide layer is mainly composed of Fe_3O_4 and Fe_2O_3 . The oxidation rate decreases significantly when a continuous Fe_2O_3 layer is formed on the metal surface. The oxidation resistance of heat-resistant steel mainly depends on the oxide layer of the Cr_2O_3 . The protective effect of Cr_2O_3 layer on the substrate involves three processes. Firstly, the oxide layer is formed; secondly, its growth and stability prevent further oxidation, and thirdly, there is adhesion between the oxide layer and the matrix.

Rare earths can increase the diffusion rate of Cr in the alloy. Oxygen layer growth is influenced by outward diffusion of metal ions and the inward diffusion of oxygen ions. The ion diffusion rate is small, the growth of the oxide layer is slow, which also hinders the development of oxidation into the interior of the matrix. Large ionic radii characterize rare earths and tend to be polarized at oxide grain boundaries, which affects the diffusion of metal ions at grain boundaries. Oxidation resistance deteriorates most significantly when the oxide layer peels off. The effect of rare earths on the adhesion of the oxide layer to the substrate is mainly the nailing action and the prevention of cavity formation, thus, improving the adhesion between the oxide layer and the matrix (Figure 8c). The results are consistent with Han Guichun [25] and others who found that the diffusion coefficient of Cr in $\text{Cr}_{24}\text{Ni}_7\text{N}$ steel is increased by four times by rare earths. The oxide skin was analyzed chemically and structurally by X-ray diffraction. The rare earth promotes the diffusion of Cr in the matrix, which increases the content of Cr in the oxide skin and enables the formation of a relatively dense and complete Cr_2O_3 oxide film on the surface, thereby preventing further oxidation of the matrix. However, adding of 0.05 wt.% Y rare earth element does not improve the oxidation resistance of heat-resistant steel, still, it accelerates the growth of oxide film, which is not conducive to the oxidation resistance of 321 steel.

Nb is considered an important micro-alloying element. Adding Nb has obvious effects on grain refinement and moderate precipitation reinforcement of steel. The nailing effect of Nb precipitates on grain boundaries can inhibit grain growth, thus improving the steel's oxidation and spalling resistance, thus improving the adhesion between the oxide layer and the substrate. Figure 5d shows that the surface oxide layer is significantly denser, and the surface of the oxide layer is distributed in a granular manner. Using scanning electron microscopy analysis, the addition of Nb element can also hinder the inward diffusion of O ions, thus slowing down the oxide layer formation. At the same time, the addition of Nb can also prevent the inward diffusion, thus slowing down the formation of the oxide layer.

4.2. Oxidation Kinetics Analysis

Figure 9 shows the mass gain curve of an alloy oxidized at 700 °C for 200 h. $\text{Nb}_{0.5}$, $\text{Y}_{0.045}$, and Y_0 alloys had lower mass gain during the oxidation process than Y_0 alloy. The mass gain of $\text{Y}_{0.015}$ alloy is greater than that of Y_0 alloy. The mass gain of $\text{Nb}_{0.5}$ alloy is the smallest. Changes in the oxidation rate of all samples were similar, as shown in Figure 9. The oxidation rate increases rapidly during the first 20 h. After 20 h, the oxidation rate gradually decreases, and the two processes are usually referred to as rapid oxidation and transition oxidation, respectively. After 160 h of oxidation, the oxidation rate tends to

stabilize, usually called the diffusion control stage. For four groups of alloys, their mass gain curves follow the parabolic rate law (Figure 9a). Parabolic law is defined as:

$$(\Delta W)^2 = Kt + C \quad (5)$$

where ΔW (mg/cm²) is oxidation gain per unit area; K is the oxidation rate constant; t (h) is the oxidation time. The fitting expression is:

$$Y_0:\Delta W^2 = 2.467 \times 10^{-4} t - 5.78 \times 10^{-12} \quad (6)$$

$$Y_{0.015}:\Delta W^2 = 3.85 \times 10^{-4} t + 4.11 \times 10^{-13} \quad (7)$$

$$Y_{0.045}:\Delta W^2 = 1.708 \times 10^{-4} t - 2.642 \times 10^{-12} \quad (8)$$

$$Nb_{0.5}:\Delta W^2 = 1.004 \times 10^{-4} t + 6.561 \times 10^{-10} \quad (9)$$

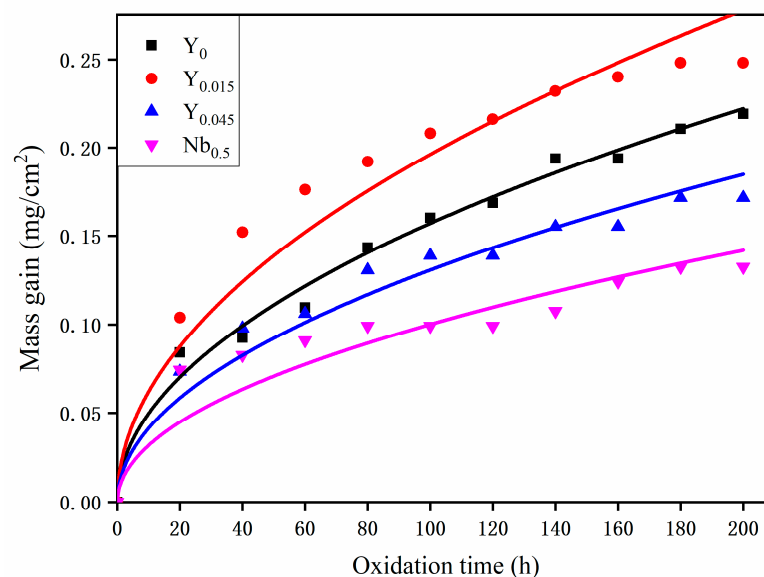


Figure 9. The oxidation kinetics curve of the sample at 700 °C for 200 h.

When fitting the data of each alloy separately, they all showed a good degree of fitting. $Y_0:R^2 = 0.987$, $Y_{0.015}:R^2 = 0.937$, $Y_{0.045}:R^2 = 0.954$, $Nb_{0.5}:R^2 = 0.848$, respectively. It can be seen from the figure that at the same temperature, the oxidation rate of $Y_{0.045}$ alloy and $Nb_{0.5}$ alloy is lower than that of Y_0 alloy, which again indicates that adding 0.045% Y or 0.5% Nb helps to enhance the alloy's high-temperature oxidation resistance. Additionally, alloy $Nb_{0.5}$ has the best high-temperature oxidation resistance, consistent with the previous testing results.

5. Conclusions

The effect of Y and Nb micro-alloying on the oxidation behavior of 321 heat-resistant steel at 600 °C, 650 °C, and 700 °C in the air was studied. The main conclusions are as follows:

- (1) $Nb_{0.5}$ alloy heat-resistant steel has the most excellent oxidation resistance at high temperatures among the five alloy groups. The oxide gradient of the alloy will not detach throughout the entire oxidation process. After oxidation at 700 °C for 200 h, the gain of the alloy is only 0.156 mg/cm². There has been a significant improvement in the alloy's resistance to oxidation at high temperatures.

- (2) Adding an appropriate amount of active elements can effectively improve the alloy's microstructure and improve high-temperature antioxidant performance. When rare earth elements are added separately, if the amount is small, it will not work, and can even accelerate the growth of the oxide film, thereby deteriorating the oxidation resistance of the alloy.
- (3) In the high-temperature oxidation process of the alloy, rare earth Y promotes the diffusion of Cr in the matrix. It increases the content of Cr on the oxide scale, which can quickly form a relatively dense and complete Cr₂O₃ oxide film on the surface. As a result of the addition of Nb, the inward diffusion of O ions can be prevented. Oxidation rate is reduced and oxide film adhesion rate is increased. The oxidation resistance of 321 stainless steel has significantly improved.

Author Contributions: X.Y.: Conceptualization, Formal analysis, Investigation, Writing—original draft, Writing—review and editing. Z.Z.: Investigation, Data curation, Writing—review and editing. X.W.: Investigation, Data curation. X.L.: Investigation, Data curation. C.G.: Conceptualization, Resources, Project administration, Funding acquisition. X.X.: Conceptualization, Supervision. B.Y.: Conceptualization, Supervision. All authors have read and agreed to the published version of the manuscript.

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