



Oxidation behavior of Hastelloy C-2000 superalloy at 800 °C and 1000 °C

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Abstract: The oxidation behavior of Hastelloy C-2000 alloy was investigated in air at 800 °C and 1000 °C for 100 h, respectively. Oxidation kinetics and oxide scales morphologies were examined by mass gain measurement, scanning electron microscopy (SEM), X-ray diffraction (XRD) and X-ray photoelectron spectroscopy (XPS). The oxidation behavior of the alloy approximately follows a parabolic rate law. Moreover, annealing twins defect structure in matrix deteriorates the oxidation resistance of alloy due to the improvement of diffusion rates for alloying elements and oxygen atoms. At 800 °C, the microstructure is primarily composed of NiO and $\text{Cr}_{1.3}\text{Fe}_{0.7}\text{O}_6$ and the initial annealing twins structure is visible and Mo-rich phases are emerged to approach boundary of oxide scales. At 1000 °C, however, the morphology microstructure of oxide scales consists of oxide particle with fine Cr oxides and large Ni oxides by inlaying each other, whilst Mo-rich phases hardly appear closing to the interface of oxide scales.

Key words: oxidation behavior; Hastelloy C-2000 alloy; parabolic rate law; annealing twins; Mo-rich phase

1 Introduction

It is well known that Ni–Cr–Mo alloys exhibit excellent corrosion resistance under extreme exposure conditions [1–4]. Therefore, an important application of Ni–Cr–Mo alloy is used for the preparation of high temperature flue gas filter with intense gas corrosion, high temperature and large diffusion rate of oxygen. With increasing complexity and rising temperature of the media atmosphere, it is a great challenge for alloys to maintain its excellent properties such as the high-temperature corrosion resistant in oxidizing and reducing medium, as well as high temperature oxidation.

A relatively new Hastelloy C-2000 alloy is a very promising candidate material as high temperature flue gas filter due to the outstanding performance in extremely corrosive environments (reducing and oxidizing acids) [5–7]. Currently, the corrosion properties of the alloy in different media have been studied extensively [8–11]; however, the corrosive effect of oxygen (i.e., high-temperature oxidation) on alloy is often overlooked. Oxide film formed on the surface of the matrix will cause the consumption of different alloying elements, which will affect or even deteriorate

corrosion resistance of alloys. XIN and WANG [12] found that evaporation rate of Mo oxides is very fast above 725 °C, causing consumption of Mo element in matrix. ZHANG et al [8] attributed excellent corrosion resistance of the C-2000 alloy to high contents of Mo element which can inhibit proliferation and spread of corrosion. Insoluble corrosion products (the oxide MoO_3 , the polymeric species $\text{Mo}_7\text{O}_{24}^{6-}$ and $\text{Mo}_8\text{O}_{26}^{4-}$) were accumulated within the corroded regions of creviced C-2000 alloy, especially within corroded grain boundaries [13]. Obviously, corrosion resistance of alloy will be deteriorated when the Mo element is continually consumed during high-temperature oxidation. We know that the grain boundaries can accelerate the oxidation rate of alloys due to their high density of defects. Although density of defects in annealing twins is lower than that of grain boundaries, it is taken for granted that twins can also provide an effective passage for diffusion of atomic when a large amount of twins appear in matrix [14–16]. Isothermal oxidation behavior of the Hastelloy C-2000 alloy is not fully understood due to the limited studies.

The purpose of this study is to investigate oxidation behavior of the Hastelloy C-2000 alloy at high temperatures. Moreover, the effect of annealing twins

and alloying elements on oxidation behavior of the Hastelloy C-2000 alloy was discussed as well.

2 Experimental

An isothermal oxidation investigation was carried out on Hastelloy C-2000 alloy supplied by Haynes International Company. Table 1 and Fig. 1 display the nominal chemical composition and the typical microstructure of the alloy, respectively. A large number of annealing twins are detected in the matrix (see Fig. 1).

Table 1 Chemical composition of Hastelloy C-2000 alloy (mass fraction, %)

Al	C	Co	Cr	Cu	Fe
0.24	0.002	0.08	22.81	1.48	0.87
Mn	Mo	P	S	Si	Ni
0.22	15.22	0.007	0.003	<0.02	Bal.

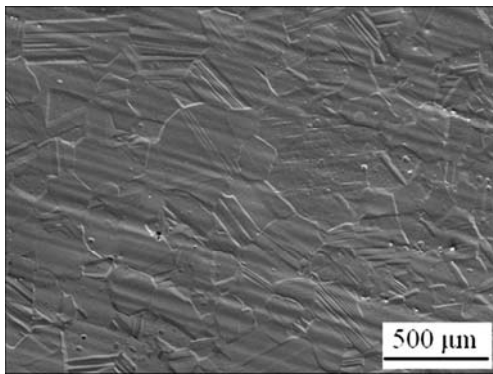


Fig. 1 Typical microstructure of Hastelloy C-2000 alloy in as-received condition

Square coupon-type samples (10 mm× 10 mm), with a thickness of 2 mm, were cut from the as-received sheet of the C-2000 alloy. The samples were polished with SiC polishing paper using successive grades up to 2000#, and then the samples were rinsed well with distilled water and then ultrasonically cleaned in ethanol before being hot air dried. After drying, the samples were stored in plastic bags. A micro-balance with a precision of 0.01 mg was used to measure mass change. Each alumina crucible was prepared at the scheduled test temperature for approximately 20 h until its mass remained at constant before the specimen was placed into it. The oxidation experiments were performed in a muffle furnace in air at 800 °C and 1000 °C for 20, 40, 60, 80 and 100 h, respectively. The mass gain change was calculated with the following equation:

$$\Delta M = \frac{m_i - m_o}{A_o t} \quad (1)$$

where ΔM is the mass gain per unit area; m_i is the instantaneous measured mass; m_o is the original mass of

the specimen; A_o is the original surface area of the specimen; t is oxidation time.

The samples were characterized by scanning electron microscopy (SEM) coupled with energy dispersive spectroscopy (EDS) analysis. In order to evaluate the surface morphology, cross-sectional microstructure, chemical composition and phase precipitation, X-ray photoelectron spectroscopy (XPS) and X-ray diffraction (XRD) were used in testing analysis.

3 Results and discussion

3.1 Isothermal oxidation kinetics

Figure 2 shows a parabolic rate law plot of the mass gain against the oxidation time. The mass gain increases with increasing oxidation temperature from 800 °C to 1000 °C. The oxidation rate and mass gain are very fast during the initial 20 h stage, which is so-called early formation stage of oxide scales. However, the mass gain rate is slow after 60 h, namely, formation stage of oxide scales has been transformed into growth stage of oxide scales. Compared with that at 800 °C, at 1000 °C, the formation rates of different oxides are all improved due to the high activity of alloying elements such as Ni, Mn, Fe and Cr, which are the primary contributor to the rapid increase in mass gain during the initial stage at 1000 °C.

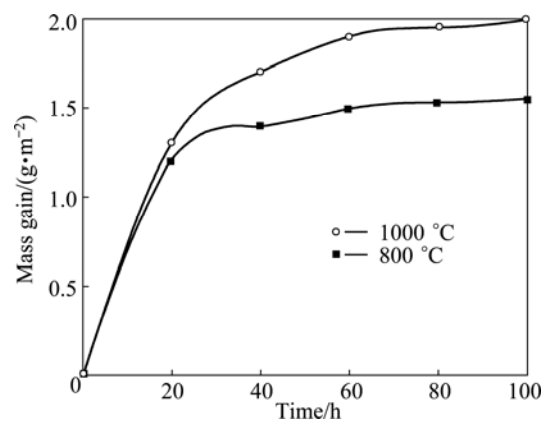


Fig. 2 Oxidation kinetic curves of C-2000 alloy oxidized in air at 800 °C and 1000 °C for 100 h

3.2 Morphology of surface oxide scales

SEM images of the surface oxide scales developed on Hastelloy C-2000 specimens after being oxidized at 800 °C and 1000 °C for 100 h are displayed in Fig. 3. At 800 °C, the surface oxide scale of Hastelloy C-2000 presents uniform granular structures (see Fig. 3(c)). In addition, initial annealing twins structure of surface is still visible (see Fig. 3(a)). At 1000 °C, however, the morphology changes to the mixture of both fine granular and large granular shape (see Fig. 3(f)). The paliform characteristic appears on the whole surface oxide scales

along grain boundaries (see Fig. 3(e)) due to the fast-diffusion paths at high temperature oxidation. YANG et al [17] reported that twins structures in alloy are generally considered preferential diffusion paths for hydrogen. Although the disorder of atoms in twins is smaller than that of grain boundaries, the contribution of abundant annealing twins in matrix to diffusion of alloying elements is not neglected. A higher oxidation rate at grain boundaries and twins leads to the rapid formation of oxide scales. As a result, initial annealing twins are difficult for observing after oxidation at 1000 °C.

Owing to Hastelloy C-2000 alloy is face-centered cubic lattice structure with low stacking fault energy, annealing twins are easily formed after being mill-annealed. Lamellar twin is formed at trigeminal grain boundary by stacking fault nucleation resulting from the existence of stacking faults, which results in the decrease of both interfacial energy and the mean free

path of dislocation [18,19]. PENG et al [20] indicated that growth of oxide scales is controlled by external diffusion of interstitial cation. The growth rate of oxide scales is much higher in annealing twins than that in other areas due to the high density of interstitial cation.

Surface oxide scales developed on Hastelloy C-2000 specimens emerge different colours and some bump structures (see Fig. 3(a)) after isothermal oxidation. Oxide scales on both annealing twins and non-annealing twins are relatively uniform resulting from a balanced growth stress and the spalling trend from alloy substrate is prevented. Table 2 shows that oxide scales are primarily composed of Cr and Ni oxides oxidized at 800 °C and 1000 °C. Meanwhile, a small quantity of Mn and Fe elements are observed at 1000 °C, indicating that Mn and Fe oxides are formed on the surface of specimens at higher oxidation temperatures. Content of Mo elements in oxide scales at 800 °C is higher than that of 1000 °C.

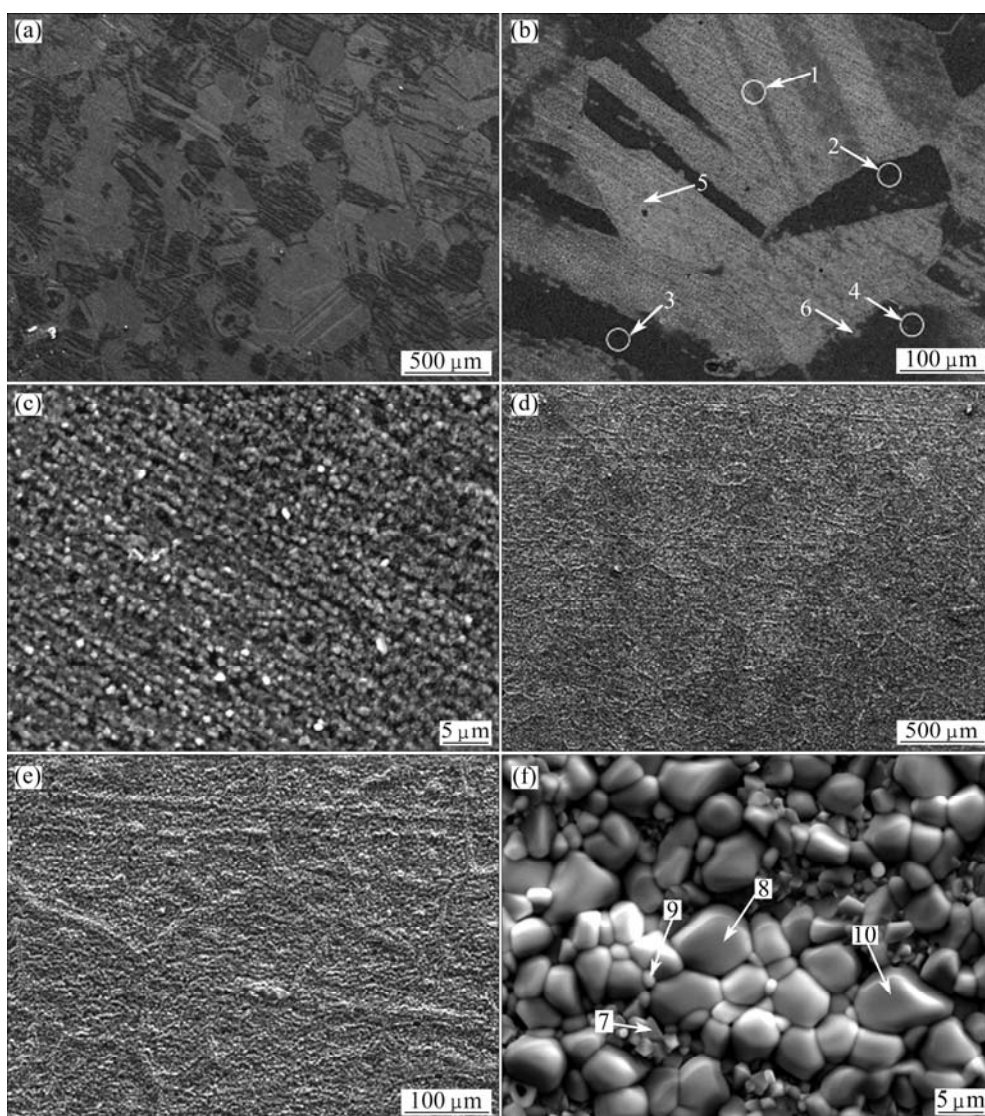


Fig. 3 SEM images of C-2000 specimen surface after oxidation at different temperatures in air for 100 h: (a)–(c) 800 °C; (d)–(f) 1000 °C

Table 2 EDS analysis results on points shown in Fig. 3

Point in Fig. 3	Mass fraction/%							
	O	Cr	Mn	Ni	Mo	Fe	C	Al
1	29.72	20.69	—	31.10	12.07	—	5.90	0.51
2	35.90	25.03	—	29.90	3.46	—	5.71	—
3	38.42	23.31	—	32.47	—	—	5.80	—
4	36.63	27.84	—	25.59	3.89	—	6.05	—
5	41.32	46.51	1.41	4.98	—	1.05	4.72	—
6	28.83	21.66	—	33.95	9.68	—	5.26	0.63
7	37.81	39.69	7.34	14.03	—	1.13	—	—
8	28.81	5.40	1.46	64.34	—	—	—	—
9	29.01	5.05	1.36	64.42	0.17	—	—	—
10	29.71	4.69	1.11	56.90	0.38	—	7.21	—

3.3 X-ray diffraction analysis

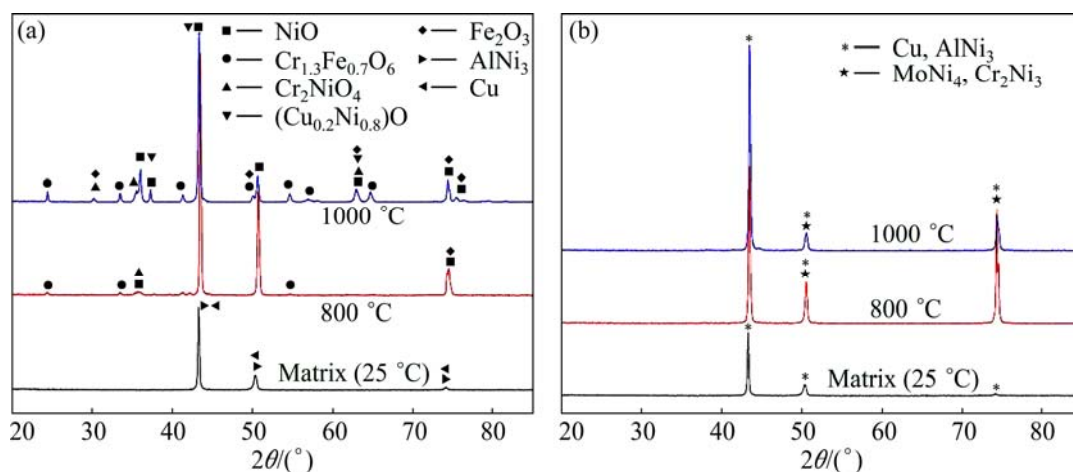
Figure 4 shows the XRD patterns of several samples of Hastelloy C-2000 oxidized at testing temperatures. At 800 °C, the XRD patterns reveal that NiO and $\text{Cr}_{1.3}\text{Fe}_{0.7}\text{O}_6$ are strongly detected as major oxide scales with a little amount of Fe_2O_3 and Cr_2NiO_4 . At 1000 °C, XRD peaks of $\text{Cr}_{1.3}\text{Fe}_{0.7}\text{O}_6$ and Fe_2O_3 strongly increase and XRD peaks of $(\text{Cu}_{0.2}\text{Ni}_{0.8})\text{O}$ are also detected (see Fig. 4(a)). The intensive peak of Fe_2O_3 indicates that a thin layer of Fe_2O_3 is formed. In addition, no XRD peaks corresponding to Mo oxides are detected. This seems to suggest that Mo oxides containing protective oxide scales are not formed due to the large volatility of Mo element at high oxidation temperatures. It seems to that the alloy matrix does not obviously change after oxidation at 800 °C and 1000 °C, as shown in Fig. 4(b).

3.4 Cross-sectional structures

Both the cross-section SEM image and elemental distribution of oxide scales structures formed on Hastelloy C-2000 specimens are shown in Fig. 5. Continuous oxide scales with 1.3 μm in thickness

oxidized at 800 °C are presented in Fig. 5(a). At 1000 °C, thickness of oxide scales is up to around 8 μm and inward oxide pegs also grow into the alloy matrix, which can improve the bonding force between oxide scales and alloy matrix [21–23]. It is well known that the thicker the oxide scales are, the larger the residual growth stress in oxide scales is. A litter amount of oxide scales spall after isothermal oxidation at 1000 °C for 100 h, indicating that the weakened effect of scales growth stress on bonding force of oxide scales is much larger than the enhanced effect of oxide pegs on bonding force of oxide scales.

It is also interesting to note that cross-section of oxide scales with partially spalling presents a bending. LIKHANSKII et al [24] investigated the structure between oxide scale and substrate interface. They discovered that a waving structure is a precursor of oxidation kinetics transformation. Since the transformation of oxidation kinetics is delayed, the compression of oxide scales will be decreased, resulting in an additional energy in alloy matrix with the bending and elastic deformation (the deformation is limited to interface). Isothermal oxidation is equivalent to long-range aging heat treatment for alloy matrix at high temperatures [25]. In this situation, there are some different variations for alloy matrix after oxidation at different temperatures. The aggregation of second phases in alloy matrix close to interface of oxide scales is observed after oxidation at 800 °C (see Fig. 5(a)). Nevertheless, it is different at 1000 °C (see Fig. 5(b)) when there are hardly any second phase close to interface of oxide scales. There is no significant distribution difference among other elements except for Mo element in the cross-sectional elemental distribution of oxide scales (see Figs. 5(c) and (d)). At 800 °C, the appearance of two small prominent peaks of Mo elements in distance of 8 and 15 μm (see Fig. 5(c)) indicates that enrichment of Mo element occurs. In addition, enrichment of Mo

**Fig. 4** XRD patterns of oxide scales (a) and alloy matrix (b) after oxidation

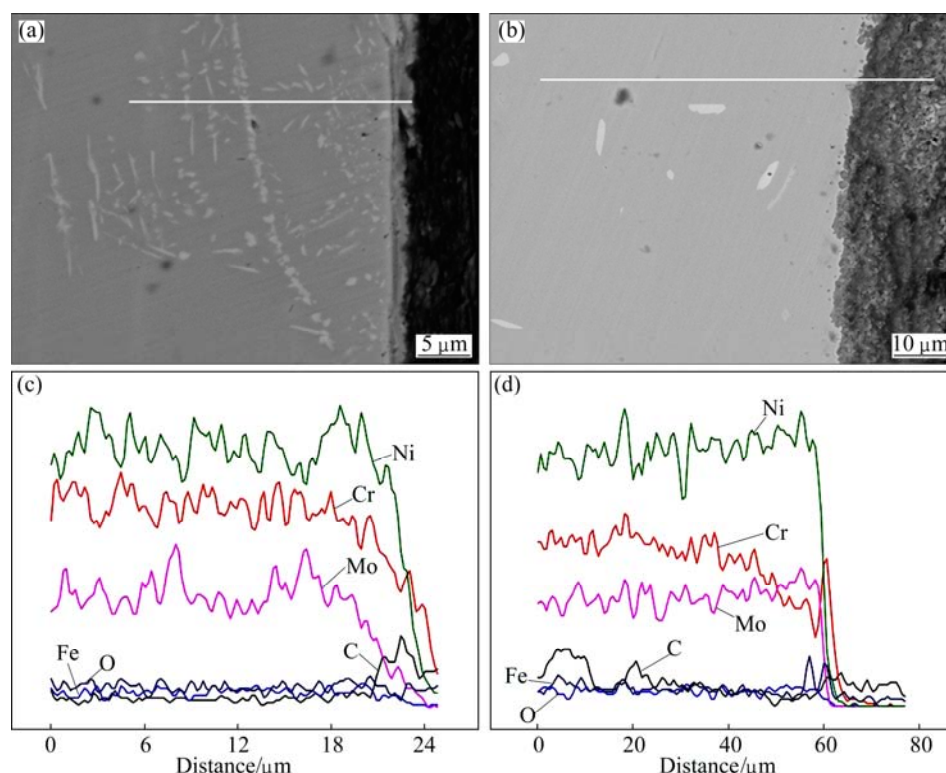


Fig. 5 Cross-section SEM images (a, b) and elemental concentration profiles (c, d) of samples after oxidation at 800 °C (a, c) and 1000 °C (b, d)

element not only appears at the critical cross-section of oxide scales, but also appears away from the cross-section (i.e. the core of the cross-section) at 800 °C. Hence, further research is necessary to be carried out on the enrichment phenomenon of Mo element in Hastelloy C-2000 superalloy. The EDS line scan shows that content of Mo element increases and that of Cr and Ni decreases in the area of second phases, respectively. Compared with spectrum 2, the content of Mo element is 45.88% in spectrum 1 by EDS in Fig. 6, which is consistent with that of the EDS line scan. XRD peaks of MoNi_4 are detected in alloy matrix after oxidation (see Fig. 4(b)).

Precipitation of Mo-rich phases occurs in alloy matrix after oxidation due to the fact that Cr and Fe oxides are formed on alloy surface at 800 °C, which can provide a protection to the matrix and decrease evaporating of Mo oxides. Therefore, a concentration gradient of Mo is established in alloy matrix when external diffusion of Mo element is inhibited. As a result, the driving force for enrichment of Mo element increases. Furthermore, the concentration of Mo at grain boundary during oxidation processes reached a minimum because the grain boundary provides a fast diffusion path of different elements. Finally, Mo element begins to transport from matrix into both areas of grain boundaries and vacancy defects, thus making enrichment of Mo element appears in above regions.

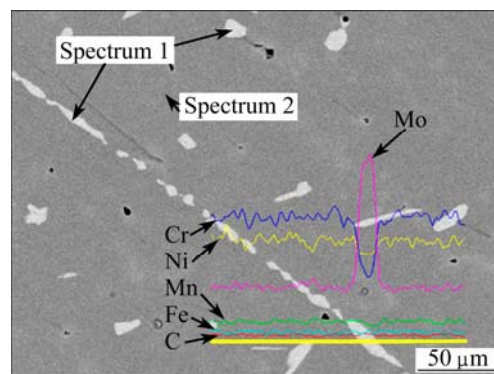


Fig. 6 Elemental concentration profile across alloy matrix after oxidation at 800 °C

TEM bright-field images and EDS patterns of samples after oxidation at 800 °C and 1000 °C are given in Fig. 7. Chemical compositions of the second phases (mass fraction, %) are 28.66 Ni, 19.60 Cr and 51.74 Mo (see Fig. 7(b)), which are consistent with the results of Fig. 6. It is determined that Mo-rich phases are $\text{Mo}_3\text{Ni}_3\text{C}$ -type carbides with a diamond cubic structure in terms of both chemical component and SAED (see Figs. 7(a) and (b)). M_6C -type carbides (chemical formula is $\text{A}_3\text{B}_3\text{C}$, where A and B are Ni and Mo elements, respectively) are easily formed in high Mo-containing nickel-based alloys during aging heat treatment [26]. Mo-rich phases with M_6C -type structure are not found

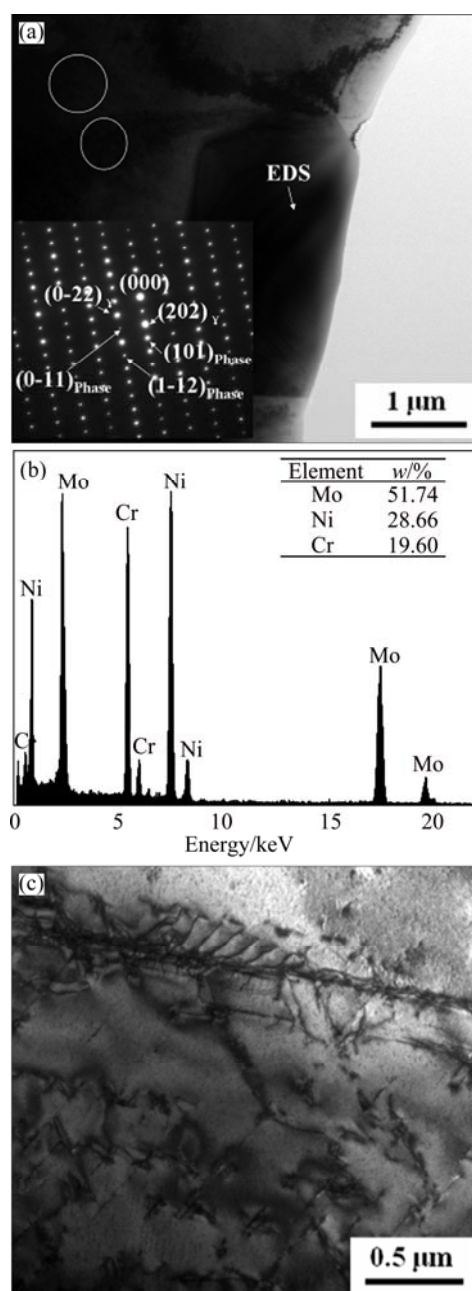


Fig. 7 TEM images (a, c) and EDS pattern (b) of sample after oxidation at different temperatures: (a), (b) 800 °C; (c) 1000 °C

after oxidation at 1000 °C, but a large number of dislocations appear in the matrix (see Fig. 7(c)). On one hand, solution of M_6C -type carbides occurred at 1000 °C. On the other hand, abundant dislocations in alloy matrix benefit diffusion of elements. As a result, the diffusion rate of Mo element increases from inward to outward. Therefore, it is difficult to form the Mo-rich phases after oxidation at 1000 °C due to the loss of Mo element.

3.5 XPS spectra of samples oxidized at 800 °C and 1000 °C

In order to make further insight to nature of oxide scales, XPS spectra of the samples oxidized at 800 °C

and 1000 °C are given in Fig. 8. It can be seen that there are O, Ni, Mo, Mn, Cr and C elements for oxide scales. Moreover, the valence state of Ni, Mo and Cr element is approximately the same at 800 °C and 1000 °C. So, the types of oxide scales formed from above elements are the same. However, there is an obvious difference in XPS peaks corresponding to Mn element at testing oxidation temperatures. Compared with that at 800 °C, two extra peaks of Mn at both 652.78 eV and 647.28 eV at 1000 °C indicated that Mn elements with different valence states are oxidized in oxide scales. ALHATAB et al [27] found that favored oxidation in the order of relevant elements is $Ni > Cr > Fe > Mn$. With increasing oxidation temperature, a small number of Mn elements start to form some Mn oxides with different valence states.

Formation rate of oxide scales is fast in early stage of oxidation. Hence, in a short period of time, overall surface of alloy is covered by an integrated protection oxide scales so that rapid evaporation of Mo oxides is suppressed. As a result, a small amount of Mo oxides remain in inner of protection oxide scales when evaporation of Mo is delayed. Finally, a small number of residual Mo oxides are less than detection range of XRD. The high evaporation rate of Mo oxides above 725 °C in pure Mo alloy was pointed out by XIN and WANG [12], at this moment, the gas diffusion becomes a rate-determining step of oxidation and oxidation is catastrophic. However, a phenomenon of catastrophic oxidation does not occur for C-2000 alloy at testing temperatures due to high contents of Ni and Cr and a certain amount of Mn and Al in the matrix. It is confirmed by the XPS and XRD testing results that a protective oxide scales would be formed on the alloy surface by interaction between elements after oxidation. Therefore, C-2000 alloy presents an outstanding oxidation resistance at testing temperatures although it is an alloy with high content of Mo.

4 Conclusions

1) Oxidation kinetics of the Hastelloy C-2000 alloy follows a parabolic rate law. At 800 °C and 1000 °C, $Cr_{1.3}Fe_{0.7}O_6$ and Fe_2O_3 oxides formed on surface of matrix have a good oxidation resistance for C-2000 alloy. However, oxidation resistance of the alloy is deteriorated by annealing twins defect structure in matrix due to the higher diffusion rate of both alloying elements and oxygen atom in defect areas.

2) There is a significant difference for morphology features of oxide scales after oxidation, namely the morphology features change from uniform granular structure at 800 °C to particle structure with fine Cr oxides and large Ni oxides by inlaying each other at 1000 °C.

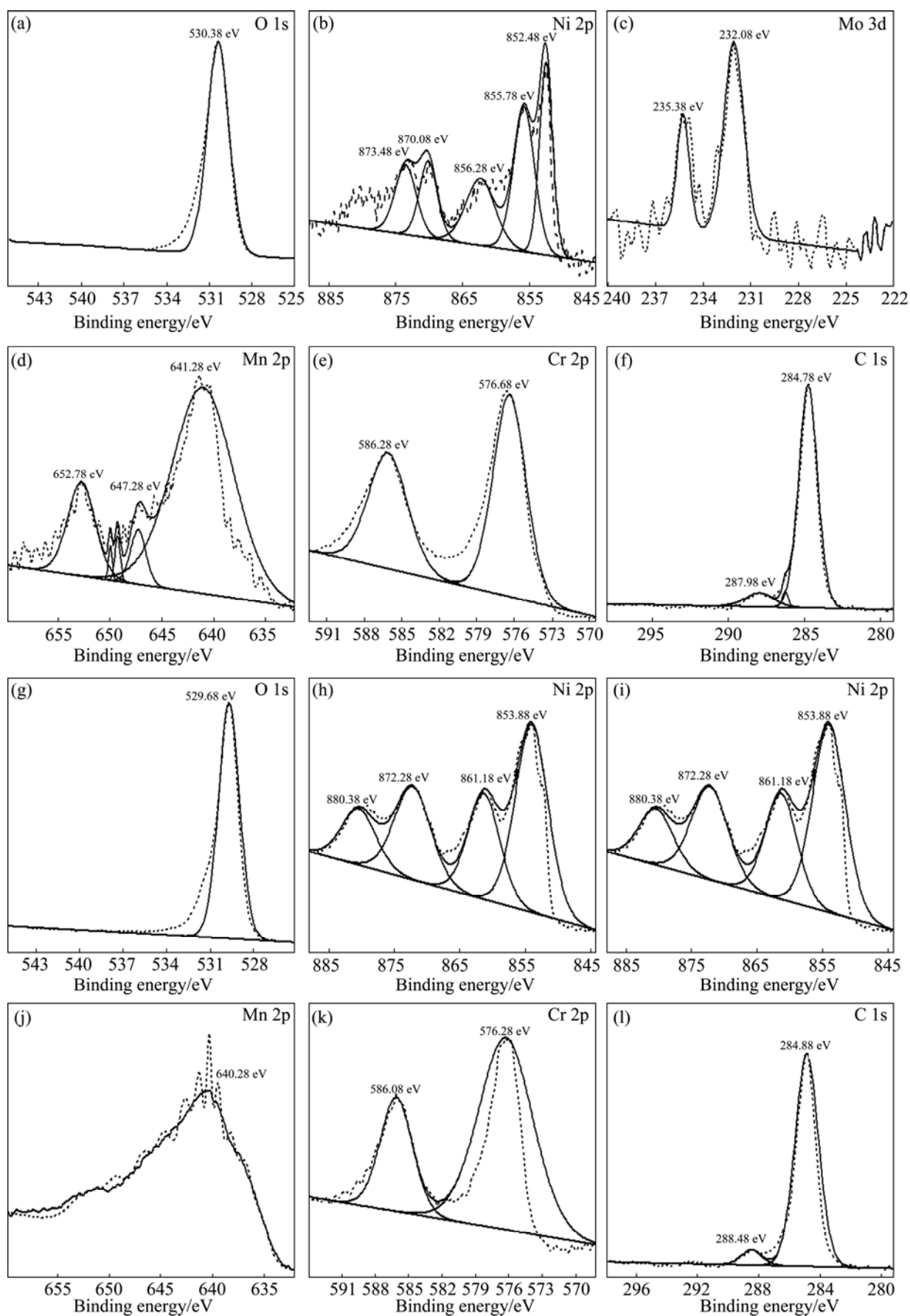


Fig. 8 XPS survey spectra of samples at different temperatures: (a)–(f) 800 °C; (g)–(l) 1000 °C

3) The Mo-rich phases with $\text{Mo}_3\text{Ni}_3\text{C}$ -type stoichiometric ratio are emerged close to boundary of oxide scales after isothermal oxidation at 800 °C. At 1000 °C, however, Mo-rich phases hardly appear close to interface of oxide scales, which is attributed to both solution of M_6C -type carbides at high temperature and acceleration effect of abundant dislocations on diffusion of Mo element.

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哈氏 C-2000 合金在 800 °C 和 1000 °C 的氧化行为

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摘 要: 研究哈氏 C-2000 合金在(800 °C, 1000 h)和(1000 °C, 100 h)时的氧化行为。分别利用增重法、SEM、XRD 和 XPS 表征氧化动力学和氧化膜的形貌特征。合金在 800 °C 和 1000 °C 时均基本满足抛物线速率规律。此外, 由于退火孪晶提高了合金化元素及氧原子的扩散速率, 因此, 降低了合金的高温抗氧化性能。在 800 °C 时, 氧化膜微观结构主要是由 NiO 和 $\text{Cr}_{1.3}\text{Fe}_{0.7}\text{O}_6$ 组成。此外, 初始退火孪晶结构在氧化后依然可见, 且临近氧化膜附近出现了富 Mo 相。然而, 在 1000 °C 时, 氧化膜的微观结构由细小的铬氧化物和粗大的镍氧化物颗粒通过相互镶嵌而构成, 同时, 在临近氧化膜处几乎无富 Mo 相出现。

关键词: 氧化行为; 哈氏 C-2000; 抛物线规律; 退火孪晶; 富 Mo 相

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