

Rare Earth Elements Enhanced the Oxidation Resistance of Mo-Si-Based Alloys for High Temperature Application: A Review

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Abstract: Traditional refractory materials such as nickel-based superalloys have been gradually unable to meet the performance requirements of advanced materials. The Mo-Si-based alloy, as a new type of high temperature structural material, has entered the vision of researchers due to its charming high temperature performance characteristics. However, its easy oxidation and even "pesting oxidation" at medium temperatures limit its further applications. In order to solve this problem, researchers have conducted large numbers of experiments and made breakthrough achievements. Based on these research results, the effects of rare earth elements like La, Hf, Ce and Y on the microstructure and oxidation behavior of Mo-Si-based alloys were systematically reviewed in the current work. Meanwhile, this paper also provided an analysis about the strengthening mechanism of rare earth elements on the oxidation behavior for Mo-Si-based alloys after discussing the oxidation process. Furthermore, the research focus about the oxidation protection of Mo-Si-based alloys in the future was prospected to expand the application field.

Keywords: Mo-Si-based alloys; Alloying; Rare earth elements; Oxidation behavior; Mechanism

1. Introduction

As the world population increases, the problem of global resource shortage has become increasingly prominent. It is well known that in addition to waste recycling, improving energy utilization and exploring new energy are effective methods to solve resource problems, and are also the main trend of future scientific and technological development [1-3]. Nowadays, the development of new energy is overwhelming. As a new high temperature structural material, the Mo-Si-based alloy is expected to replace the nickel-based alloy and play an important role in turbine engine and industrial furnace components [4-7].

A large number of studies have pointed out that Mo-Si-based alloys have outstanding high temperature performance characteristics, such as moderate density, strong electrical and thermal conductivity, ultra-high melting point, high thermal impact resistance, etc., which has been widely used in various industries [8-11]. However, these alloys also have some inherent defects that limit their generalization as structural materials

[12-14]. For example, MoSi₂-based alloys exhibit low room temperature fracture toughness, poor high temperature creep resistance, and catastrophic oxidation at 400 °C to 800 °C. Although Mo₅Si₃-based alloys have relatively strong creep resistance, they generally present accelerated oxidation or "pesting oxidation" phenomenon below 1000 °C, which is a thorny problem [15-19]. To overcome these shortcomings, relevant researchers have been working hard since the end of the 20th century [20-25]. Fortunately, people finally succeeded in improving the properties of alloy through material designs [26-29], preparation techniques [30-34] or surface modification methods [35-38]. Among them, doping second phases such as W, Nb, ZrO₂, La₂O₃, Al₂O₃, Cr₂O₃ in material designs to strengthen the performance of substrate is generally regarded as an important measure [39-41].

There is no doubt that in some refractory metal materials like niobium-based, molybdenum-based and tungsten-based materials, adding active elements may significantly improve the mechanical properties [42-44]. Rare earth or its oxides, as a kind of active element, have received particular attentions and widespread applications [45-48]. In recent years, researchers have made great breakthroughs in studying rare earth elements to enhance the mechanical properties of metal materials. However, so far, there are few reports on the oxidation behavior of Mo-Si-based alloy doped with rare earth elements. Therefore, this paper comprehensively and systematically reviewed the actions of rare earth elements such as La, Hf, Ce and Y on the antioxidant properties of Mo-Si-based alloys, especially the Mo-Si-B system, and summarized the relevant strengthening mechanisms.

2. Effects of rare earth elements on oxidation behavior of Mo-Si-based alloys

It's well known that the increase of oxide layer thickness is primarily caused by the internal diffusion of O₂ [49-52], and studies have shown that alloying with active elements can effectively reduce the internal diffusion rate of O₂ [53-55]. For example, appropriate adding rare earth elements, on the one hand, can separate oxygen atoms at the metal-oxide interfaces and oxide scale grain boundaries and react with O₂, thus hindering the diffusion of O₂ [56-59]. On the other hand, it can optimize the oxide scale microstructure and improve the scale adhesion [60-63]. Therefore, alloying with rare earth elements is of great significance to enhance the antioxidant properties. The influences of adding rare earth elements like La, Hf, Ce and Y on the oxidation behavior of Mo-Si-based alloys will be reviewed in detail below.

2.1 Effects of rare earth elements on oxidation behavior of Mo-Si-B alloys

Among all kinds of Mo-Si-based alloys, the most widely studied are Mo-Si-B alloys because of their excellent characteristics[64-70]. Therefore, it's very necessary to explore the effects of rare earth elements on

oxidation behavior of Mo-Si-B alloys.

2.1.1 Effects of La element addition

Compared with pure Mo-Si alloys, adding La element can significantly optimize the microstructure of these alloys by the means of reducing the grain size and making the intermetallic particles disperse more evenly, thus improving the fracture toughness, bending and compressive strength significantly [71-74]. Based on the existing studies of Mo-12Si-8.5B alloy (at.%) [75-79], we further analyzed the actions of doping La or La_2O_3 second phase on the oxidation behavior.

Zhang et al. [80] prepared Mo-12Si-8.5B (at.%, abbreviated as MSB) samples added with different contents of La_2O_3 through arc-melted and spark plasma sintered methods, and the specific contents were presented in Table 1. Fig. 1 (a) gives the XRD patterns of MSB+x La_2O_3 samples (x=0, 0.3, 0.6, 1.2 wt.%). It can be seen that all the samples consist of Mo_5SiB_2 , Mo_3Si and $\alpha\text{-Mo}$ three phases, which is consistent with the phase diagram of isothermal Mo-Si-B composites [76]. At the same time, it also reveals that even if La_2O_3 is added will not affect the phase composition of samples. Figs. 1 (b-e) are micrographs of the four samples prepared, where the white regions are $\alpha\text{-Mo}$ phase, and the black regions are $\text{Mo}_5\text{SiB}_2/\text{Mo}_3\text{Si}$ phases dispersed in the $\alpha\text{-Mo}$ matrix. It can also be found from micrographs that the grain size of $\alpha\text{-Mo}$ and $\text{Mo}_5\text{SiB}_2/\text{Mo}_3\text{Si}$ will be reduced after adding La_2O_3 , in which the $\alpha\text{-Mo}$ size change is more pronounced, whereas the decrease of each phase size is not sensitive to the La_2O_3 mass fraction. Moreover, the distribution of $\text{Mo}_5\text{SiB}_2/\text{Mo}_3\text{Si}$ phases is more uniform after doping La_2O_3 . This is because parts of La_2O_3 can be used as nucleation sites, which leads to the increase of nucleation density. On the other hand, La_2O_3 particles play a "pinning" role on the $\alpha\text{-Mo}$ boundary to inhibit its grains growth. The results in Table 1 further reveal the effect of La_2O_3 on grain size.

Table 1. The La_2O_3 mass fractions and grain sizes of various samples studied. Reproduced with permission [80]. Copyright 2011 Elsevier.

Materials	MSB	MSB + 0.3	MSB + 0.6	MSB + 1.2
Mass fraction of La_2O_3 (wt.%)	0	0.3	0.6	1.2
Grain sizes of $\alpha\text{-Mo}$ (μm)	19.78	10.88	9.56	9.46
Grain sizes of $\text{Mo}_3\text{Si}/\text{Mo}_5\text{SiB}_2$ (μm)	3.04	2.46	2.55	2.17

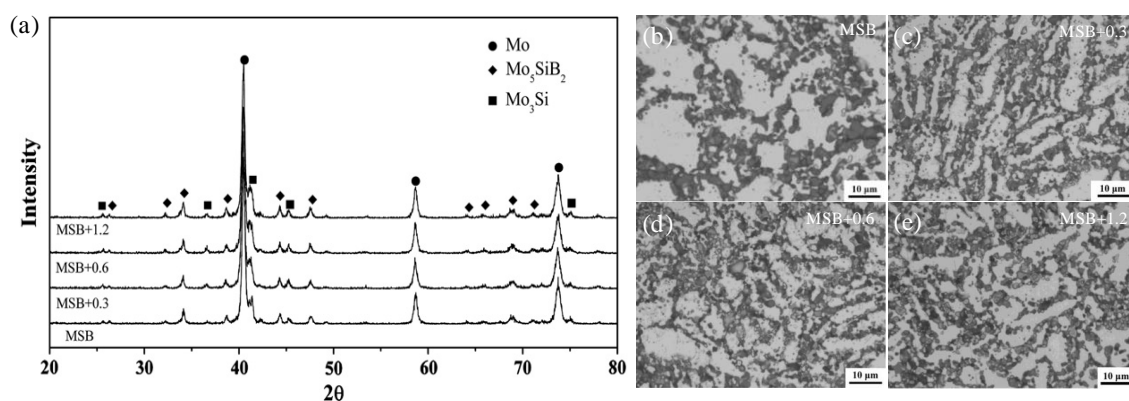


Fig. 1. XRD patterns (a) and microstructure images (b-e) of MSB+xLa₂O₃ samples (x=0, 0.3, 0.6, 1.2 wt.%) before oxidation. Reproduced with permission [80]. Copyright 2011 Elsevier.

Weight variation curves for different mass fraction La₂O₃-doped MSB samples after oxidation at 800 °C are shown in Fig. 2 (a). The results point out that the mass loss of alloy can be significantly reduced after adding La₂O₃, where MSB+0.6 sample exhibits the least mass loss during transient oxidation stage. It is due to the fact that La₂O₃-doped MSB samples present a finer grain size, which makes it faster to form a protective borosilicate scale to prevent further volatilization of MoO₃. The role of grain refinement has also been reported elsewhere [81-87]. Again, La₂O₃ in the alloy can decrease the grain boundary transport rate, leading to the reduction of weight loss rate. To determine the impact of La₂O₃ on the oxidation behavior, Zhang et al. [80] further studied the cross-sectional structure of MSB+0.6 sample oxidized at 800 °C. It has been noticed from Fig. 2 (d) that the cross section of this sample is composed of oxidation scale, interlayer and substrate. Among them, the top layer is the dense B₂O₃-SiO₂ scale and the interlayer comprised with MoO₂, which is confirmed by XRD analysis and the content of each element (i.e. Mo, Si, B, O). Furthermore, compared with MSB sample, the intensity and peaks of B₂O₃ and SiO₂ of MSB+0.6 sample are raised visibly (Fig. 2 (b)), indicating that the antioxidant capacity of MSB+0.6 sample is enhanced. Burk [88, 89] and Jéhanno [90] et al. also reported similar results.

In addition, Majumdar et al. [59, 60] also investigated the oxidizability of Mo-9Si-8B (at.%) sample doped with 2at.% La at 750-1400°C. It is established that the La-doped sample exhibits relatively good antioxidant capability below 1000 °C, which is the result of stable lanthanum oxides like 3La₂O₃·MoO₃, La₂O₃ and La₂O₃·3MoO₃ produced at the oxidation scale to inhibit the formation and volatilization of MoO₃. However, when the temperature exceeded 1000 °C, the addition of La might adversely affect the sample oxidation properties. Fig. 2 (c) displays the weight change curves of La-doped and undoped samples at 1300 °C, it can be discovered that the weight loss of La-doped sample is significantly higher than that of undoped sample.

This is because adding La makes the sample cross section present a loose and porous oxide layer structure when oxidized at 1200 °C (Fig. 2 (e)). Meanwhile, as oxidation temperature rises to 1300 °C, a large number of cracks and holes are observed on the sample surface (Fig. 2 (f)), which provides a pathway for O₂ internal diffusion and MoO₃ volatilization. In contrast, even if undoped sample is oxidized at 1300 °C for 72 h, a continuous and compact oxidation scale can be still observed in its cross section (Fig. 2 (g)) [88]. Thus, undoped sample has better antioxidant properties in high-temperature environments.

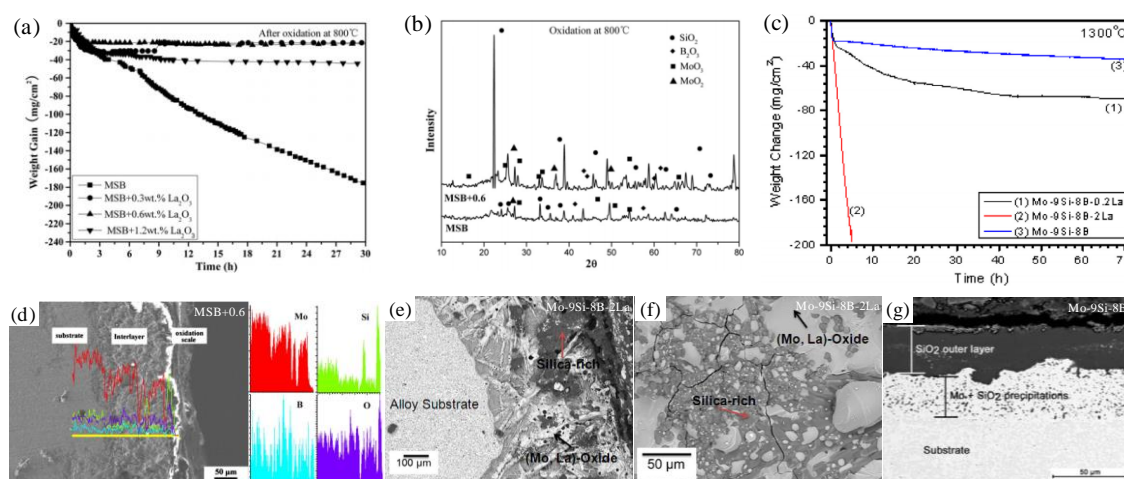


Fig. 2. Weight change curves of various samples oxidized at different temperatures (a, c); Surface and cross-sectional SEM/BSE images of various samples oxidized at different conditions: 800 °C (d) with corresponding surface XRD analysis results (b), 1200 °C for 2 h (e), 1300 °C for 23 h (f), 1300 °C for 72 h (g). (a, b, d), (c, e, f) and (g) reproduced with permissions [80], [60] and [88], respectively. Copyright 2011 Elsevier, 2014 Elsevier and 2009 Taylor & Francis.

2.1.2 Effects of Hf element addition

Extensive experiments have reported that adding Hf/HfB₂ to Mo-Si-B composites can clearly improve their performance features, such as high temperature strength, high temperature stability, creep resistance, fracture toughness, etc. [91-95]. Potanin et al. [96] discussed the oxidation behavior of MoB-HfB₂-MoSi₂ composites at 1200 °C in detail. The composition of each alloy is illustrated in Table 2, where the difference between X34₂ and X34₁ samples is that the former presents two-level structure (TLS), while the latter presents single-level structure (SLS). The microstructures of studied samples is depicted in Figs. 3 (a-c), on the whole, the three samples all contain MoB and MoSi₂ phases. The difference is that X34₁ and X34₂ samples also have additional HfSiO₄ and HfB₂ phases and their grain sizes are finer than that of X0 sample. Fig. 4 gives the oxidation kinetics curves of the three samples, it can be observed that the weight increases of X34₁ and X34₂

samples are more obvious because adding HfB_2 can make the samples generate HfSiO_4 (6.97 g/cm^3) and HfO_2 (9.68 g/cm^3), whose specific weights are greater than SiO_2 (2.36 g/cm^3) [97]. By the way, the weight gain of X34_1 sample is smaller than that of X34_2 sample owing to the fact that X34_2 sample presents special arrangement and finer grain size.

Figs. 3 (d-i) show the cross-sectional structure of X0 , X34_1 and X34_2 samples after oxidation at 1200°C for 30 h. It has been noticed that the X34_1 and X34_2 samples do produce $\text{Si}_{x-1}\text{Hf}_x\text{O}_2/\text{HfSiO}_4$ phases during the oxidation process, which is consistent with the above analysis results. Meantime, two-layered oxide films are formed on the surface of all samples, whereas there are great differences in the oxide-film composition and structure. In other words, the X0 sample oxide scale containing SiO_2 layer (top layer) and Mo_5Si_3 layer (bottom layer), the X34_1 sample oxide scale consists of $\text{Si}_{x-1}\text{Hf}_x\text{O}_2$ -doped amorphous SiO_2 layer (upper layer) and crystalline $\alpha\text{-SiO}_2$ layer (lower layer), while the X34_2 sample oxide scale is comprised with crystalline $\alpha\text{-SiO}_2$ layer (outermost layer) and HfSiO_4 layer (interlayer). Among them, the formation of amorphous SiO_2 in X34_1 sample is due to the dissolution of hafnium. Furthermore, $\text{Si}_{x-1}\text{Hf}_x\text{O}_2$ is generated because hafnium and silicon are equivalent elements leading to the incorporation of hafnium into the lattice of silica, as shown in Fig. 5. It is worth noting that the X34_2 sample oxide scale exhibits denser structure is caused by the existence of high wetting angle makes $\text{SiO}_2\text{-B}_2\text{O}_3$ melt can shrink HfSiO_4 particles together, resulting in the formation of smooth and compact oxide films [98].

To summarize, addition of HfB_2 to X0 sample can produce $\text{HfSiO}_4/\text{Si}_{x-1}\text{Hf}_x\text{O}_2$ particles dispersed in oxide film, and even form an HfSiO_4 interlayer. It has been proved that the HfSiO_4 and ZrSiO_4 particles have similar effects. On the one hand, they can promote the healing of cracks and holes in borosilicate scale [99, 100], on the other hand, they can both act as barriers and HfSiO_4 particles can also increase the crystallization temperature of amorphous scale [101]. Therefore, adding HfB_2 can enhance the alloy antioxidant effects. The research results of Sciti et al. also confirmed this conclusion [97].

Table 2. The elemental composition of various samples. Reproduced with permission [96]. Copyright 2019 Elsevier.

Samples	Composition (at.%)			
	Mo	Hf	Si	B
X0	35.0	-	60.0	5.0
X34_1	23.2	11.3	39.7	25.8
X34_2				

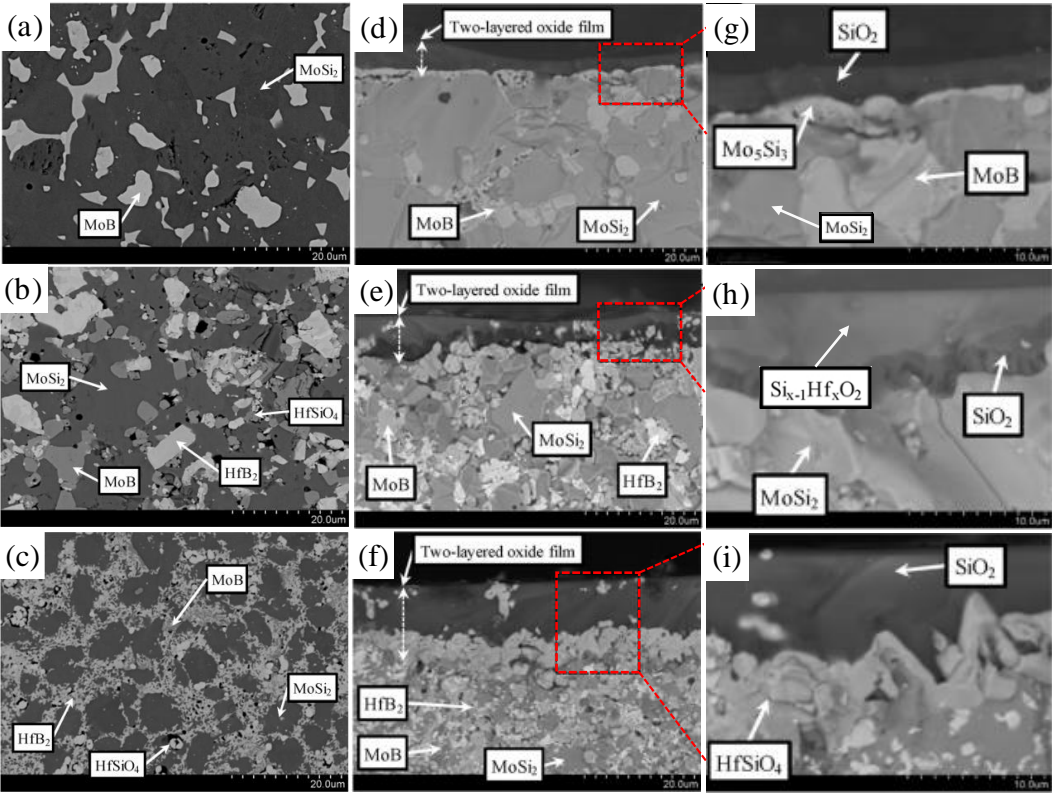


Fig. 3. Microstructure of various samples: (a) X0, (b) X34₁ and (c) X34₂; Cross-sectional SEM images of samples after oxidation at 1200 °C for 30 h: (d, g) X0, (e, h) X34₁ and (f, i) X34₂. Reproduced with permission [96]. Copyright 2019 Elsevier.

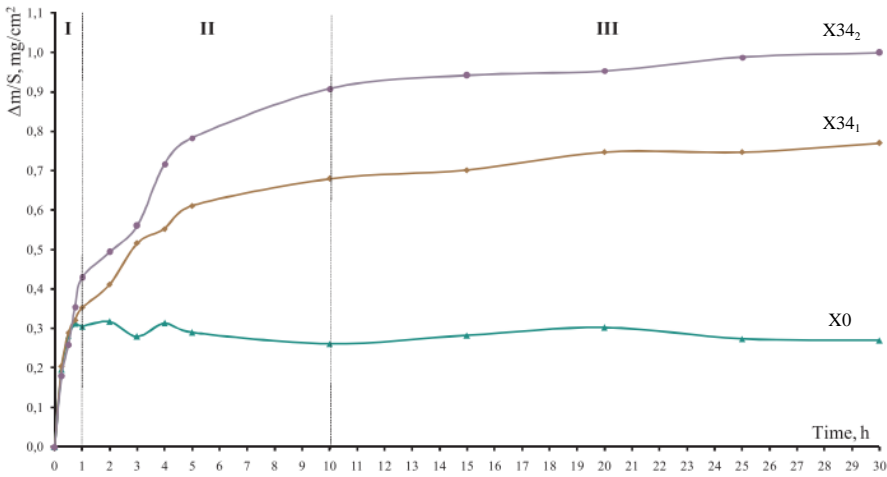


Fig. 4. Kinetic curves of the samples oxidized at 1200 °C. Reproduced with permission [96]. Copyright 2019 Elsevier.

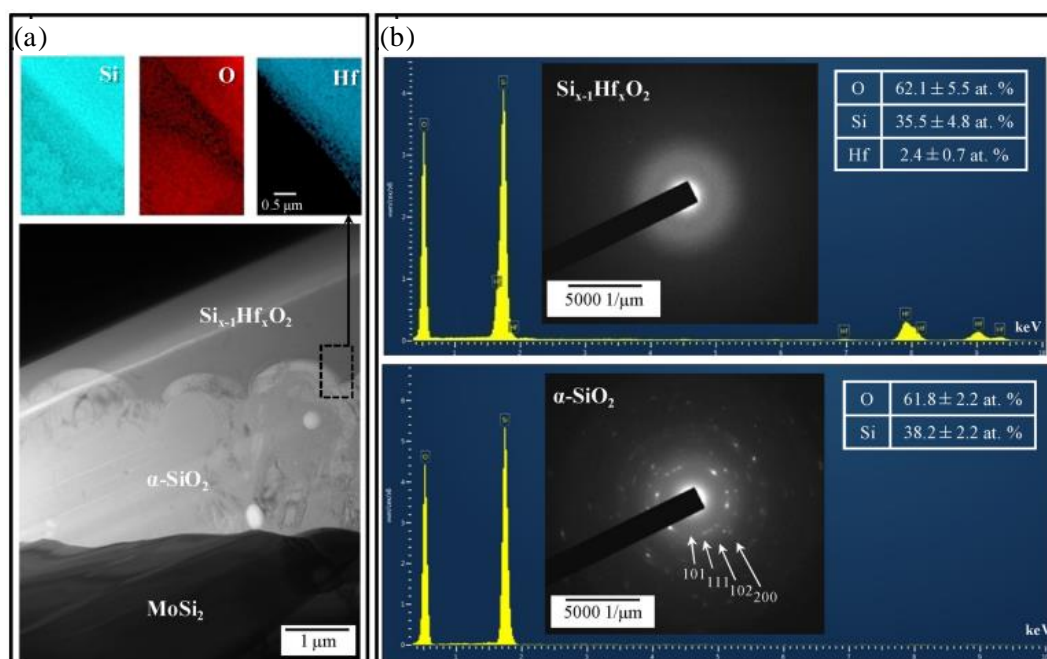


Fig. 5. (a) TEM micrograph of the oxidized X34₁ sample; (b) EDS and diffraction patterns of the oxide scale.

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2.1.3 Effects of Ce element addition

Actions of Ce on oxidation performance of Mo-Si-B alloys have been investigated deeply. Das et al. [102, 103] performed isothermal oxidation experiments about Mo-14Si-10B and Mo-13.98Si-9.98B-0.16Ce alloys (at.%, abbreviated as MSB₁ and MSBCe, respectively) synthesized by arc melting. The experiment results have suggested that doping a small amount of Ce has little effect on oxidation kinetics of the alloy at 500 °C and 700 °C (Fig. 6 (a)), while the presence of Ce presents a palpable effect on the alloy oxidation behavior at 900-1300 °C. Figs. 6 (b-d) provide the mass variation curves of MSB₁ and MSBCe oxidized at 900-1300 °C, respectively. It can be seen that the mass loss of MSB₁ increases after the addition of Ce (Figs. 6 (c, d)). Even so, MSBCe exhibits shorter transient oxidation periods as compared to MSB₁, and its steady-state stage curve almost presents a horizontal trend, revealing that MSBCe is more effectively protected than MSB₁.

At the same time, the microstructural morphology of two oxidized alloys is shown in Fig. 7. During oxidation at 900 °C, one-layered oxide film (i.e. Mo-oxide film) and flowing glassy phase are observed on the surface of both alloys (Figs. 7 (a, d)). The difference is that the glassy phase in MSBCe flows faster due to the presence of Ce, hence MSBCe presents a smaller mass variation at 900 °C (Fig. 6 (b)). However, after oxidation at 1100 °C for 24 h, the oxide-layer structure of both alloys has changed distinctly, namely SiO₂ layer is formed on top of the Mo-oxide layer (Figs. 7 (b, e)). When the oxidation temperature reaches 1300 °C, B₂O₃ has begun to evaporate from the oxide scale, resulting in increased viscosity and weak fluidity of the scale, which leads

to the deterioration of alloy antioxidation ability [104-108]. Nevertheless, the addition of Ce increases the volatilization temperature of B_2O_3 , leaving the fluidity of oxide scale largely unchanged. So the flow traces of glassy scale can still be observed on the MSBCe surface even at 1300 °C; in contrast, the MSB₁ surface has little flow traces (Figs. 7 (c, f)). There is no doubt that scale flow can heal pores and cracks on the alloy surface, thus Ce addition has a positive effect on the oxidation protection of MSB₁.

In addition, Das et al. [109] also researched the oxidation reaction of Mo-Si-B-Al-Ce alloys at 1100 °C. It is well known that adding Al to Mo-Si-B systems may lead to the failure of alloy oxidation protection owing to the formation of mullite [110-114], which is also verified by the mass loss curves of Al-doped alloy in Figs. 6 (b-d). Das [109] noted that adding Ce can further inhibit the malignant oxidation of Mo-Si-B-Al systems at 1100 °C, since the presence of Ce hindered the generation of mullite and promoted the formation of dense protective Al-oxide films on the alloy surface, thereby improving the antioxidant capacity.

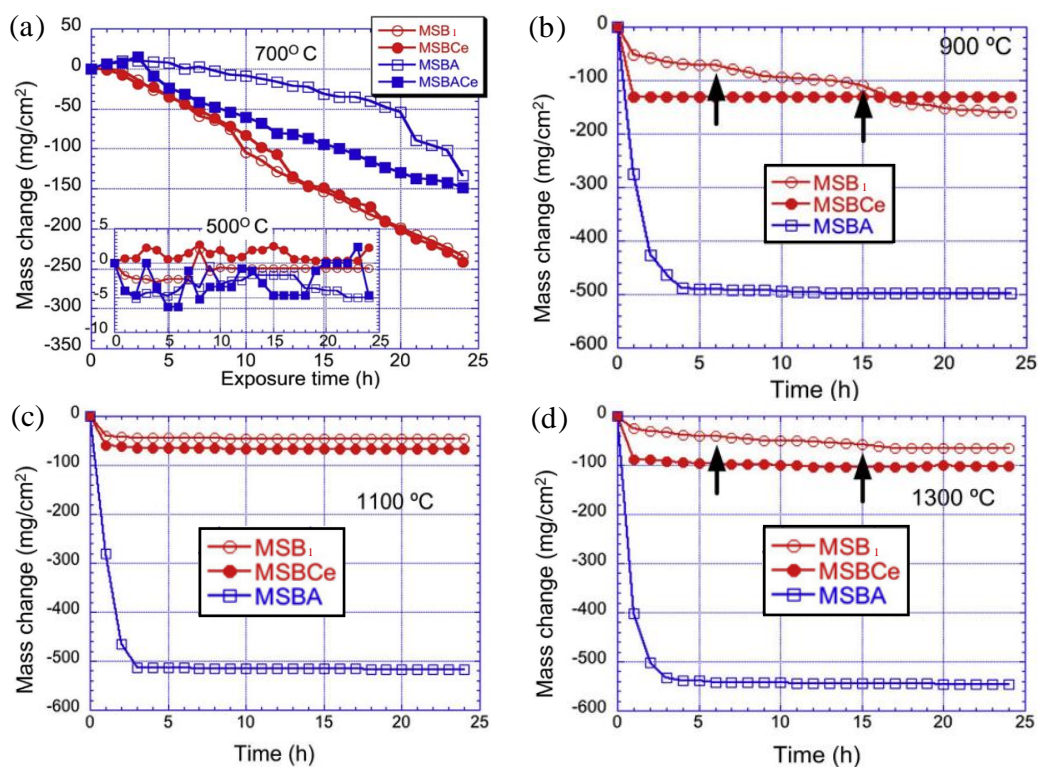


Fig. 6. Mass loss curves of various alloys at different temperatures. (a) and (b-d) reproduced with permissions [102] and [103], respectively. Copyright 2010 Elsevier and 2016 Elsevier.

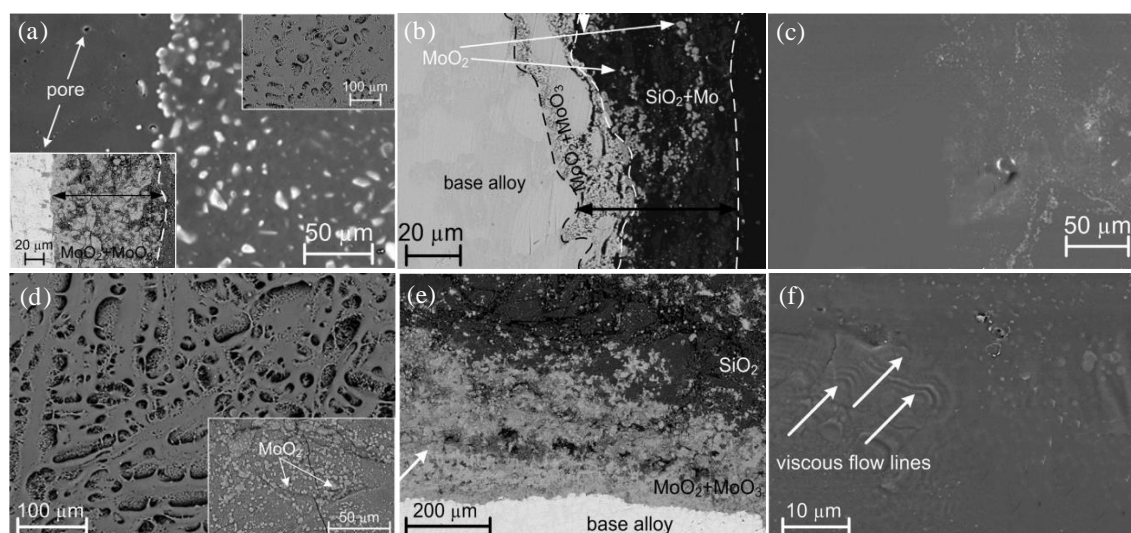


Fig. 7. Surface and cross-sectional SEM (BSE) images of MSB₁ (a-c) and MSBCe (d-f) alloys oxidized at different temperatures for 24 h: 900 °C (a, d), 1100 °C (b, e) and 1300 °C (c, f). Reproduced with permission [103]. Copyright 2016 Elsevier.

2.1.4 Effects of Y element addition

It has been sure that adding Y element can significantly prolong the service life of Mo-based alloys due to the fact that Y will exhibit higher oxygen affinity than Mo [115, 116]. Moreover, Y can also inhibit ion diffusion in grain boundaries and decrease the oxide-scale growth rate [117-119]. The presence of Y improves the adhesion between oxide film and substrate, thus improving the alloyed oxidation resistance [120, 121]. Therefore, researchers try to add Y element to Mo-Si-B alloys to obtain materials with better performance. After comparing the variation of Y-doped and Y-free Mo-9Si-8B (at.%) samples in the oxidation behavior at 650-1400 °C, Majumdar et al. [122-124] found that all samples presented a trend of transient mass increase followed by continuous rapid decrease at 650 °C (Fig. 8 (a)). Among all the samples, the 2at.% Y-doped sample had the minimum mass loss at 750-1000 °C (Figs. 8 (b-e)), and the 0.2at.% Y-doped sample presented the lowest mass loss at 1100 °C (Fig. 8 (f)). This is because the addition of Y can produce stable Y₆MoO₁₂ and Y₅Mo₂O₁₂ oxides at the initial period of oxidation, which can inhibit the generation and vaporization of MoO₃, leading to a decrease in the mass loss of the samples containing Y.

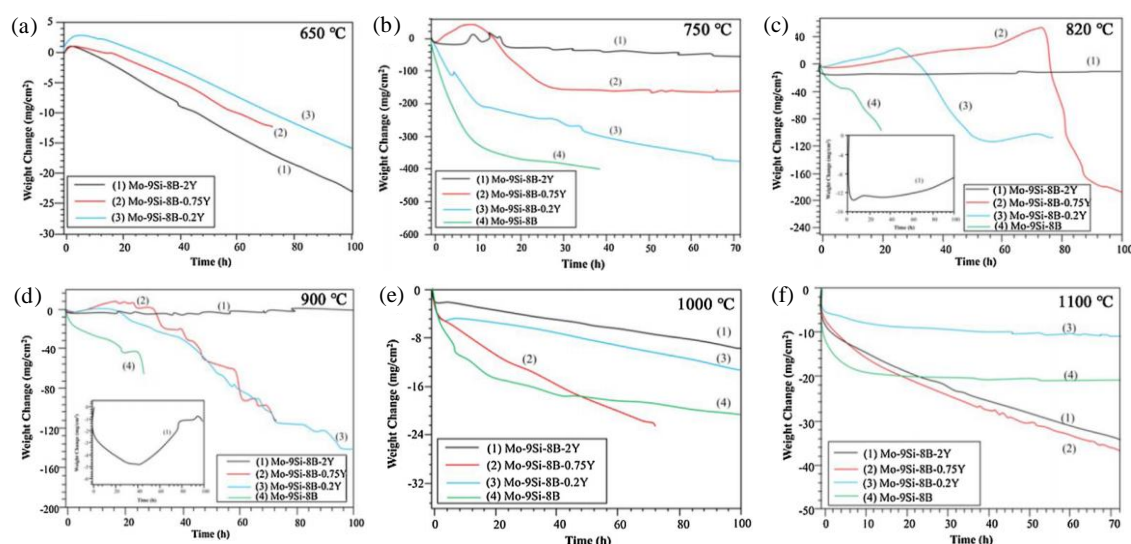


Fig. 8. Weight change curves of Y-doped and undoped samples oxidized at 650 °C-1100 °C, respectively. Reproduced with permission [122]. Copyright 2014 Elsevier.

In order to further clarify the role of Y doping on the sample antioxidation behavior, the microstructure of oxidized samples are analyzed in detail [58, 122]. Figs. 9 (a, b) give the cross-sectional micrographs of 2at.% and 0.75at.% Y-doped samples oxidized at 900 °C for 24 h, respectively. It can be seen that 0.75at.% Y-doped sample has a thicker inner MoO₂ scale than that of 2at.% Y-doped sample, which indicates that properly increasing the concentration of Y can inhibit the formation of MoO₂ to some extent. Furthermore, microcracks are also observed on the outer SiO₂ scale of 0.75at.% Y-doped sample due to the quite high growth velocity (about 7.6 μm·h⁻¹) of inner MoO₂ scale, which produces such a large tensile stress that outer SiO₂ scale breaks (Figs. 9 (b, d)). It is worth noting that the thickness of inner MoO₂ scale for 0.75at.% Y-doped sample is significantly thinner after oxidation at 1100 °C for 72 h (Fig. 9 (c)). Because SiO₂ will present viscous flow to cover holes and cracks on the alloy surface during the temperature exceeds 965 °C [125, 126], which prevents further oxidation of the substrate. Fig. 9 (e) shows the thickness changes of SiO₂ and MoO₂ layers for 0.2at.% Y-doped sample at 1100 °C and 1200 °C, which further supports the above analysis results. When the oxidation temperature is higher than 1200 °C, a thin yttrium-silicate (Y₂Si₂O₇) scale is observed on the outer surface of Y-doped samples (Figs. 10 (a-d)), and the thickness of yttrium-silicate film gradually increases as the oxidation temperature raises (Figs. 10 (c, e)) [123]. It has been proved that the outer yttrium-silicate film is conducive to the alloy oxidation protection through preventing SiO₂ from forming volatile silicon hydroxide in humid conditions above 1200 °C [127-129]. Similar studies have been reported by Gorr et al. [130]

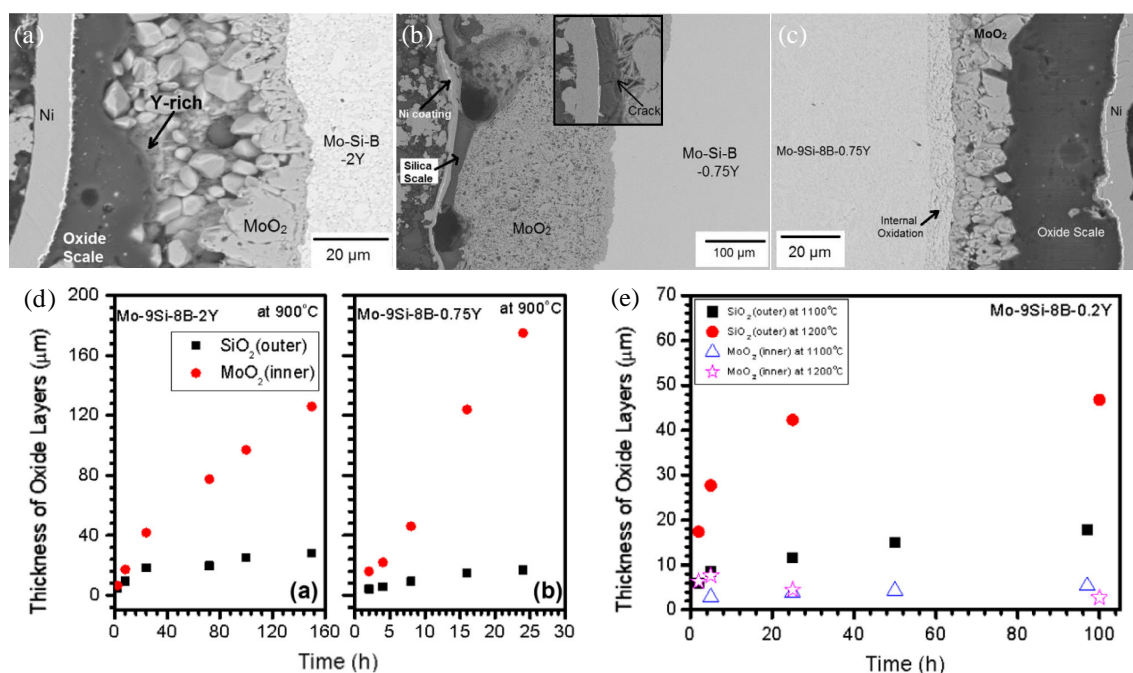


Fig. 9. Cross-sectional BSE images of 2at.% and 0.75at.% Y-doped samples oxidized at different conditions: (a, b) 900 °C for 24 h, (c) 1100 °C for 72 h; (d, e) Changes of MoO₃ layer and SiO₂ layer thickness in Y-doped samples oxidized at different temperatures. Reproduced with permission [122]. Copyright 2014 Elsevier.

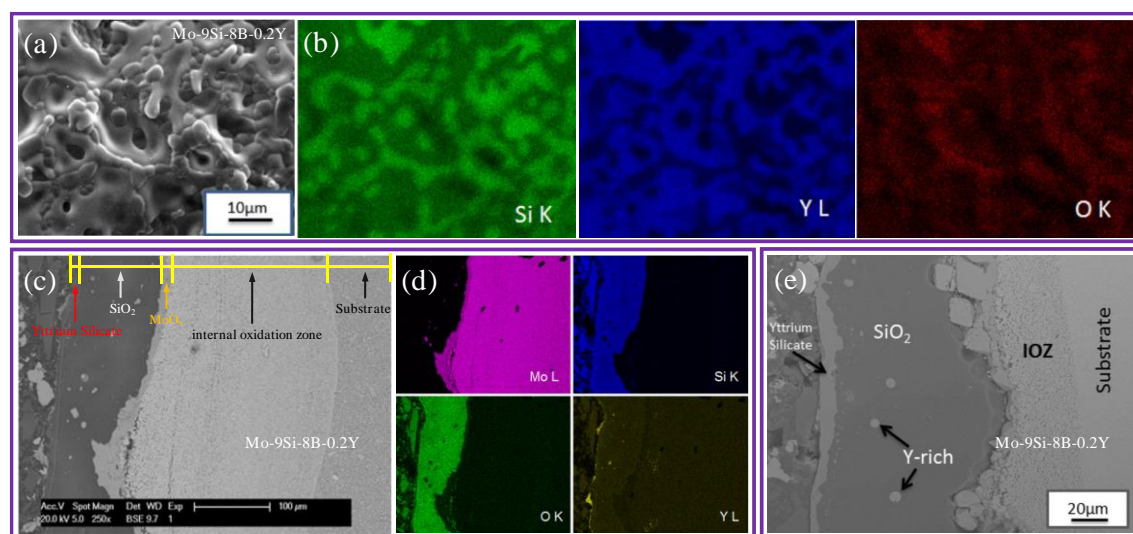


Fig. 10. Surface SE (a) and cross-sectional BSE (c, e) images of 0.2at.% Y-doped samples oxidized at different conditions: 1300 °C for 100 h (a, c) and 1400 °C for 2 h (e); (b, d) are the element mappings of (a, c), respectively.

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There is no doubt that alloying with Zr has a great impact on the antioxidant ability of Mo-Si-B materials. This is because the addition of Zr may produce polymorphic ZrO₂ or monomorphic ZrSiO₄, which mainly depends on the oxidation temperature. Among them, the ZrSiO₄ can act as an obstacle phase, which is

beneficial to improve the alloy oxidation behavior; whereas the ZrO_2 will expand in volume at high temperatures ($>1200^\circ\text{C}$), which destroys the integrity of SiO_2 scale so that it loses the protective effect [131, 132]. Therefore, inhibiting the formation of ZrO_2 phase is essential to improve the alloy oxidation resistance.

Based on the fact that yttria suppresses the zirconia phase transition [133, 134], Yang et al. [135] designed and fabricated Mo-12Si-10B-1Zr-0.3Y, Mo-12Si-10B-1Zr and Mo-12Si-10B samples (at.%, abbreviated as 1Zr-0.3Y, 1Zr-0Y and 0Zr-0Y, respectively). Fig. 11 (a) shows the mass variation of the three samples at 1250°C , it can be seen that adding 1at.% Zr to the 0Zr-0Y sample will lead to continuous and sharp mass loss. Because the 0Zr-0Y sample has formed dense protective SiO_2 films during the oxidation, which avoids the sample sustained mass loss (Fig. 12 (a)), whereas the addition of Zr causes the SiO_2 scale to become loose and porous due to the formation of ZrO_2 , and the porous structure provides channels for O_2 inward diffusion, thus accelerating the sample oxidation (Fig. 12 (b)). It is encouraging that further adding 0.3at.% Y can effectively prevent the rapid mass loss of 1Zr-0Y sample. As can be seen from Fig. 12 (c), ZrSiO_4 rather than ZrO_2 appears on the sample surface after the addition of Y, thus eliminating the adverse effect of Zr doping. Meantime, the Y-Mo-rich oxide is also observed around ZrSiO_4 phase. EDS analysis shows that the Y/Mo atomic ratio of this oxide is about 1/2, revealing that the oxide may be $\text{Y}_2\text{Mo}_4\text{O}_{15}$. Again, the XPS spectra also presents that the oxide has nearly the same Mo 3d and Y 3d bonding energies as $\text{Y}_2\text{Mo}_4\text{O}_{15}$ gauged through You et al. [136], which further verifies the above inference (Figs. 11 (b, c)). What's more, the 1Zr-0.3Y sample surface also forms a uniformly dense outer $\text{Y}_2\text{Si}_2\text{O}_7$ scale with the increase of oxidation time, which provides a better protection effect than 0Zr-0Y sample (Fig. 12 (d)). It has been observed from the cross-section enlarged Figs. 12 (e, f) that Y diffuses outward with the metastable $\text{Y}_2\text{Mo}_4\text{O}_{15}$ as the carrier and produces $\text{Y}_2\text{Si}_2\text{O}_7$ after a series of reactions at the top of SiO_2 scale, which will be accumulated and compressed to form the outer $\text{Y}_2\text{Si}_2\text{O}_7$ layer. Therefore, 1Zr-0.3Y sample presents the best antioxidant performance among the three samples.

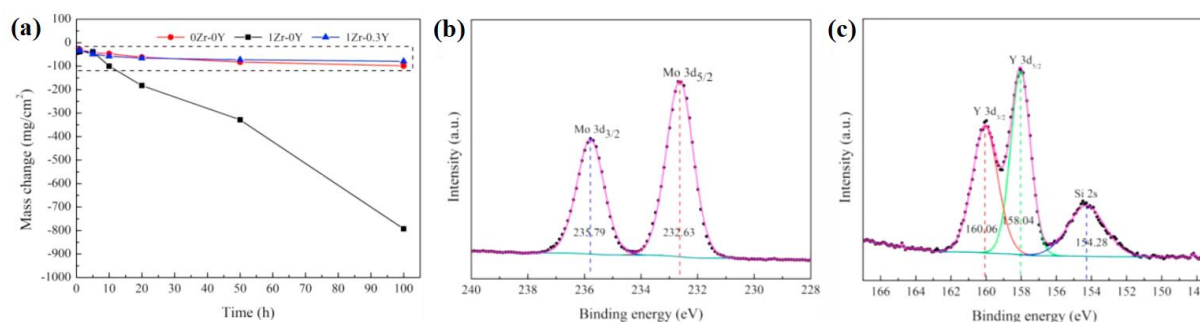


Fig. 11. Mass change curves of three studied samples at 1250°C (a); XPS analysis of Mo (b) and Y (c) characterized the 1Zr-0.3Y sample surface oxidized for 1 h. Reproduced with permission [135]. Copyright 2020 Elsevier.

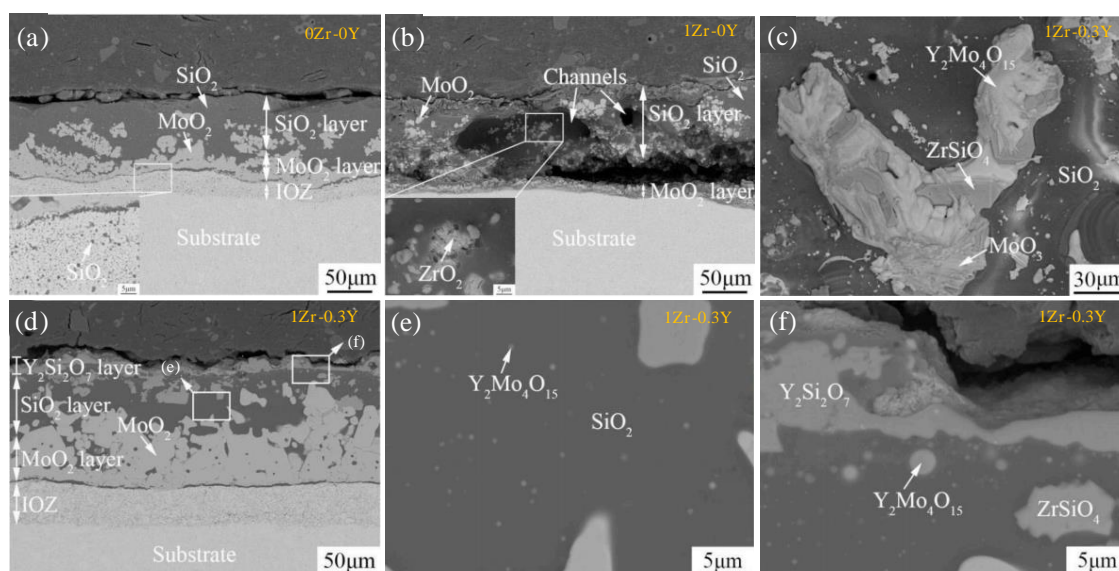


Fig. 12. Cross-sectional and surface BSE images of three studied samples oxidized at 1250 °C for different time: (a) 20 h, (b) 5 h, (c) 1 h, (d-f) 50 h. Reproduced with permission [135]. Copyright 2020 Elsevier.

2.2 Effects of rare earth elements on oxidation behavior of other Mo-Si alloys

Previous studies have pointed out that adding Nb to the Mo-Si-based materials can play a satisfactory effect in improving mechanical properties due to damaging the stability of Mo_3Si phase [137-139]. However, the presence of Nb will lead to catastrophic oxidation of the material [140-142]. Inspired by the above study that adding Y can enhance the antioxidant properties of Zr-doped Mo-Si-B alloys, we further discussed the role of adding Y on the oxidation behavior of Nb-doped Mo-Si alloys.

Majumdar [143] used the nonconsumable arc-melted method to prepare the undoped and 0.5Y-doped Mo-26Nb-19Si samples (at.%), which simply referred to as Alloy1 and Alloy2, respectively. The microstructures of both samples are shown in Figs. 13 (a, e). It can be found that they are both composed of dark and bright areas. According to XRD analysis (Fig. 14 (a)) and EBSD mappings (Figs.13 (b-d, f-h)), the dark and bright areas are $(\text{Mo, Nb})_5\text{Si}_3$ and $(\text{Mo, Nb})_{ss}$ phases, respectively. Moreover, Y_2O_3 particles are also observed on the Alloy2 grain boundaries. These particles can suppress the elongated grain growths, which results in the difference of microstructure morphology between the two samples. Meanwhile, Majumdar [143] also studied the oxidation process of Alloy2 at 1000 °C and 1300 °C. It is established that the sample exhibits continuous linear mass loss when oxidized at 1000 °C. When the oxidation temperature increases to 1300 °C, the sample is oxidized more vigorously and loses its antioxidant capacity within 2 h of oxidation, as shown in Fig. 14 (b). Fig. 15 shows the cross-section and surface micrographs of the oxidized sample. It can be discovered that the Alloy2 surface has formed a thick oxide layer after oxidation at 1000 °C for 24h (Fig. 15 (a)). As can be seen

from Fig. 15 (d), the oxide layer is mainly composed of MoO_3 , Nb_2O_5 and SiO_2 , wherein Nb_2O_5 can act as a channel for O_2 internal diffusion due to the lack of protective action, which leads to rapid oxidation of the sample. In addition, the sample surface oxide film, which consists of Y_2O_3 , Nb_2O_5 and SiO_2 , appears numerous cracks and holes during oxidation at 1300 °C for 2 h (Figs. 15 (b, c)), resulting in the loss of protection from oxidation. Therefore, adding Y to Mo-Si-Nb alloys cannot overcome the oxidizing problem.

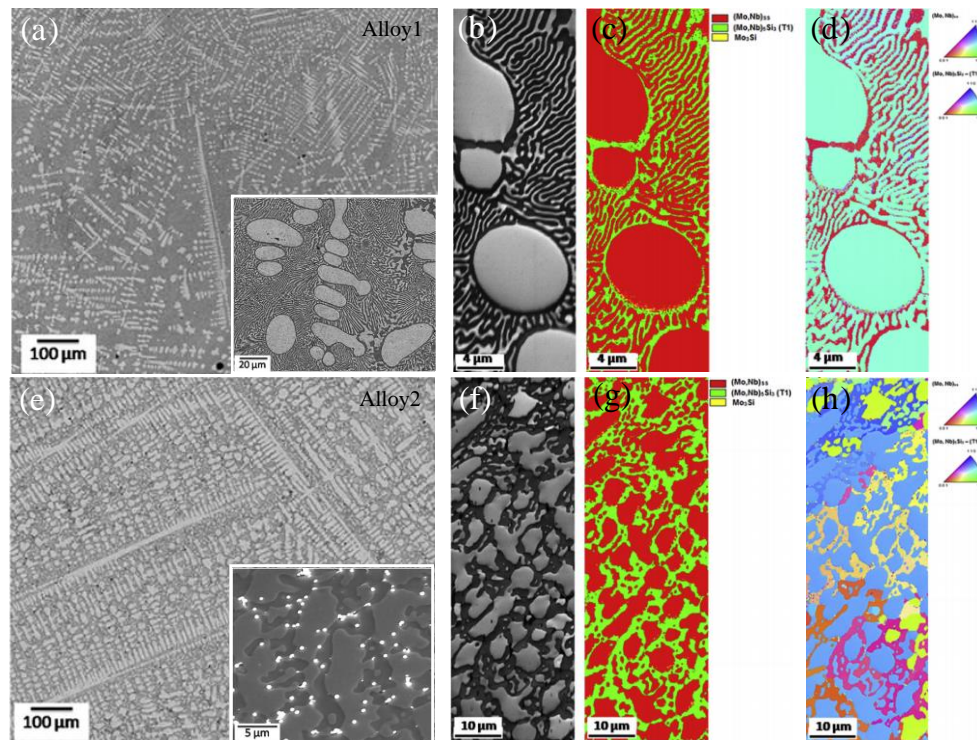


Fig. 13. Microstructure morphologies of Alloy1 (a) and Alloy2 (e); EBSD mappings of Alloy1 and Alloy2: band contrast (b, f), phase (c, g), IPF (d, h). Reproduced with permission [143]. Copyright 2018 Elsevier.

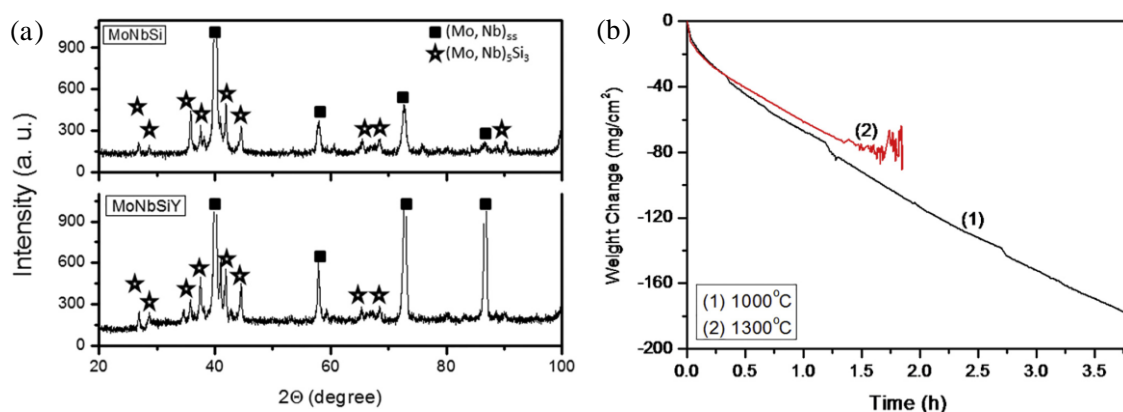


Fig. 14. (a) XRD patterns of both samples before the oxidation; (b) Weight change curves of Alloy2 oxidized at 1000 °C and 1300 °C, respectively. Reproduced with permission [143]. Copyright 2018 Elsevier.

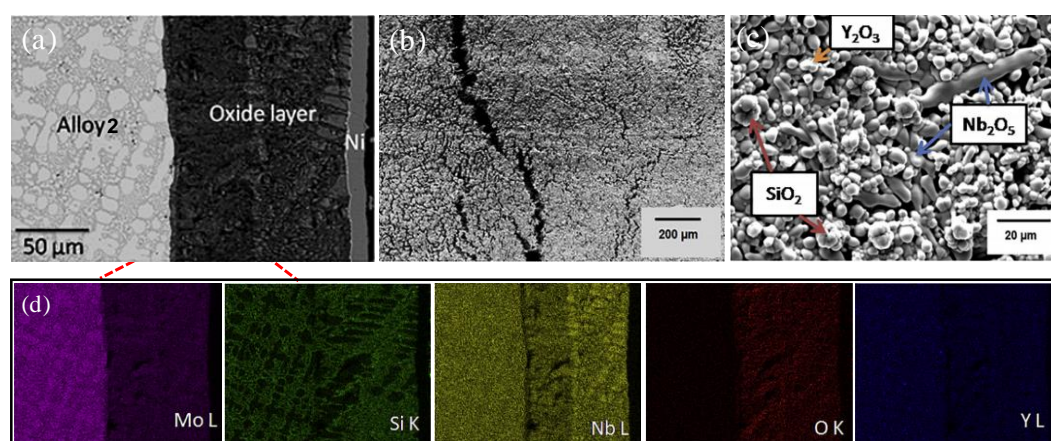


Fig. 15. Cross-section BSE micrograph of Alloy2 (a) with the corresponding EDS mappings (d) oxidized at 1000 °C for 24 h; Surface SEM/SE images of low (b) and high magnifications (c) for Alloy2 oxidized at 1300 °C for 2 h.

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3. Strengthening mechanism of rare earth elements

According to the above research, it can be determined that the improvement of oxidation behavior of Mo-Si-based alloys by rare earth elements is mainly achieved through the following three ways. First, optimizing the microstructure of the alloy is caused by refining grains or distributing phase compositions uniformly, which contributes to the rapid formation of oxide scale [80, 96, 103, 144]. Second, producing stable rare earth oxides, these oxides are dispersed in scale and act as obstacle phases or diffusion barriers, which is conducive to suppressing the MoO₃ volatilization and O₂ inward diffusion [59, 122, 123]. Third, forming an additional rare earth oxide layer, thus further improving the antioxidant capacity [96, 135].

Fig. 16 shows a schematic diagram of the oxidation process for rare earth element doped and undoped Mo-Si-based alloys at medium-high temperatures, which is helpful to further understand the strengthening mechanism of rare earth elements. It can be seen that the alloy with finer grain size can be prepared after adding rare earth elements like La, which will affect the oxidation behavior to some extent. Overall, the oxidation process of the two kinds of alloys can be divided into two stages: initial and stable oxidation stages. During the initial oxidation stage, a discontinuous SiO₂ scale is formed on the surface of alloy without rare earth doping, which cannot effectively isolate oxygen. As a result, the alloy is oxidized violently and forms a Mo-oxide (MoO₂ and MoO₃) layer below the SiO₂ scale. Among them, MoO₃ is highly volatile, which leads to a severe mass loss of the alloy and leaves some holes and cavities on the surface [145]. Fortunately, SiO₂ gradually increases and flows to heal these holes and cavities as the oxidation time increases, thus facilitating the formation of continuous SiO₂ scale [146]. During the stable oxidation stage, the complete scale can provide

sufficient protection for the substrate due to the effective restriction of O_2 internal diffusion, resulting in the reduction of oxygen pressure inside the alloy. Obviously, low oxygen partial pressure inhibits the continuous generation of MoO_2 , and the original MoO_2 will continue to oxidize to produce MoO_3 and slowly volatilize so that the Mo-oxide interlayer becomes thinner [147]. Meanwhile, the substrate below MoO_2 layer has been oxidized selectively, leading to the emergence of internal oxidation zone [122], as shown in Fig. 16 (a). In contrast, the alloy doped with rare earth can generate rare earth oxides such as La_2O_3 , Y_6MoO_{12} , $Y_5Mo_2O_{12}$, etc. in the initial oxidation stage. These stable oxides, on the one hand, promote the formation of continuous SiO_2 scale. On the other hand, they fill holes in the scale to eliminate the shortcut of O_2 inward diffusion and MoO_3 volatilization, so that the alloy can enter the stable oxidation stage faster. In addition, a double-layer protective oxide film (i.e. $Y_2Si_2O_7-SiO_2$ or $SiO_2-HfSiO_4$ duplex scales) is formed on the alloy surface during the stable oxidation stage, providing more effective protection against oxidation, as shown in Fig. 16 (b).

However, it is disappointing that sometimes the addition of rare earth elements may even lead to the deterioration of alloy oxidation behavior in high temperature environments. For example, adding La to the Mo-Si-B system above 1100 °C has led to its accelerated oxidation attribute to the formation of large amounts of cracks and holes [60]. Therefore, the challenges ahead remain severe.

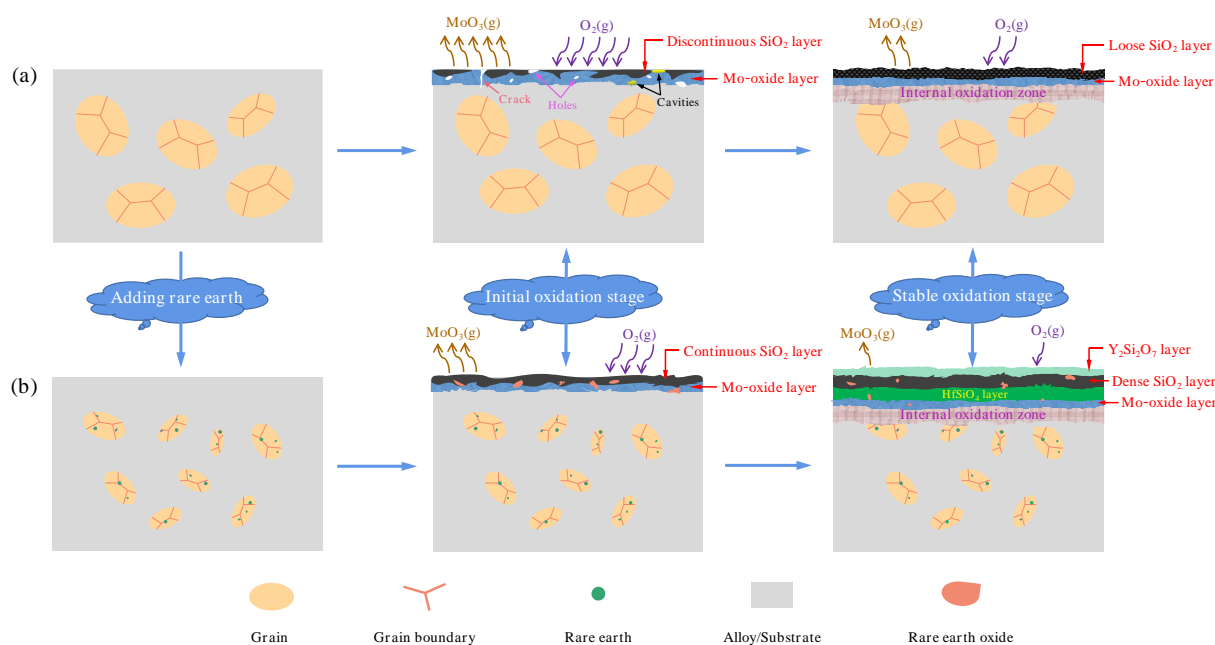


Fig. 16. Schematic diagram of the oxidation process for Mo-Si-based alloy at medium-high temperature: (a) without rare earth elements, (b) doped with rare earth elements.

4. Conclusion and outlook

This paper comprehensively reviewed the role of rare earth elements on the oxidation behavior of Mo-Si-based alloy. Based on the thorough study about the oxidation process of Mo-Si-based alloy, the strengthening mechanism of various rare earth elements such as La, Hf, Ce and Y was summarized. The addition of La to Mo-Si-B alloys can make grains become finer to promote the rapid formation of continuous boroilicate scales; Meantime, producing stable La-containing oxides with a "pinning" effect like $3\text{La}_2\text{O}_3 \cdot \text{MoO}_3$, La_2O_3 and $\text{La}_2\text{O}_3 \cdot 3\text{MoO}_3$, which limits the formation and evaporation of MoO_3 , thus significantly enhancing the alloy oxidation resistance at temperatures below 1000 °C. Mo-Si-B alloys doped with Hf can generate HfSiO_4 particles that promote the healing of holes and cracks in oxide scales; Besides, it is likely that HfSiO_4 inner layer will be formed to inhibit the MoO_3 volatilization and O_2 inward diffusion. These are helpful to improve the antioxidant capacity of the alloy. Alloying with Ce can shorten the transient oxidation period of Mo-Si-B alloys; Moreover, the presence of Ce can also raise the volatilization temperature of B_2O_3 in oxide films, which plays a positive role in maintaining the viscosity and integrity of borosilicate scale. It is worth mentioning that the addition of Ce to the Mo-Si-B-Al system can also hinder the formation of mullite and promote the emergence of protective Al-oxide scale, thus providing more effective protection against oxidation. Adding Y can passivate Mo-Si-B alloys to prevent further oxidation. On the one hand, the existence of Y will produce stable Y-Mo-rich oxides ($\text{Y}_6\text{MoO}_{12}$, $\text{Y}_5\text{Mo}_2\text{O}_{12}$, etc.), which suppresses the formation of MoO_3 and accelerates the nucleation and growth of protective SiO_2 scales; On the other hand, a $\text{Y}_2\text{Si}_2\text{O}_7$ outer layer is created which acts as a diffusion barrier. Furthermore, adding Y to the Mo-Si-B-Zr system also suppresses ZrO_2 formation, thus eliminating the adverse effect of Zr doping on oxidation behavior.

However, it is noteworthy that adding rare earth elements do not always improve the antioxidation ability of Mo-Si-based alloys in practice. For example, adding Y to the Mo-Si-Nb system cannot prevent its catastrophic oxidation; The addition of La also causes deterioration in the oxidation behavior of Mo-Si-B alloys above 1000 °C. Therefore, a further search for other oxidation protection methods is necessary. Some research schemes worth exploring in the future are listed below, hoping to help solve the problems encountered in the practical application of Mo-Si-based alloys. Before using, preoxidation treatment at an appropriate temperature can obtain protective silica scales on the alloy surface, thus effectively inhibiting the inward diffusion of O_2 and obviously extending the service life of alloy. Processing of preceramic polymers is a very promising method owing to its simple operation and low cost. Preceramic polymers, which mainly includes many silicon-containing ceramic precursors like polysilazanes, polycarbosilanes and polysiloxanes, can be decomposed into

SiC, SiCN, SiCO or Si₃N₄ to form protective silica scales at high temperature environments, thereby exhibiting the excellent antioxidant properties. Considering the type, concentration, chemical activity, selective oxidation and other factors of elements in the alloy, relevant numerical simulation or mathematical model is established to quantitatively study the relationship between oxidation behavior and microstructure. Combined with the emerging coating technology to design a suitable silicide-based coating such as MoSi₂ ceramic coating for the Mo-Si-based system, which will help to provide a superior antioxidant capability.

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The manuscript was written through the contributions of all authors. Y.Y. Zhang: Conceptualization, Investigation, and Supervision. Y.Y. Zhang and L.H. Yu: Writing original draft and image processing. L.H. Yu, T. Fu, K.K. Cui, and F.Q. Shen: Validation, Resources, Investigation, Writing- review & editing. Y.Y. Zhang, L.H. Yu and J. wang: Visualization, Writing - review & editing. All authors have given approval to the final version of the manuscript.

Notes

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