Hot corrosion behavior of Yb2Si2O7 ceramic by NaVO3 at 500 °C to 900 °C

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ABSTRACT

The hot corrosion behavior of Yb2Si2O7 bulk exposed to NaVO3 was investigated at 500–900 °C. Results indicated that the hot corrosion reaction could occur at 500 °C, the corrosion products were composed of YbVO4 and SiO2 at 500–900 °C. The content of YbVO4 and SiO2 in the corrosion product at 500 °C was small and the crystal size of YbVO4 was fine. As temperature increased, a continuous and dense corrosion layer could be formed on the bulk surface, which prevented the hot corrosion reaction from occurring dramatically. The hot corrosion reaction products mainly consisted of rod-shaped YbVO4 beyond 600 °C. Since the hot corrosion reaction driving force increased with temperature, the rod-like YbVO4 crystals tended to go from slender to plump; moreover, both ends changed from irregular lamination to regular quadrangular pyramid shape; also SiO2 crystals partly changed from flocculent to ellipsoid shape. The hot corrosion mechanisms of Yb2Si2O7 bulk exposed to NaVO3 at 500–900 °C were investigated based on the corrosion products.

1. Introduction

Environmental barrier coatings (EBCs) are widely used in gas turbines because they improve the service life of structural components of Si-based ceramic materials, such as SiC and Si3N4 at high temperatures [1–3]. Under the condition of dry oxygen, SiC and Si3N4 materials will be oxidized to form a protective layer of SiO2, which makes SiC and Si3N4 materials show a better oxidation resistance [4]. However, a large amount of water vapor in the gas would react with the SiO2 protective layer to form the gaseous product of Si(OH)4 at high temperatures, which results in the performance degradation of SiC and Si3N4 [3]. Rare-earth silicates are the third generation of EBC materials developed based on mullite and BSAS (BaO-SrO-Al2O3-SiO2) ceramics [1,5,6]. The thermal expansion coefficient of Yb2Si2O7 matches well with those of the silicon-based ceramics; moreover, Yb2Si2O7 has excellent phase stability and corrosion resistance to water-oxygen [7,8]. However, under the gas condition of turbine engines, a small number of impure elements in fuel, such as Na, S, V, and Cl, will be deposited on the substrate or EBCs of high-temperature components, and NaVO3, VO2, and Na2SO4 compounds are formed, leading to the molten salt corrosion of rare earth silicates [9–11]. The corrosion reactions of NaVO3 could cause the degradation of EBCs materials, which affects the barrier function of EBCs and reduces the service life of EBCs. Therefore, it is essential to investigate the hot corrosion behavior between the EBCs and NaVO3. Up to now, many studies have been conducted on thermal shock performance [12], calcia-magnesia-alumino-silicate (C MAS) corrosion [13,14] and water-oxygen corrosion [15,16] for Yb2Si2O7. Recently, Fan et al. [17] investigated the hot corrosion behavior of Y2Si2O7 in the molten salt of Na2SO4 and V2O5. Also, the hot corrosion behavior of Yb2Si2O7 bulk under NaVO3 salt at 1000–1500 °C was studied in our group [18]. Although the working temperature of the Yb2Si2O7 used as EBCs can exceed 1000 °C, the operating temperatures of turbine engine blades at different positions are different. Furthermore, the temperature will increase and decrease during start-up and shutdown. The melting point of NaVO3 is 630 °C. Therefore, the hot corrosion tests of Yb2Si2O7 bulk exposed to NaVO3 salt were conducted isothermally at 500–900 °C for 2 h in air in this study, and the hot corrosion behavior was investigated.

2. Experimental procedure

Yb2Si2O7 powders were synthesized by solid-state reaction using commercial powders of Yb2O3 and SiO2 (all purities >99.9%) according to Equation (1) as follows:

\[ \text{Yb}_2\text{O}_3 + 2\text{SiO}_2 \rightarrow \text{Yb}_2\text{Si}_2\text{O}_7 \]  

Yb2Si2O7 powders were uniaxially pressed into compacts with a pressure of 20 MPa for 5 min, and the compacts were pressureless sintered at 1500 °C for 12 h.
in air. The Yb$_2$Si$_2$O$_7$ bulks for hot corrosion tests were obtained with a dimension of 12.2 mm × 12.2 mm × 4.5 mm, and their relative densities were approximately 91%. Figure 1 shows the surface morphology of Yb$_2$Si$_2$O$_7$ bulk. It exhibits a relatively smooth surface. Equiaxial grains can be clearly observed on the surface. At the same time, there are only a small number of pores at the grain boundary.

NaVO$_3$ salt was uniformly coated on the surface of Yb$_2$Si$_2$O$_7$ bulk at a concentration of 20 mg/cm$^2$, and then the as-coated specimens were heated in the electric furnace at different temperatures. The specimens were removed from the electric furnace and naturally cooled down to room temperature.

After the hot corrosion tests, the samples were washed by using deionized water, removing the unreacted corrosives, and the corrosion surface of samples was white. Before and after the corrosion tests, the samples’ weight was measured by digital balance with an accuracy of 0.1 mg. Phase composition and microstructure of the samples before and after the hot corrosion tests were characterized by X-ray diffraction (XRD, Bruker D8 Advance Diffractometer, Cu Kα radiation) at a scanning rate of 6°/min and scanning electron microscopy (SEM, JSM-5310, Japan) equipped with an energy-dispersive X-ray spectrometer (EDS, Link-ISIS, England), respectively. A thin gold film was deposited on the surface of the samples for the electrical conductivity of SEM and macrographs analysis. Macrographs were conducted using an inverted metallurgical microscope (GX71).

3. Results

According to the pretest, NaVO$_3$ had no corrosive effect on Yb$_2$Si$_2$O$_7$ bulk below 480°C. Figures 2 and 3 show the macrographs and metallographic photographs of the samples before and after the corrosion tests at 500–900°C, respectively. As compared with Figure 2(a,b), it was obviously observed that there were some traces of interaction in the middle of the sample [Figure 2(b)] after the corrosion test at 500°C. Corrosives did not diffuse to the edge of the sample owing to the low test temperature. Microstructure analyses showed that no corrosion products were found in the lighter color area near the edge of the sample (see Figure 3(a)). In contrast, the corrosion products were found in the darker color area in the middle surface of the sample (see Figure 3(b)). From Figure 3(b), the corrosion products were little on the surface of sample at 500°C. The surface morphologies of the samples were relatively uniform after the corrosion tests at 700°C and 900°C, as shown in Figure 2(c,d), respectively. Microscopic analysis of the corrosion sample at 900°C indicated that the corrosion products crystals grew well, as shown in Figure 3(c).

Figure 4(a–e) show the surface SEM micrographs of Yb$_2$Si$_2$O$_7$ bulks after hot corrosion tests at 500°C, 600°C, 700°C, 800°C and 900°C for 2 h, respectively. From Figure 4, it can be seen that the corroded surfaces were mainly composed of regular rod-shaped crystals with different thicknesses and orientations. As shown in Figure 4(a), rod-shaped crystals were relatively slender (see point B), and irregular structures with porous appeared (see point A) after corrosion test at 500°C. While rod-shaped crystals obtained at other corrosion temperatures were relatively short (see points E, F, H, and K in Figure 4(b–e)). Since the surface morphology of corrosion samples was completely different from that of the Yb$_2$Si$_2$O$_7$ bulk (see Figure 1), these rod crystals can be inferred as the corrosion products. Moreover, a small number of flocules (see points D,
Figure 3. Metallographic surface photographs of (a) the non-corrosive edge and (b) corrosive zone of the sample at 500°C, and (c) the corrosion surface of the sample at 900°C.

Figure 4. SEM images on surface of Yb$_2$Si$_2$O$_7$ samples after hot corrosion tests at (a) 500°C, (b) 600°C, (c) 700°C, (d) 800°C and (e) 900°C.

Figure 5. (a) SEM image of the Yb$_2$Si$_2$O$_7$ sample exposed to NaVO$_3$ at 900°C, and (b–d) the corresponding EDS mapping.
G, and I in Figure 4(b–d)) or ellipsoidal aggregates (see points J and L in Figure 4(d–e)) existed on the surface of the samples. Figure 5 shows the SEM image of the Yb$_2$Si$_2$O$_7$ bulk after corrosion test at 900°C and the corresponding EDS mapping. The results indicated that the corrosion surface was rich in O, V and Yb elements, suggesting the existence of YbVO$_4$. The distribution of corrosion products can be observed by V element.

Figure 6 shows the XRD patterns of Yb$_2$Si$_2$O$_7$ bulks after corrosion tests at 500–900°C. All the corrosion surfaces were composed of Yb$_2$Si$_2$O$_7$, YbVO$_4$, and SiO$_2$. After the hot corrosion test at 500°C, some diffraction peaks of YbVO$_4$ and SiO$_2$ can be observed, but the peak intensities were relatively low. The main phase belonged to Yb$_2$Si$_2$O$_7$. After the hot corrosion tests at 600–900°C, corrosion products of YbVO$_4$ and SiO$_2$ became the main phase. In order to confirm the corresponding relationships between the crystals and morphologies, EDS analysis was performed for these typical morphologies of rod-shaped crystals, irregular structures with pores, and floccules or aggregates with ellipsoid in Figure 4. The EDS analysis results are shown in Figure 7. As indicated, the atomic percentage components of point A and point B in Figure 4(a) were of 28.30% Yb, 27.18% Si and 44.52% O (see Figure 7(a)) and 20.63% Yb, 21.33% V and 58.04% O (see Figure 7(b)), respectively. And that of point D in Figure 4(b) was of 2.14% Yb, 31.21% Si and 66.65% O (see Figure 7(c)). In combination with the XRD results in Figure 6, it can be confirmed that point A was Yb$_2$Si$_2$O$_7$, point B was YbVO$_4$, and point D was SiO$_2$. Moreover, EDS analysis was also performed for the other points in Figure 4(b–e). Combined with the XRD results, the analysis results are shown in Table 1. At 500°C, the corrosion sample surface was mainly composed of Yb$_2$Si$_2$O$_7$ and YbVO$_4$. 

![Figure 6](image1.png)

**Figure 6.** XRD patterns of Yb$_2$Si$_2$O$_7$ samples before and after the hot corrosion tests at different temperatures.

![Figure 7](image2.png)

**Figure 7.** EDS analysis of (a) point A in Figure 4(a), (b) point B in Figure 4(a,c) point D in Figure 4(b).
and YbVO₄ crystals were in thin-rod shape with irregular laminations at both ends. At 600°C, the corrosion sample was composed of Yb₂Si₂O₇, YbVO₄ and SiO₂, and the YbVO₄ crystals became plump. The agglomerated SiO₂ can be obviously seen. At 700–900°C, the corrosion product was relatively dense, and Yb₂Si₂O₇ was not visible on the corroded surface (see Figure 4 (c–e)). YbVO₄ crystals were in short-rod shapes, and both ends became regular quadrangular pyramid at 800°C and 900°C. Flocculent and small ellipsoidal SiO₂ appeared at 800°C, but only larger ellipsoidal SiO₂ appeared at 900°C. Obviously, with increasing temperature, the rod-shaped YbVO₄ became shorter and thicker, with ends changed from irregular lamination to regular quadrangular pyramid shape; SiO₂ changed from lamellar agglomeration flocculent to ellipsoid shape.

Yb₂Si₂O₇ bulks were washed and then weighed in order to exclude the influence of residual corrosives on the corrosion weight gain. At 500°C, 600°C, 700°C, 800°C and 900°C, the weight gains were 1.4 mg, 2.2 mg, 2.7 mg, 2.4 mg and 2.6 mg, respectively. The largest weight gain was at 700°C. Weight gains slightly decreased at 800°C and 900°C. The possible reasons are as follows: firstly, part of corrosion products fell off during the ultrasonic washing process; secondly, NaVO₃ may have a small loss due to the volatilization beyond 700°C, which led to a reduction of corrosion products.

Figure 8 shows the SEM images on the cross-section of the Yb₂Si₂O₇ bulks after the hot corrosion tests at 500°C, 600°C, 700°C, 800°C and 900°C for 2 h, and 900°C for 8 h and 16 h. The corrosive layers were formed on the top surfaces of the samples. It can be seen that the corrosion layers were relatively dense. Note that the average thickness was obtained by measuring the corrosion layer thickness at five equally spaced points in the cross-sectional image and then taking the average value. For the samples corroded for 2 h, the average thicknesses of the corrosion layers at 500°C, 600°C, 700°C, 800°C and 900°C were about 3.5 μm, 5.5 μm, 5.2 μm, 6.6 μm and 9.1 μm, respectively. The overall trend was that corrosion thickness increased with increasing temperature. Additionally, the corrosion tests were carried out at 900°C for 8 h and 16 h, and the average thicknesses of the corrosion layers were about 9.3 μm and 9.8 μm, respectively. Therefore, the thickness of the corrosion layer did not increase significantly with time at 900°C. It was worth noting that the test temperatures of 500°C and 600°C were lower than the melting point of NaVO₃, and the chemical reactions at this time were solid-solid phase reactions. However, the thickness of the corrosion layer increased from 3.5 μm to 5.5 μm. The possible reasons are as follows: 1) Contact and diffusion are necessary conditions for solid-state reaction. When the reactant reaches a certain thickness, the further reaction will depend on the diffusion of the product layer. An increase in temperature promoted the diffusion of the product layer, leading to an increase in the thickness of the corrosion layer. 2) The solid-liquid phase reaction may occur at 600°C because the melting point of NaVO₃ is nearly close to 600°C, which may result in a more efficient corrosion reaction at 600°C. Therefore, the corrosion layer thickness increased obviously from 500°C to 600°C.

### Table 1. Analysis results of phases on the corroded surfaces of samples at different temperatures.

| Test temperature | 500°C | 600°C | 700°C | 800°C | 900°C |
|------------------|-------|-------|-------|-------|-------|
| Analyzed point   |       |       |       |       |       |
| Phase and its    | A     | B     | C     | D     | E     |
| morphology       | irregular | elongated | irregular | flocculent | short rods |
|                  | F     | G     | H     | I     | J     |
|                  | Yb₂Si₂O₇ | YbVO₄ | SiO₂ | Yb₂Si₂O₇ | YbVO₄ |
|                  | K     | L     | K     | K     | K     |
|                  | SI₂O | SI₂O | SI₂O | SI₂O | SI₂O |

4. Discussion

The hot corrosion behaviors mainly include sulfidation and fluxing mechanisms [19]. The chemical reaction between vanadium compound and metal oxide follows Lewis acid-base fluxing mechanism [20]. In this study, Yb₂Si₂O₇ can be considered as the composition of Yb₂O₃ and SiO₂ in a molar ratio of 2:1. SiO₂ is an acidic oxide, and Yb₂O₃ is a basic oxide. At the same time, NaVO₃ is weakly acidic and can be decomposed into:

\[ 2NaVO₃ \rightarrow V₂O₅ + Na₂O \]  (2)

Among them, V₂O₅ is a strong acid and can directly participate in corrosion reaction. The primary corrosion reaction is:

\[ V₂O₅ + Yb₂O₃ \rightarrow 2YbVO₄ \]  (3)

In summary, the corrosion mechanism of Yb₂Si₂O₇ with NaVO₃ is:

\[ Yb₂Si₂O₇ + 2NaVO₃ \rightarrow \{Yb₂O₃ + 2SiO₂\} + (V₂O₅ + Na₂O) \rightarrow 2YbVO₄ + 2SiO₂ + Na₂O \]  (4)

From a thermodynamic perspective, the change in Gibbs free energy (Δ) for Eq. 3 is reported in Dong’s work [21], and the values of ΔG for Eq.3 are all negative at the corrosion temperatures of 500°C–900°C in this work, indicating that Eq. (3) could occur thermodynamically. Note that the thermodynamic data of Yb₂Si₂O₇ and YbVO₄ in Eq. (4) were not found in the handbooks and references. As a result, the changes in Gibbs free energy and entropy for Eq. (3) and (4) could not be calculated and displayed.
At 500°C and 600°C, which is lower than the melting point of NaVO₃ (630°C), the solid-state reaction at local contact interface between NaVO₃ and Yb₂Si₃O₇ bulk occurred, leading to the formation of YbVO₄ corrosion products. YbVO₄ nucleated and grew on the surfaces of Yb₂Si₃O₇ bulks. At the same time, some SiO₂ and Na₂O were generated. SiO₂ partly remained on the corrosion surface (see Figure 4). Na₂O were washed away during the ultrasonic washing process. The driving force of solid state corrosion reaction was low due to the low temperature, and the corrosion reaction was slow. Thus, YbVO₄ exhibited a thin and long shape, and the growth of both ends was incomplete. The amount of flocculent SiO₂ was also little. The thicknesses of corrosion layer were thin.

At 700–900°C, the molten NaVO₃ was in full contact with the Yb₂Si₃O₇ bulk. The corrosion reaction driving force increased with increasing temperature, and the corrosion products were easier to nucleate and grow up. Therefore, both ends of the short-rod YbVO₄ crystal gradually evolved from irregular lamination to regular quadrangular pyramid shape, and SiO₂ in the agglomerated state also changed from flocculent to ellipsoid shape.

The primary corrosion product is YbVO₄ in the corrosion reaction, and its crystal structure is similar to the tetragonal anatase and zircon (ZrSiO₄) [22,23]. The trunk grows in a square prism shape, and both ends in a quadrangular pyramid shape. The main driving force for grains growth and morphology changes is the reduction of the free energy of the crystal plane. For the YbVO₄ crystal, the free energy of the crystal plane follows such order: (0 0 1) > (1 1 0) > (1 1 2) [24]. Thus, the YbVO₄ crystal preferentially grows in [0 0 1] direction. The acidity of NaVO₃ is weak, the nucleation rate of the YbVO₄ in the corrosion reaction is low, but the growth rate is fast, so YbVO₄ crystals are mostly short-rod with a quadrangular pyramid at both ends.

5. Conclusions
Hot corrosion behavior of Yb₂Si₃O₇ bulks exposed to NaVO₃ was investigated at 500–900°C. The main results are as follows:

(1) Surface corrosion phenomenon was observed at the test temperatures of 500–900°C. The corrosion products were composed of YbVO₄ and SiO₂. At 500°C, the intensity of diffraction peaks of YbVO₄ and SiO₂ was relatively low. The main phase belonged to Yb₂Si₃O₇. However, corrosion products of YbVO₄ and SiO₂ became the main phase beyond 600°C.

(2) After ultrasonic washing, the surfaces of the samples were mainly composed of rod-shaped YbVO₄ crystals with different thicknesses and orientations. At 500°C, the rod-shaped YbVO₄ crystals were thin, and a large quantity of Yb₂Si₃O₇ bulk grains were exposed. The rod-shaped YbVO₄ crystals tended to be plump as the corrosion temperature increased, and both ends changed from irregular lamination to regular quadrangular pyramid shape. At the same time, there were also small amounts of flocculent and ellipsoidal SiO₂ on the corrosion surface.

(3) The weight gain of corrosion samples was 1.4 mg, 2.2 mg, 2.7 mg, 2.4 mg and 2.6 mg at 500°C, 600°C, 700°C, 800°C and 900°C for 2 h, respectively, so was the corrosion layer thicknesses 3.5 μm, 5.5 μm, 5.2 μm, 6.6 μm and 9.1 μm, respectively. The general trend was

Figure 8. Cross-sectional SEM images of the samples after hot corrosion tests at 500–900°C (a, b, c, d and e) for 2 h, and at 900°C (f) for 8 h, and (g) for 16 h.
that the weight gain and corrosion layer thicknesses increased with increasing temperature. However, the corrosion layer thicknesses at 900°C for 8 h and 16 h were 9.3 μm and 9.8 μm, respectively. The corrosion layer thicknesses did not increase significantly with time at 900°C.

(4) The primary corrosion reaction between NaVO₃ and Yb₂Si₃O₇ was V₂O₅ + Yb₂O₃ → Yb₂VO₄. Since NaVO₃ is of a low acid, Yb₂VO₄ crystals had a low nucleation rate. The driving force of corrosion reaction increased with the increasing temperature. At this time, Yb₂VO₄ crystals grew preferentially in the direction of [0 0 1], and the quadrangular pyramid shape at both ends becomes regular. Furthermore, SiO₂ in the agglomerated state also changed from flocculent to ellipsoid shape.

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Disclosure statement

No potential conflict of interest was reported by the author(s).

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