



Evaluation of hot corrosion behavior of SrHfO₃ ceramic in the presence of molten sulfate and vanadate salt



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ABSTRACT

SrHfO₃ powder was synthesized by solution combustion and SrHfO₃ ceramic was pressureless-sintered at 1600 °C for 10 h. The hot corrosion behaviors of SrHfO₃ against V₂O₅ and Na₂SO₄+V₂O₅ (molar ratio = 1:1) were investigated under 900, 1050 and 1200 °C in air. The thickness of the corrosion-layers decreases whereas the size of the corrosion products increases gradually with the increase of corrosion temperature in hot corrosion test. The chemical reactions between SrHfO₃ and the molten salts are the primary corrosion mechanisms for degradation of SrHfO₃.

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1. Introduction

Thermal barrier coatings (TBCs) are used for thermal and chemical protection of metallic substrates [1–5]. Ytria stabilized zirconia (YSZ) is prior choice due to its excellent advantages including low thermal conductivity and a relatively high thermal coefficient, which could reduce the thermal expansion mismatch with the metallic substrate [3–7]. Up to now, the state-of-the-art 6–8 wt% YSZ is widely used as the standard TBCs [6,7]. However, YSZ is limited to applications below 1200 °C and severe operating conditions can weaken the performance of YSZ coatings [6,7].

The low-quality fuels usually contain impurities, such as sodium, vanadium and sulfur, which can form sulfate and vanadate salts on the surfaces of the TBCs during long-term operation [7]. TBCs will degrade when they working in vanadium and sodium environment which comes from the combustion of low grade petroleum fuels. In which, molten sulfate-vanadate deposits resulting from the condensation of combustion products of such fuels are extremely corrosive to high-temperature materials in combustion systems [7–12]. Conventional YSZ produced YVO₄ and *m*-ZrO₂ exhibiting poor hot corrosion resistance in these molten salts. Moreover, the *t*-ZrO₂ transforms into *m*-ZrO₂, which

is accompanied by volume expansion of 3–5% resulting in increased microcracking and spallation of coating [13–15]. Hence, numerous attempts have been made to improve the phase stability and hot corrosion resistance of YSZ based TBCs [16–23]. Meanwhile, it is important to develop new TBCs materials in order to overcome the challenges of highly harsh operating environments [24–29].

The perovskites (ABO₃) possess high melting points, low thermal conductivities, and high thermal expansion coefficients (TECs), which could be considered as a new TBC material served above 1200 °C [30,31]. Furthermore, it is found that hafnate owns some interesting properties [32,33] and the earlier work has shown that SrHfO₃ possesses excellent thermal stability, low thermal conductivity and low sintering rate, which make it promising for new TBCs material [34–36]. Unfortunately, there are no reports exemplifying the hot corrosion behaviors of SrHfO₃ coatings or ceramics against V₂O₅ and Na₂SO₄+V₂O₅ corrosive environments at elevated temperature.

In the present paper, SrHfO₃ powder was prepared and hot corrosion behaviors of SrHfO₃ ceramics against V₂O₅ and Na₂SO₄ + V₂O₅ mixtures (1:1, molar ratio) corrosive salts in the temperature range of 900–1200 °C were investigated. The mechanisms of hot corrosion with regard to the corrosive product formation and microstructure change were discussed in detail.

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2. Experimental

2.1. Powder synthesis

SrHfO₃ powders were synthesized by a solution combustion method using Sr(NO₃)₂·4H₂O (99%), HfCl₄ (99%) and CH₄N₂O (99%) as raw materials (Sinopharm Group, China). Aqueous solution containing ions of Sr²⁺ and Hf⁴⁺ was prepared by dissolving stoichiometric amount of Sr(NO₃)₂·4H₂O and HfCl₄ in double distilled water. CH₄N₂O was added to the solution as fuel, whose amount was calculated based on total valence of the oxidizing and the reducing agents for maximum release of energy during combustion. The solution containing the precursor mixture at a pH of 1.0 was put in a furnace preheated up to 550 °C. After vigorous combustion, fluffy powders were obtained.

2.2. Preparation of SrHfO₃ ceramic

As-prepared SrHfO₃ powder was heated at 1200 °C for 2 h and then ball milled before being pressed. In order to obtain bulks for hot corrosion experiments, the pressed powder was further compacted by cold isostatic pressing method under a pressure of 280 MPa for 3 min. After that, the as-prepared ceramic green body was pressureless-sintered at 1600 °C for 10 h in air at a heating rate of 8 °C/min.

2.3. Hot corrosion tests

Cylinder specimens with dimensions of Φ10 mm × 3 mm were used for hot corrosion tests. At first, the V₂O₅ and Na₂SO₄ (molar ratio = 1:1) were mixed evenly by full manual grinding for 1 h. Afterwards V₂O₅ and the mixture of Na₂SO₄+V₂O₅ were strewn over the surface of SrHfO₃ specimens and then were coated uniformly by using a very fine glass rod at a concentration of 40 mg/cm², respectively. As-coated SrHfO₃ specimens put in a zirconia crucible were placed in an electric furnace. All tests were conducted

isothermally from 900 to 1200 °C in air for 4 h.

2.4. Characterization

Phase compositions of SrHfO₃ powder, SrHfO₃ ceramic and hot corrosive specimens were analyzed by X-ray diffractometer (XRD, X'Pert Pro MPD) with Cu K_α radiation at a scan rate of 3°/min. The microstructures of powder, ceramic and corrosive specimens were characterized by scanning electron microscope (SEM, JSM6460) equipped with energy dispersive spectroscopy (EDS). Before SEM observations, the prepared ceramic were polished with 1 μm diamond paste followed by thermally etched at 1500 °C for 1 h. In order to obtain high quality images, gold sputtering was used on each sample for SEM investigation. The densities ($\rho = 5.91 \text{ g/cm}^3$) and porosities ($P = 20.02\%$) of the sintered samples were measured according to the Archimedes' principle.

3. Results

3.1. Microstructures and the phase compositions of SrHfO₃ powder and ceramic

Fig. 1(a) and (b) show the XRD patterns of the synthesized SrHfO₃ powder and prepared ceramic sintered at 1600 °C for 10 h in air. The XRD patterns reveal that the products have excellent crystallization properties. The significant peaks at 21.9°, 31.1°, 44.5°, 49.8°, 55.2°, 64.6° and 73.3° for prepared powder and ceramic respectively correspond to (020), (121), (040), (042), (004), (161) and (044) planes of SrHfO₃ (JCPDS 01-089-5606) indicating the successful preparation of SrHfO₃ powder and ceramic.

As shown in Fig. 1(c), the agglomerated SrHfO₃ particles with the size of about 20 μm are fluffy and irregular. The polished surface of SrHfO₃ ceramic is quite coarse and porous (Fig. 1(d)), which will not only efficiently increase the contact area between the corrosive agents and ceramic substrate but also provide infiltration paths for molten salts during hot tests. Magnified view displays the typical

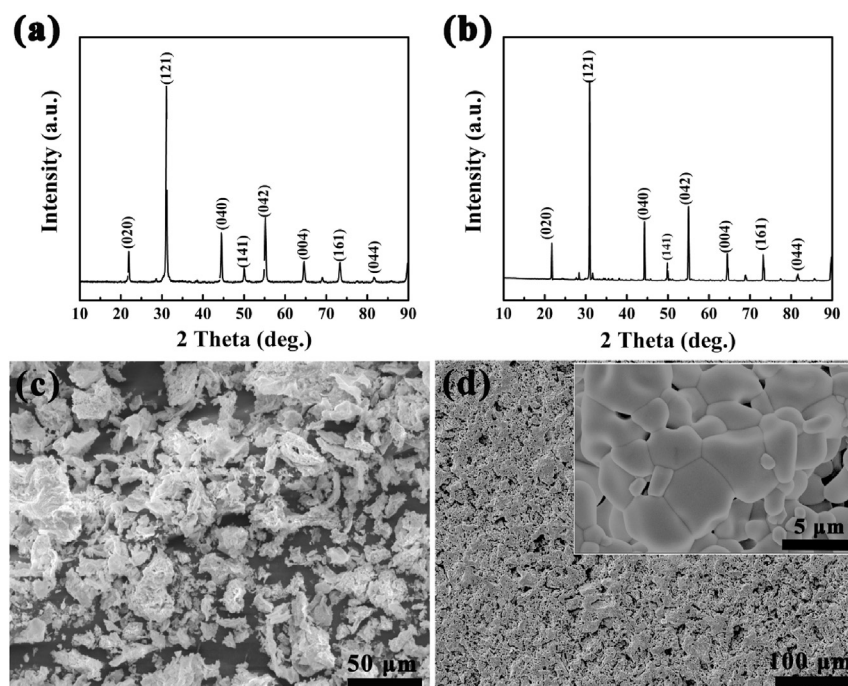


Fig. 1. XRD patterns and microstructures SEM images of the SrHfO₃ powder synthesized by combustion method (a and c) and its ceramic sintered at 1600 °C for 10 h in air (b and d).

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