



A method for assessing the volatility of oxides in high-temperature high-velocity water vapor



Robert A. Golden*, Elizabeth J. Opila

Department of Materials Science and Engineering, University of Virginia, Charlottesville, VA 22904, United States

ARTICLE INFO

Article history:

Received 28 August 2015

Received in revised form

14 November 2015

Accepted 18 November 2015

Available online 5 January 2016

Keywords:

Oxide volatility

SiO₂

TiO₂

Y₂O₃

Environmental barrier coating

ABSTRACT

A steam-jet furnace was utilized to assess the volatility of SiO₂, TiO₂ and Y₂O₃ in a simulated turbine engine environment. Specimens were exposed in the steam-jet furnace at 1 atm H₂O, steam temperatures of 1240–1300 °C, and gas velocities of 165–177 m/s. The jet of steam impinged on the specimen under study leading to surface recession via the formation of volatile hydroxide species. Linear volatilization kinetics were observed for SiO₂. The measured recession rate of SiO₂ was in reasonable agreement with the calculated recession rate based on assessed thermodynamic data for Si(OH)₄ (g). The measured recession rate of TiO₂ in this study was considerably higher than the recession rate based on measured thermodynamic data for TiO(OH)₂ (g) and lower than prior experimental results. The measured recession rate of Y₂O₃ was in fair agreement with the calculated recession rate using estimated thermodynamic data for Y(OH)₃ (g) formation and was significantly lower than prior experimental results.

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1. Introduction

Silicon carbide based Ceramic Matrix Composites (SiC CMCs) are proposed to replace Ni-base superalloys in next generation gas turbine engines to increase engine efficiency by operating at higher combustion temperatures and reducing the weight of engine components. At elevated temperatures in a dry oxygen environment, SiC CMCs oxidize to form a protective silica scale by Reaction (1) [1,2]:



However, in a combustion environment the silica scale reacts with water vapor, a product of combustion reactions, and forms a gaseous silicon hydroxide species by Reaction (2) [1,2]:



This reaction leads to unacceptable recession rates of SiC CMCs, therefore Environmental Barrier Coatings (EBCs) have been developed to limit this reaction.

There are several criteria an EBC must meet to adequately protect SiC CMCs [3]. Firstly, the coating must be thermochemically stable in combustion environments. Secondly, the coating must

possess a coefficient of thermal expansion (CTE) close to that of the substrate material to prevent delamination or cracking due to CTE mismatch stress. Thirdly, the coating must maintain a stable phase under thermal exposure to prevent disruption of the coating with the volumetric change that typically accompanies a phase transformation. Fourthly, the coating must be chemically compatible with the substrate to avoid detrimental chemical reactions. EBCs that satisfy the second and fourth requirement include mullite and barium strontium aluminosilicate (BSAS), however these silicates are not sufficiently stable due to their high rates of silica volatility [3–8]. More stable rare earth silicate EBCs have been developed, however laboratory test capabilities for determining oxide stability in simulated turbine engine environments are needed to fully assess these newer materials.

Laboratory tests suitable for characterizing the thermochemical stability of EBCs should accurately simulate the use environment and screen for measurable material recession in reasonable test times at a reasonable cost. Laboratory furnaces, such as thermogravimetric analyzers (TGAs) and cyclic furnaces, are inadequate because of the long exposure times required to achieve measurable material recession at the low steam velocities that can be achieved [1,6,9–11]. In addition, furnace ware components form volatile hydroxide species in the presence of water vapor at elevated temperatures (e.g., Al(OH)₃ (g)) which can react with the test specimen. As a result, some rare earth silicates have been observed to gain weight due to the volatile hydroxide species depositing/reacting during exposure [6,9]. Burner rigs and high pressure burner rigs

* Corresponding author.

E-mail address: rob.golden1@gmail.com (R.A. Golden).

(HPBR) provide a means of simulating a turbine engine environment however they are expensive to build and operate [10,11]. A steam-jet furnace developed by Lucato et al. [12] provides a means of simulating a turbine engine environment and is significantly more economical to build and operate compared to burner rigs and HPBR. The steam-jet furnace is capable of simulating the temperatures (1200–1500 °C) and steam velocities (150–200 m/s) of a turbine engine operating at 10 atm total pressure with 10% water vapor. In this paper, the volatility of SiO₂, TiO₂ (001) single crystals and Y₂O₃ was investigated in a high-temperature high-velocity steam-jet to validate this method for testing of EBC candidate materials.

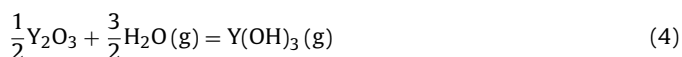
The volatility of SiO₂ in high-temperature water vapor by Reaction (2) has been well studied and is known to have linear volatilization kinetics [1,2,11]. In addition, all available thermodynamic data for Si(OH)₄ (g) have been assessed by Plyasunov [13].

The volatility of TiO₂ in high-temperature water vapor is less understood. A transpiration study by Nguyen et al. [14] concluded that TiO₂ (rutile) reacts with water vapor by Reaction (3):



The enthalpy and entropy of reaction were also determined. Weight loss rates of TiO₂ (rutile) in high-temperature water vapor have been reported by Ueno et al. [15], however they do not agree with the flux calculated using thermodynamic data of Nguyen et al. [14].

The volatility of Y₂O₃ in high-temperature water vapor is controversial. A study by Courcot et al. [16] concluded that above 1200 °C, Y₂O₃ reacts with water by Reaction (4):



Weight loss data for Y₂O₃ in water vapor at temperatures from 1000 to 1400 °C were reported. Thermodynamic data for Y(OH)₃ (g) estimated by Krikorian [17] suggest rates of Y(OH)₃ (g) formation should be orders of magnitude lower than those observed by Courcot et al.

The objectives of this paper are threefold. First, the modifications and optimization of the Lucato et al. steam-jet furnace design for testing of EBC candidate materials are described. Second, the capabilities of the steam-jet furnace are demonstrated using studies of the binary metal oxides of SiO₂, TiO₂ and Y₂O₃. Lastly, results of this work are used to resolve controversies in the literature on the volatility of TiO₂ and Y₂O₃ in high temperature water vapor.

2. Experimental procedure

2.1. Materials

Test specimens of amorphous SiO₂ (99.995% pure; Quartz Scientific Inc., Fairport Harbor, OH), single crystal TiO₂ (001) (99.99% pure; MTI Corp., Richmond, CA) and polycrystalline Y₂O₃ (99.99% pure; MaTeck, Juelich, Germany) were used for steam-jet exposures. SiO₂ specimens were prepared by cutting a 25.4 × 25.4 × 1 mm fused quartz coupon into 12.5 × 12.5 × 1 mm specimens. As-received TiO₂ (001) specimens were 10 × 10 × 1 mm and of the rutile phase. As-received Y₂O₃ specimens were 15 mm diameter × 1 mm and 94% of theoretical density. Two perpendicular cuts were made to the Y₂O₃ disc specimens to provide flat edges for insertion in an alumina specimen holder.

2.2. Steam-jet furnace

The steam-jet apparatus shown schematically in Fig. 1 uses a horizontal tube furnace (Model 1730-12HTF, CM Furnace Inc., Bloomfield, NJ) with a 3.493 cm (1.375") inner diameter alumina

tube (99.8% pure; McDanel Advanced Ceramic Technologies, Beaver Falls, PA) and an inlet stainless steel end cap (MDC Vacuum Products LLC, Hayward, CA) that accommodates feedthroughs for thermocouple, gas, and liquid water inlets.

A peristaltic pump (Reglo Analog MS 2/12, Ismatec SA, Glattburg, Switzerland) fed deionized 18.2 MΩ liquid water into a fused quartz capillary at a controlled flow rate. The liquid water was heated prior to being pumped into the capillary by flowing through copper tubing coiled in a water bath held at ~85 °C. The fused quartz capillary (1 mm ID, 3 mm OD, 99.995% pure; Quartz Scientific Inc., Fairport Harbor, OH) was inserted through an ultratorr fitting (Swagelok, Solon, OH) on the end cap and extended into the center of the hot zone. A Pt-13Rh wire (0.5 mm OD) was inserted into the capillary to assist in the heating of liquid water as it travelled down the capillary. The Pt-13Rh wire was 6–10 cm shorter than the capillary outlet. Two smaller alumina tubes (23 mm OD) were used inside the furnace tube to support and align the fused quartz capillary as shown in Fig. 1.

The flow rate of the liquid water was measured before and after each experiment to determine the average liquid water flow rate for the experiment. Average flow rates ranged from 1.55 to 1.80 mL/min. Liquid water evaporates as it enters the hot zone of the furnace resulting in a volume expansion forming a jet of high-temperature high-velocity steam. The steam-jet velocity was modeled by Computational Fluid Dynamics (CFD) analysis (ANSYS CFX, ANSYS Inc., Canonsburg, PA) (Fig. 2). Inputs to the analysis include the mass flow rate of the liquid water, the steam-jet temperature and the geometry of the fused quartz capillary relative to the test specimen. The steam-jet velocity was modeled across the surface of the specimen and the maximum surface velocity observed was chosen to represent the condition at which maximum material degradation occurs. A thermocouple (type R) inserted through a second inlet on the end cap extended to the center of the hot zone approximately 4 cm from the specimen and remained stationary during the experiment. Prior to beginning the experiment, the steam-jet temperature was calibrated by placing a second thermocouple (type R) approximately 1 mm from the tip of the capillary (the same position of the specimen during the experiment) and recording the temperature. This thermocouple was inserted through the open exhaust end of the alumina tube.

Experiments were performed at 1 atm H₂O pressure, steam-jet velocities of 165–177 m/s, and steam-jet temperatures ranging from 1240 to 1300 °C. The test specimens were mounted in an alumina holder at a 45° relative to the steam-jet and held in place by platinum foil and wire. The alumina holder was pushed into the tube furnace and locked into place in front of the capillary. The capillary was adjusted so that it was 1 mm away from the surface of the specimen. Experiments were conducted with no outlet endcap so that 1 atm air was present in the tube surrounding the steam-jet. Photographs of the specimen holder were taken before and after each experiment and were compared to confirm the specimen did not shift during the steam-jet exposure.

Although the steam-jet furnace is based on a design of Lucato et al. [12], several modifications have been made in this study. Firstly, the specimen orientation and geometry have been modified in this study. Lucato et al. directed their steam-jet onto the leading edge of sharp wedge shaped specimens whereas in this study the steam-jet was directed onto flat polished specimens which were orientated at a 45° relative to the steam-jet. This configuration allows for testing of a variety of coupon shapes as well as coated coupons. Secondly, in this study efforts were made to ensure complete vaporization of water. The liquid water was preheated prior to entering the capillary. In addition, a Pt-13Rh wire was inserted into the capillary to assist in the heating of the liquid water as it travelled through the capillary. Thirdly, Lucato et al. flowed argon gas through their mullite furnace tube whereas no argon was used

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