

## USE OF VERY HIGH WATER-VAPOR PRESSURES TO EVALUATE CANDIDATE COMPOSITIONS FOR ENVIRONMENTAL BARRIER COATINGS

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### ABSTRACT

Environmental barrier coatings (EBCs) are used to protect Si-based ceramics from accelerated oxidation and volatilization in the presence of elevated water-vapor pressures at high temperatures and high gas velocities. Previously, an analysis based on mass flux of volatilized species has shown that operating at very high  $H_2O$  pressures can be used to compensate for the low gas velocities found in many laboratory exposure facilities so as to conduct first-stage screening of EBC compositions for volatility resistance. To test this prediction experimentally, a high-temperature furnace was modified to accommodate gas pressures of up to 20 atm and bulk specimens of barium-strontium aluminosilicate (BSAS), barium aluminosilicate (BAS), and strontium aluminosilicate (SAS), silica ( $SiO_2$ ), and silicon carbide (SiC) were exposed at 1250°C in 90%  $H_2O$ -10% air. This set of materials provided an adequate spread in susceptibility to volatilization to evaluate the proposed approach because BSAS, BAS, and SAS are significantly more volatilization resistant in water-bearing environments than  $SiO_2$  and Si-bearing ceramics (such as SiC) that form silica under these conditions. The gravimetric results provided clear trends in volatilization resistance. The three aluminosilicates exhibited similar kinetic behavior and had significantly lower rates of mass losses than the  $SiO_2$  and SiC. These findings provided the experimental proof-of-principle for using high-pressure, low-gas-velocity exposures for qualitative differentiation of degrees of volatilization resistance among different candidate materials being developed for EBC applications.

### INTRODUCTION

It is now well known that the high-temperature corrosion resistance of Si-based ceramics is particularly sensitive to water vapor in the environment in terms of accelerated oxidation and/or mass loss by volatilization.[1-10] These coupled reactions can be life-limiting for the use of these materials in combustion environments, where high temperatures, elevated water-vapor pressures, and moderate to high gas-flow velocities exacerbate the process. This degradation mode has been shown to be of specific relevance for SiC and  $Si_3N_4$ , where  $SiO_2$  forms by (accelerated) oxidation and then readily reacts with environmental  $H_2O$  to form volatile species.[2,5,6,9,10] Of the various

volatilization reactions, the one forming  $Si(OH)_4$ ,



has been shown to be predominant in combustion environments.[5] Consequently, there is concern about the use of these materials as hot-section components in gas turbine and microturbine engines because, to achieve the highest energy efficiencies and lowest emission levels, the required temperatures and water-vapor pressures in the combustion zones will be such that rapid degradation of Si-based ceramics will occur. Therefore, to take advantage of the high-temperature mechanical properties of these materials for turbine engine applications, environmental barrier coatings (EBCs) [11-13] will be required on surfaces exposed to combustion gases. In a recent study, the application of a BSAS-based EBC to SiC/SiC composite combustor liners in a Solar Turbines natural-gas turbine engine significantly increased the life of the SiC/SiC composite material.[14-16] However, the long-term phase stability and oxidation/volatilization resistance of current state-of-the-art EBCs (including BSAS and SAS) are not sufficient to achieve desired engine lifetimes >30,000 h at temperatures 1200°C.[16] Therefore, the development and future implementation of improved EBCs for use in high water-vapor-containing environments has become critical for consideration of Si-based ceramic and composite materials for long-term use as hot-section components in gas turbines or microturbines.

In order to be effective for extended periods in combustion environments, an EBC must not only be substantially more volatilization resistant than the Si-based ceramic it is protecting, but also provide a permeation barrier so that oxidizing species do not readily react with the substrate and compromise properties (prevent accelerated oxidation in the presence of water vapor). In addition, the EBC must be thermally stable and relatively nonreactive with its substrate for long times at the expected use temperature (1100°C-1350°C). Efforts to develop coatings to meet these demanding characteristics have been ongoing for a number of years.[11-16] A high-temperature, high-pressure exposure facility (comprised of "Keiser Rigs"[17]), combined with detailed post-exposure microstructural analysis, has been used for first-stage evaluation of

various candidate EBCs at prototypic H<sub>2</sub>O pressures (0.3-2.0 atm) and temperatures.[16,18] Such an approach has been effective in screening potential EBCs in terms of permeation resistance and phase and interfacial stabilities, but not volatilization tendencies because, under normal operating conditions of low gas-flow velocities in the Keiser Rigs (3-20 cm/min.), the mass flux of volatile species was too low (see below) to be measured.

In a previous paper,[19] calculations based on generalized volatility reactions and mass flux of volatilized species showed that, in terms of the Keiser Rigs, high H<sub>2</sub>O pressures can be used to compensate for the low gas-flow velocities. As shown in detail for conditions relevant to the Keiser Rig,[19] the mass flux, **J**, of the volatile product A<sub>x</sub>O<sub>y+z</sub>H<sub>2z</sub> associated with the generalized reaction



can be described as

$$J \propto v^{1/2} \cdot \exp(-G_{A_xO_{y+z}H_{2z}}/RT) \cdot p_{H_2O}^z \cdot a_{A_xO_y}/(p_{tot})^{1/2} \quad (3)$$

where *v* is the gas-flow velocity,  $G_{A_xO_{y+z}H_{2z}}$  is the free energy of formation of A<sub>x</sub>O<sub>y+z</sub>H<sub>2z</sub> (equation (2)),  $p_{H_2O}$  is the partial pressure of water vapor,  $p_{tot}$  is the total system pressure,  $a_{A_xO_y}$  is the thermodynamic activity of the oxide, *T* is the absolute temperature, and *R* is the universal gas constant. A flux equation of the form shown in equation (3) has been shown to describe SiC and Si<sub>3</sub>N<sub>4</sub> recession controlled by volatilization of SiO<sub>2</sub> quite well.[6,9] However, at low gas-flow velocities and low-to-moderate pressures, as employed in most laboratory experiments, specimen mass losses due to volatilization are small and, thus, difficult to measure, particularly because of large mass gains from accelerated solid-state oxidation at elevated  $p_{H_2O}$ .[4,7,8] This was the case for most of the experiments conducted in the Keiser Rigs, which focused on the effects of elevated H<sub>2</sub>O pressures (typically 0.3-2.0 atm) on the oxidation of Si-based ceramics and composites and EBCs to evaluate permeation resistance and thermal stability.[7,8,18,20] However, as shown by Equation (3), mass fluxes for a given *v* can be increased substantially by using higher H<sub>2</sub>O pressures, particularly if *z* > 1.

An example of how higher water-vapor pressures can be used to generate more readily measurable mass fluxes can be determined from the volatilization of SiO<sub>2</sub> in the presence of H<sub>2</sub>O (Equation (1)), where, from Equations (2) and (3)

$$J \propto v^{1/2} \cdot \exp(-G_{Si(OH)_4}/RT) \cdot p_{H_2O}^2 \cdot a_{SiO_2}/(p_{tot})^{1/2} \quad (4)$$

and  $a_{SiO_2} = 1$ . Relatively high fluxes of Si(OH)<sub>4</sub> can be achieved by increasing  $p_{H_2O}$  since **J** is proportional to the square of  $p_{H_2O}$  at constant  $p_{tot}$ . Therefore, if there is no change in mechanism, low gas-flow velocities can be offset by higher  $p_{H_2O}$ . This is illustrated in Fig. 1, which plots **J**/**J**<sub>0</sub> versus  $p_{H_2O}$  for the case represented by Equation (4) where **J**<sub>0</sub> is the Si(OH)<sub>4</sub> flux for typical combustor liner conditions in land-based gas turbines (*v*=35 m/s,  $p_{H_2O}$ =1.5 atm, *T*=1200°C) and **J** is for the same temperature with *v*=6 X 10<sup>-4</sup> m/s (typical gas-flow velocity in the Keiser Rigs). Note from Fig. 1 that the normalized flux can be increased by approximately two orders of magnitude by increasing  $p_{H_2O}$  from 1.5 atm (pressure typically used in the Keiser Rigs[18]) to 20 atm.

The results shown in Fig. 1 show that the high-temperature, high-pressure capability offered by a Keiser Rig (or similar exposure facility) can be used to yield relatively high volatility fluxes by conducting experiments at substantially higher water-vapor pressures ( $p_{H_2O}$ ). As such, this approach can be used to screen the volatility resistance of candidate ceramic compositions for use in combustion environments as bulk materials or protective surface coatings (such as

needed for EBCs). However, such use is predicated on no change in the rate-controlling volatilization mechanism as the water-vapor pressure is increased. Given the nature of the process described above (diffusion of volatile product(s) across a laminar gas-boundary region), it is not expected that changes in pressures over the range indicated in Fig. 1 would result in a change in mechanism. To test this approach of using high H<sub>2</sub>O pressures to compensate for low gas-flow velocities, long-term exposure of SiO<sub>2</sub>, SiC, and currently-used EBC compositions are being conducted in a Keiser Rig under these conditions. This paper reports the most recent results, which demonstrate the utility of using this approach in evaluating the various factors involved in volatilization under oxidizing conditions as part of studies that are aimed at developing and characterizing materials that are stable at the high water-vapor pressures associated with turbine combustion conditions.

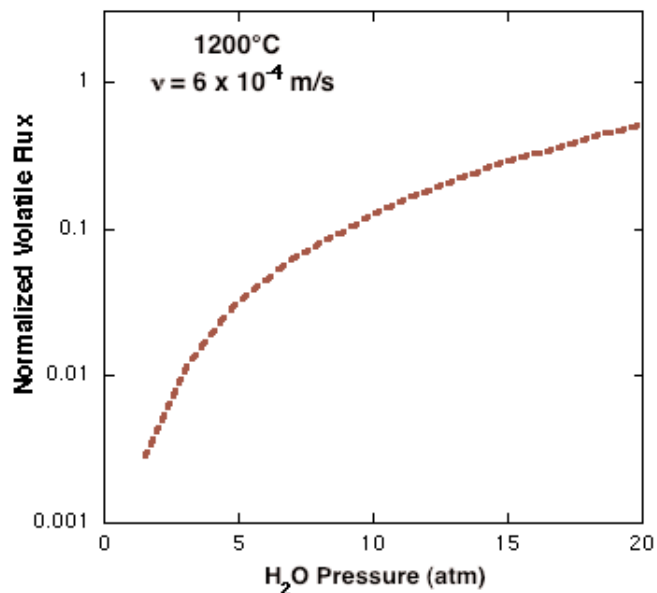


Fig. 1. Plot of flux of Si(OH)<sub>4</sub> due to volatilization of SiO<sub>2</sub> in an H<sub>2</sub>O-containing environment at 1200°C, a total pressure of 20 atm, and a gas flow velocity of 6 X 10<sup>-4</sup> m/s as a function of the partial pressure of H<sub>2</sub>O.[19]

## EXPERIMENTAL PROCEDURE

All laboratory exposures were conducted in ORNL's high-temperature, high-pressure facility, which comprises three different Keiser Rigs. The Keiser Rig used for this study consisted of a large box furnace which contained two 92-cm long by 9-cm diameter SiC pressure vessels and a multiple-flow gas-supply/manifold system.[17] Samples are hung on the outside of a slotted alumina tube using Pt wire which is suspended inside a SiC tube.[20] A slow-flow gas supply system (5-33 X 10<sup>-4</sup> m/s) provides a pressurized mixture of steam and air to each SiC tube. Previous high-temperature, high-pressure exposures of EBC-SiC/SiC materials in the Keiser Rig were conducted to evaluate the thermal-stability and protective-capability of different EBC formulations using exposure conditions which best simulated the temperature, pressure, and principal reactive gas species (H<sub>2</sub>O) of a gas-turbine combustor environments.[18,20] For example, to evaluate materials for use in a Solar Turbines Centaur 50S engine, all Keiser Rig exposures of EBC/SiC-SiC composites were conducted at ~1200°C (2200°F), 1.5 atm H<sub>2</sub>O (balance air), and 10 atm total system pressure. In the present study, an extremely high H<sub>2</sub>O pressure (18 atm) and elevated temperature (1250°C) are being utilized to

experimentally validate that (1) very high water vapor-pressures can be used to induce volatility in the Keiser Rig and (2) measure relative volatilization rates of candidate EBCs. To accommodate the much higher water-vapor pressures used in this study, several modifications were made to the Keiser Rig's gas supply and exhaust systems.[19]

There were several proof-of-principle specimens included in the initial high water-vapor-pressure exposures to demonstrate and measure volatilization in the Keiser Rig. High purity SiC (produced by chemical vapor deposition (CVD)) and fused quartz have been well-characterized after exposure to elevated water vapor-pressures[7,8] and were included as standards in the Keiser Rig runs. Also, since the primary purpose of these experiments was to measure volatilization of currently-used, state-of-the-art EBCs, hot-pressed BSAS, barium aluminosilicate (BAS), and strontium aluminosilicate (SAS) materials were exposed simultaneously with the Si-based standards. The hot-pressed oxides were prepared by United Technologies Research Center (UTRC), East Hartford, Connecticut. The 7.5 X 7.5 X 0.6 cm hot-pressed plates of the three compositions were each cut into six coupons. All of the samples to be exposed in the ORNL furnace had their surfaces mechanically ground flat and parallel and polished with 6  $\mu\text{m}$  diamond paste. The mass and dimensions of each were determined before and after each exposure period (365, 500, 1000, and 1500 h). The BSAS, BAS, and SAS coupons were heat treated at 1350°C for 48 h and microstructurally characterized prior to high-temperature exposure for comparison with the same material after each exposure period in the Keiser Rig. Pre- and post-exposure cross-section specimens were imaged in a scanning electron microscope (SEM) equipped with a backscatter electron (BSE) detector and compositionally analyzed using an electron probe microanalyzer (EPMA). In this way, reaction products (or lack thereof) that formed during exposure, as well as any other microstructural changes, were identified and characterized with respect to their spatial dimensions.

## RESULTS AND DISCUSSION

The starting microstructures of the three aluminosilicates used in this study were described in a prior paper.[19] Each exhibited a 2-phase structure consisting of a major matrix phase (of the form  $(\text{Ba,Sr})\text{Al}_2\text{Si}_2\text{O}_7$ ) and a minor distribution of (Ba,Sr)-rich particles. Each also contained moderate to extensive porosity in the as-processed state. The gravimetric data for bulk specimens of BSAS, BAS, SAS, CVD SiC, and  $\text{SiO}_2$ , after exposures at 1250°C and 18 atm  $\text{H}_2\text{O}$ , are shown in Fig. 2. All three aluminosilicates exhibited similar kinetic behavior and had significantly lower total mass losses and rates of volatilization than the  $\text{SiO}_2$  and CVD SiC. At times > 500 h, the rate of mass loss of the  $\text{SiO}_2$  was approximately an order of magnitude greater than that for the mixed oxides. This difference is about what is expected based on results from combustor liner exposures.[20]

Quantitative estimates of recession rates under conditions where there is a significant flux of volatile species will require more detailed tracking of specimen dimensions and accompanying microstructural analyses, as well as a mechanistic understanding of the volatilization process in multiphase ceramics structures (see below). Nevertheless, the volatilization trends are clearly differentiated using the gravimetric data in Fig. 2. In support of the theoretical treatment,[19] summarized by Equations (2) and (3), these results provide an experimental proof-of-principle of using very high-pressure, low-gas-velocity exposures for qualitative differentiation of volatilization resistance among different candidate materials being developed for EBC applications. An added benefit of doing such at high  $\text{H}_2\text{O}$  pressures (versus low pressures, but high gas velocities) is that permeation resistance can also be evaluated (using greater-than-worst-case conditions because the  $p_{\text{H}_2\text{O}}$  is so high) when coatings on Si-based ceramic substrates are exposed.[18]

It is important to note that the utility of using higher  $\text{H}_2\text{O}$

pressures to compensate for low gas-flow velocities is critically dependent on the details of the oxidation reaction that produces the volatile species. Referring to Equation (3), the ability to use higher  $p_{\text{H}_2\text{O}}$  to screen the volatilization resistance of candidate materials in systems using low values of  $v$  depends critically on  $z$ , the number of moles of  $\text{H}_2\text{O}$  needed to form one mole of volatile product. A lower  $z$  value than used for the present experimental calculations ( $z=2$ ) would make it more difficult to achieve easily measurable volatile fluxes by increasing the water-vapor pressure over a reasonable range. As an example, while there is about an order of magnitude increase in the volatile flux when  $p_{\text{H}_2\text{O}}$  is increased from 1.5 to 20 atm at  $v=6\times 10^{-4}$  m/s and 1200°C for  $z=1$ , this enhancement is about 10X less than for  $z=2$ . [19] Thus, it is important to understand the nature of the predominant volatility reaction to determine if substantially increasing  $p_{\text{H}_2\text{O}}$  will yield the desired outcome and to effectively compare relative Js for different EBC compositions.

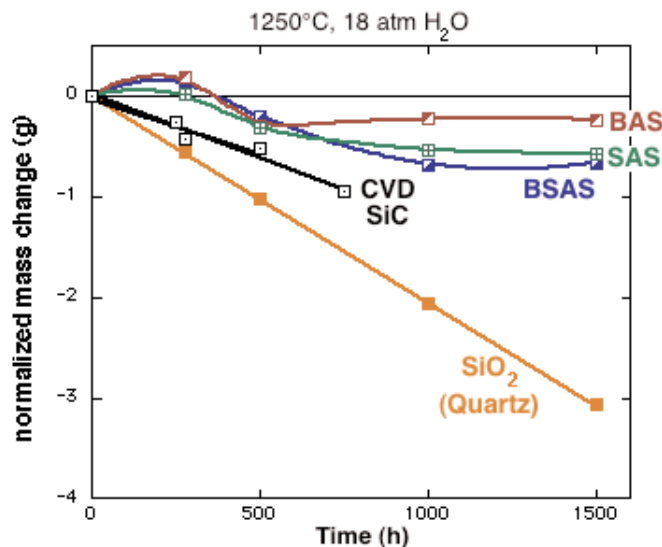


Fig. 2. Gravimetric results for exposure of all bulk specimens to 18 atm of  $\text{H}_2\text{O}$  at 1250°C.

In analyzing volatility results from experiments of the type described here, it is possible to have measured values that are not specific to one species if there are several products with comparable vapor pressures. This can be the case for complex mixed oxides with different  $z$  values and/or compounds that form gaseous oxides as well as hydroxides with high vapor pressures, such as BaO at unit activity.[21] Strict interpretation of results from multi-phase oxides will also be complicated by uncertainties in the activities of the various components (see Equation (3)) as these may deviate significantly from what is expected for ideal solution behavior and also can change upon thermal aging (see ref. 19 and below).

In considering the gravimetric data of Fig. 2, note that, while the SiC and  $\text{SiO}_2$  specimens exhibited mass losses at all exposure times, all of the aluminosilicates first showed some mass gain. Fig. 3 replots the BAS, SAS, and BSAS data from Fig. 2 on an expanded ordinate axis to more clearly observe the gravimetric behavior of these compositions as a function of time. (The curves are intended solely to suggest trends; they are fitted to the data by a smoothing function and do not represent any specific mechanistic interpretation.) Attempts at a definitive explanation of this gravimetric behavior with time await completion of the microstructural characterization and chemical analysis of each composition at the various exposures times out to 1500 h. However, it is interesting to speculate that the delay in the

onset of mass loss (volatilization) would be consistent with phase separation of a SiO<sub>2</sub>-rich constituent, which has been observed after 328 h of exposure of these aluminosilicates under the same conditions[19] as well as in the present case after 500 h (see Fig. 4). The dark phase appearing in the micrograph of Fig. 4 is enriched in silica compared to the starting phases in the BAS: EPMA yielded a composition of 5.5Ba-5.4Al-24.4Si-64.4O at.%, for this Si-enriched, Al-depleted phase while the matrix phase retained its as-processed composition. Presumably, based on what is observed gravimetrically (Fig. 3), localized enrichment of SiO<sub>2</sub> by this phase separation, which appeared to be greatest for BAS, would precede significant volatilization since recession of the aluminosilicates will occur due to preferential reaction of silica from the oxide. Furthermore, if the phase separation was sluggish relative to the rate of silica volatilization, the rate of mass loss would then decline with time and this may explain the initial higher mass loss rates of the aluminosilicates observed between 500 and 1000 h. Again, a mechanistic understanding of the oxidation processes with time in these materials depends on the completion of further microstructural analyses following additional experiments.

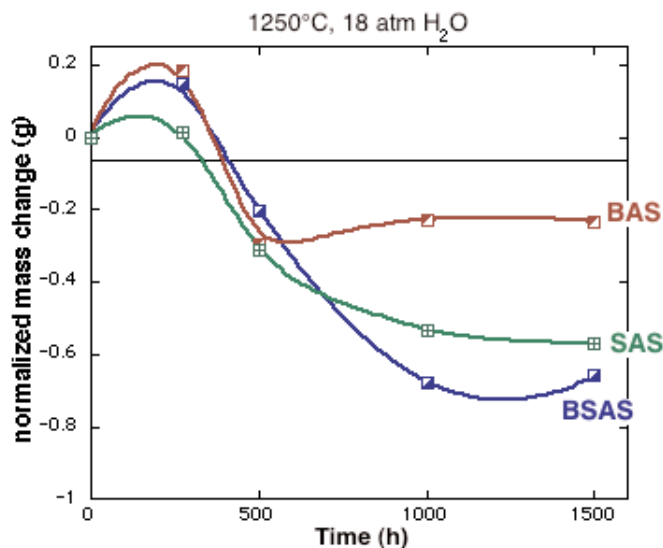


Fig. 3. Gravimetric results for exposure of bulk specimens to 18 atm of H<sub>2</sub>O at 1250°C. Subset of data shown in Fig. 2.

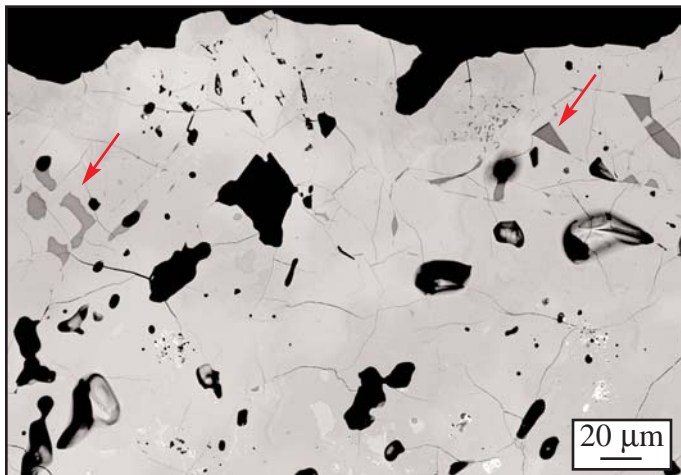


Fig. 4. Surface of BAS coupon after 500 h exposure at 1250°C at 18 atm of H<sub>2</sub>O. Dark second phase (as indicated by arrows) is enriched in silica and was not present in starting material.

## SUMMARY

A high-temperature furnace with a water-vapor pressure of 18 atm was used to examine whether very high-gas pressures can compensate for the low-gas velocities so as to conduct first-stage screening of EBC compositions for volatility resistance. Bulk specimens of barium-strontium aluminosilicate, barium aluminosilicate, and strontium aluminosilicate, SiO<sub>2</sub>, and CVD SiC were exposed at 1250°C in 90% H<sub>2</sub>O-10% air at a total pressure of 20 atm. The aluminosilicates are known to be significantly more volatilization resistant in water-bearing environments than Si-based materials that form SiO<sub>2</sub> under these conditions and thus provided an adequate spread in susceptibility compared to quartz and CVD SiC to evaluate the proposed approach. The gravimetric results showed clear differentiation in volatilization resistance: all three aluminosilicates exhibited similar kinetic behavior and had significantly lower rates of mass losses than the SiO<sub>2</sub> and SiC. As such, these findings provided the experimental proof-of-principle for using simple high-pressure, low-gas-velocity exposures for qualitative evaluation of volatilization resistance among different candidate materials being developed for EBC applications. Additional experimental work, including extensive microstructural and microchemical analyses, will be required to fully elucidate the degradation/volatilization mechanisms for candidate oxide-based EBC compositions.

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