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Thermal Conductivity and Stability of HfO_2-Y_2O_3 and $La_2Zr_2O_7$ Evaluated for 1650 °C Thermal/Environmental Barrier Coating Applications

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THERMAL CONDUCTIVITY AND STABILITY OF HFNIA- AND ZIRCONATE-BASED MATERIALS FOR ADVANCED 1650 °C THERMAL/ENVIRONMENTAL BARRIER COATING APPLICATIONS

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ABSTRACT

HfO_2-Y_2O_3 and $La_2Zr_2O_7$ are candidate thermal and environmental barrier coating (T/EBC) materials for gas turbine ceramic matrix composite (CMC) combustor applications because of their relatively low thermal conductivity and high temperature capability. In this paper, thermal conductivity and high temperature stability of hot-pressed and plasma-sprayed specimens with representative partially-stabilized and fully-cubic HfO_2-Y_2O_3 compositions and $La_2Zr_2O_7$ were evaluated at temperatures up to 1700 °C using a steady-state laser heat-flux technique. Sintering behavior of the plasma-sprayed coatings was determined by monitoring the thermal conductivity increases during a 20-hour test period at various temperatures. Durability and failure mechanisms of the HfO_2-Y_2O_3 and $La_2Zr_2O_7$ coatings on mullite/SiC hexoloy or SiC/SiC CMC substrates were investigated at 1650 °C under thermal gradient cyclic conditions. Coating design and testing issues for the 1650 °C thermal/environmental barrier coating applications are also discussed.

INTRODUCTION

Thermal and environmental barrier coatings (T/EBCs) will play a crucial role in advanced gas turbine engine systems because of their ability to significantly increase engine operating temperature and reduce cooling, thus greatly help to achieve engine low emission and high efficiency goals. In particular, T/EBCs are being developed for low emission SiC/SiC ceramic matrix composite (CMC) combustor applications by extending the CMC liner and vane temperature capability to 1650 °C (3000 °F) in oxidizing and water vapor containing combustion environments. In order to achieve the engine design and performance goals, the coating systems under development must provide vital thermal and environmental protections for the Si-based components at the gas temperatures exceeding 1760 °C (3200 °F) in the harsh combustion environments, while maintaining the CMC substrate below its temperature limit of 1316 °C (2400 °F). The advanced 1650 °C T/EBC system is required to have significantly increased phase stability, lower lattice and radiation thermal conductivity, and improved sintering and thermal stress resistance under the engine high-heat-flux and severe thermal cycling conditions. Advanced heat-flux testing facilities [1–3] and approaches [3–7] have been established for the 1650 °C coating development. The simulated combustion water-vapor environment is also being incorporated into the heat-flux test capabilities [3].

In order to develop a T/EBC system for the 1650 °C Si-based CMCs, a multi-functional coating concept may be considered. Figure 1 shows an advanced coating design concept for the 1650 °C T/EBC system for CMC combustor applications. The top layer is a high-temperature capability ceramic thermal barrier coating. This top coating layer will provide the major thermal protection for the sub-coating systems and CMC substrate, and also act as the first-stage radiation barrier by reducing the transmission of the infrared thermal radiation from the combustion gas environment and form the higher temperature coating surface. The second layer is an energy dissipation layer that will act as a “bond coat” to tailor the thermal expansion mismatch and reduce the thermal stress of the coating system. The third layer is a secondary radiation barrier and thermal control layer. The sophisticated coating compositions and structures will provide combined high reflectivity and diffusion barrier functions. The fourth layer will be an improved stability environmental barrier coating system for the CMC substrate.

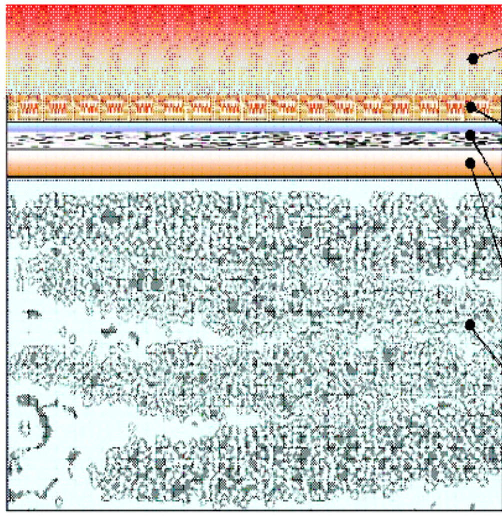

Figure 1.—An advanced coating design concept for the 1650 °C T/EBC system for CMC combustor applications.

The purpose of this paper is to investigate the thermal conductivity and stability of candidate T/EBC top-layer materials, HfO_2-Y_2O_3 and $La_2Zr_2O_7$, under high temperature thermal gradient conditions using a steady-state heat-flux test approach. Both the hot-pressed and plasma-sprayed HfO_2-Y_2O_3 and $La_2Zr_2O_7$ were investigated. The emphasis is placed on the lattice and radiation thermal conductivity, sintering, and cyclic durability of the HfO_2-Y_2O_3 and $La_2Zr_2O_7$ under the 1650 °C thermal gradient test conditions. The temperature capability of HfO_2-Y_2O_3 (with the Y_2O_3 dopant concentration ranging from 5 to 25 mol%) and $La_2Zr_2O_7$ systems on thermal conductivity and the phase stability is evaluated. Coating design and testing issues for the 1650 °C thermal/environmental barrier coating applications will also be discussed.

MATERIALS AND EXPERIMENTAL METHODS

Materials

Thermal conductivity of five HfO_2-Y_2O_3 compositions, i.e., HfO_2 doped with 5, 10, 15, 20, and 25 mol%Y_2O_3, respectively, and $La_2Zr_2O_7$ were investigated at high temperatures using a steady-state laser heat flux test technique. The test specimens were either hot-pressed disks (25.4 mm in diameter, 2-4 mm in thickness), or plasma-sprayed coatings (254-400 μm in thickness). The hot-pressed specimens were fabricated at 1500 °C for an hour under 30 MPa pressure primarily by using spray-dried powders. For the $La_2Zr_2O_7$ material, sol-gel derived, fine particle sized powder was also used to make very high density hot-pressed specimens.

The coating specimens were prepared by plasma-spraying the spray-dried, and plasma-reacted powders on either metallic or CMC substrates. For thermal conductivity measurements, the coating specimens with the low-pressure-plasma-sprayed NiCrAlY bond coated René N5 nickel-base superalloy substrates (25.4 mm in diameter and 3.2 mm in thickness) were used. On the other hand, for sintering and 1650 °C cyclic durability testing, complete T/EBC coating systems (including the top oxide coating layer + the mullite/Si or mullite+BSAS/Si) were prepared with either the melt-infiltrated (MI) SiC/SiC CMC (25.4 mm in diameter, 2.2 mm in thickness) or Hexoloy SiC (25.4 mm in diameter, 3.17 mm in thickness) substrates. The phase structures of the coating materials were examined using X-ray diffraction.

Thermal Conductivity Testing

Thermal conductivity testing of the HfO_2-Y_2O_3 and $La_2Zr_2O_7$ hot-pressed and coating specimens was carried out using a 3 kW CO_2 laser (wavelength 10.6 μm) high-heat flux rig. A schematic diagram of the test rig is illustrated in

Figure 2. The general test approaches have been described elsewhere [1–4]. In this steady-state laser heat-flux thermal conductivity test, the specimen surface heating was provided by the laser beam, and backside air cooling was used to maintain the desired specimen temperatures. A uniform laser heat-flux was obtained over the 23.9 mm diameter aperture region of the specimen surface by using a beam integrating ZnSe lens with either the specimen or the lens rotation. Platinum wire flat coils (wire diameter 0.38 mm) were used to form thin air gaps between the top aluminum aperture plate and stainless-steel back plate to minimize the specimen heat losses through the fixture. The thermal conductivity of the hot-pressed and coating specimens can be determined from the pass-through heat flux and measured temperature difference through the ceramic specimen (or the ceramic coating) thickness under the steady-state laser heating or thermal cycling conditions by a one-dimensional (one-D) heat transfer model.

RESULTS AND DISCUSSION

Thermal conductivity of hot-pressed specimens

HfO_2-Y_2O_3

Figure 3 shows a typical thermal conductivity measurement for the hot-pressed specimens. As can be seen in the example of the HfO_2-20mol%Y_2O_3 coating case, the specimen pass-through heat flux was first slowly increased and then decreased during a heating and cooling cycle by programming the delivered laser power. During the test, the heat flux and cooling can be adjusted to control the temperature difference across the specimen for the conductivity measurements. The corresponding specimen temperature changes, and the temperature difference across the specimen were continuously recorded under given heat flux and cooling conditions. The specimen thermal conductivity was thus obtained as a function of time and temperature.

Figure 4 shows the temperature dependence of the thermal conductivity for the HfO_2-Y_2O_3 hot-pressed specimens. As expected, the HfO_2 materials generally showed the conductivity decrease with increase in temperature. In addition, the thermal conductivity typically showed less temperature dependence with a higher Y_2O_3 dopant content. For the specimens tested at the surface temperature above 1400-1450 °C (such as in the cases of ZrO_2-20mol%Y_2O_3 and ZrO_2-25mol%Y_2O_3), slight conductivity increases were observed when the temperature increases. The conductivity increases with temperature in the high temperature regime can be attributed to the increased radiation heat fluxes, which will result in the increased apparent thermal conductivity [3].

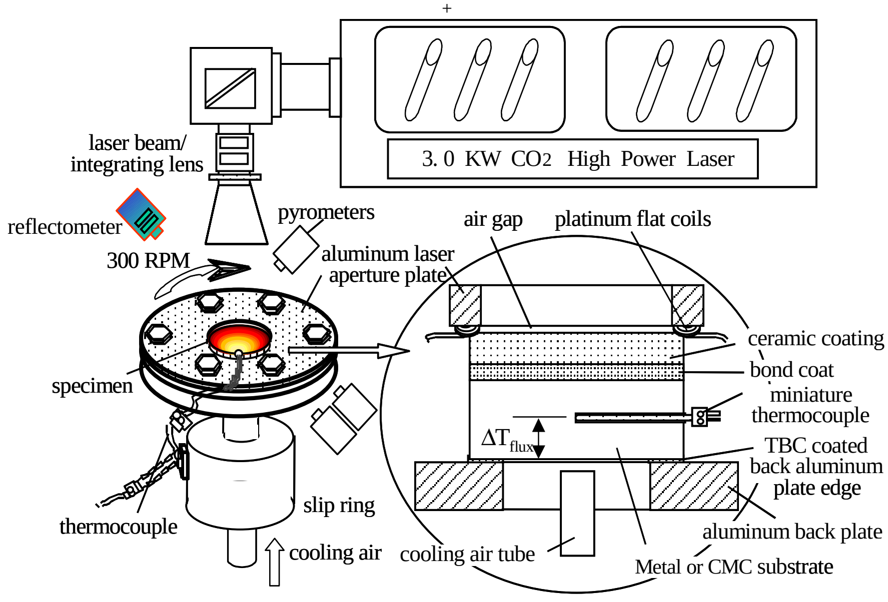

Figure 2.—Schematic diagram showing the laser high-heat-flux rig for determining thermal conductivity and cyclic durability of T/EBC materials. During the test, the ceramic surface and the substrate backside temperatures are measured by infrared pyrometers. The thermal conductivity of the ceramics can be determined from the pass-through heat flux and measured temperature difference through the ceramic specimen (or the ceramic coating) thickness under the steady-state laser heating or thermal cycling conditions by a one-dimensional (one-D) heat transfer model.

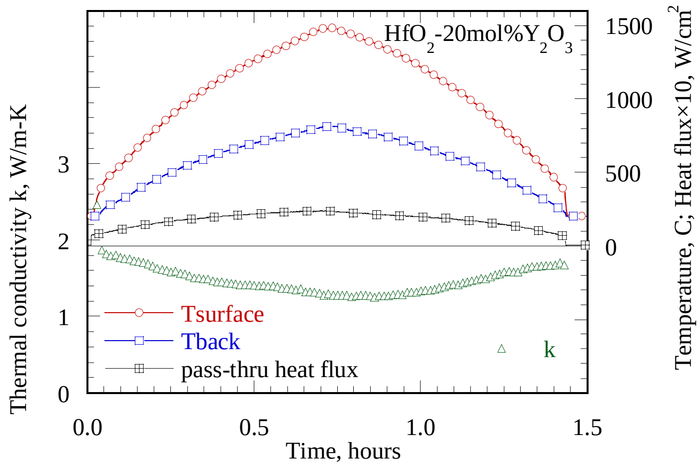

Figure 3.—Typical thermal conductivity measurement for the hot-pressed specimens. During test, the corresponding specimen temperature changes, and the temperature difference across the specimen are continuously recorded. The specimen thermal conductivity is thus obtained as a function of time and temperature.

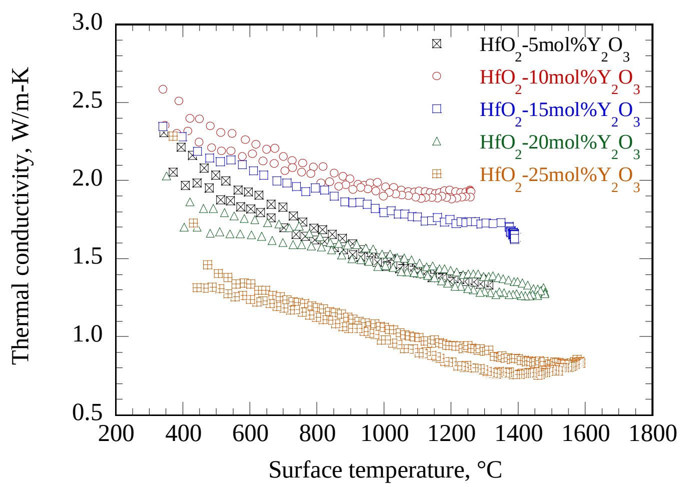

Figure 4.—Thermal conductivity of HfO_2-Y_2O_3 hot-pressed specimens as a function of temperature.

Figure 5 shows measured density and thermal conductivity at 1400 °C of the HfO_2 hot-pressed specimens as a function of Y_2O_3 concentration. The density of the HfO_2 specimens was relatively low (i.e., the porosity was relatively high) due to the relatively large particle size used in this study. The conductivity data scatter was also observed due to the variation in porosity in these hot-processed specimens, with the lower density specimens having lower measured conductivity. Nevertheless, the specimen thermal conductivity showed a general trend to decrease with increase in the yttria-dopant concentration.

$La_2Zr_2O_7$

Figure 6 shows thermal conductivity of $La_2Zr_2O_7$ hot-pressed specimens as a function of surface test temperature. The $La_2Zr_2O_7$ specimens had very low porosity due to the small particle sizes of $La_2Zr_2O_7$ powder used. In particular, the specimens fabricated with the sol-gel method derived powder achieved the theoretical density. These high density, low porosity $La_2Zr_2O_7$ specimens showed significant thermal conductivity increases starting at the temperature of 900 °C because of the increased radiation contribution with increasing temperature. The small amount of micro-porosity in the spray-dried powder processed specimen did not affect the lattice thermal conductivity dramatically. However, the micro-porosity in the specimen significantly reduce the radiation conductivity as compared to the almost pore-free, sol-gel powder processed specimens. The conductivity can increase by more than 100% for the very dense specimens because of the increased radiation heat-transfer under thermal gradient conditions.

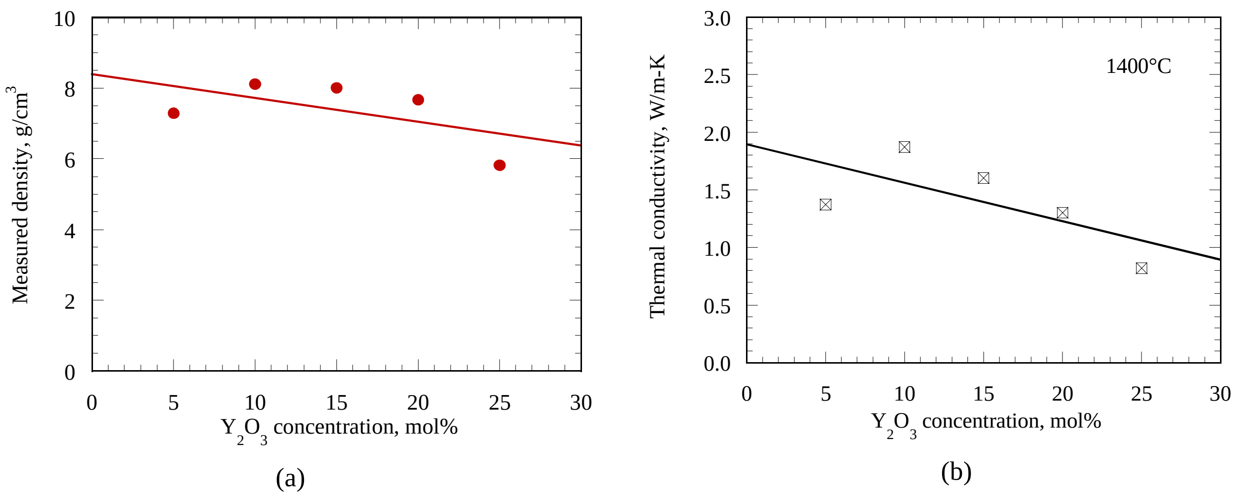

Figure 5.—The measured density (a) and thermal conductivity at 1400 °C (b) of the HfO_2 hot-pressed specimens as a function of Y_2O_3 concentration.

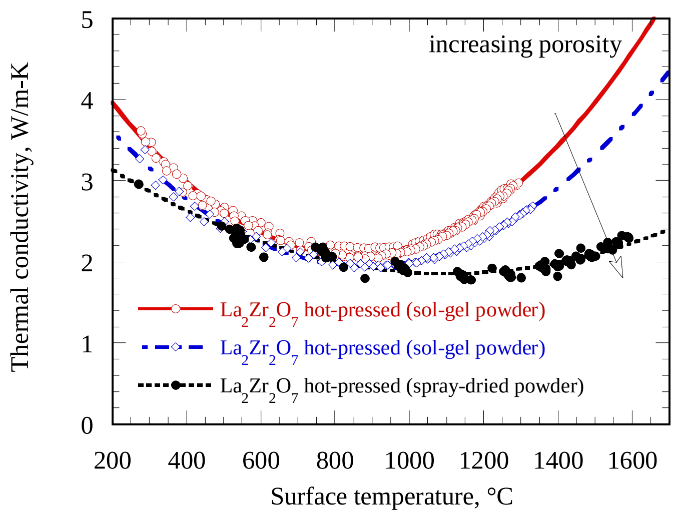

Figure 6.—Thermal conductivity of dense $La_2Zr_2O_7$ hot-pressed specimens as a function of surface test temperature. The conductivity can increase by more than 100% at high temperature for the very dense, sol-gel powder processed specimens because of the increased radiation heat-transfer under thermal gradient conditions.

Thermal conductivity of plasma-sprayed specimens

HfO_2-based coatings

Figure 7 shows the thermal conductivity change kinetics of plasma-sprayed HfO_2-Y_2O_3 coatings as a function of temperature tested at 1650 °C with the pass-thru heat flux in the range of 95 to 100 W/cm^2. It can be seen that HfO_2-5mol%Y_2O_3 (5YSHf) had significant conductivity increase upon the 1650 °C thermal exposure even after a couple of hours testing. On the other hand, the HfO_2-15mol%Y_2O_3 (15YSHf) and HfO_2-25mol%Y_2O_3 (25YSHf) coatings showed lower initial and 20-hour sintered thermal conductivity, indicating the better temperature stability of the higher dopant coatings. The X-ray diffraction results showed that the as-sprayed 5YSHf coating initially had partially-stabilized tetragonal phase structure with a small amount of the monoclinic phase (2-3 mol%). The as-sprayed 15YSHf and 25YSHf had stabilized cubic structure. The monoclinic phase content in the 5YSHf coating increased to 10-12 mol% after the 1650 °C testing. Therefore, the conductivity increase for the 5YSHf coating is not only due to the significant sintering of the coating, but also due to the increased amount of the higher conductivity monoclinic phase in the coating.

Figure 8 shows the initial and 20-hour sintering thermal conductivity of the HfO_2-Y_2O_3 coatings tested at 1650 °C as a function of the Y_2O_3 concentration. It can be seen that thermal conductivity decreases with increase in

Y_2O_3 dopant. HfO_2-5mol%Y_2O_3 (5YSHf) showed the highest conductivity and also conductivity increases after the 1650 °C testing. The more stable cubic HfO_2-15mol%Y_2O_3 (5YSHf) and HfO_2-25mol%Y_2O_3 (25YSHf) showed lower conductivity and less conductivity increase. Advanced multi-component HfO_2 coatings have achieved even lower thermal conductivity and better thermal stability.

Figure 9 shows the 1650 °C sintering and cyclic testing results of HfO_2 baseline coatings (HfO_2-5mol%Y_2O_3 or 5YSHf, and HfO_2-15mol%Y_2O_3 or 15 YSHf), and a multi-component HfO_2 coating, coated on mullite EBC/Si on SiC substrates. The coatings were initially tested under the standard 20 hour steady-state 1650 °C sintering condition to assess the sintering behavior; the coatings were then thermal cyclically tested at 1650 °C to evaluate the cyclic durability. The two HfO_2 baseline coatings showed significant conductivity increases due to the extensive sintering, and later conductivity reductions due to coating cracking and delamination. The 5YSHf showed severe spallation after the testing partially due to the large amount of monoclinic phase formation (>25mol%) [3]. In contrast, the advanced multi-component HfO_2 coating had relatively low conductivity increase during the steady-state testing, indicating its good sintering resistance. The coating also showed essentially no conductivity reduction during thermal cycling, demonstrating its good cyclic durability.

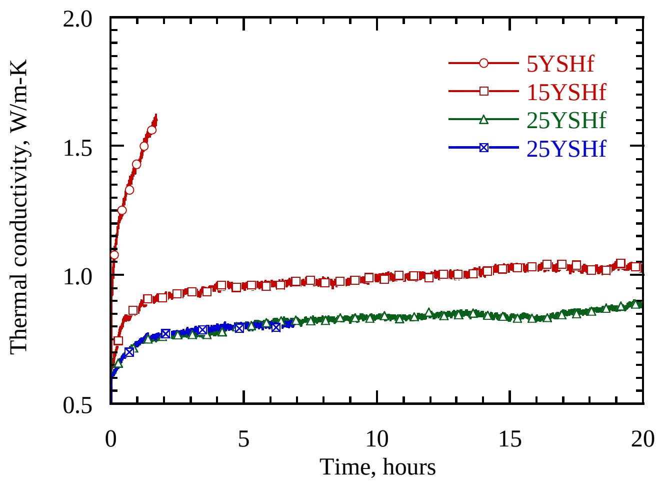

Figure 7.—Thermal conductivity of plasma-sprayed HfO_2-Y_2O_3 coatings tested at 1650 °C as a function of time (test pass-through heat flux 95-100 W/cm^2).

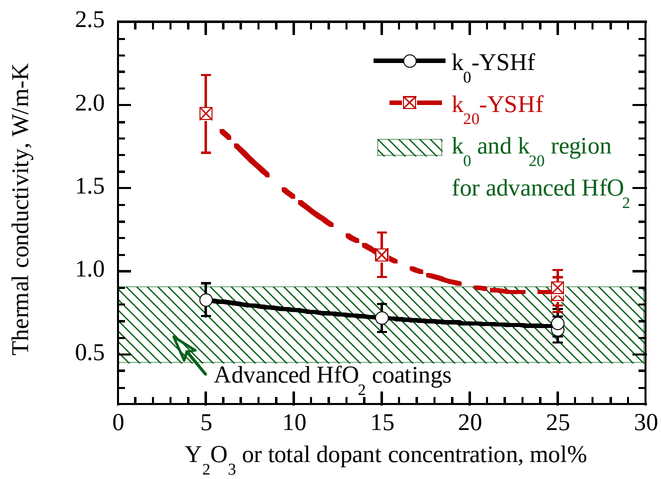

Figure 8.—The initial and 20-hour sintered thermal conductivity of plasma-sprayed HfO_2-Y_2O_3 coatings, tested at 1650 °C with the pass through heat flux 95-100 W/cm^2, as a function of the Y_2O_3 concentration. The k_0 and k_{20} denote the initial and 20 hour sintered thermal conductivity of the HfO_2-Y_2O_3 system. As also indicated in the plot, advanced multi-component HfO_2 coatings have achieved even lower thermal conductivity and better thermal stability.

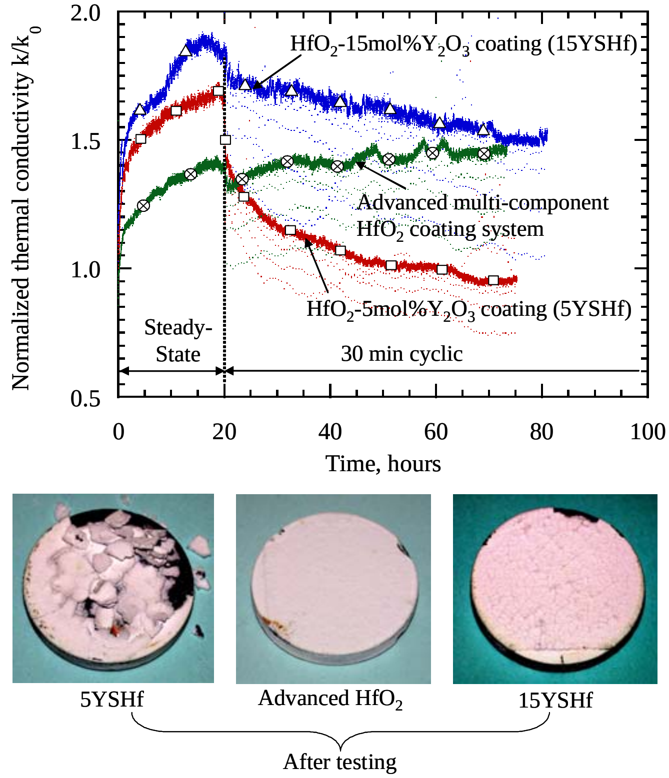

Figure 9.—Sintering and cyclic response of HfO_2 coating systems on mullite EBC/Si on SiC substrates tested at 1650 °C (3000 °F) with the pass-through heat flux of 95-100 W/cm^2. The advanced HfO_2 coating system showed excellent performance after the total 70 hours testing, with low sintering conductivity increase and essentially no delamination induced conductivity reductions.

$La_2Zr_2O_7$ coatings

Plasma-sprayed $La_2Zr_2O_7$ coatings were also tested under laser heat flux conditions. Fig. 10 shows thermal conductivity change kinetics of a plasma-sprayed $La_2Zr_2O_7$ tested at 1575 °C (with the pass-through heat flux of 80 W/cm^2). The $La_2Zr_2O_7$ coating showed significant thermal conductivity increase (the conductivity increased from 0.55 W/m-K to 0.95 W/m-K in 20 hours), indicating the coating was undergoing substantial sintering at the test temperature. Fig. 11 shows the cyclic result of a $La_2Zr_2O_7$ TBC/mullite EBC/Si system on the SiC/SiC substrate at 1450 °C (pass-thru heat flux 50 W/cm^2). The $La_2Zr_2O_7$ coating system demonstrated 150, 30 min hot-time cycles (or 75 hot hours) durability, and had the predominant coating sintering conductivity increase with only minor delamination related conductivity reduction. The coating had no visual damage after the testing. The plasma-sprayed $La_2Zr_2O_7$ /mullite/Si system on the SiC/SiC substrate showed severe spallation upon heating under the higher heat flux (95-100 W/cm^2), 1650 °C test conditions, as shown in Figure 12. The development of advanced pyrochlore coatings is also in progress to further improve the coating toughness, thermal stress resistance and overall performance.

CONCLUSIONS

In this study, a laser sintering-and-cyclic-durability test approach has been demonstrated for advanced 1650 °C (3000 °F) T/EBC development. The test results showed that HfO_2-Y_2O_3 and $La_2Zr_2O_7$ are promising candidate 1650 °C coating materials due to their low thermal conductivity and good sintering resistance. For the HfO_2-Y_2O_3 based coatings, the composition optimization can improve phase stability, and further reduce thermal conductivity. For the pyrochlore oxide coatings, further processing and coating composition modifications are needed in order to improve toughness for the 1650 °C application. The lattice and radiation conductivity of the advanced coating systems was also evaluated using the laser heat flux technique. The results showed that the radiation contribution can become significant at high temperature, especially for very dense materials. The phase stability, and sintering and thermal stress resistance are crucial for coating 1650 °C long-term cyclic durability.

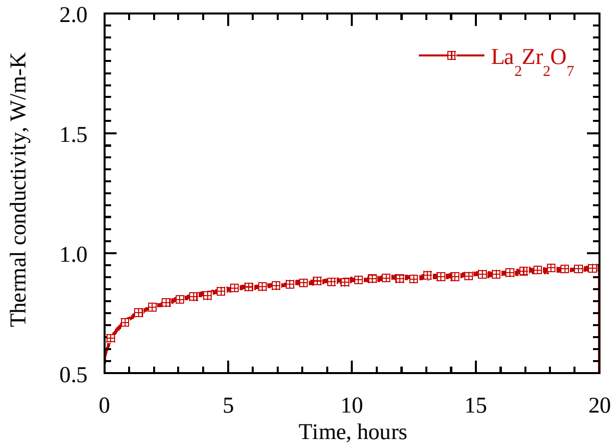

Figure 10.—Thermal conductivity change kinetics of a plasma-sprayed $La_2Zr_2O_7$ coating tested at 1575 °C (with the pass-through heat flux of 80 W/cm^2).

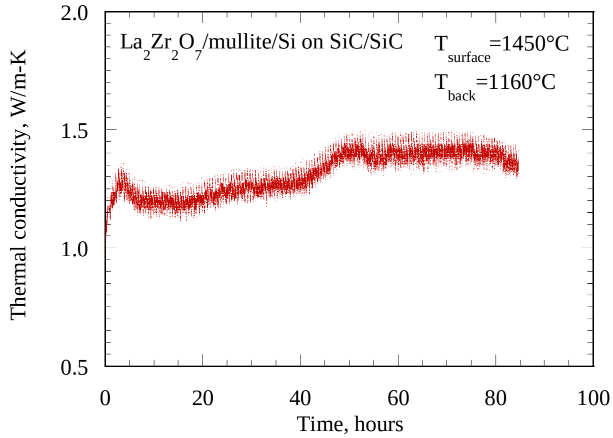

Figure 11.—The cyclic result of a $La_2Zr_2O_7$ TBC/mullite EBC/Si system on the SiC/SiC substrate at 1450 °C (pass-thru heat flux 50 W/cm^2).

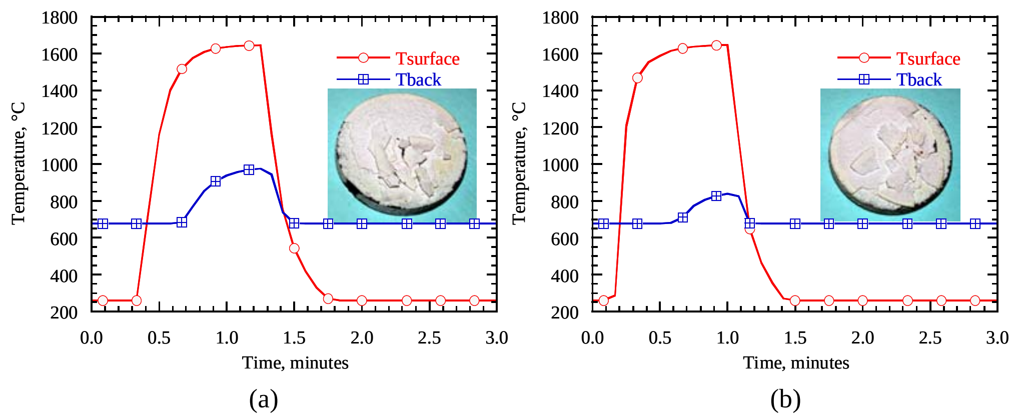

Figure 12.—The plasma-sprayed $La_2Zr_2O_7$/mullite/Si coating system on the SiC substrates showed severe spallation upon heating under the heat flux of 95-100 W/cm^2, 1650 °C test conditions after the 1650 °C exposure. (a) and (b) The temperature profiles and failure morphologies of two similarly processed $La_2Zr_2O_7$ coating specimens.

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13. ABSTRACT ***(Maximum 200 words)***

HfO_2-Y_2O_3 and $La_2Zr_2O_7$ are candidate thermal and environmental barrier coating (T/EBC) materials for gas turbine ceramic matrix composite (CMC) combustor applications because of their relatively low thermal conductivity and high temperature capability. In this paper, thermal conductivity and high temperature stability of hot-pressed and plasma-sprayed specimens with representative partially-stabilized and fully-cubic HfO_2-Y_2O_3 compositions and $La_2Zr_2O_7$ were evaluated at temperatures up to 1700 °C using a steady-state laser heat-flux technique. Sintering behavior of the plasma-sprayed coatings was determined by monitoring the thermal conductivity increases during a 20-hour test period at various temperatures. Durability and failure mechanisms of the HfO_2-Y_2O_3 and $La_2Zr_2O_7$ coatings on mullite/SiC hexoloy or SiC/SiC CMC substrates were investigated at 1650 °C under thermal gradient cyclic conditions. Coating design and testing issues for the 1650 °C thermal/environmental barrier coating applications are also discussed.

14. SUBJECT TERMS

Thermal barrier coating; Environmental barrier coating; Thermal conductivity; Thermal stability; Ceramic matrix composites

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