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Prepared for the
27th International Thermal Conductivity Conference (ITCC27)
sponsored by the High Temperature Materials Laboratory
at Oak Ridge National Laboratory
Knoxville, Tennessee, October 26–29, 2003

National Aeronautics and
Space Administration

Glenn Research Center

Acknowledgments

This work was supported by NASA Ultra-Efficient Engine Technology (UEET) Program. The authors are also grateful to George W. Leissler, QSS Group, Inc., at the NASA Glenn Research Center for his assistance in the preparation of plasma-sprayed thermal barrier coatings, and to Robert W. Bruce at General Electric Aircraft Engines and Kenneth S. Murphy at Howmet Research Corporation for EB-PVD coating processing.

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Abstract

The development of low conductivity and high temperature capable thermal barrier coatings requires advanced testing techniques that can accurately and effectively evaluate coating thermal conductivity under future high-performance and low-emission engine heat-flux conditions. In this paper, a unique steady-state CO₂ laser (wavelength 10.6 μm) heat-flux approach is described for determining the thermal conductivity and conductivity deduced cyclic durability of ceramic thermal and environmental barrier coating systems at very high temperatures (up to 1700 °C) under large thermal gradients. The thermal conductivity behavior of advanced thermal and environmental barrier coatings for metallic and Si-based ceramic matrix composite (CMC) component applications has also been investigated using the laser conductivity approach. The relationships between the lattice and radiation conductivities as a function of heat flux and thermal gradient at high temperatures have been examined for the ceramic coating systems. The steady-state laser heat-flux conductivity approach has been demonstrated as a viable means for the development and life prediction of advanced thermal barrier coatings for future turbine engine applications.

Introduction

Ceramic thermal and environmental barrier coatings (T/EBCs) have received increasing attention for advanced gas turbine engine applications. The advantages of using T/EBCs include potentially higher engine efficiency by increasing gas temperatures, and improved engine reliability by reducing engine hot-section component temperatures. The development of advanced ceramic barrier coatings is aimed at significantly increasing engine operating temperature and simultaneously reducing air cooling, in order to meet future engine low emission, high efficiency and improved reliability goals. The future ceramic coating systems must be designed with increased high temperature stability, lower thermal conductivity, and improved thermal stress and erosion resistance.

Advanced low conductivity and high temperature capable T/EBC development requires testing techniques that can accurately and effectively evaluate coating thermal conductivity and stability under expected engine high-heat-flux and thermal cycling conditions. In this paper, a unique steady-state CO₂ laser (wavelength 10.6 μm) heat-flux approach is described for determining the thermal conductivity and the conductivity-deduced cyclic durability of T/EBC systems at high temperatures under large thermal gradients within the coating that are encountered in advanced engine systems. The laser heat-flux thermal conductivity approach emphasizes the real-time monitoring and assessment of the coating thermal conductivity under steady-state, cyclic, and engine-like heat-flux thermal gradient conditions. The

conductivity increases due to coating sintering and the conductivity decreases due to coating delamination have been demonstrated.

The thermal conductivity behavior of advanced thermal and environmental barrier coatings has also been investigated using the laser conductivity approach for metallic turbine airfoil and Si-based ceramic matrix composites (CMC) combustor liner and vane applications. The coating internal and external radiation flux resistance has been evaluated by measuring the apparent coating conductivity response under large coating internal thermal gradients, and by using a laser-heated external radiative-flux approach, respectively. The relationships between the lattice and radiation conductivities as a function of heat flux and thermal gradient at high temperatures have been examined for the ceramic coating systems.

Laser Steady-State Heat Flux Thermal Conductivity Measurement Technique

The CO₂ laser (wavelength 10.6 μm) can efficiently deliver well-characterized and well-controlled heat energy to the material surface to investigate the thermal fatigue and sintering behavior of ceramic thermal barrier coatings under simulated engine heat-flux conditions [1–3]. The steady-state CO₂ laser heat-flux thermal conductivity technique is developed based on laser thermal fatigue testing, but the modified test capability, using a uniform laser heat-flux distribution and continuous real-time monitoring of both the front heating surface and the backside surface temperatures, allows one to characterize the temperature difference across the ceramic coating system or monolithic ceramic specimen under a given delivered laser heat flux [4–5].

A schematic diagram showing the laser thermal conductivity rig is given in figure 1. This test rig consists of a high power laser system, 3.0 to 3.5 kW CO₂ continuous-wave laser, a motor-driven rotating test station, and temperature measurement instruments such as a thermography system and infrared pyrometers. In the laser steady-state heat-flux thermal conductivity test, the specimen surface heating was

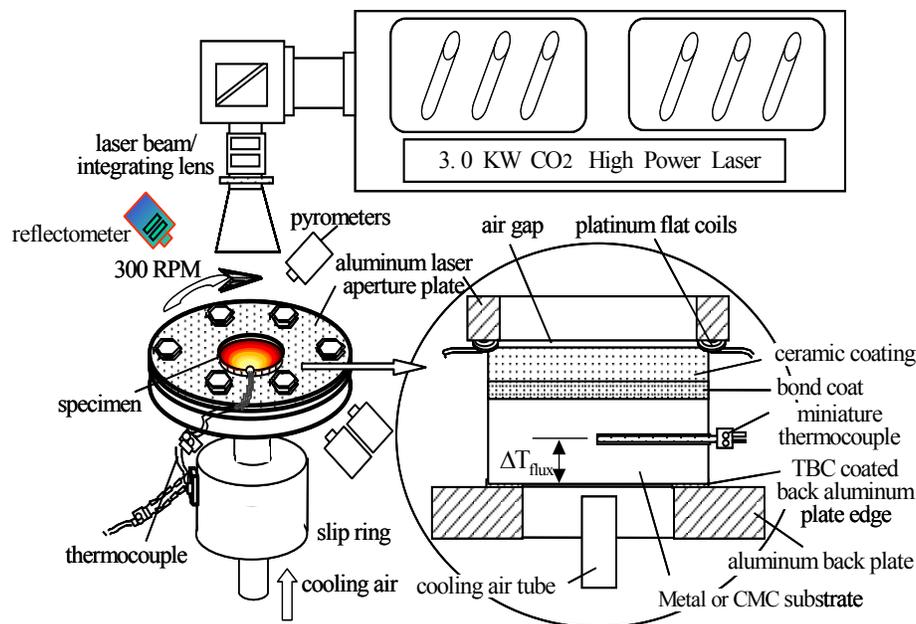


Figure 1.—Schematic diagram showing the laser heat-flux rig for determining thermal conductivity of thermal and environmental barrier coatings or coating materials. During the test, the ceramic surface and the metal backside temperatures are measured by infrared pyrometers. The metal substrate mid-point temperature can be obtained by an optional, embedded miniature type-K thermocouple. The interfacial temperatures, and the actual heat-flux passing through the thermal barrier coating system, are therefore determined under the steady-state laser heating conditions by a one-dimensional (one-D) heat transfer model.

provided by the laser beam, and backside air cooling was used to maintain the desired specimen temperatures. The laser surface heating and the backside air cooling determine appropriate steady-state temperature gradients across the coating systems. A uniform laser heat flux was obtained over the 23.9 mm diameter aperture region of the specimen surface by using an integrating ZnSe lens combined with the specimen or laser beam rotation. Platinum wire coils (wire diameter 0.38 mm) were used to form 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 $k_{ceramic}$ of ceramic materials can be determined from the pass-through heat flux q_{thru} and measured temperature difference $\Delta T_{ceramic}$ across the ceramic specimen (or the ceramic coating) thickness $l_{ceramic}$ under the steady-state laser heating conditions

$$k_{ceramic} = q_{thru} \cdot l_{ceramic} / \Delta T_{ceramic} \quad (1)$$

The actual pass-through heat flux q_{thru} for a given ceramic specimen can be obtained by subtracting the laser reflection loss (measured by a 10.6 μm reflectometer) and the radiation heat loss at the ceramic coating surface from the laser delivered heat-flux. Note that the non-reflected laser energy is absorbed at the specimen surfaces because of the high emissivity and absorption values at the 10.6 μm laser wavelength region for the oxides and silicates typically used as thermal and environmental barrier coatings at high temperatures [6]. The pass-through heat flux q_{thru} was also verified with an internal heat flux gauge within the substrates (instrumented specimens) via an embedded miniature thermocouple. For the hot pressed bulk specimens, the temperature difference $\Delta T_{ceramic}$ in the ceramic was directly measured by using surface and backside pyrometers. For the coating specimens, the temperature difference in the ceramic coating $\Delta T_{ceramic}$ was obtained from the measured coating surface and substrate backside temperatures using an 8 μm pyrometer and a two-color pyrometer, respectively, and subtracting the temperature drops in the substrate and bond coat

$$\Delta T_{ceramic} = T_{ceramic-surface} - T_{substrate-back} - \int_0^{l_{bond}} \frac{q_{thru} \cdot dl}{k_{bond}(T)} - \int_0^{l_{substrate}} \frac{q_{thru} \cdot dl}{k_{substrate}(T)} \quad (2)$$

where $T_{ceramic-surface}$ and $T_{substrate-back}$ are measured ceramic surface and substrate backside temperatures, l_{bond} , $l_{substrate}$, and $k_{bond}(T)$ and $k_{substrate}(T)$ are the thicknesses and the temperature-dependent thermal conductivity of the bond coat and substrate, respectively.

Thermal Conductivity of ZrO₂-8wt%Y₂O₃ Thermal Barrier Coatings

Thermal Conductivity Increase Due to Sintering

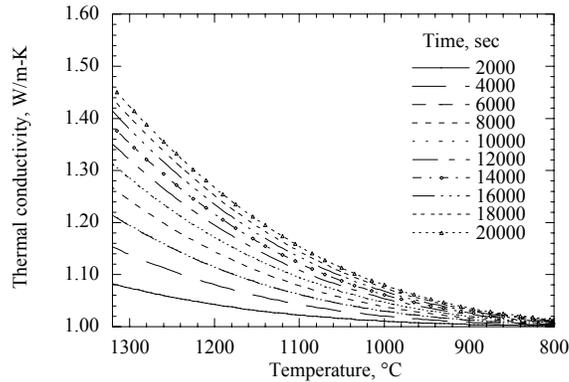
The coating thermal conductivity kinetics of plasma-sprayed ZrO₂-8wt%Y₂O₃ thermal barrier coatings have been determined using the steady-state laser heat-flux testing under a fixed heat flux (64W/cm²). Because a thermal conductivity gradient is expected across the ceramic coating under the high thermal gradient conditions (due to the more rapid thermal conductivity increase near the ceramic surface as compared to near the ceramic/bond coat interface), the observed ceramic thermal conductivity increase reflects an overall effect of the conductivity change in the coating. The coating shows faster initial conductivity increases, presumably due to the fast microcrack sintering rate at the initial stage. After long-term testing, the coating conductivity seems to reach saturation conductivity values. The experimental coating thermal conductivity change kinetics for the ZrO₂-8wt%Y₂O₃ can be expressed as [7]

$$\frac{k_c - k_c^0}{k_c^{\text{inf}} - k_c^0} = 102.2 \cdot \exp\left(-\frac{68228}{RT}\right) \left\{1 - \exp\left[-\frac{t}{\tau}\right]\right\} \quad (3a)$$

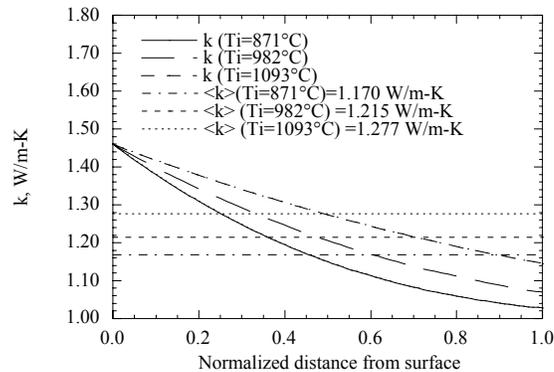
$$\tau = 572.5 \cdot \exp\left(\frac{41710}{RT}\right) \quad (3b)$$

where k_c is the coating thermal conductivity at any given time t , k_c^0 and k_c^{inf} are ceramic coating thermal conductivity values at the initial time and at infinitely long time, respectively, R is gas constant, and τ is relaxation time.

From the temperature dependence of the thermal conductivity change kinetics, the thermal conductivity gradients in the coatings as a function of time can be derived. Typical thermal conductivity distributions in a plasma-sprayed $\text{ZrO}_2\text{-8wt\%Y}_2\text{O}_3$ thermal barrier coating, modeled based on the laser heat flux thermal conductivity test data, are given in figure 2. It can be seen that the conductivity increase near the surface is more significant than that near the interface under the thermal gradient testing conditions.



(a)



(b)

Figure 2.—Thermal conductivity distributions in a plasma-sprayed $\text{ZrO}_2\text{-8wt\%Y}_2\text{O}_3$ thermal barrier coating, modeled based on the laser heat flux thermal conductivity test data. (a) The ceramic thermal conductivity as a function of temperature and time; (b) Thermal conductivity gradients and corresponding average thermal conductivity values $\langle k \rangle$ in the ceramic coating tested at three different interface temperatures after 20000 seconds (surface temperature $T_s = 1315.5^\circ\text{C}$; interface temperatures T_i are 871, 982, and 1093 °C, respectively).

Thermal Conductivity Reduction Due to Coating Delamination

Figure 3 shows thermal conductivity changes of a plasma-sprayed $\text{ZrO}_2\text{-}8\text{wt}\%\text{Y}_2\text{O}_3$ coating under a combined steady-state and cyclic laser heat-flux test conditions, measured in-situ by the laser heat-flux technique. As expected, the coating conductivity increases with time due to coating sintering during the steady-state testing. However, upon the cyclic testing, the coating conductivity continuously decreases with time. The conductivity reduction indicates the accumulated damage with the cycle number until the final coating spallation. In this particular test, the large initial conductivity drop may suggest that significant coating delamination occurred after the first sintering cycle, which may have accumulated the highest strain energy after the long test cycle.

The laser thermal fatigue and crack propagation study can also be based on a modified laser thermal conductivity test for a pre-center-penny-shape cracked coating specimen. Figure 4 shows typical test results for a $127\ \mu\text{m}$ thick, pre-cracked TBC specimen under the laser cyclic testing. The surface temperature increases continuously due to the crack initiation and propagation. The metal backside temperature and the predicted metal/ceramic interface temperature, however, remains relatively constant or a slightly decreasing trend. The initial rise in the measured conductivity is attributed to the ceramic sintering effect. It is also noted that a sudden drop in thermal conductivity that is corresponding to a large surface temperature jump, was observed at about 30 hr, due to a coating spallation event which occurred at that time. The coating propagation process, confirmed independently by a high sensitivity video camera, has been used to evaluate the coating thermal fatigue behavior [9].

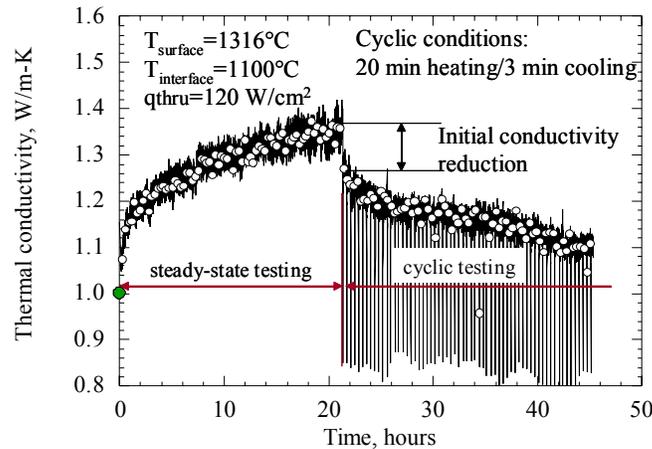


Figure 3.—Thermal conductivity changes of a $127\ \mu\text{m}$ plasma-sprayed $\text{ZrO}_2\text{-}8\text{wt}\%\text{Y}_2\text{O}_3$ coating under a combined steady-state and cyclic laser heat-flux test conditions, measured in-situ by the laser heat-flux technique. The coating showed the thermal conductivity increase due to coating sintering and the coating conductivity decrease due to accumulated delamination (pass-through heat flux $q_{thru} = 120\text{W/m-K}$).

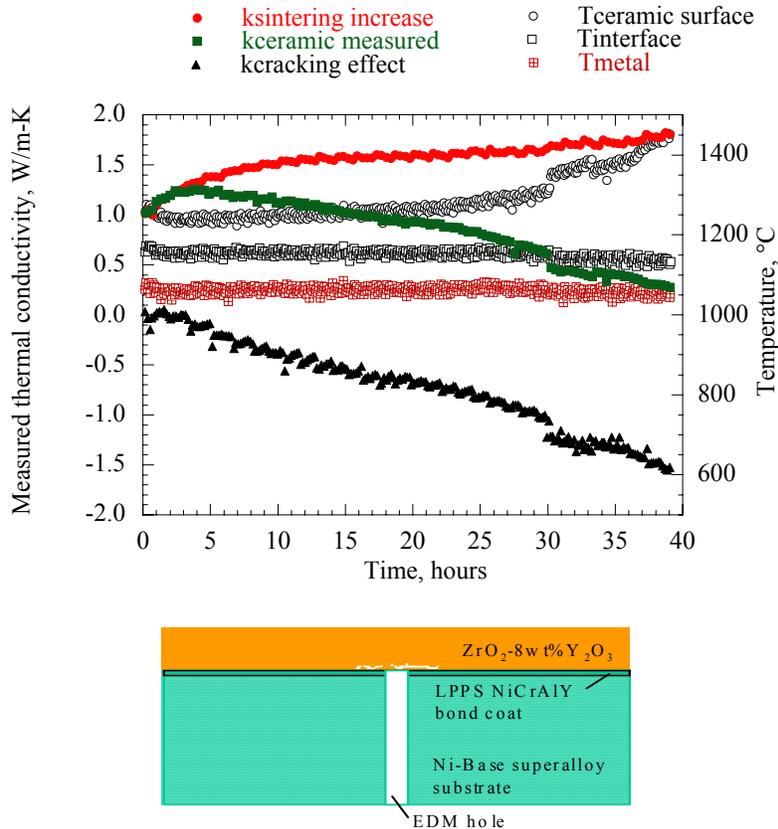


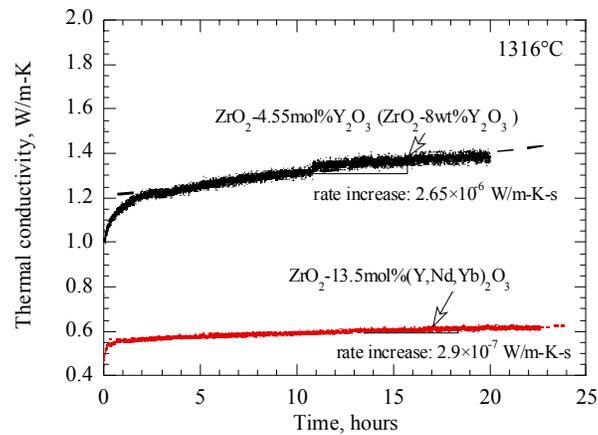
Figure 4.—Laser thermal fatigue test result of a 127 μm thick, pre-center penny-shape-cracked (diameter about 1 mm) coating specimen showing the coating temperature and thermal conductivity changes as a function of cycle number under 10 min heating and 2 min cooling laser cycling. The ceramic surface temperature increases and the metal backside temperature slightly decreases as the delamination crack is propagated. The effective ceramic coating conductivity shows an initial increase due to the coating sintering, and then a monotonic decrease due to the crack propagation. The insert is a schematic diagram showing the coating specimen.

Advanced Thermal Barrier Coatings Development Using Laser Heat Flux Approaches

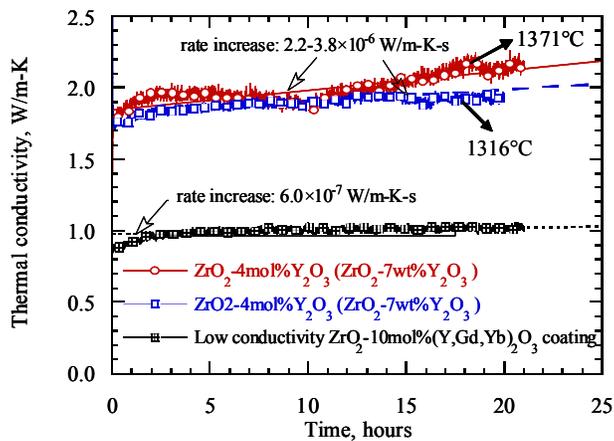
The laser heat-flux testing approach can play a significant role in the development of advanced low conductivity and high thermal stability coatings. Since the coating conductivity increases with time due to ceramic sintering, the coating surface temperature will continuously drop under the fixed laser heat-flux condition. The measured initial coating conductivity (k_0), the conductivity at 20 hr (k_{20}), and the conductivity rate of increase have been used for evaluating the candidate coating performance. It should be mentioned that for some of the EB-PVD oxide coating systems, the coating conductivity after 5 hr testing (k_5) can be used for characterizing the coating behavior, because the EB-PVD coatings can usually reach a steady-state sintering conductivity increase stage after 5 hr of testing.

Figure 5 illustrates typical high temperature thermal conductivity behavior of advanced multi-component plasma-sprayed and EB-PVD oxide cluster thermal barrier coatings as a function of test time, determined by the laser heat-flux technique at temperature of 1316 $^{\circ}\text{C}$. The advanced oxide coatings consist of $\text{ZrO}_2\text{-Y}_2\text{O}_3$, and also are co-doped with additional paired rare earth oxides $\text{Nd}_2\text{O}_3\text{-Yb}_2\text{O}_3$ or $\text{Gd}_2\text{O}_3\text{-Yb}_2\text{O}_3$ (i.e., $\text{ZrO}_2\text{-Y}_2\text{O}_3\text{-Nd}_2\text{O}_3\text{-Yb}_2\text{O}_3$ and $\text{ZrO}_2\text{-Y}_2\text{O}_3\text{-Gd}_2\text{O}_3\text{-Yb}_2\text{O}_3$ coating systems). These

advanced coating systems are found to possess much lower thermal conductivity and better temperature stability than the conventional $\text{ZrO}_2\text{-8wt\%Y}_2\text{O}_3$ coatings [10–12]. From figure 6, it can be seen depending on the test temperature and coating composition, the conductivity reduction of 50 to 66% can be achieved for the advanced coating systems as compared to the $\text{ZrO}_2\text{-8wt\%Y}_2\text{O}_3$ coatings. Both the advanced low conductivity plasma-sprayed and EB-PVD coatings have demonstrated 100 to 200 hr long-term cyclic durability in the temperature range of 1360 to 1540 °C testing temperatures [11].



(a) Plasma-sprayed coating



(b) EB-PVD coating

Figure 5.—Thermal conductivity of advanced multi-component plasma-sprayed (a) and EB-PVD (b) oxide cluster thermal barrier coatings, as compared to determined by laser heat-flux technique.

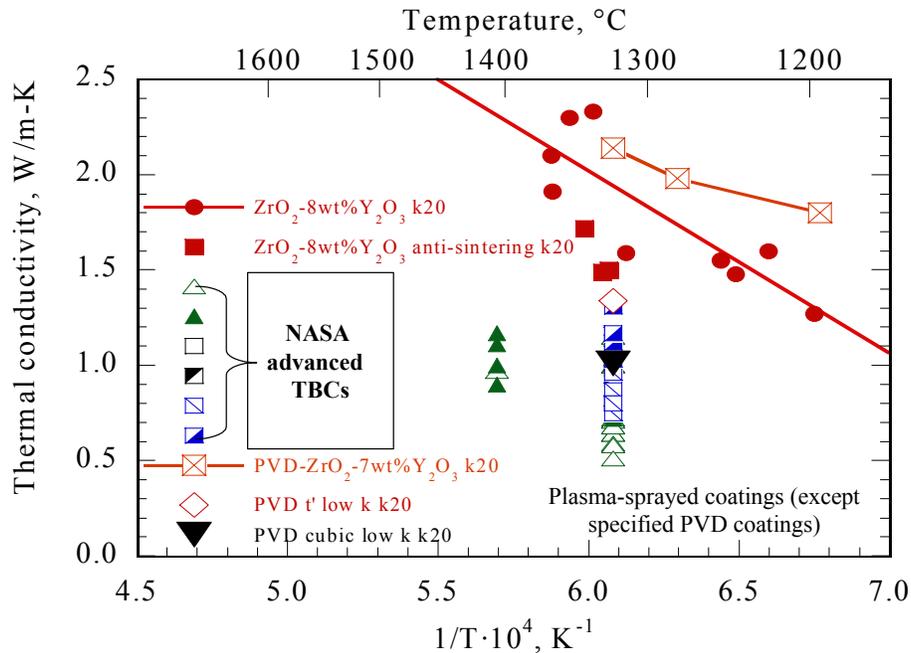


Figure 6.—Thermal conductivity of advanced multi-component, zirconia-based thermal barrier coatings as a function of test temperature after 20 hr testing (k_{20}), determined by the laser heat-flux technique tested at the surface temperature. Depending on the test temperature and coating composition, the conductivity reduction of 50 to 66% can be achieved for the advanced coating systems as compared to the $ZrO_2-8wt\%Y_2O_3$ coatings after the 20hr sintering test.

Advanced Thermal/Environmental Barrier Coating Applications

Advanced T/EBCs are being developed for the low emission SiC/SiC CMC combustor liner and vane applications by extending the component temperature capability to 1650 °C (3000 °F) in oxidizing and water vapor containing combustion environments [8]. The coating system is required to have increased phase stability, lower lattice and radiation thermal conductivity, and improved sintering and thermal stress resistance under the future engine high-heat-flux and thermal cycling conditions. The simulated combustion water-vapor environment is also being incorporated into the laser heat-flux testing capabilities.

Figure 7 shows thermal conductivity and durability of the 1650 °C plasma-sprayed HfO_2 -based thermal/environmental barrier coatings using the laser heat-flux technique. From figure 7(a) it can be seen that the initial and 20-hr sintering thermal conductivity of the $HfO_2-Y_2O_3$ coatings generally decreases with increase in Y_2O_3 dopant content. The more stable cubic phase structured $HfO_2-15mol\%Y_2O_3$ (15YSHf) and $HfO_2-25mol\%Y_2O_3$ (25YSHf) have been shown to have lower conductivity and less conductivity increases as compared to the tetragonal phase structured $HfO_2-5mol\%Y_2O_3$ (5YSHf) [8]. However, the advanced multi-component rare earth doped $HfO_2-Y_2O_3-Gd_2O_3(Nd_2O_3)-Yb_2O_3$ coatings have achieved even lower thermal conductivity and better thermal stability. Figure 7(b) shows the 1650 °C sintering and cyclic behavior of a multi-component $HfO_2-Y_2O_3-Gd_2O_3-Yb_2O_3$ coating that was coated on the mullite-based EBC/Si on SiC substrates. The advanced multi-component HfO_2 coating had relatively low conductivity increase during the first 20hr steady-state testing, and also showed essentially no cracking and delamination during the subsequent 100, 30 min cyclic testing at 1650 °C, indicating its excellent sintering resistance and cyclic durability. The 5YSHf showed severe spallation partially due to the large amount of monoclinic phase formation (>25 mol%) due to the phase destabilization [8].

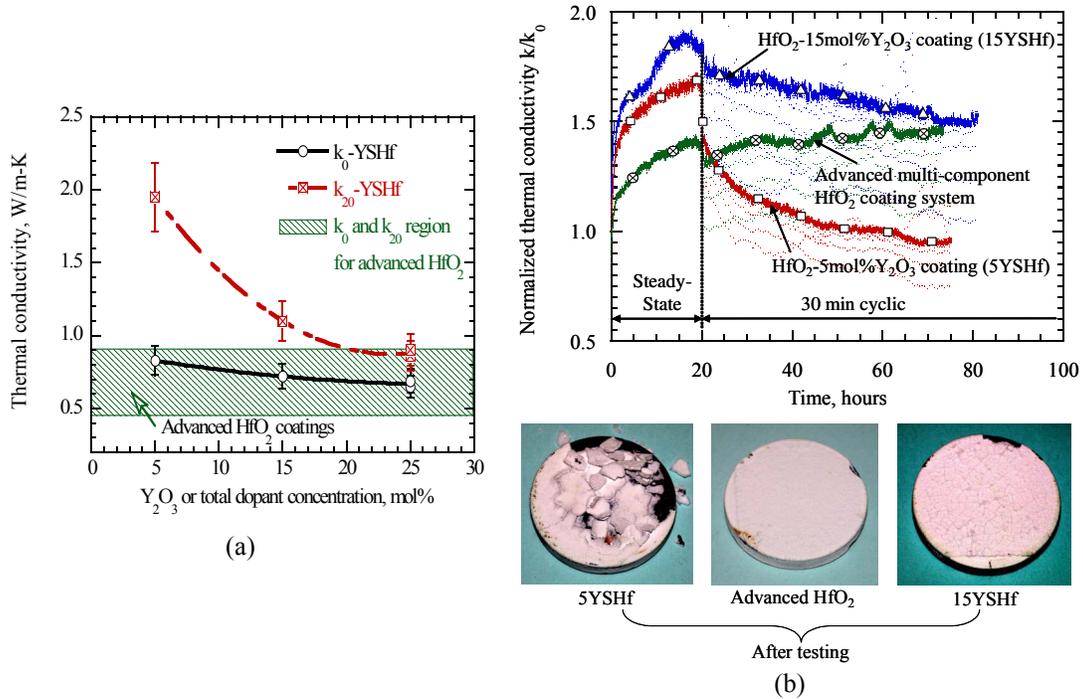


Figure 7.—Thermal conductivity and durability evaluation using the laser heat flux technique for 1650 °C thermal and environmental barrier coatings. (a) The initial and 20-hr sintered thermal conductivity of plasma-sprayed HfO₂-Y₂O₃ coatings, tested at 1650 °C with the pass through heat flux 95 to 100 W/cm², as a function of the Y₂O₃ concentration. The k_0 and k_{20} denote the initial and 20 hr sintered thermal conductivity of the HfO₂-Y₂O₃ system. As also indicated in the plot, advanced multi-component HfO₂ coatings have achieved even lower thermal conductivity and better thermal stability. (b) The 1650 °C sintering and cyclic behavior of a multi-component HfO₂-Y₂O₃-Gd₂O₃-Yb₂O₃ coating that coated on the mullite-based EBC/Si on SiC substrates, as compared to the baseline 5YSHf and 15YSHf coatings.

Figure 8 shows the internal radiation conductivity component of the ZrO₂-based coatings and monolithic hot-pressed ceramic specimens, determined by the laser heat-flux technique, as a function of the imposed thermal gradient (and thus heat flux) and surface temperature. The internal radiation component is defined as the ratio of the radiation thermal conductivity $k_{radiation}$ ($k_{radiation} = k_{apparent} - k_{lattice}$, where $k_{apparent}$ is the measured apparent conductivity and $k_{lattice}$ is the lattice conductivity, respectively) to the lattice thermal conductivity $k_{lattice}$. The radiation conductivity contribution generally increases with increasing the thermal gradient and surface temperature. The dense hot-pressed specimens showed significantly higher internal radiation conductivity as compared to the plasma-sprayed coatings due to the lack of the micro-crack and micro-porosity scattering effect in these dense materials.

Figure 9 shows the external radiation flux resistance $\ln(q_{rad}/q_{rad0})$, defined as the ratio of the pass-through radiation heat-flux q_{rad} to the imposed radiation flux q_{rad0} , of a plasma-sprayed HfO₂-Y₂O₃-Nd₂O₃-Yb₂O₃ coating, as a function of coating thickness, as determined by a laser activated black-body emitting source-flux technique [13]. It can be seen that compared to the baseline plasma-sprayed ZrO₂-8wt%Y₂O₃ coating, the advanced HfO₂-Y₂O₃-Nd₂O₃-Yb₂O₃ coating showed the significantly improved radiation resistance. The advanced high stability and low conductivity 1650 °C HfO₂ coatings will be expected to significantly impact future low emission combustor technology.

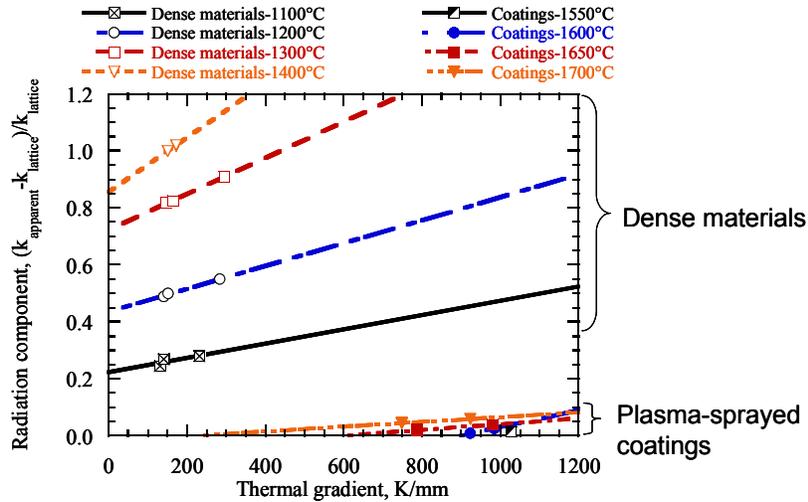


Figure 8.—The internal radiation component, determined for the ZrO_2 -based coating and monolithic ceramic specimens using the laser heat-flux technique, increases with increasing the thermal gradient (and thus heat flux) and surface temperature. The dense hot-pressed specimens show significantly higher internal radiation conductivity contribution compared to the plasma-sprayed coatings due to the lack of the micro-crack and micro-porosity scattering.

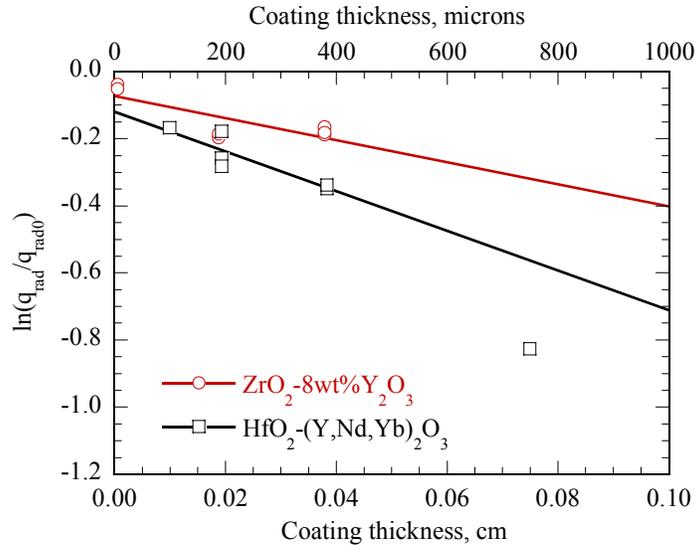


Figure 9.—Significantly improved radiation resistance is demonstrated for an advanced plasma-sprayed HfO_2 - Y_2O_3 - Nd_2O_3 - Yb_2O_3 coating as compared to a baseline plasma-sprayed ZrO_2 -8wt% Y_2O_3 coating.

Conclusions

1. A laser steady heat-flux approach has been established for ceramic thermal and environmental barrier coating thermal conductivity measurements and advanced coating development.
2. Real-time monitoring of coating thermal conductivity has been demonstrated as an effective technique to assess coating performance under simulated engine conditions.
3. The multi-component ZrO₂-based thermal barrier coatings, as compared to the baseline ZrO₂-8wtY₂O₃ coating, have demonstrated significantly lower thermal conductivity, improved long-term high temperature stability and cyclic durability required for advanced turbine airfoil and combustor liner and vane applications.
4. The multi-component HfO₂-based T/EBC systems have shown the great potential for future 1650 °C (3000 °F) CMC and metallic combustor coating applications.
5. The lattice and radiation conductivity of 1650 °C (3000 °F) T/EBC systems at high temperatures have been evaluated using the laser heat-flux techniques.

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1. AGENCY USE ONLY (<i>Leave blank</i>)	2. REPORT DATE July 2004	3. REPORT TYPE AND DATES COVERED Technical Memorandum	
4. TITLE AND SUBTITLE Thermal Conductivity of Advanced Ceramic Thermal Barrier Coatings Determined by a Steady-State Laser Heat-Flux Approach		5. FUNDING NUMBERS WBS-22-714-30-09 1L162211A47A	
6. AUTHOR(S) Dongming Zhu and Robert A. Miller			
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) National Aeronautics and Space Administration John H. Glenn Research Center at Lewis Field Cleveland, Ohio 44135-3191		8. PERFORMING ORGANIZATION REPORT NUMBER E-14474	
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) National Aeronautics and Space Administration Washington, DC 20546-0001 and U.S. Army Research Laboratory Adelphi, Maryland 20783-1145		10. SPONSORING/MONITORING AGENCY REPORT NUMBER NASA TM-2004-213040 ARL-TR-3262	
11. SUPPLEMENTARY NOTES Prepared for the 27th International Thermal Conductivity Conference (ITCC27) sponsored by the High Temperature Materials Laboratory at Oak Ridge National Laboratory, Knoxville, Tennessee, October 26-29, 2003. Dongming Zhu, U.S. Army Research Laboratory, NASA Glenn Research Center; and Robert A. Miller, NASA Glenn Research Center. Responsible person, Dongming Zhu, organization code 5160, 216-433-5422.			
12a. DISTRIBUTION/AVAILABILITY STATEMENT Unclassified - Unlimited Subject Categories: 23, 26, and 27 Available electronically at http://gltrs.grc.nasa.gov This publication is available from the NASA Center for AeroSpace Information, 301-621-0390.		12b. DISTRIBUTION CODE	
13. ABSTRACT (<i>Maximum 200 words</i>) The development of low conductivity and high temperature capable thermal barrier coatings requires advanced testing techniques that can accurately and effectively evaluate coating thermal conductivity under future high-performance and low-emission engine heat-flux conditions. In this paper, a unique steady-state CO ₂ laser (wavelength 10.6 μm) heat-flux approach is described for determining the thermal conductivity and conductivity deduced cyclic durability of ceramic thermal and environmental barrier coating systems at very high temperatures (up to 1700 °C) under large thermal gradients. The thermal conductivity behavior of advanced thermal and environmental barrier coatings for metallic and Si-based ceramic matrix composite (CMC) component applications has also been investigated using the laser conductivity approach. The relationships between the lattice and radiation conductivities as a function of heat flux and thermal gradient at high temperatures have been examined for the ceramic coating systems. The steady-state laser heat-flux conductivity approach has been demonstrated as a viable means for the development and life prediction of advanced thermal barrier coatings for future turbine engine applications.			
14. SUBJECT TERMS Thermal barrier coatings; Thermal conductivity; High-heat-flux testing; Thermal radiation effect		15. NUMBER OF PAGES 17	
		16. PRICE CODE	
17. SECURITY CLASSIFICATION OF REPORT Unclassified	18. SECURITY CLASSIFICATION OF THIS PAGE Unclassified	19. SECURITY CLASSIFICATION OF ABSTRACT Unclassified	20. LIMITATION OF ABSTRACT

