



# Thermal Conductivity and Thermal Gradient Cyclic Behavior of Refractory Silicate Coatings on SiC/SiC Ceramic Matrix Composites

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# THERMAL CONDUCTIVITY AND THERMAL GRADIENT CYCLIC BEHAVIOR OF REFRACTORY SILICATE COATINGS ON SiC/SiC CERAMIC MATRIX COMPOSITES

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## ABSTRACT

Plasma-sprayed mullite and BSAS coatings have been developed to protect SiC/SiC ceramic matrix composites from high temperature environmental attack. In this study, thermal conductivity and thermal barrier functions of these coating systems are evaluated using a laser high-heat-flux test rig. The effects of water vapor on coating thermal conductivity and durability are studied by using alternating furnace and laser thermal gradient cyclic tests. The influence of laser high-thermal-gradient cycling on coating failure modes is also investigated.

## INTRODUCTION

Environmental barrier coatings (EBCs) have been developed to protect SiC-based ceramic components in gas turbine engines from high temperature environmental attack [1-3]. With continuously increasing demands for significantly higher engine operating temperature, fuel efficiency and better engine reliability, future EBC systems must be designed for both thermal and environmental protections of the engine components in gas turbine combustion gas environment. In particular, thermal barrier functions of EBCs become a necessity for reducing the engine component thermal loads and chemical reaction rates, thus maintaining required mechanical properties and durability of these components. The advances in EBC development will directly impact the successful use of ceramic components in advanced engine systems.

Plasma-sprayed BSAS and mullite EBC coatings have been successfully used as protective coatings for SiC/SiC ceramic composite systems. In this study, thermal conductivity of the two EBC coating systems is evaluated using a laser high-heat-flux test rig. The effects of water vapor on coating thermal conductivity and durability are studied by using alternating furnace and laser thermal gradient cyclic tests. The influence of laser high-thermal-gradient cycling on coating failure modes is also discussed.

## EXPERIMENTAL MATERIALS AND METHODS

Three EBC systems, namely, BSAS, mullite-20wt%BSAS, and BSAS/mullite-20wt%BSAS two-layer coatings, were air plasma-sprayed (APS) onto the 25.4 mm-diameter and 2.3 mm-thick MI SiC/SiC ceramic composites substrates. The coating thicknesses of the BSAS and mullite-20wt%BSAS coating systems were approximately 254  $\mu\text{m}$ . For the BSAS/mullite-20wt%BSAS two-layer coating system, the thickness of each layer was about 127  $\mu\text{m}$ . A high power CO<sub>2</sub> laser was used to test the EBC specimens under a high thermal gradient with 60 min heating and 5 min cooling temperature cycles. 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 rotation. The uniformly distributed laser beam provided the specimen surface heating. The required specimen temperatures and thermal gradients for this study were achieved by controlling the delivered laser heat flux and backside air-cooling. Thermal conductivity of the ceramic coating systems was also measured in-situ as a function of the laser cycle number using the steady-state laser heat flux test approach [4-6]. During the laser thermal cycling test, the ceramic surface temperature was measured by an 8  $\mu\text{m}$  infrared pyrometer, and the backside CMC surface was measured by a two-color pyrometer. The laser test surface temperatures were set at 1482°C and 1371°C, respectively, while the interface temperature was approximately controlled at 1300°C. Besides the pure laser thermal cyclic testing, a set of specimens were also tested using alternating furnace cycling in a 90% water vapor environment and the laser thermal gradient cyclic testing in air, to study the possible water vapor effect. The furnace test temperature was at 1300°C with 60 min hot time temperature cycles.

## EXPERIMENTAL RESULTS AND DISCUSSION

### Thermal Conductivity of Environmental Barrier Coatings

Fig. 1 shows the thermal conductivity test results of the EBC coatings on SiC/SiC CMC substrate using the laser heat flux technique. For the as-sprayed EBC coatings, average initial coating thermal conductivity varies from 1.4 to 2.2 W/m-K in the temperature range of 900 to 1350°C. Conductivity increases were observed during the heating/cooling cycles due to the ceramic sintering. The BSAS coating showed the largest increase in the conductivity. After the first test cycle, the conductivity values were approximately 2.2 W/m-K for BSAS coating, and 1.7-1.8 W/m-K for mullite-20wt%BSAS and the BSAS/ mullite-20wt%BSAS two-layer coatings.

Thermal conductivity of the EBCs further increased in the first 10 hour furnace water vapor cyclic testing. As shown in Fig. 2, the conductivity of the EBCs increased by as high as 30% after the furnace test. The increase in the conductivity is attributed to the coating sintering. The subsequent 10 hr laser cyclic testing, however, reduced the conductivity of the coatings. This may be due to coating cracking and micro-delaminations under the high temperature, high thermal gradient cyclic testing.

Fig. 3 shows thermal conductivity changes of the BSAS coatings as a function of cycle number. In the pure laser test case shown in Fig. 3 (a), the conductivity had a minimum value of 1.5 W/m-K at about 20 cycles. The conductivity then increased, and maintained at about 2.5-2.6 W/m-K with further thermal cycling. In the combined furnace and laser thermal cycling case, significant thermal conductivity increase was also observed. As shown in Fig. 3 (b), the measured conductivity increased to above 3.0 W/m-K. Substantial glass phase formation was observed at both coating surfaces after the high temperature cyclic testing, as shown in Fig. 4.

Fig. 5 shows the thermal conductivity changes of the BSAS/mullite-20wt%BSAS two-layer coating systems as a function of the cycle number. For both pure laser tested and combined furnace and laser tested specimens, initial thermal conductivity increased due to the coating sintering. However, the later coating conductivity decreased due to the coating cracking and delaminations under the further testing. From the tests, it can be seen that both the coating sintering and coating delaminations occurred much faster for the combined furnace water vapor and laser cyclic test, as compared to the pure laser test. The preliminary test results indicated that the water vapor has a detrimental effect on coating durability, especially under the thermal gradient cyclic test conditions. The coating failure can be greatly accelerated, by the interface pore formation in the water vapor environments, and by subsequent coating cracking and delaminations under the severe laser thermal transient and thermal gradient loadings. Typical EBC failure modes and cracking morphologies of the BSAS and BSAS/mullite-20wt%BSAS two-layer coatings under the combined furnace and water vapor cycles exposure are shown in Fig. 6.

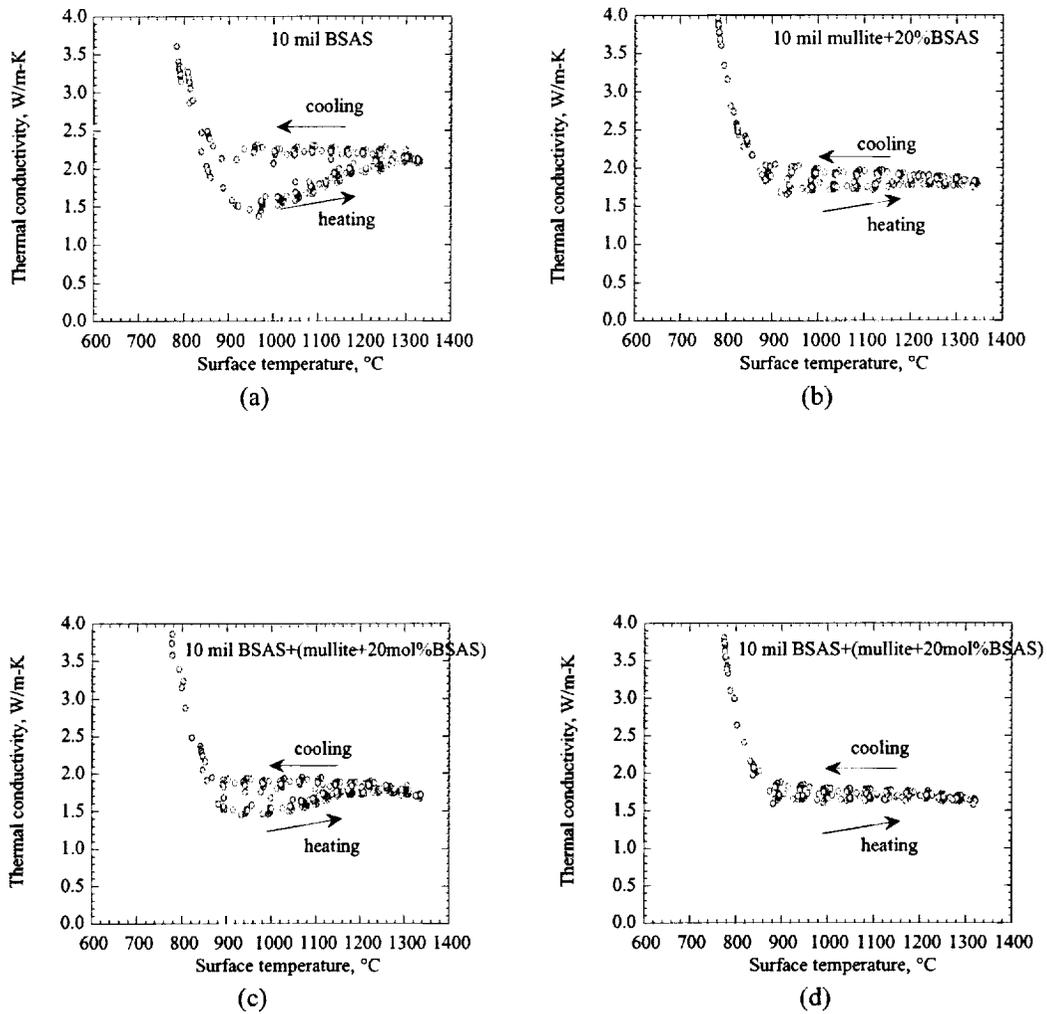


Fig. 1 Thermal conductivity of EBCs on SiC/SiC CMC substrate as a function of coating surface test temperature. Conductivity increases were observed during the heating/cooling cycles due to ceramic sintering. (a) BSAS coating, the first heating/cooling cycle; (b) mullite-20wt%BSAS coating, the first heating/cooling cycle; (c) and (d) BSAS/mullite-20wt%BSAS two-layer coating, the first, and the second heating/cooling cycles, respectively.

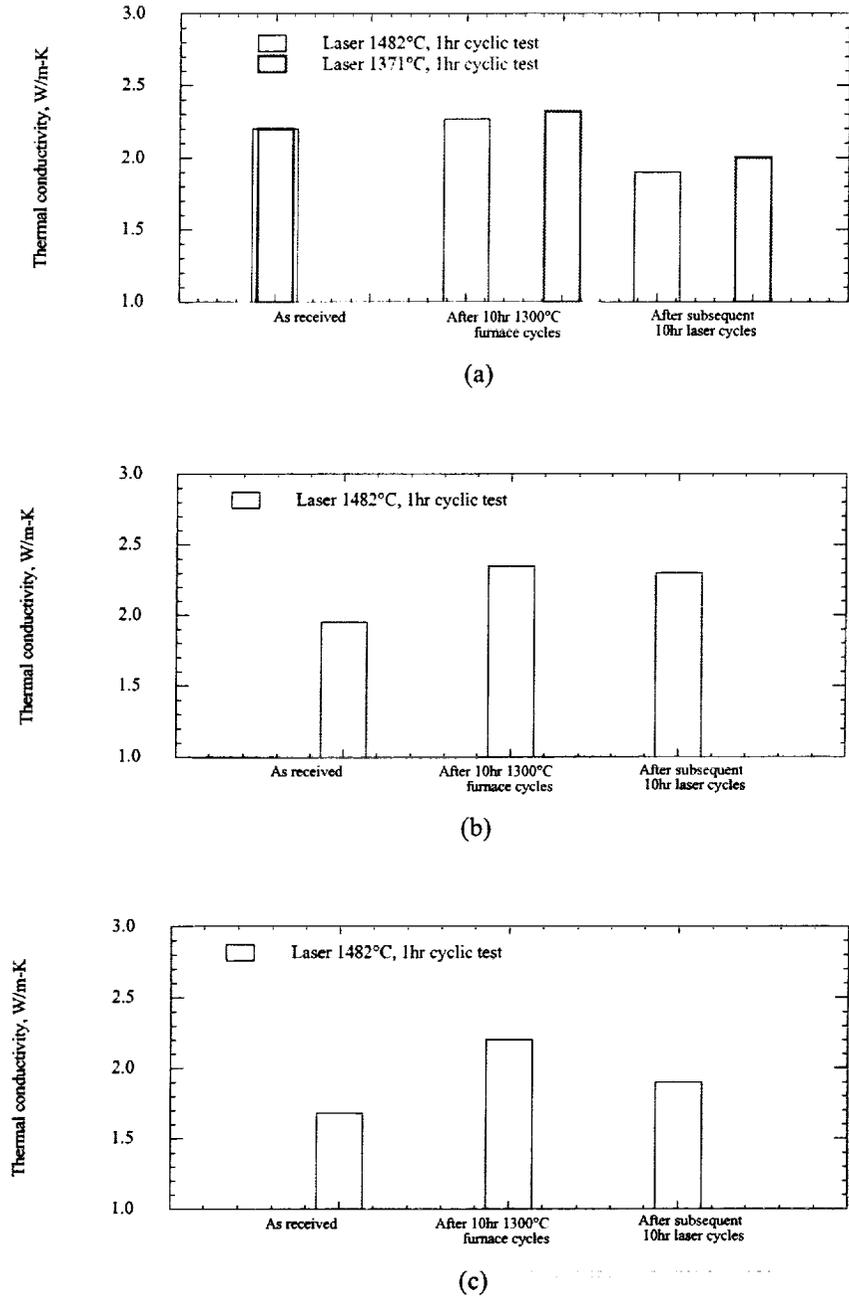
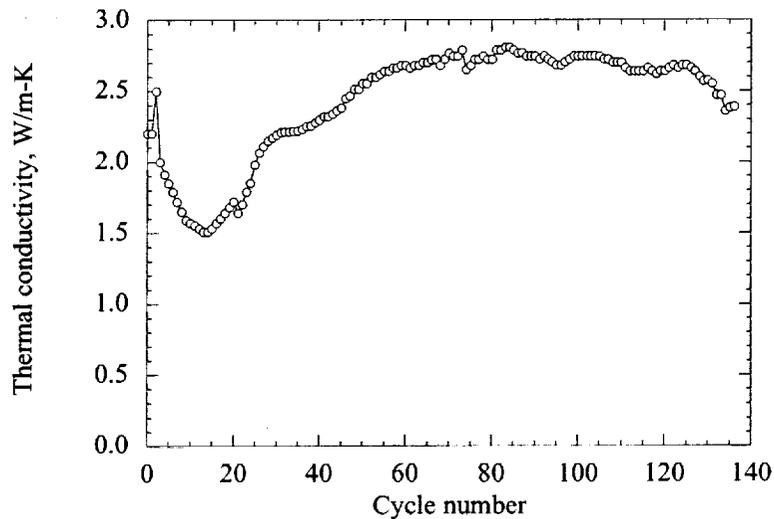
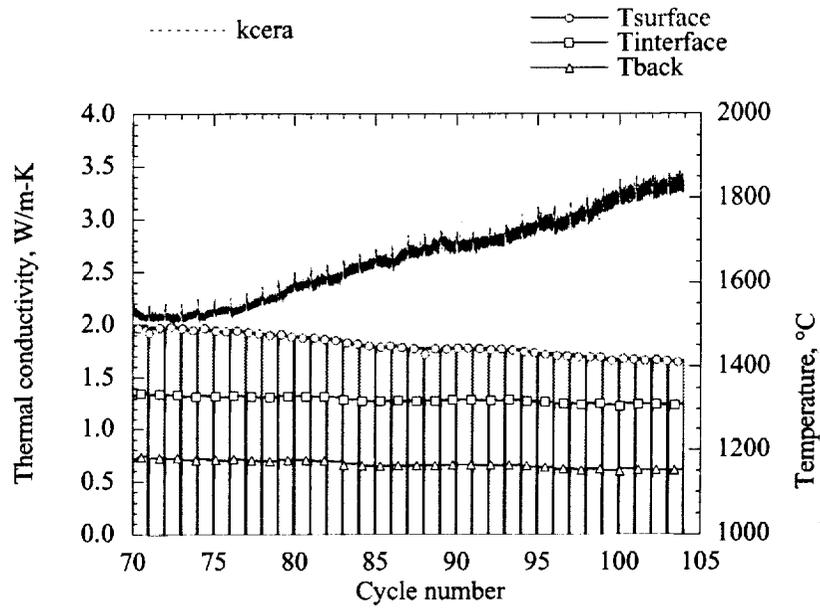


Fig. 2 Thermal conductivity of EBCs after the first 10 hr furnace cyclic testing, and a subsequent 10 hr laser cyclic testing. (a) BSAS coating; (b) mullite-20wt%BSAS coating; (c) BSAS/mullite-20wt%BSAS two-layer coating.

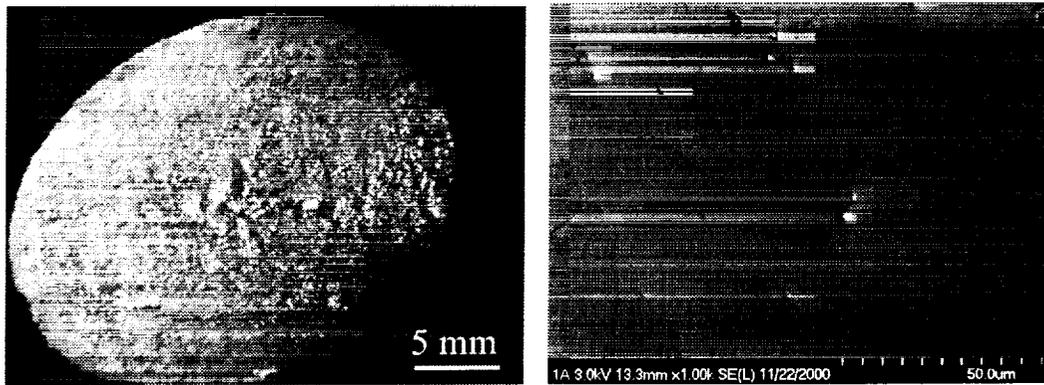


(a)

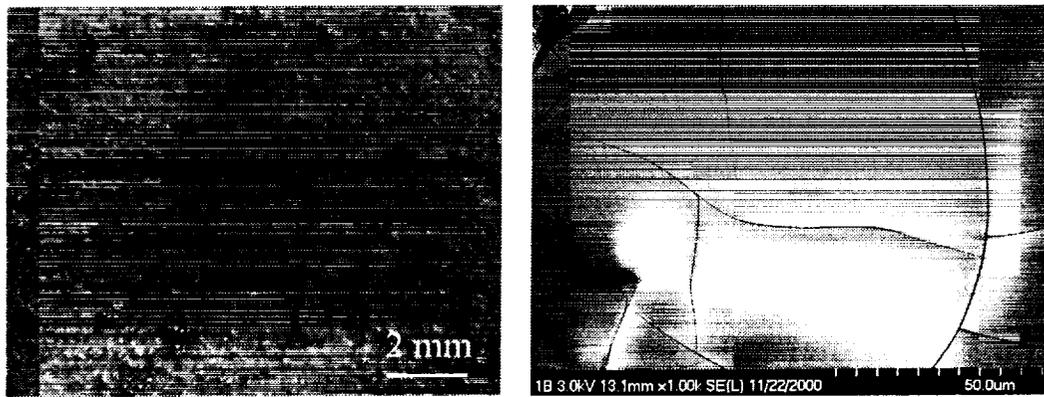


(b)

Fig. 3 Thermal conductivity changes of the BSAS coatings as a function of cycle number. (a) Laser thermal cycling testing (surface temperature 1482°C and interface 1300°C); (b) Combined furnace water vapor and laser thermal cyclic testing during the 70-104 cycles.

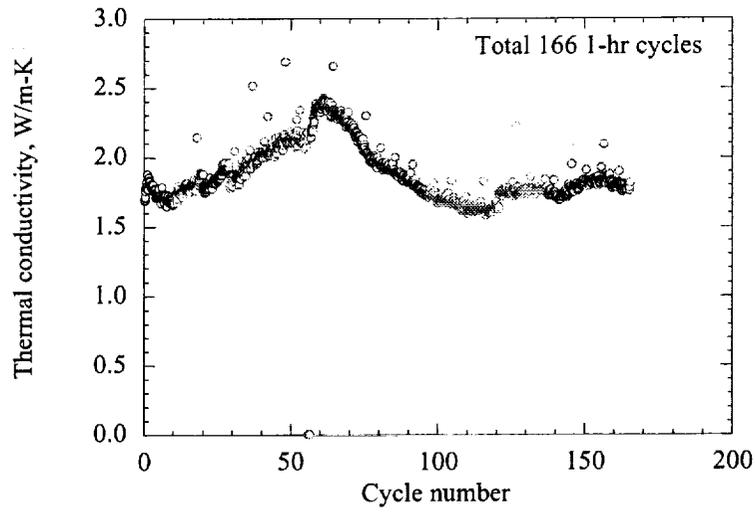


(a)

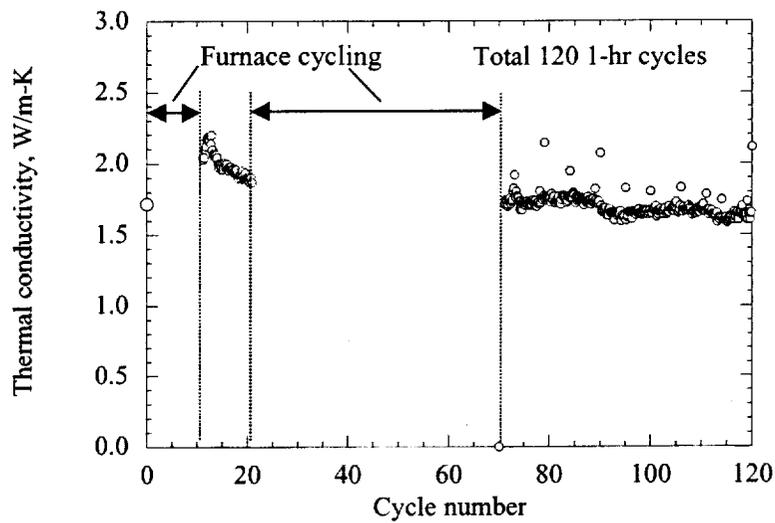


(b)

Fig. 4 Surface morphologies of the glass phase formed on the BSAS coating surface. (a) Laser tested specimen after 136 cycles; (b) Combined furnace water vapor and laser thermal cyclic tested specimen after 120 cycles.



(a)



(b)

Fig. 5 Thermal conductivity changes of the BSAS/mullite-20wt%BSAS two-layer coating systems as a function of the cycle number. (a) Laser thermal cycling testing (surface temperature 1482°C and interface 1300°C); (b) Combined furnace water vapor and laser thermal cyclic testing.

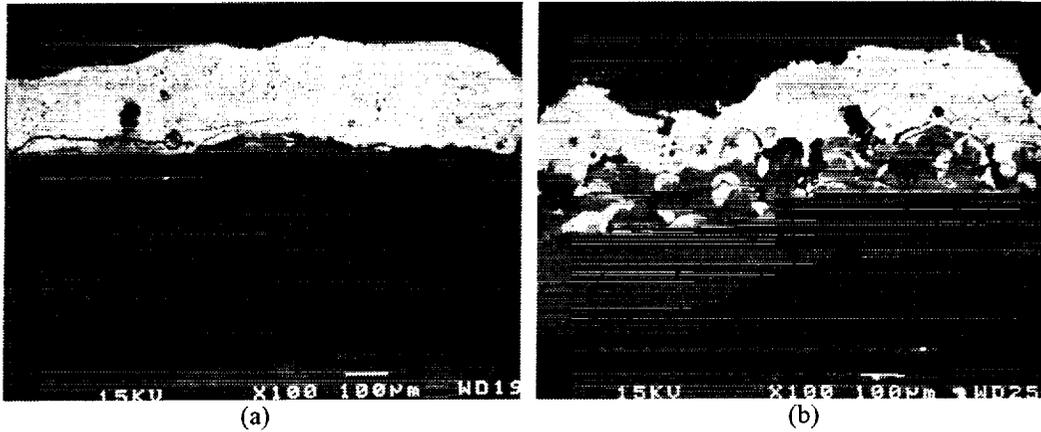


Fig. 6 Micrographs of cross-sections of BSAS and BSAS/mullite-20wt%BSAS two-layer coating coatings after the combined laser and furnace water vapor cycle testing, showing the coating interface pore structures and coating cracking and delaminations after testing. (a) BSAS Coating; (b) BSAS/mullite-20wt%BSAS two-layer coating.

#### Thermal Barrier Considerations of Environmental Barrier Coatings

Thermal barrier functions of the EBCs depend on the coating thermal conductivity, thickness and heat flux conditions encountered in an engine. Fig. 7 (a) and (b) shows calculated and experimentally measured temperature distributions across a coating system as a function of the heat flux and coating thickness. For the given conductivity values of the coating and substrate, in order to maintain the interface temperature of approximately 1300°C under the fixed surface temperature 1450°C, either a higher heat flux (100 W/cm<sup>2</sup>) or a thicker coating (0.508 mm thick) is required. The effect of EBC conductivity on the coating temperature reduction is illustrated in Fig. 8 (a) and (b), for the heat fluxes of 50 W/cm<sup>2</sup> and 100 W/cm<sup>2</sup>, respectively.

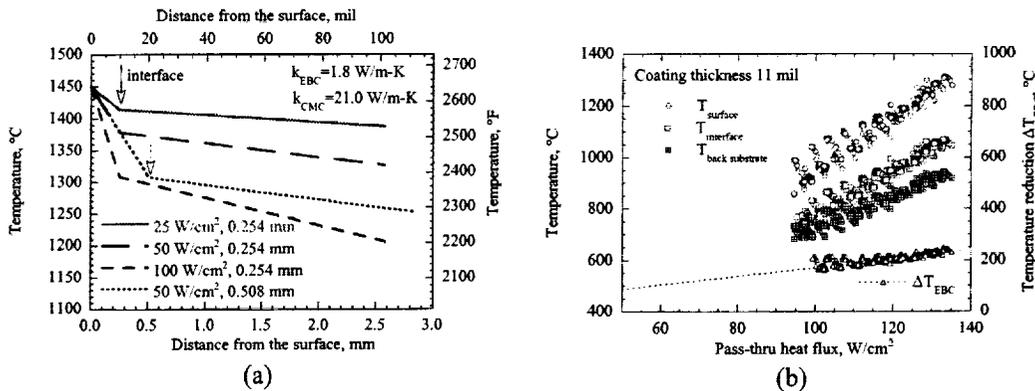
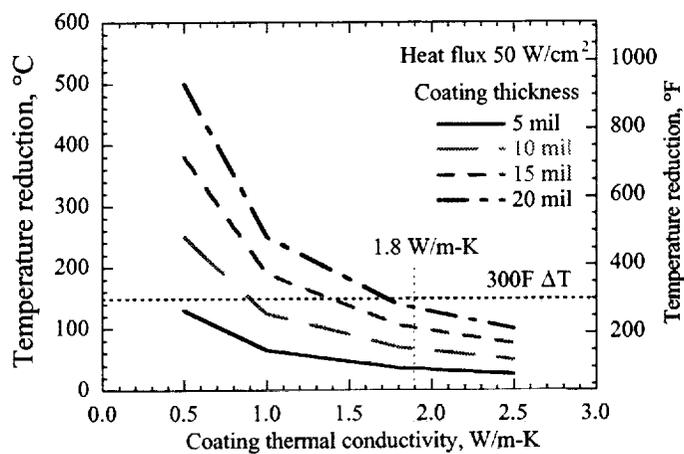
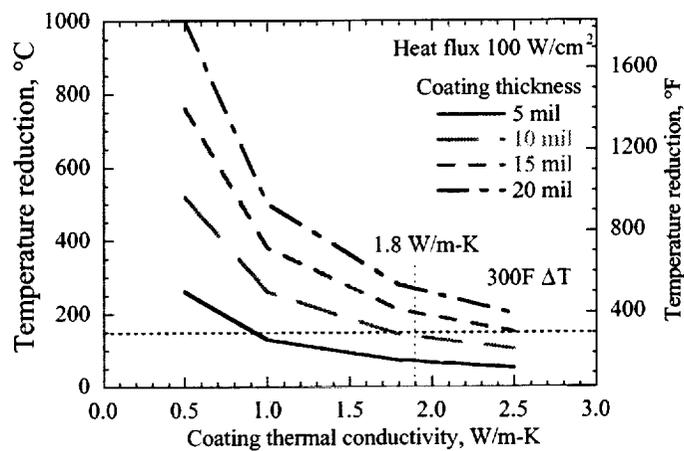


Fig. 7 Calculated (a) and experimentally measured (b) temperature distributions across a coating system as a function of heat flux.



(a)



(b)

Fig. 8 The effect of EBC conductivity and thickness on the coating temperature reduction. (a)  $50 \text{ W/cm}^2$ ; (b)  $100 \text{ W/cm}^2$ .

## CONCLUSIONS

A laser steady-state heat flux rig has been used to investigate thermal conductivity of several environmental barrier coating systems. Thermal conductivity values of the current EBC coatings were in the range of 1.4-3.0 W/m-K. Significant conductivity increase was observed for the coating systems after the laser and furnace water vapor cyclic testing. The preliminary test results showed that the coating failure can be greatly accelerated by enhanced interface pore formation in water vapor environments and the promoted coating cracking and delaminations under the severe laser thermal transient and thermal gradient loadings.

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