Mechanical Properties and Durability of Advanced Environmental Barrier Coatings in Calcium-Magnesium-Alumino-Silicate Environments

Daniel S. Miladinovich¹, Dongming Zhu²

1. Illinois Institute of Technology, Chicago IL, 60616
2. NASA John H. Glenn Research Center

Abstract

Environmental barrier coatings are being developed and tested for use with SiC/SiC ceramic matrix composite (CMC) gas turbine engine components. Several oxide and silicate based compositions are being studied for use as top-coat and intermediate layers in a three or more layer environmental barrier coating system. Specifically, the room temperature Vickers-indentation-fracture-toughness testing and high temperature stability reaction studies with Calcium Magnesium Alumino-Silicate (CMAS) and SiC/SiC CMC substrate are being conducted using advanced testing techniques such as high pressure burner rig tests as well as high heat flux laser tests.

Introduction

Advanced SiC/SiC ceramic matrix composites (CMCs) developed for gas turbine engine hot section component applications are susceptible to environmental attack from harsh combustion and general operation. This is why it is necessary to apply layers of environmental barrier coatings (EBCs) to protect SiC/SiC CMCs. EBCs need to improve the chemical and environmental stability for CMC systems in the presence of CMAS. The stability of candidate EBC material and coating systems in the presence of CMAS is also investigated.

Experimental Procedure

• Surface preparation
• Sintering
• Polishing
• Indentation
• CMAS reaction
• SEM EDS
• X-ray diffraction
• Vickers indentation fracture toughness tests
• Laser high heat flux testing
• High pressure burner rig

EBC Materials Tested

• Vickers Indentation Fracture Toughness
  - Oxides: SiO₂, TiO₂, ZrO₂, HfO₂, Y₂O₃, Gd₂O₃, Yb₂O₃
  - Silicates: Er₂SiO₅, Al₆Si₂O₁₃ (Mullite), Al₂SiO₅ (Silicate)

CMAS Reaction

• Oxides: SiO₂ + 5%Y₂O₃, TiO₂, HfO₂, 70% Er₂O₃, 30% Al₂O₃
• Silicates: Er₂SiO₅, Al₆Si₂O₁₃ (Mullite), Al₂SiO₅ (Silicate)

Sample Configurations

• 1X1 in. disk, 0.010 in. thick disc specimen
• Hybrid Air EB-PVD coatings on SiC substrate for laser high heat flux tests

Results

Fracture Toughness

Hardness Vickers

Laser Heat Flux Testing

CMAS Reaction and Behavior With Coatings

Figure 2: The initial vickers indentation results of non-CMAS-reacted coatings. (a) the mean value of each samples Hv was measured from the crack lengths. ZrO₂ is the toughest material. (b) the sample HVs as measured using the length of the diagonals. Hardness data was easily taken from the fracture toughness tests so (b) shows the hardest material as the HfO₂ + 5wt%Y₂O₃.

Figure 3: SEM images that span ~10µm. Right, secondary electron image showing the topology of HfO₂ + 5wt%Y₂O₃, 20wt%Gd₂O₃, 5wt%Y₂O₃. Left, two EDS map images showing Hf (green) and Ca (yellow) locations. This image illustrates the effect of penetration of CMAS into the material.

Figure 4: (above) Laser-high-heat flux tests were conducted on three structurally identical samples. With surface concentration increasing from left to right it is evident that the CMAS is damaging and melting of the coatings. (Left)increase in conductivity is due to the continued egressing from the high temperatures.

Figure 5: (above) is a hybrid EB-PVD coating system that consists of ZrO₂ and silicate system. The HBPR tests showed that the CMAS-reacted Er₂SiO₅ seemed to be damaged twice for a total of 100 hr. This also shows the increasing thickness of various layers of mullite. Each layer represents a different effect CMAS had. Time in hours is on the x axis. Depth is on the y axis.

Figure 6: (right) Shows the increasing thickness of various layers of mullite. Each layer represents a different effect CMAS had. Time in hours is on the x axis. Depth is on the y axis.

Concluding Remarks

Initial fracture toughness testing using the Vickers indentation approach has shown that the ZrO₂ and HfO₂ coating materials are the most fracture resistant. Further testing is being considered to determine the strength and fracture toughness of materials tested with CMAS.

Seven mechanisms of CMAS interactions with the coating materials have been identified. While the oxides were more stable than the silicates, they were still affected by penetration and void generation from CMAS especially when initial high porosity is present. Silicates react strongly with the CMAS generally decreasing the melting point and causing the coating to change phases as evidenced from X-ray diffraction such as in Y₂O₃ case. Some coating materials experienced combinations of all of the effects. The laser-high-heat flux tests showed the damage and potentially reduced temperature capability caused by the CMAS on a single-layer hybrid EB-PVD Plasma spray oxide-silicate EBC. This can be fatal to the coating structure as the operating temperature approaches the melting point of material after reacting with CMAS. The HBPR tests showed that the CMAS-reacted Er₂SiO₅ seemed to be damaged (delamination) and reduced recession rates compared to the non-reacted Er₂SiO₅.

While these studies are not complete, the current results easily showed that CMAS can cause serious damage to the coating or coating materials. Coating that have improved resistance to CMAS must be designed and tested for advanced EBC systems.

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