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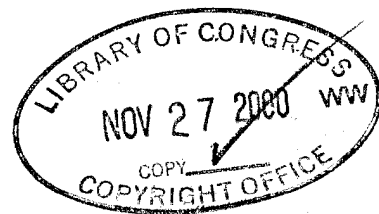


Preface: Special ICE Section

In recent years, the ever-increasing socioeconomic pressures for developing cleaner and more efficient means for converting chemical fuel energy into useful mechanical work have resulted in a dramatic growth in internal combustion engine research and development activities. As a natural outcome of this vigorous activity, a large number of technical papers dealing with various mechanical aspects of engine design, as well as with the basic thermo-fluid engine processes have been presented at the Fall and Spring Technical Conferences organized by the Internal Combustion Engine (ICE) Division of ASME. In 1999, the ICE Division has renewed its commitment to identify quality papers of long-term reference value for the journal. Therefore, a major section of this issue of the ASME JOURNAL FOR GAS TURBINES AND POWER is devoted to a selection of the highest quality ICE Papers presented at those meetings, and other papers of long-term reference value which were submitted directly to the journal. The contributions of the ICE Division Associates, the support of the ICE Executive Committee, and the ASME Editorial Staff, and above all the contributions of the authors and referees are gratefully acknowledged.

The papers that have been included in the special ICE section of this volume have been arranged in four topical areas: engine design, intake and exhaust system dynamics, in-cylinder processes, and alternative fuels combustion and emissions. As a special note, the lead paper for the ICE section, authored by Dan E. Richardson and entitled "Review of Power Cylinder Friction for Diesel Engines," was presented at the 1999 Spring Technical Conference, Columbus, Indiana. This paper was selected as the most valuable technical paper presented at an ASME-ICE Division Meeting during 1999.

Dennis N. Assanis
Associate Editor
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Key Durability Issues With Mullite-Based Environmental Barrier Coatings for Si-Based Ceramics

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Plasma-sprayed mullite ($3Al_2O_3 \cdot 2SiO_2$) and mullite/yttria-stabilized-zirconia (YSZ) dual layer coatings have been developed to protect silicon-based ceramics from environmental attack. Mullite-based coating systems show excellent durability in air. However, in combustion environments, corrosive species such as molten salt or water vapor penetrate through cracks in the coating and attack the Si-based ceramics along the interface. Thus the modification of the coating system for enhanced crack-resistance is necessary for long-term durability in combustion environments. Other key durability issues include interfacial contamination and coating/substrate bonding. Interfacial contamination leads to enhanced oxidation and interfacial pore formation, while a weak coating/substrate bonding leads to rapid attack of the interface by corrosive species, both of which can cause a premature failure of the coating. Interfacial contamination can be minimized by limiting impurities in coating and substrate materials. The interface may be modified to improve the coating/substrate bond. [S0742-4795(00)03203-8]

Introduction

Silicon-based ceramics are promising candidates for hot section structural components of heat engines and heat exchanger tubes for industrial furnaces. One potential barrier to such applications is their environmental durability. The excellent oxidation resistance of silicon-based ceramics at high temperatures in clean, dry oxygen is due to the formation of a solid, protective external silica scale. However, the normally protective silica scale can be degraded by reacting with impurities, such as alkali salts [1,2] or water vapor [3–5].

Molten Na_2SO_4 can deposit in gas turbine engines operating near marine environments or from contaminants in the fuel [6]. The Na_2SO_4 then reacts with the silica to form liquid sodium silicate, leading to accelerated degradation of Si-based ceramics [1]. In coal-fired combustion environments, combustion gases contain low levels of alkali salts because of naturally occurring minor alkali components in the coal. These alkali salts can dissolve in the silica scale and enhance the transport of oxygen, leading to drastically enhanced oxidation [2]. In heat engines, substantial amounts of water vapor, typically about 10 percent, is produced from burning hydrocarbon fuels in air [7]. The water vapor reacts with silica, forming gaseous hydroxide species, such as $Si(OH)_4$ [3–5]. In high-pressure combustion environments, the higher water vapor pressure generates even higher levels of gaseous hydroxide species, resulting in linear volatilization of silica. The linear volatilization of silica coupled with the parabolic oxidation of Si-based ceramics results in overall parabolic kinetics [4], causing rapid degradation of Si-based ceramics. Therefore, the realization of the full potential of silicon-based ceramics in high temperature structural applications depends on the development of environmental protection schemes.

An external environmental barrier coating is a promising ap-

proach to protect Si-based ceramics from environmental attack. Mullite is a promising candidate coating material because of its close coefficient of thermal expansion (CTE) match and good chemical compatibility with Si-based ceramics. Researchers at Solar Turbines, Inc., San Diego, CA, and Oak Ridge National Laboratory, Oak Ridge, TN, have done pioneering work on applying refractory oxide coatings such as alumina, zirconia, yttria, mullite, cordierite, etc., on SiC [8,9]. In those studies, mullite was found to be adherent and offer the best protection of all the refractory coatings tested. However, those plasma-sprayed mullite coatings tended to crack on thermal cycling. Researchers at NASA Glenn Research Center, Cleveland, OH, identified the crystallization of amorphous phase mullite, which accompanies a volumetric contraction, as the main source for the cracking of plasma-sprayed mullite coatings [10]. Based on this finding, researchers at NASA GRC successfully eliminated most of the amorphous phase mullite from the coating by spraying the mullite while heating the SiC substrate above the crystallization temperature of amorphous mullite ($\sim 1000^\circ C$) [10].

These second-generation mullite coatings provide excellent protection in air and molten salt environment [10–14]. Mullite coatings, however, suffer selective vaporization of silica in the presence of water vapor because of its high silica activity (0.3–0.4) [11,15,16]. Thus, an environmental overlay coating is required when protection from water vapor is needed. Yttria-stabilized zirconia (YSZ) was selected as a baseline overlay coating because of its proven performance as a thermal barrier coating (TBC) in combustion environments. The mullite coating in the mullite/YSZ coating system is somewhat analogous to the bond coat in conventional TBC's, in the sense that it provides bonding as well as oxidation protection. This paper will discuss the current durability issues of second generation mullite-based environmental coatings on Si-based ceramics and future research directions in this area.

Experimental

Mullite and YSZ coatings were applied by atmospheric pressure plasma spraying onto $2.5 \times 0.6 \times 0.15$ cm sintered α -SiC coupons (Hexoloy™, Carborundum, Niagara Falls, NY) and reaction bonded silicon nitride (RBSN R. Bhatt, NASA GRC). The SiC

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substrates were roughened ($R_a^2=5-6\ \mu\text{m}$) by etching in Na_2CO_3 to achieve a good mechanical bond [10], whereas RBSN was used as processed. Fused mullite powder with the particle size of $44-74\ \mu\text{m}$ was used (Cerac, Inc., Milwaukee, WI). Typical coating thickness was $100-200\ \mu\text{m}$ for the mullite coating and $50\ \mu\text{m}$ for the YSZ coating. Details of the coating parameters are described elsewhere [10].

Coated coupons were annealed in air at $1300\ ^\circ\text{C}$ for 100 h, prior to the environmental exposure. Environmental exposures were thermal cycling in air, thermal cycling in 90 percent $\text{H}_2\text{O}/\text{O}_2$ at 1 atm (simulated lean combustion environments) or isothermal exposure in high-pressure burner rigs with or without molten salt. Thermal cycling tests in water vapor were to evaluate the long-term behavior of coatings in lean combustion environments because high-pressure burner rigs are not suitable for long-term tests due to their high operating cost. Thermal cycling was performed using an automated thermal cycling furnace. Each thermal cycle consisted of 2 h at temperature, rapid cooling to room temperature, and 20 min at room temperature. Typically, samples reached the high temperature within 2 min and the low temperature within 5 min in each cycle. Molten salt environments were generated using a high-pressure burner rig with Jet A fuel containing 2 ppm Na at 4 atm.

Tested samples were mounted in epoxy, polished to $1\ \mu\text{m}$ using diamond suspension, and examined using Scanning Electron Microscopy (SEM) and Energy Dispersive Spectroscopy (EDS).

Environmental Durability

Air. Mullite/YSZ-coated SiC was exposed to a 2 h thermal cycling exposure in air at $1300\ ^\circ\text{C}$. Figure 1 shows the cross-section after 500h exposure. Mullite coatings typically developed through-thickness cracks, however, they maintained excellent adhesion and provided excellent oxidation protection. Mullite-coated SiC exhibited a similar behavior under the same exposure, indicating that the presence of YSZ overlay coating did not affect the coating durability despite the large CTE mismatch between the two layers.

Mullite/YSZ-coated RBSN was exposed to an isothermal oxidation at $1300\ ^\circ\text{C}$ in air for 50 h. A thick oxide scale and large pores developed at the mullite/RBSN interface (Fig. 2). EDS showed a significant amount of Mg in the scale. This contamination by MgO, which is from the RBSN, is responsible for the enhanced oxidation and pore formation. Similar enhanced oxidation and pore formation of mullite-coated SiC was observed when the system was contaminated by Na_2O or K_2O from the coating processed with a low purity mullite powder [17]. Alkali and alkaline earth metal oxides are known to degrade the oxidation resis-

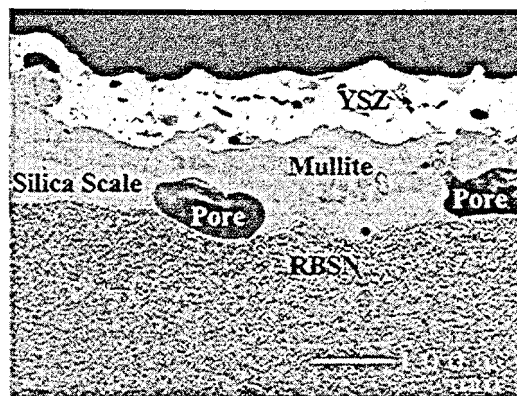


Fig. 2 Mullite/YSZ-coated RBSN after 50 h at $1300\ ^\circ\text{C}$ in air

tance of Si-based ceramics by enhancing the oxygen transport through silica by altering the silica network [18].

Combustion Environments

High Pressure Burner Rig. Uncoated, mullite-coated, and mullite/YSZ-coated SiC was exposed to high-pressure burner rig (HPBR) under a rich burn condition (equivalence ratio=1.9) at 6 atm and $1230\ ^\circ\text{C}$. Figure 3 shows the plot of weight change vs. time. Uncoated and mullite-coated SiC showed weight loss due to the volatilization of silica. The lack of weight change in the mullite/YSZ-coated SiC indicated that the YSZ overlay coating provided the protection from water vapor. Figure 4(a) shows the cross-section of mullite-coated SiC after the high-pressure burner rig exposure. Pores are observed at the interface where cracks intersected the SiC interface. Enhanced oxidation was observed around pores, indicating that water vapor penetrated through the cracks and reacted with SiC. The pore formation is attributed to the generation of gaseous silicon hydroxide species. The selective volatilization of silica from mullite left a porous layer of alumina on the surface of mullite (Fig. 4(b)).

Water Vapor Cyclic Furnace. Mullite-coated SiC/SiC (Dupont Lanxide, Newark, DE) exposed to 2 h cycle exposure in 50 percent $\text{H}_2\text{O}/\text{O}_2$ at $1300\ ^\circ\text{C}$ showed weight loss, whereas mullite/YSZ-coated SiC/SiC exhibited parabolic oxidation [19]. The weight loss of mullite-coated SiC/SiC was attributed to the selective volatilization of silica from the mullite. This result is consistent with the weight change behavior of mullite and mullite/YSZ coatings in high pressure burner rig (Fig. 3). A similar weight

²Average distance from the roughness profile to the mean line

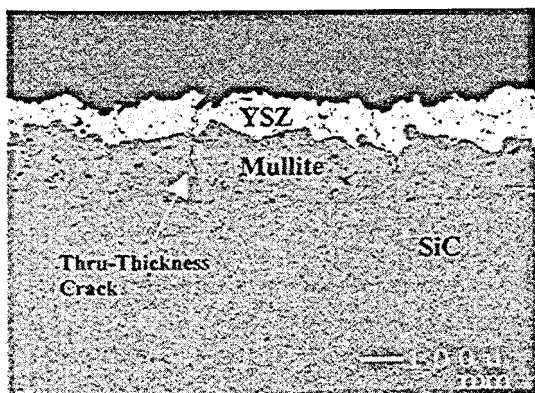


Fig. 1 Mullite/YSZ-coated SiC after 500 h with 2 h cycles at $1300\ ^\circ\text{C}$

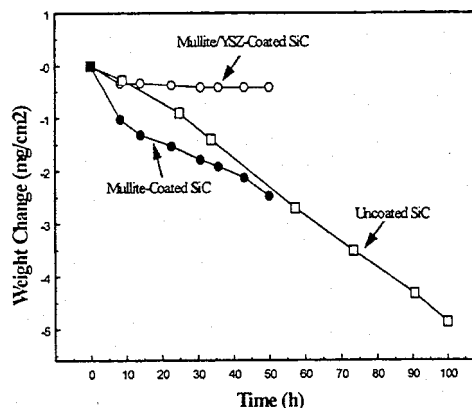


Fig. 3 Weight change versus time for coated and uncoated SiC

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