



# Plasma spray deposition of tri-layer environmental barrier coatings

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## Abstract

An air plasma spray process has been used to deposit tri-layer environmental barrier coatings consisting of a silicon bond coat, a mullite inter-diffusion barrier, and a  $\text{Yb}_2\text{SiO}_5$  top coat on SiC substrates. Solidified droplets in as-deposited  $\text{Yb}_2\text{SiO}_5$  and mullite layers were discovered to be depleted in silicon. This led to the formation of an  $\text{Yb}_2\text{SiO}_5 + \text{Yb}_2\text{O}_3$  two-phase top coat and 2:1 mullite ( $2\text{Al}_2\text{O}_3 \cdot \text{SiO}_2$ ) coat deposited from 3:2 mullite powder. The compositions were consistent with preferential silicon evaporation during transient plasma heating; a consequence of the high vapor pressure of silicon species at plasma temperatures. Annealing at 1300 °C resulted in internal bond coat oxidation of pore and splat surfaces, precipitation of  $\text{Yb}_2\text{O}_3$  in the top coat, and transformation of 2:1 mullite to 3:2 mullite +  $\text{Al}_2\text{O}_3$ . Mud-cracks were found in the  $\text{Yb}_2\text{SiO}_5$  layer and in precipitated  $\text{Al}_2\text{O}_3$  due to the thermal expansion mismatch between these coating phases and the substrate.

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## 1. Introduction

Improvements to the fuel efficiency and specific thrust of *future* gas turbine engines will require advances in many areas including: (i) increasing the gas turbine inlet temperature, (ii) higher overall pressure ratios of engines, (iii) increasing the by-pass ratio and (iv) incorporation of more efficient thermodynamic cycles into novel engine core designs.<sup>1–3</sup> Developments will be paced by the emergence of new propulsion materials, protective coatings, thermal management concepts, and manufacturing tools for economical component fabrication. Arguably the most difficult materials challenge will be encountered in the engine's hot core section.<sup>2,4–6</sup> Here, severely stressed rotating components will be subjected to ever increasing temperatures whose upper limit is set by stoichiometric combustion of the Jet-A fuel. At a combustor pressure of 1 MPa and an air temperature of 500 °C, the NASA CEA code<sup>7</sup> predicts this fuel burn temperature to be 2255 °C.

Recent increases to the turbine inlet temperature have been achieved by the use of internal air and surface film cooled superalloy components protected against oxidation and hot corrosion by thermal barrier coating (TBC) systems, [Fig. 1](#). This strategy is approaching its limit because coating sintering leads to a loss of compliance and increased thermal conductivity,<sup>9–13</sup> the bond coat oxidation rate (and thus delamination risk) increase rapidly with temperature,<sup>9,14–16</sup> and calcium–magnesium–aluminum–silicate (CMAS) melting and infiltration cause premature coating failure.<sup>17–19</sup> Even if these issues were resolved, TBCs can be eroded by fine (dust) particles<sup>19–22</sup> or chipped away by larger foreign object damage (FOD).<sup>19,22,23</sup>

These considerations have stimulated the development of turbine components made from ceramic materials with much higher temperature capability.<sup>24–27</sup> Monolithic ceramics have insufficient fracture toughness, so development has focused on damage tolerant fiber reinforced ceramic matrix composites (CMCs) with weak fiber/matrix interfaces.<sup>28–31</sup> The most promising CMCs are based upon woven fabrics of BN coated SiC fibers (such as Hi-Nicalon S and Sylramic fibers) and SiC matrices incorporated by chemical vapor infiltration with residual pores filled by silicon slurry infiltration followed by carburization. While oxide–oxide CMC systems are chemically

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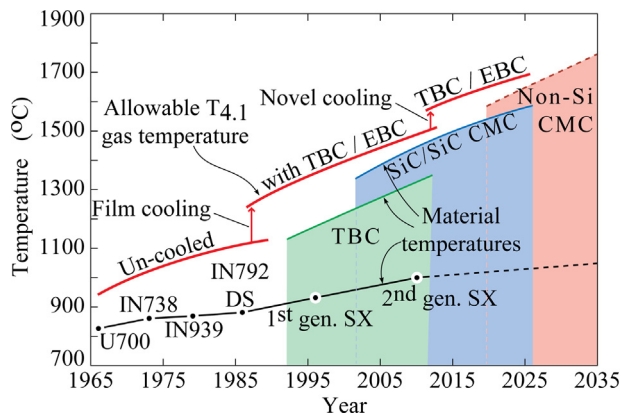


Fig. 1. The evolution of turbine blade materials, coatings, cooling concepts and turbine inlet  $T_{4.1}$  gas temperature plotted against the year of entry. Current engines utilize internal and film cooled single crystal (SX) superalloy blades protected by thermal barrier coatings in the hottest regions of the engine (adapted from Ref. 8).

inert in oxidizing environments, the current fibers have insufficient creep rupture strengths at temperatures of interest,<sup>32–34</sup> and are only used in lightly loaded applications.<sup>35–38</sup> The most heavily stressed components in future engines are likely to utilize SiC fiber/SiC matrix CMC materials for robust mechanical performance.<sup>27,39–43</sup>

When SiC is exposed to oxidizing environments a protective silica scale forms with a thickness that exhibits parabolic oxidation kinetics.<sup>44</sup> Unfortunately, the protective scale reacts with water vapor in hydrocarbon combustion environments to create gaseous silicon hydroxides via several reaction pathways.<sup>45–48</sup> The temperature, pressure, incident water vapor flux, and local flow conditions during exposure determine the observed parabolic oxidation kinetics.<sup>46–49</sup> SiC recession rates significantly greater than  $1 \mu\text{m/h}$  have been observed in the  $1300\text{--}1350^\circ\text{C}$  range.<sup>45</sup> Consequently, CMC components need to be protected by environmental barrier coatings (EBCs) to prevent component recession during operational lifetimes of tens of thousands of hours.

The first EBCs consisted of a 3:2 mullite ( $3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$  or  $\text{Al}_6\text{Si}_2\text{O}_{13}$ ) coating applied directly to the substrate.<sup>50</sup> During prolonged high temperature exposure, silica was volatilized from the mullite leaving an alumina scale that readily spalled, offering only short protection times.<sup>51</sup> The next EBC development focused on bi-layer concepts in which an yttria-stabilized zirconia (YSZ) top coat was used to prevent volatilization and chemical attack of the underlying mullite.<sup>52</sup> Thermal cycling tests in water vapor revealed that the coating quickly cracked and delaminated at either the YSZ/mullite or the mullite/SiC interfaces. The rapid failure was attributed to the substantial CTE mismatch between YSZ, the underlying mullite layer and the substrate.<sup>51,53–56</sup> Subsequently, several alkaline earth and rare earth silicates were identified with well-matched CTE's that exhibit little weight change during high temperature, high water vapor pressure environmental exposure,<sup>57,58</sup> Table 1.

Barium–strontium aluminosilicate (BSAS) has a low silica activity and a CTE that is well matched with substrates, Table 1. However, BSAS EBCs react with thermally grown silica,

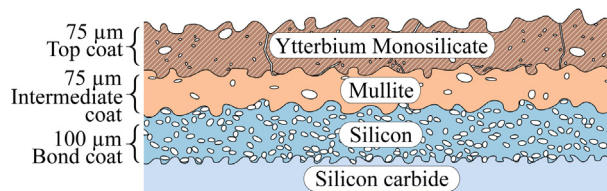


Fig. 2. Schematic illustration of an APS deposited tri-layer EBC structure.

necessitating a tri-layer BSAS/mullite/silicon EBC architecture<sup>65</sup> to eliminate failure at the coating/substrate interface and increase EBC life.<sup>72</sup> Unfortunately, low melting eutectics occur near  $1300^\circ\text{C}$  and limit the useful lifetime to under 1000 h for higher temperatures,<sup>65</sup> indicating the need for top coats with low volatility and high temperature phase stability. Ytterbium and lutetium silicates exhibit some of the lowest volatilities and are monomorphic. Ytterbium has monosilicate ( $\text{Yb}_2\text{SiO}_5$ ) and disilicate ( $\text{Yb}_2\text{Si}_2\text{O}_7$ ) variants whereas lutetium has only a monosilicate variant. The reported CTE of these and other candidate materials are summarized in Table 1. The data for  $\text{Yb}_2\text{SiO}_5$  is inconsistent. One report<sup>58</sup> gives a value of  $3.5\text{--}4.5 \times 10^{-6}^\circ\text{C}^{-1}$  which is reasonably matched to SiC. However, a more recent report<sup>57</sup> gives a higher value of  $7\text{--}8 \times 10^{-6}^\circ\text{C}^{-1}$ . Like BSAS, the RE-silicates are unstable as single layer coatings and so a tri-layer RE-silicate/mullite/silicon coating approach analogous to the BSAS top coat EBC has been proposed.<sup>58</sup>

There have been few studies of the tri-layer EBC deposition process or investigations of the relationships between coating deposition conditions, coating structure, and thermo-cyclic durability. To gain insight into these relationships, we describe an air plasma spray (APS) process to deposit tri-layer coatings on silicon carbide substrates with  $\text{Yb}_2\text{SiO}_5$  top coats, and have used a variety of methods to characterize the coatings. We report the presence and identify the formation mechanism of previously unidentified phases in the  $\text{Yb}_2\text{SiO}_5$  and mullite layers, and discuss the implications of their presence upon the propensity for cracking.

## 2. Experimental

### 2.1. Coating deposition

Three-layer  $\text{Yb}_2\text{SiO}_5$ /mullite/silicon EBCs were deposited onto SiC substrates using an APS approach. The general coating architecture is schematically illustrated in Fig. 2. Coatings were deposited on  $2.54 \text{ cm} \times 1.27 \text{ cm} \times 0.32 \text{ cm}$   $\alpha$ -SiC coupons obtained from Hexoloy<sup>TM</sup> (Saint Gobain Ceramics, Niagara Falls, NY). Prior to EBC deposition, the substrates were grit blasted using  $\sim 270 \mu\text{m}$  diameter SiC grit (Black SiC, White Abrasives, Niagara Falls, ON CA) resulting in an average surface roughness  $R_a = 1 \mu\text{m}$ , as measured by surface profilometry using a  $500 \mu\text{m}$  sampling distance. The substrates were then ultrasonically cleaned in ethanol to remove surface contamination.

The silicon powder used for bond coat deposition was supplied by Micron Metals (Bergenfield, NJ) with a particle

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