



Influence of microstructure on the durability of gadolinium zirconate thermal barrier coatings using APS & SPPS processes



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ABSTRACT

Excellent CMAS resistance in Gadolinium Zirconate, $Gd_2Zr_2O_7$ (GZO) coatings is attributed to vigorous reactions between the CMAS melt and the GZO that form secondary phases that rapidly crystallize and block further infiltration of CMAS. In this study, $Gd_2Zr_2O_7$ (GZO) coatings were deposited using air plasma spraying (APS) and solution precursor plasma spraying (SPPS) processes. The relative performance of GZO in furnace thermal cycle tests with and without CMAS was determined. SPPS GZO TBCs showed $8 \times$ longer lives in thermal cycling but $10 \times$ shorter lives in CMAS testing, as compared to APS GZO TBCs. Superior performance of the SPPS coatings without CMAS can be attributed to characteristic stress relieving vertical cracks in the microstructure, which are absent in APS TBCs. These vertical cracks also act as channels to CMAS melt infiltration, which causes the loss of strain compliance and hence poorer CMAS resistance. X-ray diffraction of the failed SPPS GZO coatings revealed the formation of apatite phases, but the majority of vertical cracks were not sealed. The denser than typical APS coatings on the other hand effectively blocked the CMAS infiltration with CMAS arrest observed at a depth of $\sim 25 \mu\text{m}$. The SPPS results suggest that the width of vertical cracks is an important parameter for sealing and hence impeding CMAS infiltration. Sealing was observed in cracks with $< 1 \mu\text{m}$ in width. Wider cracks completely defeat the beneficial CMAS blocking behavior of GZO.

1. Introduction

Thermal barrier coatings (TBCs) are commonly employed in gas turbine engines for thermal protection of superalloy components providing a temperature drop of $100 \text{ }^\circ\text{C}$ to $300 \text{ }^\circ\text{C}$ depending upon operating conditions, TBC material and thickness [1–4] and hence allowing the engines to operate at higher temperatures. They are viewed as necessary components for achieving high performance and energy-efficient systems. The current material of choice, commonly used in TBC applications such as hot sections of gas turbines and aero-engine components, is 6–8 wt% yttria stabilized zirconia (YSZ), owing to its favorable properties such as high thermal expansion coefficient, high fracture toughness (due to ferroelastic toughening [5]) and low thermal conductivity [1,6].

Higher operating temperatures result in greater engine efficiency which has inevitably led to an increasing demand for higher operating temperatures [7,8]. However, at temperature of $1200 \text{ }^\circ\text{C}$ and above, the metastable YSZ t' -phase starts to convert to cubic and tetragonal and also monoclinic phases accompanied by volumetric expansion leading to crack formation in the coatings and premature spallation [6,9]. Also

near $1200 \text{ }^\circ\text{C}$, atmospheric contaminants, usually dust, sand and even runway debris start to adhere to TBC surfaces, melt and attack YSZ-based TBCs. The deposited contaminants are generally calcium-magnesium-alumino-silicates (CMAS) because of aforementioned being the major constituents, upon melting form a glassy material which degrades the durability and thermal properties of the coating through chemical and mechanical interactions. CMAS infiltrates the coatings, fills up the pores and cracks which essentially densifies the microstructure and eliminates stress relieving features, thereby inducing rise of internal stresses and hence premature failures during thermal cycling [10,11], e.g. spallation. [4].

Therefore, the limitations of 8YSZ and the increasing temperature demands for next-generation gas turbines have promoted the search for alternative materials. Several potential materials systems have already been reported so far, which include modified YSZ systems [12], pyrochlores [6,13], garnets [14–16] and perovskites [17,18]. Gadolinium zirconate (GZO) has been one of those materials that have been extensively studied and deployed in service engines owing to its low thermal conductivity [6], high temperature stability [19] and enhanced resistance to molten silicate deposits [20–27]. This is achieved by

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vigorous reaction between GZO and siliceous melt and formation highly stable crystalline apatite phase ($\text{Ca}_2\text{Gd}_8(\text{SiO}_4)_6\text{O}_2$) that prevents further penetration of the melt. The arrest mechanism has been observed in different TBC microstructures (EB-PVD [20] and APS [23,26,27]) and seems to be relatively insensitive to the type of molten silicate composition (CMAS [26], volcanic ash [27] and coal fly ash [23]). However, GZO has lower fracture toughness than YSZ due to absence of ferroelastic toughening mechanism [28], thus may exhibit poorer performance as compared to YSZ in thermal cyclic experiments, erosion and damage occurring from foreign objects. To counteract this and deal with compatibility issues with the TGO, double layer TBCs are often used with the inner layer being YSZ and the top coat being GZO [29]. Since the failure during thermal cycling usually happens near or at the ceramic to thermally-grown-oxide interface due to formation and propagation of microcracks, an inner layer of high-toughness materials helps mitigate this problem.

In the current paper, we examine the effectiveness of microstructural variables on CMAS resistance and compared the performance of APS GZO coatings to that of SPPS GZO coatings. Cyclic furnace tests with and without CMAS were carried out. It is noted that GZO has been extensively studied from a potential TBC material standpoint and the blocking reaction has been clearly identified and studied [20–23,26,27]. In this paper, we will examine the effects of microstructure geometry only, using two different GZO microstructures. The effects considered will be thermal cyclic durability with and without CMAS. We also note that a) the APS coatings that were made for the study is relatively denser than a standard APS coating and, b) the SPPS structure produced has unusually open structure with extensive stress relieving through thickness cracks. These two microstructures are highly contrasting and thus are helping to more strongly illustrate the important difference between them making clearer the trends shown.

Both APS and SPPS GZO coatings were deposited on OEM superalloy coupons with bond coats and APS 8YSZ inner layers. These superalloy samples were tested for thermal cycling as well as CMAS resistance performances.

2. Materials and methods

2.1. Substrates

The substrates used for process optimization of the coatings were 304 stainless steel coupons with diameters of 25.4 mm and thicknesses of approximately 3 mm. Prior to deposition, the SS 304 coupons were grit blasted with 80 mesh alumina grit at 60–70 psi resulting in a surface roughness (R_a) of 4–5 μm . For furnace cycle tests, GZO was deposited on superalloy substrates provided by industrial partners with a dimension of 25.4 mm diameter and ~ 3.5 mm thickness. These superalloy substrates already had a 100 μm NiCrAlY APS bondcoat and $\sim 25 \pm 10$ μm of APS 8YSZ as the inner layer.

2.2. TBC deposition process and parameters

All APS GZO coatings were sprayed using commercial powders with hollow sphere morphology (HOSP) obtained from Saint-Gobain ceramics (Worcester, MA), with a particle size ranging between 10 μm –50 μm . A Metco 9 MB Plasma Gun with a GH nozzle (9 mm) with a powder feeding port was used to produce all samples. A matrix of 12 different sets of spray parameters was sprayed to obtain a TBC with no through thickness cracks and a relatively dense structure to contrast the SPPS coatings. The spray parameters used to obtain the APS samples for testing is given in Table 1.

For the SPPS GZO coatings, a solution precursor was prepared by mixing 2.4 mol of gadolinium nitrate hydrate (Alfa Aesar, Ward Hill MA) in 1-l zirconium acetate solution (22.8 wt% ZrO_2 in diluted acetic acid, MEL Chemicals Inc., Flemington NJ). A Metco 9 MB plasma gun with a GP nozzle (6 mm) was employed and the precursor was fed

Table 1
Spray parameters for the deposition of APS and SPPS GZO coatings.

Spray Parameters	APS Process	SPPS Process
Plasma gun	Metco 9 MB	Metco 9 MB
Gun nozzle	GH	GP
Gun power (kW)	36	45.5
Primary/secondary gas	Ar/ H_2	Ar/ H_2
Gas flow rate (L/min)	Ar: 47, H_2 : 8	Ar: 61, H_2 : 10
Precursor injection mode	–	Atomization: BETE FC4 nozzle, 20 psi pressure
Powder/precursor feed rate	4.5 kg/h	24 mL/min
Standoff distance (mm)	90	41
Gun scan speed (mm/s)	550	450
Raster step size (mm)	5	2
Number of passes	10	25

radially in the plasma plume using an atomizing nozzle. The spray parameters used for the process were adopted from a previous study [29] and are shown in Table 1.

2.3. Materials analysis

Samples were sectioned at 0.015 mm/min using a Struers Accutom-50 Precision Saw, mounted in epoxy resin (Allied High Tech Products Inc., Rancho Dominguez CA), placed under a vacuum to remove air bubbles and left to cure at the room temperature. The mounted samples were ground, polished and sputter-coated with Pd/Au (Polaron E5100 SEM Coating Unit) for metallography. Field emission scanning electron microscope (JSM-6350/5F, JEOL USA, Peabody MA) for SEM and BSE, and energy dispersive X-ray spectrometer (Noran system six EDS, Thermo, Waltham MA) were utilized to analyze coatings cross-sectional microstructures and element distribution. For determining porosity of the coatings, image analysis was conducted using ImageJ software. X-ray diffraction (XRD) was carried out using X-ray diffractometer (D2 Phaser, Bruker AXS, Madison, WI) to determine the phases present in the coatings. To determine Vickers hardness, micro indentation tests were conducted on the cross section of the polished samples that were previously prepared for SEM using microindentation hardness testing system (LM-248AT, LECO Corporation, Saint Joseph, MI). For analysis of cross sections of samples that were partially delaminated from the substrates, the entirety of the samples was first embedded in epoxy before sectioning and all the aforementioned steps were followed for samples preparation. SEM of GZO powder was done by sprinkling little amount of the powder on a sample mount with carbon tape pasted on it. The entire fixture was sputter coated for making the powder conductive and reducing the charging effects.

2.4. CMAS powder synthesis

Synthesis of 4 component CMAS (4-CMAS) and the testing methodology was kept similar to a previous study [16]. The CMAS composition was adopted from the study conducted by Drexler et al. [21]. Firstly, stoichiometric amounts of hydrated nitrates salts of calcium, aluminum and magnesium ($\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$, $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$, $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, Alfa Aesar, Ward Hill, MA), and Silica (LUDOX® TMA colloidal silica, Sigma-Aldrich Co. LLC, Spruce Street, MO) was added and dissolved in de-ionized (DI) water. The pH of the solution was constantly monitored to the ensure stability of colloidal silica as per manufacturer's recommendation and adjusted using nitric acid. The solution was converted into a gel by drying overnight at 120 °C, later heat treated at 600 °C in a tube furnace (CM 1600 Tube Furnace, Bloomfield N.J.) to form CMAS powder. The chemical composition of the CMAS is given in Table 2.

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