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### Feasibility of luminescent multilayer sol-gel thermal barrier coating manufacturing for future applications in through-thickness temperature gradient sensing

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### ABSTRACT

This paper investigates the feasibility of manufacturing sol-gel multilayer thermal barrier coatings (TBC) functionalized with different lanthanide ions  $Ln^{3+}$  having distinct photo-luminescence emission wavelengths (Ln = Sm, Eu, Dy, Er, Tm) for future applications in temperature gradient sensing.  $Ln^{3+}$  doped 9.75 mol% yttria stabilized zirconia (YSZ) powders were produced to study the effect of activator concentration on luminescence intensity and host matrix crystal structure. Self-quenching was found to limit the maximum signal-to-noise ratio achievable with  $Sm^{3+}$ ,  $Dy^{3+}$ ,  $Er^{3+}$  and  $Tm^{3+}$  activators, which was not the case for  $Eu^{3+}$  in the 1–10 mol% range. The increase in activator was found to affect the crystal structure of YSZ. A solution was proposed that suppressed this effect while significantly increasing the luminescence intensity of all activators. Finally a TBC sensor prototype integrating  $Eu^{3+}$ ,  $Er^{3+}$  and  $Dy^{3+}$  doped layers distributed throughout the thickness was successfully deposited by a dip-coating sol-gel process and showed promising through-thickness luminescence sensing capabilities. © 2014 Published by Elsevier B.V.

### 1. Introduction

The progressive use and improvement of thermal barrier coatings (TBCs) to protect critical components from heat damage in gas turbines have allowed significant increase in turbine inlet temperature over the last 30 years [1,2]. Today's jet engines' efficiency and durability therefore greatly depend on TBC's reliability and performance. The degradation during service of the current standard 6-8 wt% yttria stabilized zirconia (YSZ) based TBCs is mainly caused by the initiation and the propagation of microcracks at the interface between either the bond coat and the thermally grown oxide (TGO) or the TGO and the ceramic overcoat resulting from the thermal expansion coefficient mismatch between coatings and substrates [3,4]. This sub-surface mode of failure makes an early detection particularly difficult. The development of reliable predictive models for YSZ overcoat spallation is indeed limited by the difficulty of accessing the effective interface temperature that governs this process through conventional means without compromising the integrity of the coating. This results in strongly conservative margins being imposed to allow safe operation of jet engine turbine components.

From this perspective there has been a growing interest in the application of phosphor thermometry methods for the diagnostic of TBCs as

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http://dx.doi.org/10.1016/j.surfcoat.2014.08.077 0257-8972/© 2014 Published by Elsevier B.V. the partial transparency of YSZ in the visible range of the spectrum allows to collect local information conveyed by the luminescence emissions from optically excited luminescent layers integrated throughout the depth of the TBC [5,6]. This functionalization can be obtained by the introduction of optically active components such as trivalent lanthanide ions directly into the crystal structure of YSZ, thus without any detrimental alterations of the coating properties [6–8]. Such "sensor TBCs" has shown high potential for measuring substrate/TBC interface temperature [9,10] or investigate local interface delamination [11,12] by non intrusive optical sensing.

Reported here is a feasibility study for the design and manufacturing of multilayer functionalized TBCs, deposited by a dip-coating process from sol-gel precursors, alternative to the standard electron beam physical vapor deposition (EB-PVD) and atmospheric plasma spraying (APS) methods used for YSZ coatings, for future applications in through thickness measuring of temperature gradients. This cost effective process allows one to deposit 9.75 mol% porous YSZ TBCs presenting the metastable t' phase with performance under cyclic oxidation comparable to that of EB-PVD coatings [13,14]. The easy composition control offered by the technique has already generated interest for manufacturing some high purity YSZ:Er and YSZ:Sm phosphor coatings and powders [15]. Therefore the effect of additions of several dopants including Sm, Eu, Dy, Er and Tm in 9,75 mol% YSZ was investigated, and a solution is proposed for the optimization of YSZ luminescence properties. A prototype of multilayer TBC with three luminescent layers with distinct

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luminescence emissions embedded throughout the thickness, respectively doped with Eu, Er and Dy, was deposited by dip-coating and characterized to assess the feasibility of the process.

### 2. Thermal barrier coating sensors

The health monitoring issues brought by the critical role played by TBCs in the durability of jet engines led to the development of the concept of multifunction TBCs, that combine thermal insulating and optical sensing capabilities. The latter are based on the semi-transparency of zirconia in the visible range and its ability to accept small amounts of trivalent lanthanide ions that substitute for Zr<sup>4+</sup> cations in its lattice and act like luminescent activators under appropriate UV or visible illumination (Fig. 1), thus providing information about their local environment. First introduced by Amano et al [16] in 1988, multilayer TBCs alternating various luminescent and undoped layers allow one to evaluate the progression of TBC erosion from the appearance/ extinction of luminescent peaks or directly from evolutions of intensity maps [17,18]. Eldridge et al. [11,12] later showed that the effect of reflectance enhanced luminescence, which creates an intensity contrast between metal/YSZ:Eu and air/YSZ:Eu interfaces, can reveal localized delaminated areas at the interface region in APS-TBCs integrating a 7-10 µm luminescent sublayer. Finally the application of phosphor thermometry methods [19,20], mainly based on the dependence of luminescence intensity and decay time on temperature, allows nonintrusive optical sensing of buried luminescent layers to determine through-thickness temperature. Application to bilayer systems such as YSZ:Dy/YSZ [9,21] and YSZ:Eu/YSZ [10] has thus allowed measurements of substrate/YSZ interface temperature. The existence of several dopants with distinct luminescence emission peaks suggests that more complex layered TBC architectures, also referred by Gentleman and al. [6] as "rainbow sensors", could be powerful tools to measure the complete temperature gradient or heat flux across TBCs. Fig. 1 presents the luminescence spectra of the various luminescent activators in 9.75-YSZ selected for this work on the basis of their luminescence properties and performance as thermographic phosphors either in YSZ or other matrices. Several doublets and triplets of compatible activators such as Tm-Er-Eu or Dy-Er-Eu are thus indentified as valid candidates to build multiwavelength TBCs sensors for through thickness health and temperature monitoring. It should be born in mind that although Dy<sup>3+</sup> and Tm<sup>3+</sup> appear to have distinct emissions, thermalization of the  ${}^{4}I_{15/2}$  level of  $Dy^{3+}$  at high temperature will generate an additional peak at 458 nm potentially overlapping the peaks of Tm<sup>3+</sup> [22], limiting to three the number of functionalized



**Fig. 1.** Luminescence spectra in the visible range for various lanthanide-doped 9.75 mol%-YSZ:Ln phosphors (Ln = Sm, Eu, Dy, Er and Tm) under long wave UV illumination from a mercury vapor lamp (365 nm). The content of Sm<sup>3+</sup>, Dy<sup>3+</sup>, Er<sup>3+</sup> and Tm<sup>3+</sup> activators correspond to the concentration for which the maximal intensity is achieved.

luminescent layers that could be used for this range of activators. Of all dopants presented in Fig. 1,  $Eu^{3+}$ ,  $Er^{3+}$  and  $Dy^{3+}$  exhibit the brightest luminescence in YSZ in addition to having distinct emission peaks in the red, green and blue region of the visible spectrum respectively, that therefore make them the best candidates for the design and the manufacturing of a multilayer TBC sensor prototype.

### 3. Experimentation

#### 3.1. Synthesis of YSZ phosphors

YSZ phosphor powders (9.75 mol%  $YO_{1.5}$ ) with contents of  $LnO_{1.5}$ (Ln = Eu, Er, Dy, Sm or Tm) between 0.15 and 10 at% were synthesized using the sol-gel route [23]. The sols were prepared by mixing zirconium (IV) propoxide (Zr(OPr)4) (Sigma Aldrich) and appropriate contents of yttrium (III) and Ln (III) nitrates (Sigma Aldrich) as precursors in a solution of 1-propanol (Sigma Aldrich) and ultrapure water. Acetylacetone (AcAc) (Sigma Aldrich) was used as a complexing agent to control the kinetics of hydrolysis of the zirconium alkoxide [24]. The volume rates of [AcAc/Zr(OPr)<sub>4</sub>] and [H<sub>2</sub>O/Zr(OPr)<sub>4</sub>] were kept constant at 0.8 and 9.5 respectively. More details about the preparation of the mixtures from the reagents can be found in reference [23]. After 30 min of mechanical stirring the solutions were then held for one night (~16 h) at 50 °C to accelerate the hydrolysis and condensation of the sols into bright monolith gels free of precipitates. The solvent was then evaporated in a drying oven at 70 °C. The resulting dried gels, called xerogels, were subsequently calcined for 2 h at 800 °C to remove the remaining traces of solvents and then manually ground to powders for 4 min. The products obtained at this stage are white powders consisting in 2–50 µm particles of partially crystallized YSZ. An additional heat treatment of the powders at 1100 °C for 2 h ensured the full crystallization of the YSZ into metastable quadratic phase t' or cubic phase c depending on their composition.

### 3.2. Deposition of TBC sensors

Multilayer TBC sensor prototypes containing luminescent layers of composition  $Ln_xY_{0.0975-x}Zr_{1-0.092-x}O_{1.951-2x}$  (Ln' = Eu, Er or Dy) with distinct emissions wavelengths were shaped by a dip-coating process previously developed [14,15].

First YSZ aerogel powders were synthesized from a sol-gel protocol adapted from Lecompte et al. [25]. Rare earth doped YSZ gels were prepared as described in part 3.1 but were dried above the supercritical point of 1-propanol ( $T_c = 261$  °C;  $P_c = 5.1$  MPa) in a stainless steel autoclave (Paar Instrument 4621) instead of being dried in free air. Temperature was kept constant at 270 °C and pressure at 9 MPa for 1 h, afterwards the solvent was slowly released in isothermal conditions. The resulting brittle monolithic aerogels whose structure is similar to that of the wet gels were calcined for 2 h at 700 °C and ball milled at 250 rpm for 1 h in an agate mortar to obtain highly porous powders having morphological and grain size characteristics more suitable for dispersion and deposition by dip-coating than xerogel powders obtained through conventional drying at atmospheric pressure [23,26].

The substrates used for the depositions were AM1 single crystal nickel based superalloy coupons coated with Ni(Pt,Al) bondcoat, previously sand blasted with 10  $\mu$ m corindon particles and preoxidized for 2 h at 950 °C under a O<sub>2</sub> pressure of 0.05 Pa. Slurries were prepared by mixing 20 wt% of YSZ sol with YSZ aerogel powder previously dispersed in 1-propanol with polyvinylpyrrolidone 3500 (PVP) (Acros Organics) dispersing agent. The depositions were carried out at room temperature by dipping the substrates in the slurry with a controlled withdrawal rate of 250 mm/min to ensure the deposition of a ~10–15  $\mu$ m thick layer at each dip procedure. The deposited films were then dried for 5 min at 50 °C before implementing the next dip-coating. Coatings of final thickness in the range of 100–150 microns were obtained after 13 successive dips. Multilayer prototypes alternating

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