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High temperature mechanical characterization of plasma-sprayed zirconia–yttria from conventional and nanostructured powders

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ABSTRACT

Plasma-sprayed yttria partially stabilized zirconia (YSZ) may experience densification during high temperature service life affecting those properties that are directly related to porosity. Nanostructured YSZ has received specific attention due to peculiar properties that are supposed to properly counteract the effects of densification.
The present study investigates and compares thermo-mechanical properties of plasma sprayed YSZ obtained from conventional and nanostructured powders, in order to appraise whether and how microstructure affects high temperature mechanical behavior.
Porosity measurements, Knoop hardness measurements, Young's modulus evaluation by dynamic indentation, dilatometric analysis and X-ray diffractometric analysis were carried out both on as-sprayed samples and on samples previously exposed at high temperature.
Four-point bending tests were carried out from room temperature to 1500 °C for both types of materials.
Results show that nanostructured YSZ coatings tend to preserve their porosity for higher temperatures than the conventional coatings. High temperature mechanical tests indicate analogous increases of Young's modulus with test temperature up to 1000 °C for nanostructured and conventional coatings (with higher stiffness for nanostructured deposits), but remarkably different behavior when approaching the sintering temperature.

1. Introduction

Thermal barrier coatings (TBCs) are commonly employed as an insulation system for hot sections both in aerospace and land-based turbine components, in order to protect metallic parts from high temperature degradation [1,2].

State-of-the-art TBCs consist of a metallic bonding layer (MCrAlY, with M═Ni and/or Co) and a ceramic top coat made of 7–8 wt.% yttria partially stabilized zirconia (YSZ) with a thickness of about 100–500 μm [3]. Air plasma spray (APS) and electron beam physical vapor deposition (EB-PVD) are the two main process techniques used to deposit ceramic coatings [3].

The successful use of YSZ as the top insulating layer is due to beneficial properties such as a relatively high coefficient of thermal expansion, thermodynamic and chemical stability at high temperature and low thermal conductivity [4]. However, YSZ undergoes phase transformations that may cause a volume expansion detrimental to the in-service behavior and durability of the top coat, possibly leading to its early failure [4–7]. In addition to phase transformation, progressive sintering occurring with high temperature exposure may negatively affect both mechanical and thermal properties of the selected top coat. In fact, an increase in thermal conductivity may be detrimental to the coated component, since a thermal conductive barrier would fail in insulating the metallic turbine blade from the hot gases [8–10].

In recent years nanostructured coatings have received wide attention due to potentially superior mechanical and thermal properties [4, 11–15]. Because of better wear resistance, lower thermal conductivity and higher coefficient of thermal expansion, coatings obtained from nanostructured powders are expected to provide improved performance of the ceramic topcoat as compared with the conventional TBCs [4,10]. Some authors [11] have shown that a refined agglomerate morphology with a particle size below 1 μm may increase the strain accommodation ability at room temperature, decreasing the elastic modulus. Literature also shows that the non-molten nanostructured zones present in YSZ coatings obtained by atmospheric plasma spraying (APS) may decrease the thermal conductivity and increase the thermal shock resistance of the thermal barriers [16].

Limited information is available in the literature concerning high temperature mechanical properties and microstructural evolution of conventional and nanostructured coatings [16,17]. Knowledge and control of these features are essential requirements to further optimization of the performance and service life of zirconia-based thermal barriers.

The study of the effect of temperature on mechanical properties of plasma sprayed self-standing coatings produced from nanostructured

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and conventional feedstock materials at temperatures ranging from 20 °C to 1500 °C is the object of the present investigation. The limit of 1500 °C was selected because it approximates the sintering temperature of zirconia [18,19], and the evaluation of mechanical properties as the material approaches an almost total sintering can provide valuable information for the design of TBCs and, in general, more insight for the characterization of high temperature ceramics.

Results of bending tests, porosity measurements, Knoop hardness tests, elastic modulus evaluation by dynamic indentation, phase analysis by X-ray diffractometry and dilatometric analysis of the coatings are reported and discussed.

2. Experimental

Yttria partially stabilized zirconia (ZrO_2–8 wt.%Y_2O_3) thick coatings from conventional (Metco 204 NS 110, Sulzer-Metco AG, Winterthur, Switzerland) and nanostructured (Nanox S4007, Inframat Corp., North Haven, CT, USA) powders were deposited onto AISI 316 stainless steel by air plasma spray using an Ar/H_2 APS torch (Sulzer-Metco F4MB).

Operating parameters for conventional and nanostructured zirconia coatings, selected from pre-optimization procedures based on coating cohesion, are reported in Table 1.

The substrate was mechanically removed after the deposition and the freestanding ceramic was cut in 32 prismatic bars (45 × 4 × 3 mm), in the number of 16 per each material.

A subset of the samples was exposed in furnace in air at 500, 1000 and 1500 °C for 30 min and then cooled in air. The heating ramp and dwell time of thermal treatment were set in order to simulate the high temperature exposure during the bending test, and are reported in Table 2.

Samples were prepared for porosity and microhardness measurement by mounting in epoxy resin and polishing with a sequence of 400, 600, 800 and 1200 μm SiC papers and 9, 6, 3, and 1 μm diamond suspensions (Presi Mecatech P 120 Polishing Machine, Presi SA, Tavernolles, France).

Porosity analysis was performed on digital images of coating cross sections (ASTM E-2109-01) [20,17] at 200× magnification, with a Nikon Eclipse L150 optical microscope (Nikon Instruments B.V., Amsterdam, The Netherlands) and using the LUCIA Measurement image analysis software. Porosity was calculated as an average of 10 values measured on 10 different images for each cross section.

Knoop microhardness measurements were performed on cross section of samples at 25, 50 and 100 gf (maximum load was reached in 10 s and maintained for 15 s), followed by optical evaluation of indentation size (ASTM E-384-89) [21] using a Leica VMHT Microhardness Tester (Leica Microsystems GmbH, Wetzlar, Germany). Thirty indentations were carried out for each load.

Evaluation of Young's modulus by dynamic indentation (ISO 14577:2007) [22] was performed using an instrumented indentation system (Nanotest, MicroMaterials Ltd., Wrexham, UK) [22]. A maximum load of 500 mN was applied for 10 s, using a Berkovich 3-sided indenter. Both loading and unloading velocity was fixed at 50 mN/s. Post-test analysis of load/penetration curves was carried out using the approach described by Oliver and Pharr [23]. Values for Poisson ratio for plasma-sprayed YSZ were selected from the data base for thermal sprayed YSZ proposed in [24] and were assumed variable from 0.05 for as-sprayed samples to 0.2 for heat treated samples. Indentation tests were carried out both on as-sprayed samples and on samples exposed at high temperature in air atmosphere [25].

Table 1
Operating parameters for the deposition of yttria stabilized zirconia coatings from conventional (samples A) and nanostructured (samples B) feedstock materials.

Samples	Spray distance (mm)	Gas flow rate (slpm)		Current (A)	Voltage (V)	Carrier gas flow rate (Ar) (slpm)
		Ar	H_2			
Micro	110	50	14	510	73	3.05
Nano	110	37	13.5	750	73–74	2.7

Table 2
Heating ramps and dwell times for heat treatment of both conventional and nanostructured samples. T_f is the final temperature of the heat treatment.

Heating ramp step	1	From room temperature up to 40 °C at 5 °C/min
	2	From 40 °C up to T_f-50 °C at 15 °C/min
	3	From T_f-50 °C up to T_f-5 °C at 10 °C/min
	4	From T_f-5 °C up to T_f at 5 °C/min
High temperature dwell time (minutes)	500 °C	30
	1000 °C	30
	1500 °C	30

Dilatometric analysis (ASTM E-228-06) [26] was performed on as-sprayed coatings from room temperature up to 1500 °C with a gradient of 3 K/min, in order to compare the thermal expansion trends for both type of YSZ coatings. The test was performed with a LINSEIS L76 linear dilatometer (LINSEIS GmbH, Selb, Germany) with an alumina gauge.

X-ray diffractometry (XRD) was performed with a Philips X'Pert^3 device (PANalytical B.V., Almelo, The Netherlands) on pulverized samples in order to investigate whether phase transformation occurred during high temperature exposure. The XRD device operated at 40 kV and 40 mA with Cu Kα radiation ($\lambda_{K\alpha1}$ = 1.540598 Å, $\lambda_{K\alpha2}$ = 1.544426 Å), with a scan range of 10–80° (2θ), step size of 0.02° and a counting time of 2 s.

A scanning electron microscope (SEM) analysis was carried out to investigate microstructure and morphology of materials using a FESEM Zeiss Auriga 405 (Carl Zeiss Microscopy, Oberkochen, Germany).

Finally, mechanical characterization at high temperature was carried out performing four-point bending tests in air (according to ASTM C1211 and C1161) [27,28] at room temperature, 500, 1000 and 1500 °C on the remaining samples. The elaboration of stress–strain curves allowed the evaluation of Young's modulus, modulus of rupture (MOR) and deformation at break. Four-point bending tests were performed with a Zwick-Roell Z 2.5 testing machine (Zwick GmbH, Ulm, Germany) equipped with a Maytec furnace (up to 1600 °C), a 3-point-contact extensometer, and a silicon carbide fully-articulated flexure device (Maytec GmbH, Singen, Germany) (Fig. 1). Stress–strain curves elaboration was performed by Zwick-Roell TestXpert II software. The measurement of sample deflection on three separated points allowed to exclude the undesired contribution of thermal expansion both of alumina extensometer rods and loading tubes: the described experimental setup enables a direct and reliable measurement of sample curvature, with appreciable improvements as compared to the control of cross-head travel or the direct LVDT measurement of sample deflection performed only in the central section of the sample [29–31].

3. Results and discussions

3.1. Microstructural evolution

The evolution of the microstructure of both types of coatings for increasing exposure temperature offers important elements for the interpretation of the overall physical and mechanical characterization results.

Fig. 2 shows SEM images of cross sections of conventional YSZ coatings after different heat-treatments. The as-sprayed microstructure (Fig. 2a) is porous, and lamellae of molten and semi-molten particles typical of APS deposits are shown. A gradually denser structure can be observed in the samples exposed at 500 °C and 1000 °C (Fig. 2b and c), suggesting that the densification processes are active at these temperatures. After heat treatment at 1500 °C (Fig. 2d) the densification process has further advanced, the amount of pores is strongly reduced