



Characterization of microstructure and thermal properties of YCSZ coatings obtained by suspension plasma spraying

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ABSTRACT

The paper describes the procedure of determination of thermal properties (thermal diffusivity and thermal conductivity) of yttria/ceria stabilized zirconia coatings. The coatings were deposited by suspension plasma spraying method using SG-100 torch. The commercial powder with a chemical composition $\text{ZrO}_2 + 24 \text{ wt.}\% \text{ CeO}_2 + 2.5 \text{ wt.}\% \text{ Y}_2\text{O}_3$ was used and coatings were sprayed onto stainless steel substrates. The suspensions consisted of zirconia powder, water, ethanol and small addition of dispersant. The design of spray experiments included the following variable process parameters: (i) spray distance; and (ii) torch scan linear speed. The microstructure of coatings was determined by scanning electron microscopy (SEM) and X-ray diffraction (XRD). The phase composition and the crystallite size of coatings were estimated by Rietveld method. The different methods were used to find out the thermal properties values up to 573 K. Firstly, thermal diffusivity of YCSZ coatings was determined by using a commercial setup and the obtained results were used to calculate the values of thermal conductivity. Then, to check the validity of the measurements and calculations, the simulations of laser flash experiment were made using COMSOL Multiphysics software. The least square method enabled to find the optimized value of thermal conductivity. Finally, the use of another setup enabled to find out the thermal transport properties in the temperature up to 873 K.

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1. Introduction

Thermal barrier coatings (TBC) are important elements of gas turbine technology applied mainly in aviation industry [1,2]. Two different reasons of their application can be distinguished. First is the possibility of extending lifetime at the same operating temperatures. The second one is the rise of the operating temperature resulting in better turbine efficiency.

The turbine blades work in demanding conditions and increasing of temperature inside gas turbine causes further increasing of requirements for TBC's materials [3]. Consequently, the research of new deposition technologies and new materials has been continuing [4].

A part of them is research of new material for overlay coatings which includes the rare earth zirconates having also additions of elements like gadolinium, lanthanum, ytterbium or cerium [5–7]. On the other hand, new technologies have been developed to produce the coatings with improved parameters. One of these technologies is suspension plasma spraying (SPS). The SPS process may enable modifying the microstructure of coatings, e.g. thickness, porosity, morphology, etc. [8–10].

The presented experiments are devoted to yttria and ceria stabilized zirconia (YCSZ) coatings produced by SPS method. The previous studies on this material showed that YCSZ can work in higher temperatures and is characterized by higher resistance to high temperature corrosion than YSZ [11–13]. The present study focuses on the thermal transport properties of YCSZ coatings.

2. Experimental material

The commercial powder Metco 205NS (YCSZ) prepared by spray drying and heat treatment was used to produce coatings by SPS method. The initial volume-surface mean diameter of this powder was equal to $d_{vs} = 38 \mu\text{m}$. The powder was crushed mechanically to decrease the particles sizes. The mechanical ball milling was realized with the use of moliNEx system (NEZTSCH, Germany), the zirconia balls used as grinding medium and ethanol as cooling medium. Finally the powders were sieved prior to suspension formulation.

After milling process resulting powder had a mean size equal to $d_{vs} = 3.91 \mu\text{m}$ and monomodal sizes' distribution. The distribution was measured with the use of laser diffraction technique (Partica LA-950V2, Horiba). Moreover, SEM micrographs of the milled powder (Fig. 1) showed finely grained particles, confirming the granulometry tests.

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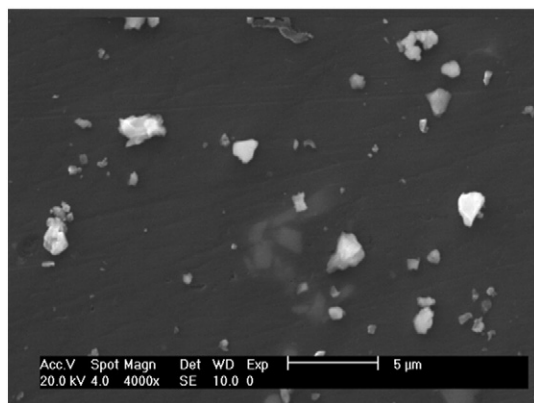


Fig. 1. SEM image (secondary electrons) of Metco 205NS powder after milling process.

The suspension used to spray experiments consisted of 20 wt.% of milled zirconia powder with 40 wt.% of distilled water and 40 wt.% of ethanol. The dispersant agent, phosphate ester (Beycostat C213, CECA), was added to prevent particles agglomeration. The zeta potential measured using Zetasizer Nano ZS (Malvern, England) of these suspensions was equal to $\zeta = -6.2$ mV. The value is relatively low.

3. Experimental methods

3.1. Spray process

Plasma spraying process was performed using a SG-100 torch (Praxair S.T., Indianapolis, In, USA) installed on a 5-axis IRB-6 robot of ABB (Zürich, Switzerland). The working gas mixture of Ar + H₂ (45 + 5 slpm) and electric power of 40 kW were used. The experiments were made using 2^k ($k = 2$) full factorial design, where the variable parameters were robot scan speed (from 300 mm/s and 500 mm/s) and spray distance (from 40 mm and 60 mm). The suspension feed rate was of about 39 g/min. The suspension was introduced via internal injection mode using a continuous-stream injector (with an internal diameter of 0.5 mm) installed inside the anode-nozzle of the plasma torch. The suspension spraying was performed in order to obtain coatings with a thickness up to 70 μm. The operational spray parameters are collected in Table 1.

Stainless steel 304L cylinders (diameter 25 mm and thickness about 8 mm) were used as the substrate material. They were sand blasted using corundum grit (F36 according to the FEPA standards) under a pressure of 0.04 MPa and cleaned in ethanol before spray process. Furthermore the thickness of substrates was controlled before and after spraying to ensure the same deposition conditions for all samples and finally of thermal transport properties measurements also.

3.2. Coating characterization

The basic characterization of coatings, including the determination of their thicknesses, was made with the use of the light microscopy

Table 1
Variable spray parameters and the values of thickness and porosity of coatings.

Run	Scan velocity [mm/s]	Spray distance [mm]	Range of maximal surface temperature [°C]	Thickness [μm]	Thickness, standard deviation [μm]	Porosity [%]
1	300	40	668–736	53	2.9	21.5
2	500	40	509–643	90	8.1	18.7
3	400	50	453–603	78	1.6	20.3
4	300	60	422–563	101	2.7	15.1
5	500	60	393–474	88	1.1	20.6

(Nikon Eclipse LV100). The procedure of thickness measurement was as follows—for each sample 3 regions were chosen and in each region 5 local thicknesses measurements were made. Finally, the average value for each region and the average value for these 3 regions were calculated.

The morphology of obtained ceramic coatings YCSZ was characterized with the scanning electron microscope Philips 515 (Eindhoven, Netherlands) using a secondary electron detector. The micrographs were made on the top of surfaces and on the cross-sections of the coatings. To estimate the porosity of coatings SEM images were taken at 10,000 × magnification. Then, the images were edited using ImageJ free software for the porosity calculations.

The phase composition was estimated by X-ray diffraction analysis using a D8-Bruker apparatus (Bruker AXS, Karlsruhe, Germany) with Cu-Kα1 radiation. The measurements were made in the wide range of 2θ angles from 15° to 120°. The phases were identified using Diffrac + Eva software. The quantitative analysis of coating composition was made with the use of Rietveld method. In the same way the lattice parameters of coatings were determined.

3.3. Thermal diffusivity measurements

Thermal diffusivity of YCSZ coatings was determined by using laser flash method. It is one of the most popular methods that enable thermal diffusivity characterization, where the bottom side (the substrate) of the sample is heated by energy pulse and the change of the temperature versus measurement time is collected by the detector on the top of the sample (the TBC coating). The sample is coated few times with the thin graphite layers prior to measurements to increase signal-noise ratio and provide good quality of analysis.

During the research two devices LFA 447 and LFA 427 both of Netzsch, Germany, were used. The LFA 447 setup, equipped in a xenon lamp allowed analyzing the coatings up to 573 K. The use of LFA 427, which used the laser as the heat source enabled obtaining the values of thermal diffusivity in higher temperatures. Because the measurements were made with the use of the coatings attached directly to the substrates (without the intermediate layer), the measurements in this case were made up to 873 K to avoid the effect of the difference of linear expansion coefficients. The chosen range of the temperature did not require the use of shielding gas or vacuum—the measurements were made in the air in both cases. The two measuring systems required also two different sample shapes, for LFA 447 it was necessary to prepare 25 mm discs and for LFA 427 10 mm squares. Moreover the setups have installed the same kind of infrared detector—InSb. Finally the 2-layer numerical model was developed to determine thermal diffusivity of the coatings. The model included corrections resulting from: (i) heat losses; (ii) pulse duration, and (iii) contact resistance between substrate and zirconia coating.

3.4. Thermal conductivity calculations

To calculate thermal conductivity of sprayed coatings, it was necessary to determine the values of: (i) thickness, (ii) density, (iii) specific heat, (iv) thermal dilatation, and (v) thermal diffusivity for each layer of material.

The thickness of coatings was found in using observation of the cross-section with the optical microscope. The density values were corrected for porosity basing on following expression:

$$\rho = \rho_0 \cdot (1 - P) \quad (1)$$

where: ρ_0 is the density of tetragonal phase ZrO₂, equal to 6050 kg/m³ and P is porosity.

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