



Thermal diffusivity of TBC: Results of a small round robin test and considerations about the effect of the surface preparation and the measuring approach

F. Cernuschi ^{a,*}, P. Bison ^b, J.G. Sun ^c

^a RSE – Ricerca sul Sistema Energetico, Via Rubattino, 54, 20134 Milano, Italy

^b CNR ITC, C.so Stati Uniti, 4, 35127 Padova, Italy

^c Argonne National Laboratory, 9700 South Cass Avenue, Argonne, IL 60439, USA

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ABSTRACT

Among the techniques used to measure the thermal diffusivity of TBC, the Laser Flash is a standard. Nonetheless, this technique shows two main limitations related to the size and the well-defined geometry of the specimens. Furthermore the most reliable data can be typically obtained only on freestanding coatings. On the contrary, other photothermal and thermographic techniques in reflection configuration (the same side is heated and temperature detected) can overcome these limitations. One aspect, only partially studied in the literature, is common to most of the photothermal and thermographic techniques. It concerns the effect of the blackening coating used for guaranteeing the absorption of the heating radiation just within a very shallow outer layer and to make opaque the TBC in the sensitivity range of the IR detector/camera.

For this purpose, an inter-laboratory round robin has been promoted for comparing the thermal diffusivity in dependency of the blackening layer deposition technique, the TBC microstructure (in particular porous APS, columnar EB-PVD and PS-PVDTM) and the three different experimental set-ups in terms of spectral range and frame rate of the IR cameras, heating source and data reduction.

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1. Introduction

Thermal barrier coatings (TBCs) are widely applied for protecting hot path components of gas turbines from combustion gases. By this way, the temperature on metallic substrates can be reduced by 30 °C to 100 °C, depending on the thickness and on the specific properties of the insulating layer [1,2]. The state-of-the-art of these TBCs is represented by yttrium oxide partially stabilized zirconium (YPSZ) oxide (7–8 wt.% Y₂O₃ + ZrO₂) deposited onto the components either by atmospheric plasma spray (APS) or by electron beam physical vapor deposition (EB-PVD) [1,2].

The thermal insulating ability of the TBC is related to its thermal conductivity and thickness. The thermal conductivity of TBC is the result of the combined effect of the thermal conductivity of the bulk material and its peculiar porous microstructure. As discussed in the literature, the same temperature drop across the TBC thickness could be achieved depositing onto the component either a thin TBC with a very low thermal conductivity or a thicker TBC with higher thermal conductivity [3–5].

In any case, independently from the followed approach, the TBC microstructure should be correctly designed and deposited by properly choosing the deposition parameters to obtain the desired thermal

conductivity and elastic modulus, according to the operating conditions of the specific component the TBC will be sprayed onto. Furthermore, it is also important to study the evolution of the TBC microstructure along the operation owing to the sintering phenomena occurring at high temperature. In fact, sintering promotes a significant reduction of both the insulating ability and the strain compliance of the TBC [6–14].

A very wide range of thermal diffusivity values of TBCs are reported in the literature [7,8,11,14–17] owing to the several microstructures that can be obtained in dependency of powder, production routes, deposition techniques and process parameters.

Possible additional causes of the great variability of the TBC thermal diffusivity reported in the literature, that rarely are taken into account, are related to the experimental testing conditions (not always fully provided in the literature), such as for example the measuring atmosphere (i.e. gas and pressure), the sample preparation, the measuring technique, the experimental set-up and the algorithm used for parameter estimation. To give an example of this, the thermal diffusivity of a porous APS TBC could be reduced by a factor close to 2, when the measurement is carried out in a vacuum instead of in air at atmospheric pressure and room temperature [14,18].

Among the techniques used to measure the thermal diffusivity of TBC, the Laser Flash is a standard [19,20]; the main limitations are the size and the well-defined geometry (disks or parallelepipeds) of the specimens. Moreover, although this technique can be applied also on

* Corresponding author. Tel.: +39 02 3992 4577; fax: +39 02 3992 5626.
E-mail address: federico.cernuschi@rse-web.it (F. Cernuschi).

two layer samples, the most reliable data can be typically obtained on freestanding coatings.

In the last decades, photothermal¹ techniques in reflection configuration (i.e. the heating and temperature detection confined to the same side of the sample) such as Pulsed Photothermal Technique [21], Time Resolved Infrared Radiometry (TRIR) [22], Thermal Waves Interferometry (TWI) [23] and PopTea [24,25], have demonstrated their ability to measure the thermal diffusivity on TBC deposited onto substrates without any special requirement on the sample size and geometry. In particular, measurements on real components have been recently reported [25,26].

In the last fifteen years, the availability of high resolution focal plane array IR cameras with performances comparable to single IR detectors allowed reliable mapping of the thermal diffusivity of TBC deposited onto samples and components [14,27–30].

One aspect only partially studied in the literature [31,32] common to most of the photothermal and thermographic techniques concerns with the effect of the blackening coating used for guaranteeing the absorption of the heating radiation just within a very shallow outer layer and to make opaque the TBC in the sensitivity range of the IR detector/camera.

The most applied coatings consist in thin graphite, Au or Pt layers [33,34], these latter deposited typically by sputtering. It has been reported that a large difference (>10%) in measured TBC properties may be observed when using different graphite coatings for certain TBCs [35].

For this purpose, an inter-laboratory round robin has been promoted for comparing the thermal diffusivity in dependency of the blackening layer deposition technique, the TBC microstructure (in particular porous APS, columnar EB-PVD and PS-PVDTM) and the three different experimental set-ups in terms of spectral range and frame rate of the IR cameras, heating source and data reduction. Three laboratories participated to the round robin and they are referred in the following with the acronyms ANL (Argonne National Laboratory, USA), CNR (Consiglio Nazionale delle Ricerche, Italy) and RSE (Ricerca sul Sistema Energetico, Italy).

2. Experimental

2.1. The samples

The experimental activity has been performed on three TBC samples: one standard porous $369 \pm 7 \mu\text{m}$ thick APS (APS163), one $175 \pm 2 \mu\text{m}$ thick EB-PVD (EB-PVD1) and one $140 \pm 3 \mu\text{m}$ thick Plasma Spray – PVDTM (SYS3) TBCs. CMSX4 single crystal Ni-base superalloy was used as substrate (4 mm thickness 25 mm diameter disks), APS TBC has been sprayed onto a $\approx 300 \mu\text{m}$ CoNiCrAlY bondcoat (Amdry 995) deposited by LPPS (Low Pressure Plasma Spray) technique. For both PVD TBCs, the bondcoat was a diffusion Pt–Al coating of about $50 \mu\text{m}$ thick. Fig. 1 shows the microstructure of all the three TBC systems as seen from the top surface and along the section.

In the following, a short description of each TBC system is given.

2.1.1. APS standard porous TBC

The ceramic top coating has been deposited by air plasma spray, using ARTEC SpA F4A type torch and YPSZ commercial powder (Amperit 827.7 produced by H. C. Starck) with a particle size distribution in a range from 45 to $90 \mu\text{m}$. The cubic and tetragonal phase contents are 98 and 1 wt.% respectively; whereas the monoclinic phase content is 1 wt.% in the as-received feedstock.

2.1.2. PS-PVDTM TBC

The PS-PVD process is based on the ChamProTM technology of Sulzer Metco which comprises all those thermal spray processes performing under a defined and controlled atmosphere like LPPS, VPS and LVPS [36]. The PS-PVDTM process operates at lower pressures down to 0.5–

2 mbar. Under these conditions, the properties of the plasma jet change substantially [37]. Even though the PS-PVDTM work pressure (~ 1 mbar) is still much higher than the one used in conventional PVD processes ($\sim 10^{-3}$ mbar), the combination of a high energy plasma gun operated at a low pressure environment enables a defined evaporation of the injected powder material. This allows one to produce a controlled deposition out of the vapor phase. In EB-PVD, the transport of the vaporized coating material towards the substrate is done through a diffusion process having a limited throughput and producing coatings at low growth rates. In contrast to that, in the PS-PVDTM process the vaporized coating material is transported in a hot and supersonic gas stream ($2000\text{--}4000 \text{ ms}^{-1}$, $6000\text{--}10,000 \text{ K}$) which is expanding in a 1 mbar atmosphere. This leads to high growth rates and the possibility to coat undercuts and areas which are not in the line-of-sight. More details about the PS-PVDTM deposition technique can be found in the literature [38].

2.1.3. EB-PVD TBC

The topcoat has been deposited via the EB-PVD process. The substrate is mounted on a rotating holder positioned perpendicular to the ingot axis and heated in the range of $900\text{--}1000 \text{ }^\circ\text{C}$. The evaporation source material comes from an ingot with standard ZrO_2 7 to 8 wt.% Y_2O_3 chemical composition. Evaporation of the ingot is achieved by applying an electron power gun. During deposition the chamber pressure is kept in the 10^{-3} mbar range.

2.2. The approach

To guarantee the highest level of significance of the results it is essential to design the experimental activity carefully. The main issue to consider in designing the round robin to investigate the effect of the different blackening layers concerns the following alternative choices:

- to use the same TBC sample (for each deposition technique, i.e. plasma spray, PS-PVD, EB-PVD) with the consequent needs of removing the old blackening coating before to apply the new one;
- to coat different TBC samples (even though obtained with the same deposition technique) with the different blackening layers selected for the experimental activity.

In order to answer this question, the spread of thermal diffusivity values of three sets of TBC (deposited in the frame of the European Project TOPPCOAT) similar to those selected for the experimental activity was considered, as summarized within the probability plot in Fig. 2.

It is worth noting that the relative spread of thermal diffusivity (i.e. $(\text{max} - \text{min}) / \text{max}$) ranges from 9% to 18% depending on the TBC set. Since the aim of this experimental activity is to detect small thermal diffusivity variations caused by the blackening layer, it is essential to minimize all the error sources. The results of the probability plot suggest the use of the same sample for each TBC deposition technique to get rid of the variability of thermal diffusivity caused by the small microstructural variations from sample to sample.

This choice requires the consideration on how to remove the blackening layers without significantly altering the sample microstructure, and thus its thermal diffusivity by a significant amount. Since the selected blackening layers are all based on graphite, the candidate solution to remove graphite consists in burning it in a furnace at a temperature slightly higher than the $650 \text{ }^\circ\text{C}$. Based on previous trials, the suggested heat treatment consists in heating the sample at $700 \text{ }^\circ\text{C}$ for 1 h.

A preliminary study of the effect of this heat treatment on the TBC thermal diffusivity is thus required.

Since no literature on the effect of heat treatment at $700 \text{ }^\circ\text{C}$ on thermal diffusivity of TBC is available, some considerations have been done starting from data referring to higher temperatures.

Fujii and Takahashi [39] and Dinwiddie et al. [40] report thermal diffusivity variations at temperatures higher than $950 \text{ }^\circ\text{C}$ and $871 \text{ }^\circ\text{C}$, respectively.

¹ Photothermal means that an electromagnetic radiation in visible or near infrared range is used to heat a sample and the related temperature variations are detected to estimate thermo-physical properties of the sample.

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