Active thermography technique for barrier coatings characterization

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Abstract. Aim of this paper is to define and set up an experimental procedure, based on active thermography, for the characterization of thermal barrier coatings for industrial applications, above all in aerospace field. The developed procedure is intended to be a fast and reliable method, alternative to the consolidated one described in International Standards as ISO 18755 and ISO 18555. In particular, this approach consists of a pulsed active thermography, in transmission configuration, obtained by means of a laser excitation. Temperature data processing, according to and adapting Standard procedures, allowed us to determine thermal parameters as conductivity and diffusivity. Obtained results were compared in terms of thermal properties variation with respect to base and coated materials, and in term of different coating procedures. These results were also compared to those available in literature.

1. Introduction

In industrial aerospace field, coatings are widely applied and a typical application is as thermal barrier coating (TBC) for underneath metallic layers. In particular, Ceramic Thermal Barrier Coatings (TBCs) on superalloy components are used successfully in land-based gas turbine and aircraft engines.

These coatings are generally made by either air plasma spraying (APS) or electron beam physical vapour deposition (EB-PVD) [1]. In general, EB-PVD TBCs show a superior durability due to the columnar structure, but they are very expensive compared to APS TBCs. Nowadays, Suspension Plasma Spraying (SPS) technology provides a promising methodology to produce advanced thermal barrier coatings using nanopowders [2]. This process is capable of producing coatings with different types of structure, from very dense and compact ones to columnar with high volume of vertical intercolumnar voids. As a matter of fact, SPS coatings represent a challenging field of research, both for academic interest and commercial applications. Increased coating characteristics, such as low thermal conductivity and high level of thermal cycling resistance, may be actually reached by SPS technology; SPS coatings can generate columnar microstructures typical of electron-beam physical vapor deposition (EB-PVD) TBC, but at a much lower cost [3].

To this aim, the characterization of thermal diffusivity and conductivity properties is critical for process and material selection in industrial manufacturing of turbines and other components. Recently, thermographic techniques have been developed to characterize mechanical and thermal properties of components with coatings.

Aim of this paper is to define and set up an experimental procedure, based on active thermography, for the characterization of coatings for industrial applications. This procedure is intended to be a fast...
and reliable method, alternative to the consolidated one described in International Standards. In this research activity, it was applied to different industrial coating processes to investigate the corresponding thermal properties. In particular, in the present paper two coating processes (Atmospheric and Suspension Plasma Spray, APS and SPS) applied to the same base material and the same coating material were investigated.

The active thermography analysis provided the measurement of surface temperature of specimens undergoing a thermal excitation, applied by means of a laser pulse (Pulsed Technique). Temperature data processing, according to and adapting Standard procedures, allowed obtaining thermal conductivity and diffusivity parameters. These results were compared in terms of thermal properties variation with respect to base and coated materials, and in term of different coating procedures.

2. Materials and Methods

Thermal barrier coatings investigated in this study are made of Yttria stabilized Zirconia powder. Two types of deposition process were utilised, respectively Suspension Plasma Spray (SPS) and Atmospheric Plasma Spray (APS). The base material (substrate) was an Inconel alloy 601.

Figure 1 shows the specimens adopted in this experimental activity.

![Figure 1. Specimens.](image)

Shape and dimension of specimens were chosen according to Standards [4] and [5]. A SEM analysis was performed to evaluate both structure and thickness of coatings, generated by SPS and APS deposition processes. Thickness values were necessary to calibrate experimental set up parameters. Figure 2 shows SEM results of SPS and APS specimens, emphasising both coatings structure and their thickness. A microscope analysis was also performed to evaluate both thickness of Inconel 601 specimen and of metallic layer (Inconel 601) in coated specimens.

Physical characteristics values (density and specific heat) adopted for the estimation of thermal conductivity may be found in [6] for substrate (Inconel 601) and in [7] for coatings (see Table 1).

The experimental equipment was composed by a thermal camera, a laser excitation source and a PC control unit. The IR thermo camera is a FLIR A6751sc with sensitivity lower than 20 mK and 3-5 μm spectral range, while the laser source can generate a maximum power of 50 W concentrated in a small surface.

The experimental configuration was in “transmission mode”. Figure 3 shows both equipment and configuration. More in detail, Inconel 601 was excited with an heating period of 50ms at the maximum power, while specimens coated were excited with an heating period of 5ms at the maximum power.

| Material                                  | $\rho$ [kg/m$^3$] | $c$ [J/kg °C] |
|-------------------------------------------|------------------|---------------|
| Inconel 601 (substrate)                   | 8110             | 448           |
| Yttria Stabilized Zirconia (coating)      | 5200             | 467           |

Table 1. Physical characteristics of materials.
Figure 2. SEM results of SPS and APS specimens.

Figure 3. Experimental equipment.

Thermal characterization methods for barrier coatings follow Standards [4] and [5]. ISO18755 Standard [5] allows to determine the thermal parameters (thermal diffusivity and thermal conductivity) of a single material by using the *half rise time method*. ISO18555 Standard [4] may be used for multilayer materials (as metallic coatings), allowing to determine the thermal parameters (thermal diffusivity and thermal conductivity) of each layer by using the *areal heat diffusion time method*. 
Standards [4] and [5] are generally also used to compute the apparent thermal diffusivity of multilayer materials.

Figure 4 shows an example of “Temperature rise curve” and the related parameters useful for the computation of thermal diffusivity values from experimental data (areal heat diffusion time “A” and half rise time “t1/2”).

In this work, the temperature rise curve was obtained from the temperature profile acquired by the IR thermo camera.

Standards [4] and [5] also propose different corrections to consider particular phenomena occurring during the experimental test. Centroid method (to correct the finite pulse period of the excitation source) and the Cowen method (to correct the heat losses) were adopted.

3. Results
Thermograms of all specimens were acquired during the tests. Temperature profiles utilised in the procedure were related to a box located in the centre of each specimen.

A dedicated filtering process (Gaussian filter) was performed to reduce the noise due to the high frame rate acquisition (786 Hz for the Inconel 601 and 600 Hz for the specimens with coating). In particular, 50 points were used to filter Inconel 601 thermal profiles and 20 points were used to filter coatings thermal profiles. This procedure allowed us to better identify the starting point of the heating profile.

Figure 5 shows the relative temperature \( \Delta T \) (difference between measured temperature, \( T \), and ambient temperature, \( T_{amb} \)) of each specimen. A specific thermal excitation was tuned for the two different types of samples (substrate and substrate+coating), according to their different thermal behaviour (thermal conductivities). Five test replications were considered for Inconel 601 specimen (substrate material) (Test 1, 2, …,5), to better compute the heat diffusion time, useful for coatings thermal characterization. Three test replications were taken into account for APS and SPS specimens (Test 1, 2, 3).

Figure 6 shows a comparison between the relative temperatures of APS and SPS, measured with the same thermal excitation.
By analysing Figures 5 and 6, it may be pointed out that Inconel 601 base material and Inconel 601 with coatings show a different thermal behaviour. As expected, the base material emphasises a substantially higher relative temperature respect to the corresponding of coated specimens (see Figure 5). On the other hand, specimens with TBC, regardless of the production process, show a similar relative temperature trend during the tests (see Figure 6).

**Figure 5.** Relative temperature $\Delta T$ of each specimen.

**Figure 6.** SPS and APS: relative temperature $\Delta T$.

All temperature profiles were processed, according to Standards [1] and [2], by considering the temperature increment up to the maximum temperature value. Figure 7 shows the normalized relative temperature profile of each test with respect to its maximum value. This elaboration allowed us to
determine the parameters of interest (*areal heat diffusion time* “A” and *half rise time* “t_{1/2}”) and to realize the thermal characterization of both substrate and coatings.

![Figure 7. Normalized relative temperature ΔT of each specimen.](image)

Table 2 illustrates the results concerning the thermal characterization of the substrate (averaged values of all replications) (Inconel 601) by applying Standards [4] and [5] formula. Ideal thermal diffusivity and thermal conductivity values calculated by ISO18755 Standard [5] (monolayer ceramic material model) are presented in the left side of the table (*α_{ideal}* and *k_{ideal}*), while the corresponding values obtained by ISO18555 Standard [4] (multi layer material model) are shown in the right side one (*α_{1-ideal}* and *k_{1-ideal}*). Ideal thermal parameters [5] were also corrected by both Centroid Method (*α_{tc} and k_{tc}) and Cowen Method (*α_{Cowen5}, k_{Cowen5}, α_{Cowen10} and k_{Cowen10}*) and represented in the same table (Table 2, left side). Finally, Table 2 reports the calculated heat diffusion time of the substrate (*τ_{A}*).

| Thickness: 2 mm | ISO 18755 [5] | ISO 18555 [4] |
|-----------------|---------------|---------------|
|                 | *α_{ideal}* [m^2/s] | *k_{ideal}* [W/m°C] | *α_{tc}* [m^2/s] | *k_{tc}* [W/m°C] | *α_{Cowen5}* | *k_{Cowen5}* [W/m°C] | *α_{Cowen10}* | *k_{Cowen10}* [W/m°C] | *α_{1-ideal}* | *k_{1-ideal}* [W/m°C] | *τ_{A}* [s] |
| Inconel 601     | 3.31E-06      | 12.02         | 3.82E-06      | 13.90         | 3.84E-06      | 13.96         | 3.83E-06      | 13.90         | 3.53E-06      | 12.82         | 1.2504         |

Table 3 illustrates the corresponding results related to coated specimens (substrate and TBCs), as for base material the results are averaged on all replications. Tests performed on coated specimens allowed us to characterize both SPS and APS coatings, once the heat diffusion time (*τ_{A}* of the substrate was calculated (see Table 2). The layout of Table 3 (SPS and APS coatings) is the same already utilised for Table 2, the left side refers to the ISO18755 Standard [5] approach and the right side to the ISO18555 Standard [4] one. In particular, respectively for APS and SPS coated specimens, the following results
are shown in the left side of the table: ideal apparent thermal diffusivity values ($\alpha_{\text{app-idea}}$), apparent thermal diffusivity values corrected by both Centroid Method ($\alpha_{\text{app-c}}$) and Cowen Method ($\alpha_{\text{app-cowen5}}$ and $\alpha_{\text{app-cowen10}}$), respectively 5 and 10 times the half rise time "t1/2"). The right side of the table shows: ideal apparent thermal diffusivity values ($\alpha_{\text{app-idea}}$) of substrate+coatings, ideal thermal diffusivity and conductivity values of coatings ($a_2$ and $k_2$).

### Table 3. Thermal parameters for SPS and APS coatings.

| Mean thickness [mm] | $a_{\text{app-ideal}}$ [m$^2$/s] | $a_{\text{app-c}}$ [m$^2$/s] | $a_{\text{app-cowen5}}$ [m$^2$/s] | $a_{\text{app-cowen10}}$ [m$^2$/s] | $a_2$ [m$^2$/s] | $k_2$ [W/m°C] |
|-------------------|-----------------------|------------------|---------------------|-----------------------|----------------|----------------|
| APS 0.1868        | 3.18E-06              | 3.09E-06         | 2.67E-06            | 2.58E-06              | 0.32           |
| SPS 0.1759        | 2.64E-06              | 2.63E-06         | 2.58E-06            | 1.24E-07              | 0.30           |

From the analysis of Table 2, it can be observed that for Inconel 601 both Standard analytical formula ([4] and [5]) provide similar diffusivity and conductivity values. These values are in a good agreement with the corresponding provided by the material producer [6] ($k = 11.2$ to $12.7$ W/m°C).

A similar consideration can be stated referring to TBCs results, basing on the analysis of Table 3. More in detail, it can be concluded that obtained thermal parameters values for SPS and APS coatings are comparable to those available in literature [7] as order of magnitude. A great tuning of data is difficult to be reached due to the variability of TBC thickness that strongly influences the obtained values.

### 4. Conclusions

The results obtained in the present work allow us to draw the following conclusions.

The experimental procedure defined and set up for thermal characterisation of industrial TBCs, based on active thermography, provided very good results comparable to those available in literature. As wished and hypothesised, this methodology can be thought as a fast and reliable method, alternative to the consolidated one described in International Standards ISO18755 and ISO18555, for which it is required a dedicated equipment.

Furthermore, this developed technique, together with its processing algorithms, provides a robust method to be used in industrial environment. As a matter of fact, from coatings point of view, it can be conclude that, despite of the difficulty in define specimens with quite constant thickness, obtained thermal diffusivity and conductivity values are representative of the actual behaviour of TBCs obtained by different production processes.

Finally, particularly for as concerns the present industrial application, it can be concluded that SPS and APS coatings show a substantial identical thermal behavior and the choice to employ one of these can be left to cost considerations.

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