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## IR thermography applied to flash experiments of semitransparent TBCs.

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#### Abstract

Thermal Barrier Coatings (TBC) are applied to protect components of gas turbines from high temperature of combustion gases.  $ZrO_2$ , the material mainly used in TBCs, is semitransparent to near IR radiation that is typically delivered by the laser in the laser flash equipment and also radiated at working temperatures that are grater than 1000 °C. Thermography and radiometers are utilized to measure thermal diffusivity of TBCs. Discussion is done on the countermeasures to obtain correct results in case of semitransparency.

#### 1. Introduction

Thermal Barrier Coatings (TBCs) are applied to protect components of gas turbines from high temperature combustion gases (see in Fig. 1 a gas turbine system for electrical power production under maintenance). Typically, they are metalceramic multi-layers structures made up of a yttria partially stabilized zirconia (7-8 wt.% Y<sub>2</sub>O<sub>3</sub>+ZrO<sub>2</sub>) deposited either by Air Plasma Spray (APS) or Electron Beam-Physical Vapour Deposition (EB-PVD) on a high temperature oxidation/corrosion resistant metallic Bond Coat (BC) (see Fig. 2).



*Fig. 1. A gas turbine system for electrical power production, under maintenance.* 

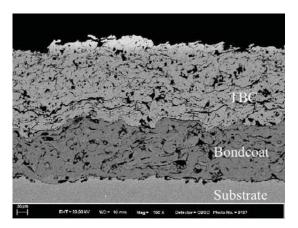
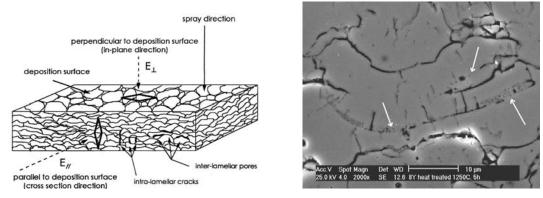


Fig. 2. A typical metal-ceramic multi-layers structures of a gas turbine combustion chamber: yttria partially stabilized zirconia (7-8 wt.%  $Y_2O_3+ZrO_2$ ) deposited either by APS or EB-PVD on a high temperature oxidation/corrosion resistant metallic bond coat (BC) to protect the metallic substrate.

The refractory ceramic porous layer can reduce the temperature of the metallic substrate by 30°C to 100°C, depending on the thickness and on the specific microstructural properties of the coating. Under typical working conditions, gas turbines are subjected to several switching on and off with consequent cyclic variations of temperature of TBCs, from ambient to working temperature (>1000 °C). That leads both to mechanical stress and to sintering phenomena that changes the characteristics of TBCs reducing the mechanical compliance and the insulating properties (see Fig. 3).

## (Air Plasma Spray deposition)



## Sketch of TBC deposition

## SEM micrograph of the heat treated TBC

Fig. 3. Scheme of TBC deposition by Air Plasma Spray technology and SEM micrograph showing the sealing of microcracks (see the arrows) due to sintering phenomena promoted by high temperature exposition. Reduction of microcracks reduces the strain compliance of the TBC under temperature variation and thermal expansion.

Measuring the variation of thermal parameters on TBCs subjected to artificial cyclic temperature variation is a way to assess the ageing effects [1]. Typically, thermal diffusivity of TBCs is determined by a laser flash apparatus [2] and IR thermography as well, both in transmission and reflection scheme [3-5]. Unfortunately, ZrO<sub>2</sub> is semitransparent to near IR radiation that is typically delivered by the laser in the laser flash equipment. Moreover, the characterization of TBCs at high temperatures is particularly interesting as the typical working temperature of gas-turbine is >1000 °C. At these temperatures heat transfer is no more limited to conduction and the radiative heat transfer becomes paramount [6, 7]:

- $Y_2O_3 + ZrO_2$  is fairly transparent to radiation with wavelength  $\lambda < 5\mu m$ 
  - i.e. typical laser shot (λ = 1064nm) pass through
  - heat is transmitted by radiation for T >> RoomTemp
- it is semitransparent for 5  $\mu$ m <  $\lambda$  < 8 $\mu$ m
- it is almost opaque for  $\lambda > 8\mu m$ 
  - heat is not transmitted by radiation around RoomTemp
- being a white oxide its reflectance is quite high for  $1 < \lambda < 5 \mu m$

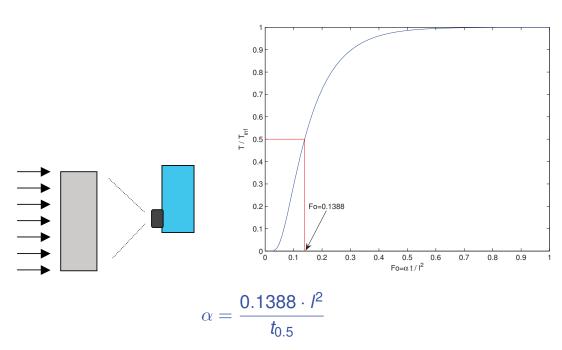
The evaluation of effective heat conduction is carried out in this work by laser flash equipment and IR thermography as well. The effects of blackening the surfaces by a thin layer of graphite is considered [8]. The role of heat conduction and radiation is also taken into account trying to separate each contribution. Experiments are carried out at ambient temperature by means of a thermographic camera and, at higher temperature, in vacuum until 1200 °C by means of a laser flash equipment and in argon atmosphere by means of another laser flash system (from another manufacturers) until 900 °C. Data are treated according to classical scheme taking into account the exchange with the environment [9]. Successively the data are considered by using the possibility of radiative exchange between the two blackened sides of the sample [10]. The possibility of simultaneous heating of the two sides of the sample, due to the semitransparency of the material to the laser shot is introduced, giving rise to a new model that explains in some conditions anomalous immediate heating of the side facing the detector. Results are shown and explained by using all the mathematical models presented.

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### 2. Modeling

Nowadays the Laser Flash method [2, 11, 12] is the most frequently used photothermal technique to measure thermal diffusivity. This method consists of heating the front face of a sample (typically a small disk-shaped specimen) by a short laser pulse and detecting the temperature evolution on its rear surface. The scheme is described in Fig. 4. The main advantages of this method are its simplicity and rapidity of measurement, and the possibility to measure the thermal diffusivity on a wide range of materials at different temperatures. The mathematical model utilized to describe the heat conduction problem that arises in the Laser Flash experiment is the following :

- a slab of thickness L
- heat flux prescribed on one side of the slab, consisting of a pulse of finite duration  $t_h$
- · heat exchange with the environment according to the linear Newton law on the heated side, adiabatic on the back side
- (z = L) where temperature is measured.



# Parker's method

Fig. 4. Scheme of Laser Flash technique: a slab is heated on one side by a short pulse while the temperature rise is collected by radiometer (or an IR camera) on the back side. The temperature rise is depicted on the right and the formula to obtain the thermal diffusivity is given below (Parker's formula) under adiabatic conditions.

The solution for the temperature on the back side of the slab is given by:

$$T(t) = \frac{Q}{h} \sum_{i=1}^{\infty} \frac{2\sin\mu_i}{\mu_i + \sin\mu_i \cos\mu_i} \left[ \exp\left(-\mu_i^2 \frac{\alpha}{L^2}(t-t_h)\right) - \exp\left(-\mu_i^2 \frac{\alpha}{L^2}t\right) \right]$$

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- Q power of the pulse during the heating time  $[Wm^{-2}]$
- h heat exchange coefficient  $[Wm^{-2}K^{-1}]$
- $\alpha$  thermal diffusivity  $[m^2 s^{-1}]$
- L specimen thickness [m]
- t time [s]
- $t_h$  pulse duration [s]
- $\mu_i$   $i_{th}$  positive root of the trascendental equation  $\mu \tan \mu = Bi$
- $Bi = \frac{h L}{\lambda}$  dimensionless Biot number
- $\lambda$  thermal conductivity  $[Wm^{-1}K^{-1}]$

Because of the semitransparency of ZrO<sub>2</sub> it is common practice to deposit a thin graphite layer (few microns thickness) both on the heated side, in order to increase the absorption coefficient, and on the rear side to increase the emissivity. Nonetheless, especially at high temperature, it is expected that heat is transferred from the heated graphite layer towards the back one not only by conduction but also by radiation due to the semitransparency of the TBC. In order to take into account this phenomenon Mehling [10] proposed to modify the boundary conditions of the slab according to the following formulae:

$$\lambda \frac{\partial T}{\partial x}\Big|_{x=0} = hT(0,t) + h\eta \left[T(0,t) - T(l,t)\right]$$
$$\lambda \frac{\partial T}{\partial x}\Big|_{x=l} = -hT(l,t) - h\eta \left[T(0,t) - T(l,t)\right]$$

Finally, in order to take into account the possibility of transparency of TBC to the laser source (that is not completely absorbed by front graphite layer) the model is hypothesized as schematized in Fig. 5 where  $I_0$  is the energy delivered by the laser and *a* is the absorption coefficient of the graphite layer.

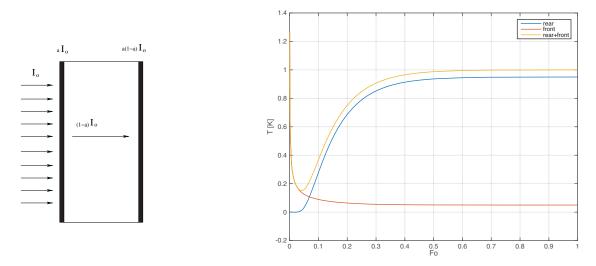


Fig. 5. Scheme of Laser Flash technique with the possible partial absorption of the laser pulse on the front graphite layer, the perfect transparency to laser radiation through the material and the absorption of the pulse on the back side. The

solution on the back side (yellow) is due to the superposition of a front side solution (red) plus a classical rear side solution (blue).

### 3. Experimental

At room temperature IR thermography has been utilized, taken advantage of its imaging feature. Indeed by means of a mirror it is possible to capture temperature evolution both of front and rear side of the sample (see Fig. 6 for the experimental scheme and Fig. 7 for a IR image captured during a sequence).

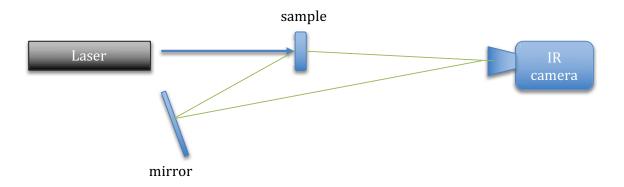


Fig. 6. Scheme of Laser Flash technique with IR camera allowing to view at the same time both front and rear side.

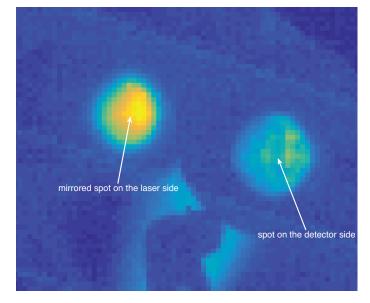


Fig. 7. IR image by thermography. It allows to view the mirrored image of the laser spot imprint after the shot on the heated sample side and, at the same time, the rising temperature of the spot on the rear surface, facing the detector.

In Fig. 8, 9 and 10, some data, fitting function and results as well are shown, proving the increasing accuracy of the models described in the previous chapter. In Fig 11 (left) the laser flash data of the same sample at increasing temperature are reported while the results are summarized in Fig. 11 (right) showing the difference in the thermal diffusivity obtained adopting the Cowan's and the proposed model.

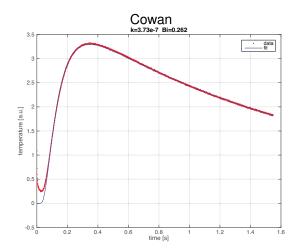


Fig. 8. Data and fitting model according to Cowan's model

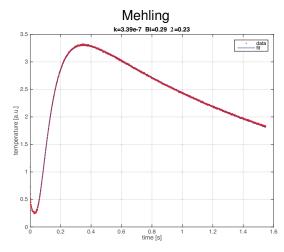


Fig. 9. Data and fitting model according to Mehling model

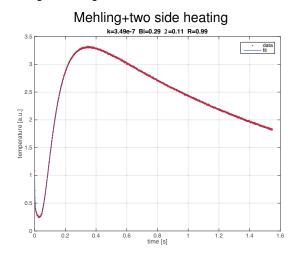


Fig. 10. Data and fitting model according to Mehling + the two side heating due to the transparency of TBC to laser light.

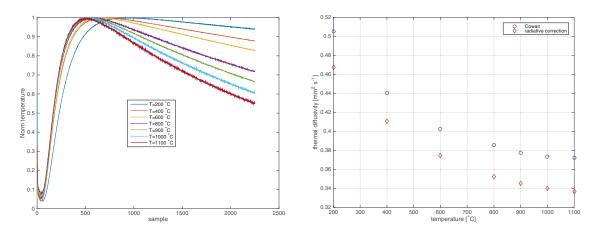


Fig. 11. On the left the temperature profile obtained on the rear side of the sample after a laser pulse. The same sample is tested at increasing temperature. On the right the results of thermal diffusivity are reported for different temperature, both according to the classical Cowan's model and to the proposed one, taking into account both the radiative exchange and the semitransparency of the sample to the laser source.

#### Conclusion

A new model that account for the semitransparency of  $ZrO_2$  is proposed. It allows for taking into account both the effect of radiative transfer and the semitransparency of the material to the laser source when the material is blackened by a graphite layer. Results are given for thermal diffusivity of a sample at increasing temperature showing the difference in the results adopting the classical models for Laser Flash experiments and the proposed one.

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