The Future of Gas Turbine Technology
8th International Gas Turbine Conference
12-13 October 2016, Brussels, Belgium

Paper ID Number (*36-IGTC16*)

A NOVEL APPROACH FOR NON-DESTRUCTIVE TESTING OF THE ADHESION OF THERMAL BARRIER COATINGS

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ABSTRACT

The operation temperatures of gas turbine engines have been increased significantly to optimize their efficiency factor. To protect the metallic blades from these high temperatures, thermal barrier coatings (TBCs) are applied onto the turbine blades. These layers must have a good adhesion to the supporting turbine blade. A poor adhesion may lead to a delamination of the layer during operation and finally to a destruction of the turbine blade and eventually the complete turbine.

It is therefore necessary, to check the quality of the layer adhesion regularly during service or preferably during operation. Approaches for non-contact and non-destructive techniques by using optical or infrared radiation are not sophisticated up to now.

Hence in this paper a new attempt to improve these optical or infrared-optical methods is described. The presented idea relies on the application of different wavelengths for the used measurement system. Using a short wavelength range, where the TBC is semi-transparent, allows the measurement of the temperature of the turbine blade. Using a second, long wavelength range where the TBC is non-transparent, the temperature of the surface of the TBC can be determined. As the thermal contact is usually correlated with the mechanical adhesion such measurements can be a possible tool for non-destructively testing the adhesion of TBCs.

KEYWORDS

thermal barrier coating, non-destructive testing, adhesion, radiative thermometry, infrared-optical properties

INTRODUCTION

For the conversion of energy from fossil fuels to electrical energy or mechanical energies in turbines of stationary power plants or in turbines for aero planes usually temperatures in excess of the melting or softening temperatures of the applied metallic or superalloy material are obtained. Therefore, the metallic parts inside the combustion chamber have to be protected to withstand such hostile environmental conditions. Generally they are protected with a so-called thermal barrier coating (TBC) made of ceramic materials, mostly yttrium stabilized zirconium oxide [1, 2, 3]. Such layers have a low thermal conductivity and a significant temperature drop of about 200 K to 300 K occurs via a layer thickness of some hundreds of micrometers [4]. Beside the thermal conductivity the mechanical adhesion of the TBC on the metallic substrate is of evidential importance. It is therefore necessary that the adhesion of these TBCs has to be checked routinely to assure a safe operation of the turbines. Up to now such a check can only be performed visually during a shutdown of the turbine and a direct inspection of the turbine blade. Such a shutdown is quite costly and a final check, whether the turbine blade is still ok or has reached the end of its operation time, has to be performed invasive, i.e. the turbine blade has to be destroyed for testing the interface and adhesion. The aim of the present approach is to check the operation ability during operation by an in-situ optical inspection. The intention of this inspection is twofold. In a first step a control of the adhesion during and directly after the fabrication process of the turbine blade is set-up. If this approach is successful it is intended to apply a similar approach to observe changes in the adhesion during operation and to evaluate a criteria, at which degradation stage the turbine blade has to be substituted. As the infrared-optical characteristics of the ceramics are strongly wavelength dependent, it is possible to monitor on the one side the temperature of the surface of the TBC. This is usually the case at long wavelengths, where the ceramic is opaque. At short wavelengths, the ceramics are semi-transparent, allowing a partly temperature measurement of the surface of the covered metallic substrate. Using these two different wavelengths regions a synergetic check of the adhesion properties and the thermal barrier possibility is possible. In this paper the theoretical background for this approach is presented together as well as the necessary experimental setup. Finally first simplified measurements will be presented.

BACKGROUND

TBCs are mainly based on oxide ceramics which are usually semi-transparent in near infrared (NIR) [5]. Therefore a considerable amount of thermal radiation can pass through the TBC [6]. Hence the heat transfer through the TBC is not only caused by solid conduction but also by the radiative transfer which especially has to be considered at high temperatures. The solid thermal conductivity and the radiative thermal conductivity can be described by the heat transfer equation and the equation of radiative transfer, respectively. Furthermore both quantities and the total thermal conductivity (which is the sum of the solid thermal conductivity and the radiative thermal conductivity) can be measured with different methods. This work focuses on the determination of the radiative thermal conductivity and the infrared-optical properties with the main objective to determine the adhesion of thermal barrier coating using non-destructive testing techniques.

In principle the microscopic infrared-optical properties of a TBC or more general of a sample are the scattering and the absorption coefficient [7] which are closely correlated on the one hand with macroscopic infrared-optical properties such as the transmittance, reflectance and emittance of the sample and on the other hand correlated with the refractive index and the structure of the sample. Thus it is possible to determine the scattering and the absorption coefficient from experimentally measured transmittance and reflectance using for example the three-flux-calculation which is a certain solution of the equation of radiative transfer. From the derived scattering and the absorption coefficient it is finally possible to derive information about the structure of the sample using scattering-theory or more precise Mie-theory. The principle procedure or reverse procedure for determining the different quantities from infrared-optical characterizations is depicted in Fig. 1.

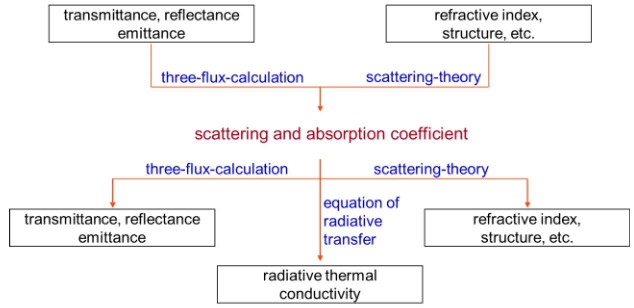

Fig. 1: Scheme of the different procedures for determining infrared-optical, thermal and structural properties from infrared-optical or structural measurements.

A delamination of the TBC may for example occur due to a thermally grown oxide (TGO) layer as described in [8] and depicted in Fig. 2. This delamination provokes a change of the structure and thus of the infrared-optical properties as well as of the temperature gradient. The correlation of the structure and the infrared-optical properties is illustrated in Fig. 3.

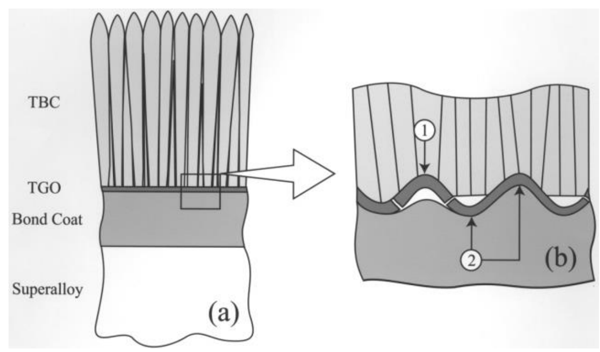

Fig. 2: Partially delamination of a TBC which causes an additional boundary TBC – air – bond coat or superalloy, respectively (picture taken from [9]).

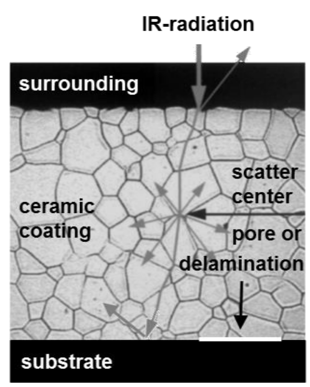

Fig. 3: Radiative transfer through a ceramic layer: infrared radiation is partially absorbed within the layer and partially scattered at pores, grain boundaries or gaps caused by delamination processes.

These correlations lead to the basic idea to determine the adhesion or more precisely delamination processes by a non-destructive testing method using infrared radiation.

EXPERIMENTAL CHARACTERIZATION

For determining the infrared-optical properties of TBCs in dependence on the wavelength two spectrometric setups are used for performing measurements at ambient temperature and high temperatures. Additionally two radiation thermometers for the near infrared (NIR) around 1 µm and for the long wavelength infrared (LWIR) around 10 µm are used.

Integrating Sphere Setup

At ambient temperature a FTIR-spectrometer with an integrating sphere setup can be used for measuring the spectral directional-hemispherical reflectance R_{dh} and transmittance T_{dh} of the free standing ceramic samples (Fig. 4). The directional spectral emittance ε_λ is than calculated from the spectral directional-hemispherical reflectance R_{dh} and transmittance T_{dh} [10, 11]:

$$\varepsilon_\lambda = 1 - R_{dh} - T_{dh}\,. \qquad (1)$$

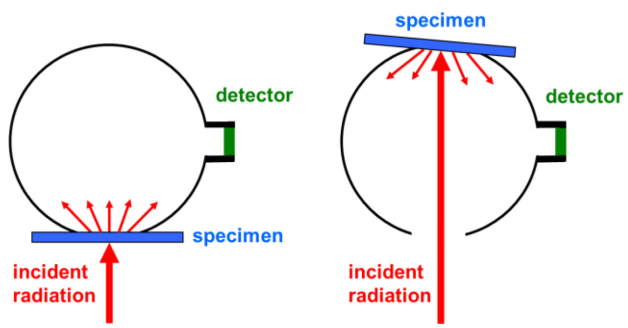

Fig. 4: Measurement of the directional-hemispherical transmittance T_{dh} (on the left side) and the directional-hemispherical reflectance R_{dh} (on the right side) using an integrating sphere.

Black Body Boundary Conditions Apparatus

At high temperatures the black body boundary conditions (BBC) method can be used for directly determining the directional spectral emittance ε_λ. With the BBC it is also possible to determine the spectral directional-hemispherical transmittance and reflectance. The basic idea of the BBC is to derive the infrared-optical properties of the sample at elevated temperature T_{sp} by varying the boundary conditions [12]. As boundary conditions for the front side and the back side of the sample a black enclosure is used. This black enclosure emits like a black body. These boundary conditions are then varied by changing the temperature of the black enclosure between the higher temperature T_b and the lower temperature T_a. A sketch of the BBC method is shown in Fig. 5. The sample is heated in a furnace to the desired temperature T_{sp} with a homogenous temperature distribution inside, and then placed between the two black enclosures.

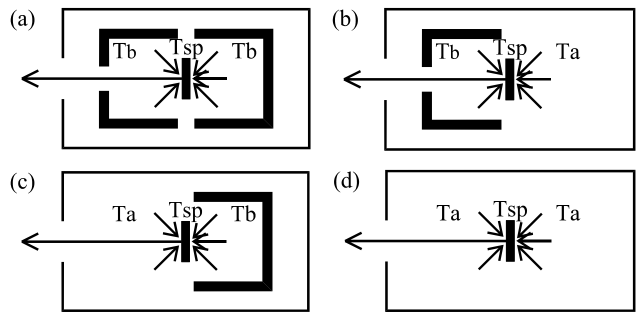

Fig. 5: Black body boundary conditions (BBC) setup with its various boundary conditions for the determination of ε_λ, T_{dh} and R_{dh}.

In Fig. 5a the sample at the temperature T_{sp} is surrounded by two black enclosures at the temperatures T_b. The detected radiation (long arrow) is the sum of the emitted radiation at the sample temperature T_{sp} and the black body radiation at the lower temperature T_b transmitted through the sample from the rear side and reflected from the sample from the front side:

$$I_{meas}\left(T_{sp}, T_b\right) = \varepsilon_\lambda\left(T_{sp}\right)\cdot I_b\left(T_{sp}\right) + \left[R_{dh}\left(T_{sp}\right) + T_{dh}\left(T_{sp}\right)\right]\cdot I_b\left(T_b\right)\,. \qquad (2)$$

With Eq. (1) one gets the directional spectral emittance ε_λ at the sample temperature T_{sp}:

$$\varepsilon_\lambda\left(T_{sp}\right) = \frac{I_{meas}\left(T_{sp}, T_b\right) - I_b\left(T_b\right)}{I_b\left(T_{sp}\right) - I_b\left(T_b\right)}\,. \qquad (3)$$

Together with the configurations displayed in Fig. 5b and Fig. 5c the estimation of the directional-hemispherical transmittance and reflectance are also possible. The configuration Fig. 5d is equal to Fig. 5a, with a lower temperature of the black enclosures.

RESULTS AND DISCUSSION

At first the possibility of determining the structure of ceramics from infrared-optical measurements is demonstrated for alumina (Al_2O_3) ceramics. For experimental characterization a cylindrical alumina sample has been prepared by a sintering process at about 1900 K from a Al_2O_3-powder which was pressed isostatically at 250MPa before [13]. Finally slices with different thicknesses d have been cut from this cylinder which all exhibit the same structural properties as illustrated in Fig. 6. The porosity of the samples has been derived to be $\Pi = 2\,\%$.

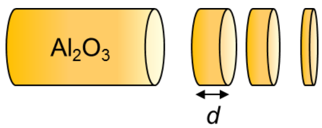

Fig. 6: Slices with different thicknesses d have been cut from the alumina cylinder which has been prepared by a sintering process.

The spectral directional-hemispherical transmittance T_{dh} and reflectance R_{dh} of these slices have been measured as described above at ambient temperature. The resulting spectra are depicted in Fig. 7 and Fig. 8, respectively. Alumina is semi-transparent for wavelengths below 8 µm as the transmittance does not vanish in this wavelength region, even for samples with thicknesses of some millimeters. Furthermore, the transmittance decreases with increasing sample thickness as expected. The reflectance in Fig. 8 correspondingly increases with increasing thickness. Moreover, the reflectance vanish around 10 µm which corresponds with a high emittance near to one.

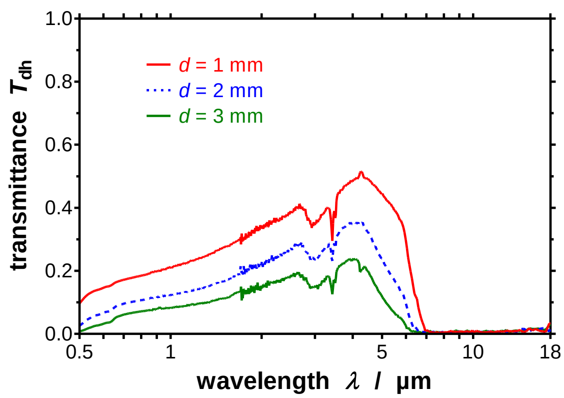

Fig. 7: Directional-hemispherical transmittance T_{dh} of slices with different thicknesses d, but with identical structural properties, versus wavelength from 0.5 µm to 18 µm.

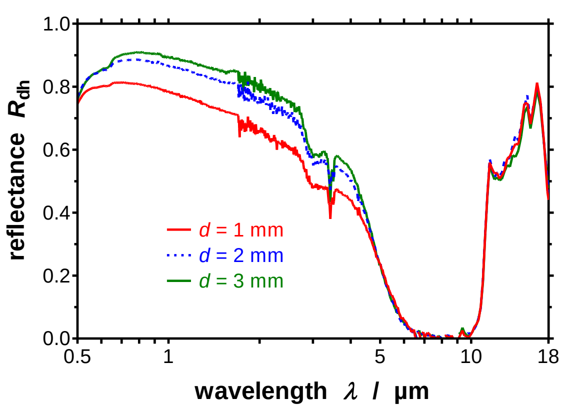

Fig. 8: Directional-hemispherical reflectance R_{dh} of slices with different thicknesses d, but with identical structural properties, versus wavelength from 0.5 µm to 18 µm.

From the measured spectral transmittance and reflectance the structural properties of the alumina ceramic have been derived using scattering-theory. As sintered ceramics with a low porosity exhibit air-filled pores which serve as scatter center, the determination of the pore sizes from infrared-optical measurements is possible. For this purpose the scattering coefficient of the three slices has been calculated from the measurements using three-flux-calculation. The derived scattering coefficient can be found in Fig. 9 as a function of the wavelength. It can be seen that the scattering coefficient is similar for all three slices which is due to the fact that these slices exhibit nearly identical structural properties as explained above. For obtaining the pore sizes of the investigated samples the scattering coefficient has been fitted using Mie-theory by varying the average pore size of the sample. The resulting fit curves are given in Fig. 10 for the set of samples discussed above and depicted in Fig. 7 to Fig. 9 (sample set 1) together with another set of samples (sample set 2) which exhibits the same porosity ($\Pi = 2\,\%$) but a different structure due to different sintering conditions. This results in a different scattering coefficient and finally in different pore sizes. In both cases the fit curves meet the curves which have been derived from the measurements quite well (see Fig. 10). For sample set 1 an average pore size of 0.4 µm results whereas for sample set 2 an average pore size of 2.2 µm results which both have been validated by scanning electron microscope (SEM) micrographs.

Hence the determination of structural properties of ceramic materials from infrared-optical measurements has been demonstrated.

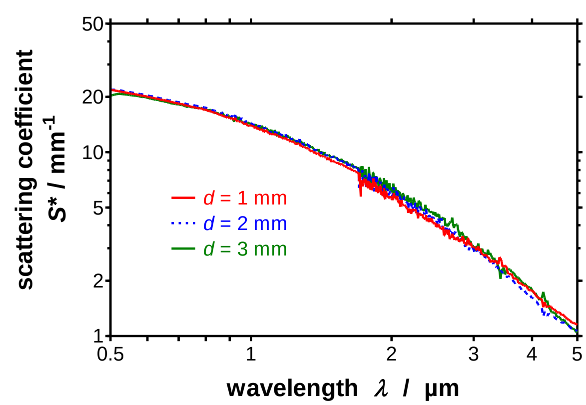

Fig. 9: Scattering coefficient S^* of slices with different thicknesses d, but with identical structural properties, versus wavelength from 0.5 µm to 5 µm.

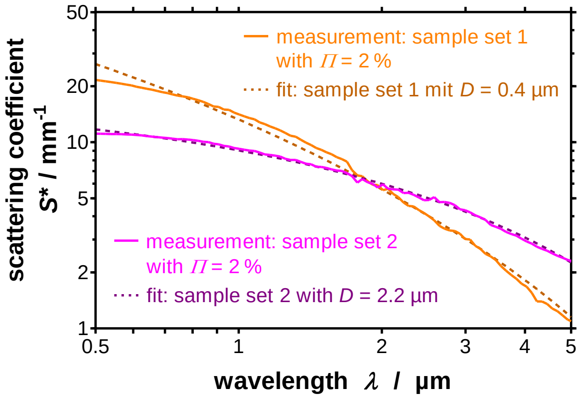

Fig. 10: Spectral scattering coefficient S^* of two different sets of samples which exhibit a different structure although the porosity is identical. Beside the scattering coefficients derived from measurements (solid lines) the theoretically fitted scattering coefficients (dotted lines) are shown.

Subsequently further investigations have been performed on thermal barrier coatings which have been prepared by electron beam physical vapor deposition (EP-PVD) and which have been exposed to a heat treatment. For the experimental determination of the transmittance and reflectance of the TBCs the coatings have been applied on a substrate and separated afterwards [14], as illustrated in Fig. 11, in order to determine the transmittance as well as the reflectance. As TBC material zirconia (ZrO_2) which was stabilized with 7 wt.-% yttria (Y_2O_3) and which is abbreviated as PYSZ (partially yttria stabilized zirconia) has been chosen. For determining the change of the infrared-optical and thermal properties at high temperatures during operation the coating has been exposed for $t = 100$ h at a temperature of $T = 1400$ K. The transmittance of the layer before and after the heat treatment has been measured at 1100 K using the BBC apparatus. The resulting spectral transmittance can be found in Fig. 12 for both states (before and after the heat treatment). It is clearly visible that the transmittance of the layer after the heat treatment is higher than before the heat treatment. This is caused by a structural change of the layer due to a sintering process which reduces the number of boundaries which serve as scatter center. A SEM-micrograph of the layer is given in Fig. 13.

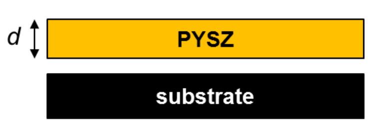

Fig. 11: A PYSZ coating with a thickness d has been applied onto a substrate by EP-PVD and separated afterwards.

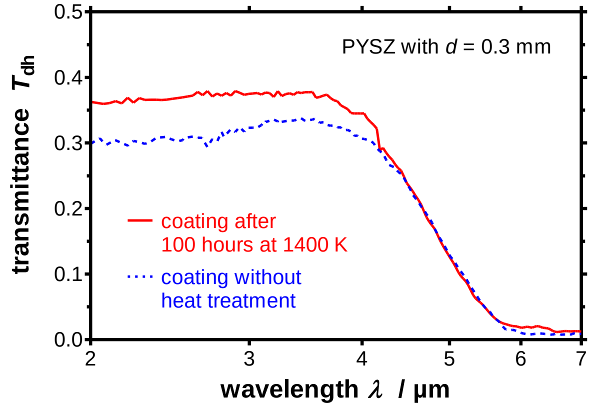

Fig. 12: Directional-hemispherical transmittance T_{dh} of a free standing PYSZ-TBC without and with heat treatment versus wavelength from 2 µm to 7 µm.

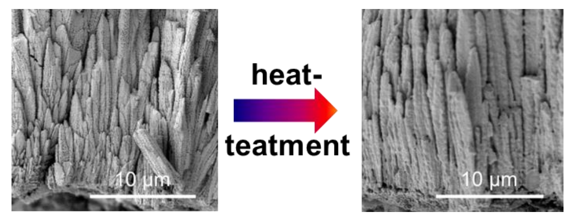

Fig. 13: SEM-micrograph of the PYSZ-layer before (on the left) and after (on the right) a heat treatment for 100 h at 1400 K.

Such a heat treatment not only causes changes in the structural and infrared-optical properties but also in the thermal properties, as all three properties are correlated. Using the equation of radiative transfer it is possible to determine the radiative thermal conductivity from the scattering and absorption coefficient or the transmittance and reflectance of the layer, respectively (see Fig. 1). The radiative thermal conductivity caused by the transfer of thermal radiation through the layer is depicted in Fig. 14 for the layer before and after the heat treatment. The radiative thermal conductivity increases with increasing temperature. Furthermore, the radiative thermal conductivity after the heat treatment is higher than before the heat treatment which is due to a higher transmittance which causes a higher transfer of thermal radiation through the layer. For comparison an approximate value of the solid thermal conductivity caused by the heat transfer through the solid skeleton is given in Fig. 14 for comparison. The total thermal conductivity is the sum of the radiative thermal conductivity and the solid thermal conductivity. Hence the radiative thermal conductivity significantly contributes to the total thermal conductivity at temperatures around 1400 K. A change of the solid thermal conductivity after heat treatment would also be expected, but has not been investigated in this work.

Altogether this investigation demonstrates on the one hand the evidence to characterize TBCs not only in the as-coated state but also after heat treatment and on the other hand the existence of a correlation between infrared-optical, structural and thermal properties.

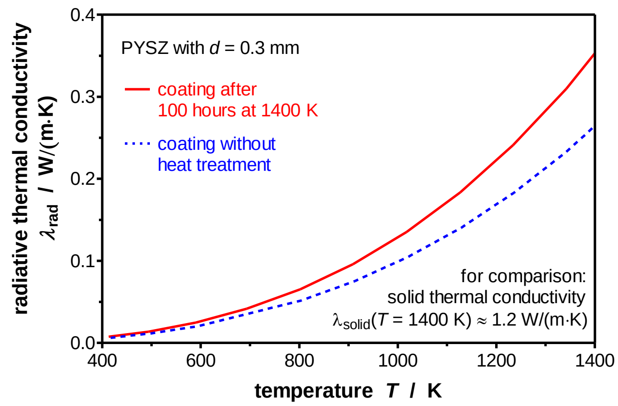

Fig. 14: Radiative thermal conductivity of the PYSZ-layer in the as-coated state and afterheat treatment versus temperature from 400 k to 1400 K.

The derived correlation between infrared-optical, structural and thermal properties can be finally used for non-destructive testing of the adhesion of thermal barrier coatings. A delamination of the TBC causes additional boundaries which change the structure and which influence the infrared-optical and thermal properties. Therefore IR-spectrometers or radiation thermometers can be used to determine the infrared-optical properties or the temperature gradient in order to correlate the derived values with changes in the structural properties or the delamination, respectively.

For this purpose at first a simplified measurement setup has been chosen in order to demonstrate and test the method. A sapphire sample has been placed directly onto a substrate and with a gap above the substrate as illustrated in Fig. 15. Sapphire as non-porous alumina has been chosen due to its high transmittance in the NIR and its high emittance in the LWIR which equals one around 10 µm. The spectral transmittance of sapphire can be found in Fig. 16. The measurements have been performed with two radiation thermometers which are sensitive at 1 µm (NIR-pyrometer) and 10 µm (LWIR-pyrometer), respectively. At a wavelength of 1 µm mainly radiation from the substrate is detected by the NIR-pyrometer whereas at a wavelength of 10 µm only radiation from the sapphire surface is detected by the LWIR-pyrometer.

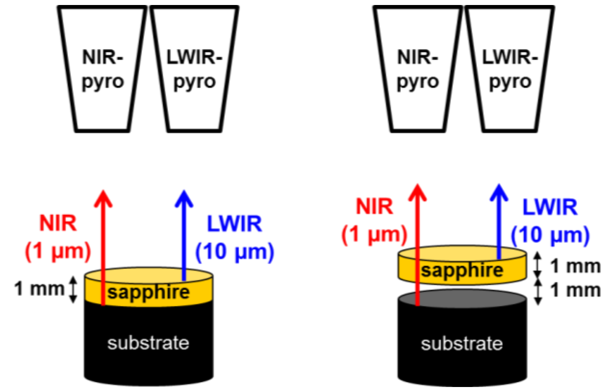

Fig. 15: Simplified test setup for determining the influence of a gap onto the temperature gradient and the contactless determination of the temperatures of the substrate and sapphire surface.

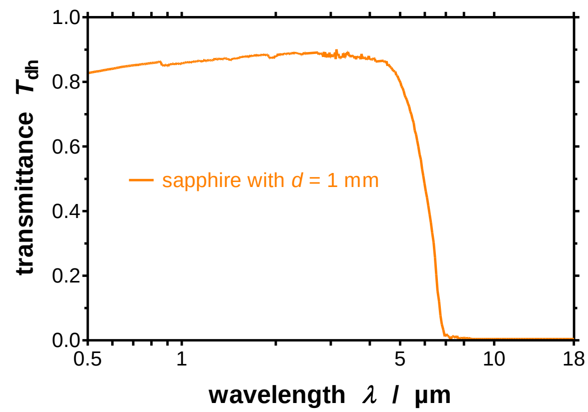

Fig. 16: Directional-hemispherical transmittance T_{dh} of s sapphire sample with a thickness of 1 mm versus wavelength from 0.5 µm to 18 µm.

The substrate has been heated up to a temperature of 900 K and 1500 K and the temperatures of the substrate and the sapphire surface have been determined using the NIR- und LWIR-pyrometer. The detected temperatures of the sapphire surfaces for the two cases (without and with gap) are shown in Fig. 17. It is clearly visible that the temperature of the sapphire sample without gap is significantly higher than the temperature of the sapphire sample with gap. Obviously the gap increases the thermal resistance and therefore the temperature gradient between the substrate and the sapphire surface. Hence the simplified measurement setup demonstrates this well-known behavior which of course could also have been measured with other equipment, such as thermocouples. But the infrared-optical characterization provides the potential of non-contact measurements during application or in harsh environment even for complex TBCs.

As explained above for TBCs with a certain structure and porosity the infrared-optical properties correlate with the structure and a delamination only slightly influences the temperature gradient. But the additional boundaries which occur due to a delamination change the structure and therefore the infrared-optical properties due to an additional scattering. With the characterization methods and the analyzing procedures described above it would be possible to detect such a delamination.

For this purpose a project is planned with partners who provide coatings with defined structural properties on different types of substrates and partners who provide relevant IR-measurement techniques.

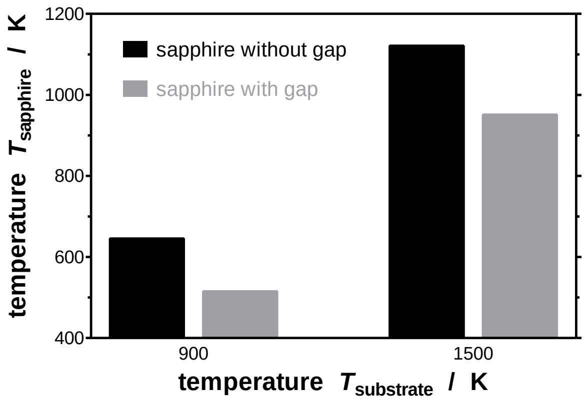

Fig. 17: Detected temperatures of the sapphire surface versus the temperature of the substrate for two cases: sapphire without gap and sapphire with gap.

CONCLUSIONS

Altogether it has been shown that the existing correlation between infrared-optical, thermal and structural properties of TBCs can be used for a non-contact determination of the properties which provides the possibility for a non-destructive testing of the adhesion of thermal barrier coatings.

To demonstrate these correlations three investigations have been presented exemplary:

- Determination of the structure and especially the pores of sintered alumina ceramics.
- Investigation of the properties of PYSZ thermal barrier coatings and its changes due to heat treatment.
- Detecting of the substrate and layer temperature for a setup without and with gap between substrate and layer using NIR and LWIR radiation thermometers, respectively.

These investigations and correlations finally lead to the result that a non-destructive determination of delamination is possibly as a delamination provokes additional boundaries within the thermal barrier coating which causes changes of the infrared-optical properties and the temperature gradient, respectively. Hence the delamination can be detected contactless by using different wavelength regions as demonstrated for a simplified setup. For real TBC the procedure will be more complicated as scattering effects occur, but an analysis can be done using the presented theoretical procedures and experimental devices. This will be the subject of further activities.

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