Layer thickness measurement of ceramic systems with a numerical model for flash thermography

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Abstract

Thermography is a non-destructive testing technique which is non-contacting as well as imaging and therefore advantageous for delicate materials. It is more widely used for qualitative evaluation but can also provide data for a quantitative analysis when the heat source is actively controlled. With flash thermography, fast diffusion or conduction processes can be monitored, providing an effective method for the inspection of metals and thin coatings.

Ceramic layered systems are used as thermal barrier coatings, for example on turbine blades. Gas turbines with such blades can be operated at higher temperatures, which leads to an increase in efficiency. To fully exploit this advantage, the properties and thickness of the coating must be within a predefined parameter range. Due to the challenging and hard to monitor manufacturing process, the coating must be examined afterwards.

With the help of a numerical model using a finite difference solution, multiple material parameters such as thermal conductivity and layer thickness can be determined simultaneously with nonlinear regression fitting.

During the research presented herein, a four-stepped sample of ceramic coatings on an Inconel steel basis is examined and the thermographic data analysed. After the thermographic measurements, the sample was cut, embedded in resin, and polished. The scanning electron microscopy images and measurements serve as the reference values to compare the extracted parameters to.

KEYWORDS: Non-destructive testing, active thermography, flash thermography, thermal barrier coating, finite difference solution

1. Introduction

Thermal barrier coatings (TBCs) are of integral importance for the operation of turbines. This form of ceramic coating on turbine blades enables the turbine to work at high temperatures, increasing its efficiency and therefore power output. Since the material parameters such as thickness and thermal conductivity determine the effectiveness of the TBC and the coating process is challenging, these parameters must be monitored.
Thermography is already employed qualitatively for detection of debonds of the TBC. Hence, a thermographic setup is already present at most manufacturing sites. A quantitative evaluation with thermography resulting in these material parameters would therefore be economically desirable. Flash or pulse thermography (PT) uses a short optical flash as excitation to heat a sample and observe the temperature evolution of the surface from the infrared (IR) radiation recorded with an IR camera. This temperature evolution is governed by the heat diffusion process inside the sample and depends in turn on the thermophysical properties and the structure of the sample. Therefore, this correlation can be exploited to extract different parameters and information on the sample by evaluating the thermographic data.

Building upon two existing studies by Frisch et al. (1) and Burger et al. (2) on the same sample, a numerical flash model (NFM) with the finite difference solution technique for PT is used to extract the thickness and thermal conductivity of a layered TBC system with additional input from scanning electron microscopy (SEM) analysed micrographs.

2. Experimental

2.1 Thermography

The thermographic setup is the same as for the study by Frisch et al. (1). The IR radiation in the mid-wave IR band was recorded between 2.5 and 5 µm with a mid-wave broadband camera lens on a FLIR SC5000 (FLIR Systems Inc., Portland, OR, USA). In quarter frame mode with an integration time of 2000 µs a resolution of 160 x 128 pixels and a frame rate of 469 Hz were achieved. The arbitrary intensity values are used without a temperature calibration for the evaluation in this study.

A Broncolor Grafit A4 (Bron Elektronik AG, Allschwil, Switzerland) photographic flash was used as the excitation source with a polymethyl methacrylate (PMMA) filter, a flash energy of 1054 J, and a flash duration of 1/750 s. This flash series makes use of built-in shut-off electronics enabling an exact adjustment of the pulse duration.

The measurements were performed with the camera and the flash positioned both on the ceramic-coated side of the sample, resulting in a reflection arrangement.

2.2 Investigated sample

The ceramic coating sample investigated in this study is the same as in the previously published studies by Frisch et al. (1) and Burger et al. (2). It consists of three layers: TBC, bond coat (BC), and substrate (see also figure 2). The basis of the sample is a 9.0 mm thick substrate of Inconel 738 (IN 738), a nickel-based superalloy. To ensure strong bonding of the TBC layer, first, a bond coat was applied. Afterwards, the sample was coated with yttria-stabilised zirconia (YSZ, Y2O3+ZrO2) by electron beam physical vapour deposition. The deposition was performed in four thickness steps resulting in a ceramic step-wedge with four thickness variations. Each step of the sample is named after its nominal thickness ("6 mils", "7 mils", "9 mils", "11 mils"), which corresponds to the thickness specified for the coating process (see also table 2). 1 mil is an American unit for measuring length and corresponds to 25.4 µm. The sample measured 89 mm × 19 mm × 9 mm (L×W×H) in its entirety. The material parameters of the TBC sample are collected in table 1 (1).
Table 1. Parameters of the different layers of the TBC sample as input for the NFM. Table from (1). Values were either estimated and validated with the NFM or \(^1\) from (3)

<table>
<thead>
<tr>
<th>Layer</th>
<th>Density (\text{1e-3 kg/m}^3)</th>
<th>Heat capacity (\text{1e-2 J/(kgK)})</th>
<th>Thermal conductivity (\text{W/(mK)})</th>
</tr>
</thead>
<tbody>
<tr>
<td>TBC</td>
<td>6.05</td>
<td>5.37</td>
<td>-</td>
</tr>
<tr>
<td>BC</td>
<td>7.00</td>
<td>4.80</td>
<td>5.0</td>
</tr>
<tr>
<td>Substrate</td>
<td>8.11</td>
<td>4.19</td>
<td>10.0</td>
</tr>
</tbody>
</table>

YSZ is partially transparent in the mid-wave infrared radiation region and has low emissivity. To counteract this problem, the upper half of the sample was coated with graphite absorbing coating (Graphit 33, Kontakt Chemie) before performing the PT measurements (visible in figure 3 on the left side). The influence of this additional graphite coating is negligible for thermography, as previously verified by Sun (4).

To gain a reference to compare the results of the NFM to, the TBC sample was cut into four pieces and embedded in resin. The cross-section cut was polished, and the micrograph was prepared for evaluation with optical microscopy and SEM. Figure 1 shows the micrograph of the sample “11 mils” embedded in resin and polished. Before performing the SEM measurements, each sample was coated with a very thin graphite layer by vapour deposition to minimize the electrostatic charging of the sample.

Figure 1. Photograph of micrograph of sample “11 mils” embedded in resin. The white layer on top is the TBC system, and the polished base is the IN 738 substrate

2.3 SEM

The SEM measurements were performed on a JEOL JSM-6010 Plus (JEOL Ltd., Tokyo, Japan) of the InTouchScope series. The images were captured, and the thicknesses were analysed with the tool-included measurement software. With the setting high vacuum and an acceleration voltage of 20 kV, backscattered electron (BSE) images with different magnification were recorded. For the thickness measurement, all samples were evaluated with a magnifications of x50 and therefore a region of 1.8 mm to average the boundaries between the different layers by eye was possible. For each sample, only one measurement series of TBC, BC, and the entire coating thickness was possible due to an increasing electrostatic charging of the sample. A possible reason for the fast charging is air bubbles around the cross-section cut and inside the resin as visible in figure 1. The measurement error was estimated to be ± 10 \(\mu\)m.

3. Theory and modelling

The model employed in this study has already been described by Sripragash et al. (5) and Frisch et al. (1). For more extensive information on the theory, please refer to these publications.

The finite difference solution technique of the Crank-Nicholson algorithm was employed to solve the one-dimensional Fourier heat equation for a three-layered system containing
of TBC, BC, and substrate. The NFM makes use of several assumptions: Instantaneous excitation and absorption, uniform heating of the sample in the x-y-plane, adiabatic conditions on the back of the sample, negligible interface resistance, a very thick substrate in comparison to the coating, and no coating translucency in the considered wavelength range. Via regression fitting, the numerical data was then fitted to the second derivative of the logarithmic intensity of the recorded thermographic data. As previously shown by Sun (6), only two of the three important TBC parameters – thickness, thermal conductivity, and heat capacity – can be determined independently. For this study, the evaluation of thickness and thermal conductivity were chosen.

4. Results

In the previous study by Frisch et al. (1), the NFM was used with input parameters measured and calculated from eddy current testing and with the help of a calliper. The results were compared to eddy current testing and terahertz time domain spectroscopy (THz-TDS). After these measurements, the sample was cut and polished and micrographs of each step of the sample could be analysed with SEM and optical microscopy. Figure 2 corresponds to a SEM image at 20 kV of the sample “9 mils” with a schematic drawing of the layer system of the TBC sample.

![Figure 2. Left: Section of a SEM BSE image at 20 kV of sample “9 mils” with a magnification of x50. Right: Schematic drawing of the TBC sample](image)

The extracted BC thickness and the substrate thickness measured with an optical microscope are compiled in table 2. The nominal thickness values of the coating process (cf. chapter 2.2) are listed there as well. The TBC thickness was also measured as a reference and is included in table 3 with the results of the NFM. The BC thickness and the substrate thickness are used as input for the evaluation of the flash thermographic data with the NFM. The nonlinear regression fitting of the experimental data was performed for eleven data points for each step. Figure 3 shows a photograph and a thermogram of the sample “9 mils” with the locations of the eleven data points.

### Table 2. Nominal and measured thickness values for the different layers of the TBC sample

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Step 1 “6 mils”</th>
<th>Step 2 “7 mils”</th>
<th>Step 3 “9 mils”</th>
<th>Step 4 “11 mils”</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nominal TBC thickness (mils)</td>
<td>6.0</td>
<td>7.0</td>
<td>9.0</td>
<td>11.0</td>
</tr>
<tr>
<td>Nominal TBC thickness (µm)</td>
<td>152.4</td>
<td>177.8</td>
<td>228.6</td>
<td>279.4</td>
</tr>
<tr>
<td>BC thickness with SEM (µm)</td>
<td>134.0</td>
<td>120.0</td>
<td>114.0</td>
<td>108.0</td>
</tr>
<tr>
<td>Substrate thickness with opt. microscope (mm)</td>
<td>9.0</td>
<td>9.0</td>
<td>9.0</td>
<td>9.0</td>
</tr>
</tbody>
</table>
Figure 3. Left: Photograph of sample “9 mils”. Right: Thermogram of sample “9 mils” with evaluation points for NFM

For each point, the temporal evolution of the IR intensity, or rather the second derivative of its logarithm, was fitted and the results of all eleven locations were averaged. Figure 4 plots these results as well as the average of the second logarithmic derivative of the experimental data for the eleven data points. The fit was able to match the minimum of the experimental data very well. With increasing coating thickness, the minimum of the curve shifts to the right and therefore to later times. This correlates to the heat having to travel a larger distance with a thicker layer.

![Figure 4. Experimental (continuous line) and numerical fit (dashed line) data of the second derivative of the logarithmic intensity. Each curve is the average of eleven data points](image)

The values of the TBC thickness and the TBC thermal conductivity evaluated with the model are compared to the SEM determined TBC thickness in table 3. Included as the error is the standard deviation (SD) of the averaged values.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Step 1 “6 mils”</th>
<th>Step 2 “7 mils”</th>
<th>Step 3 “9 mils”</th>
<th>Step 4 “11 mils”</th>
</tr>
</thead>
<tbody>
<tr>
<td>Averaged TBC thermal conductivity and SD (W/(mK)) with NFM</td>
<td>0.427 ± 0.037</td>
<td>0.506 ± 0.101</td>
<td>0.707 ± 0.408</td>
<td>0.760 ± 0.362</td>
</tr>
<tr>
<td>Nominal TBC thickness (µm)</td>
<td>152.4</td>
<td>177.8</td>
<td>228.6</td>
<td>279.4</td>
</tr>
<tr>
<td>TBC thickness with SEM (µm)</td>
<td>206.0 ± 10.0</td>
<td>236.0 ± 10.0</td>
<td>330.0 ± 10.0</td>
<td>380.0 ± 10.0</td>
</tr>
<tr>
<td>Averaged TBC thickness and SD (µm) with NFM</td>
<td>126.5 ± 3.9</td>
<td>163.83 ± 11.7</td>
<td>261.5 ± 39.6</td>
<td>309.0 ± 61.6</td>
</tr>
<tr>
<td>Difference between TBC thickness SEM and NFM (µm)</td>
<td>79.5</td>
<td>72.1</td>
<td>68.5</td>
<td>71.0</td>
</tr>
<tr>
<td>Difference between TBC thickness SEM and NFM (%)</td>
<td>38.6</td>
<td>30.6</td>
<td>20.7</td>
<td>18.7</td>
</tr>
</tbody>
</table>
The thermal conductivity increases with the thickness of the TBC layer, but the SD also increases. Burger et al. (2) analysed the porosity and surface roughness of the sample under investigation from the SEM measurements. They identified a porosity ranging from 14.9 to 24.5 area% and a surface roughness varying between 10.8 and 21.5 µm. Since porosity influences thermal conductivity, this could cause an increase and a large deviation between measurement points.

The TBC thickness values also show an increase in the SD with increasing thickness. Here, the surface roughness mentioned above (and the likely similar interface roughness TBC-BC) obviously could be a reason. Overall, the values differ less from the SEM reference than in the previous study by Frisch et al. (1). Noticeable is the difference between reference and NFM-determined TBC thicknesses of about 70 µm too thin for each sample. This could also hint at a systematic error within the NFM or of some input parameters listed in table 1. The density and heat capacity therein were assumed to be independent of the coating thickness which is not the case for a varying porosity.

5. Conclusions

In this work, the potential of a numerical model using finite difference solutions and nonlinear regression fitting for the determination of material parameters of a layered TBC system with PT was verified. The thermal conductivity as well as the thickness of the TBC layer could be extracted from the thermographic data by fitting the model to the second derivative of the logarithmic intensity. With better input for the model from SEM measurements, the accordance of the resulting values with the reference could be increased in comparison to previous studies.

References