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Measurement of direct strain optic coefficient of YSZ thermal barrier coatings at GHz frequencies

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Abstract: We report the first observation of stress induced birefringence in air plasma sprayed (APS) thermal barrier coatings (TBCs) using a reflection based polariscope and GHz illumination. Strain optic coefficients of \((-0.0133 \pm 0.0102) \times 10^{-9}\) and \((-0.0190 \pm 0.0043) \times 10^{-9}\) were measured for yttria-stabilized zirconia (YSZ) APS coatings of the same thickness deposited on substrates of 3 mm and 1 mm mild steel. The reflection measurement approach was validated by additional measurements of the stress optic coefficient of bulk yttria-partially stabilized zirconia (YTZP) ceramic that were in agreement with previously reported transmission measurements. The ultimate application of this technique is the prediction of remaining life in TBCs.

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1. Introduction

Ceramic thermal barrier coatings (TBCs) are routinely used to protect critical components in gas-turbines from extreme environmental conditions such as high temperature and pressure [1–3]. The continuous drive for increased gas-turbine efficiency and power output is resulting in higher operating temperatures that reduce the lifetime of existing TBCs and require new coating technologies to be developed [1–4]. Lifetime prediction is a major challenge for TBC manufacturers but there is currently no effective non-destructive evaluation method that can identify and discard coatings with expected shorter lifetime prior to their going into service [3]. In this paper we demonstrate the first measurement of a direct stress optic coefficient in ceramic thermal barrier coatings using a reflection polariscope and GHz illumination, and discuss how it might lead to a lifetime prediction tool for the manufacture of TBCs.

A typical TBC system consists of four parts; a super alloy substrate (typically nickel or cobalt based), a bond coat, a thermally grown oxide (TGO) layer and the ceramic top coat. Ytria-stabilized zirconia (YSZ) is the most common ceramic used for the top coat because of its low thermal conductivity and high thermal expansion coefficient. The ceramic top coat is typically deposited either by electron beam physical vapor deposition (EBPVD) or air plasma spray (APS) [5]. Both methods produce significantly different morphologies, leading to different failure mechanisms [3]. This paper focuses on APS manufactured coatings. Figure 1 shows a scanning electron microscope image of a typical cross section of an APS coating system and the four constituent parts outlined above.

The failure of both EBPVD and APS TBC systems results in spallation of the ceramic top coat, exposure of the unprotected substrate to elevated temperatures and ultimately catastrophic failure of the gas-turbine. Hence, TBCs are considered ‘prime reliant’ within the gas-turbine, meaning that in addition to enabling increased performance they are critical to safe turbine operation. As a result commercial turbines are only designed to utilize a small percentage of total TBC temperature capacity [2], and in-service TBC lifetimes are severely underestimated in order to maximize the margin of safety.

For APS TBCs, the principal failure mechanism is oxidation of the bond coat at high operating temperatures of the gas turbine, resulting in growth of the TGO layer. This growth
causes increased tensile stress along the crests of the undulating bond coat and TGO interface due to the mismatch in the respective thermal expansion coefficients [1,2,6,7]. Ultimately, local cracks and debonds form larger networks and TBC spalling occurs. Understanding TBC failure is ultimately a matter of understanding strains and stresses within the coating system [1,3,6–8].

Several non-destructing testing methods have been used to inspect TBC properties. These include, acoustic emission [9–11], impedance and Raman spectroscopy [12], as well as thermography [13], photoluminescence [14,15], infrared imaging [6,15] and x-ray diffraction [16,17]. Some of these methods are used to determine material properties of the TBC [18], while others study the effect of thermal-gradients on thermal-fatigue [13,14] or overall coating wear [19]. X-ray diffraction has been used to measure residual and thermal stresses in TBCs, however this technique is limited to measurements over small areas near the top coat surface due to poor x-ray penetration [17]. THz-time domain spectroscopy (TDS) has been used to inspect for damage on TBC coatings but not to measure stress or strain distributions [15,20,21]. In Ref [20], the reflected signals from the interface of YTZP ceramic coatings on a metal substrate were compared between their initial and aged condition after 1350 hours in a furnace. Increased delays in the arrival times of terahertz pulses, as well as changes in the width and shape of the pulses, were observed and attributed to the growth of the TGO and the development of air gaps that would ultimately lead to spalling of the coating. However, these relative changes were not related to any physical property of the ceramic, such as its refractive index.

We recently reported the first measurement of stress induced birefringence for bulk YTZP ceramic, using illumination between 260 and 380 GHz in a transmission polariscope [22]. YTZP exhibited a linear change in refractive index as a function of applied stress. Materials that exhibit changes in the real component of their refractive index as a function of stress, are said to show stress induced birefringence [23,24]. In a material’s elastic regime, the stress optic coefficient describes the linear relationship between changes in refractive index and stress [22],

\[ \Delta n_t = c_1 \Delta \sigma_t \]  
(1)

where \( c_1 \) is the direct stress optic coefficient, \( n_t \) is the refractive index and \( \sigma_t \) is the principal stress.

In this paper, we demonstrate for the first time that APS thermal barrier coatings also exhibit stress induced birefringence for GHz illumination. The difficulty with using Eq. (1) for coating systems is that the in situ Young’s modulus for the TBC must be known in order to calculate the stress from the applied load. For a material that exhibits stress induced birefringence, the strain optic law relates the change in refractive index to the applied principal strain, \( \varepsilon_t \). In a material’s elastic regime, this relation is linear and is written as [23,25–28],

\[ \Delta n_t = b_1 \Delta \varepsilon_t \]  
(2)

where \( b_1 \) is the direct strain optic coefficient. Hence, for the thermal barrier coatings in this paper, we report the strain optic coefficient rather than the stress optic coefficient. If the Young’s modulus of the coating is known, the change in strain can be converted to a change in stress and a stress optic coefficient may be calculated via the stress optic law, Eq. (1).

The approach taken in this paper is to measure the refractive index of the material at different loads, from which the direct strain optic coefficient is determined. Clearly, a TBC is deposited on a metal substrate that is reflective to GHz illumination which requires measurements to be made in reflection. Therefore, reflection measurements were first made from bulk YTZP to confirm that the same strain optic coefficient was obtained as previously obtained in transmission. We then measured the strain optic coefficient of APS thermal
barrier coatings. The experimental system to make measurements in reflection is described in Section 2. Results are provided in Section 3. Finally, in Section 4 we discuss how strain distributions in TBCs could be measured from variations in refractive index after manufacture, potentially leading to an approach for quality control that is not currently available.

Fig. 1. Scanning electron microscope image of an air plasma sprayed (APS) thermal barrier coating. The thermally grown oxide (TGO) develops between the bond coat and the ceramic top coat.

2. Experimental arrangement

A schematic of the reflection polariscope is shown in Fig. 2(a). The source was a synthesizer and amplifier/multiplier chain (Virginia Diodes) that produced a vertically polarized, divergent electromagnetic beam, tunable between 260 and 400 GHz. A pair of plano-convex lenses (L) was used to focus the beam on to the specimen with a spot diameter of approximately 5 mm. A linear polarizer (P) was placed in front of the specimen, oriented so that the vertically polarized component of the incident beam was allowed to pass. A Deben 2kN dual leadscrew tensile testing rig was used to load the specimens, Fig. 2(b). Specimens were oriented at 45° to the optical axis of the source in order to reflect the incident beam into the detector. A second polarizer, used as an analyzer (A), was placed after the specimen, again with its polarization axis orientated vertically to produce a bright field polariscope configuration. The specimens were loaded parallel to the polarisation axis of the illumination. Finally, second pair of plano-convex lenses focused the beam to a pyroelectric detector (D) (Gentec-eo THZ9D).
Fig. 2. (a) Schematic of the reflection polariscope. Source (S), detector (D), plano-convex lens (L), polarizer (P) and analyser (A). (b) Deben tensile stage with dogbone specimen. The polarizer (P) and the mount for the analyzer (A) can be seen on the left and right hand sides respectively of the specimen, both tilted with respect to the optical axis to reduce standing wave interference.

All the measurements were conducted in an anechoic chamber comprising of RF absorbing tiles, which can be seen in the background of Fig. 2(b). RF absorbing tiles were also placed around the feed horn and the detector. To further reduce standing waves between the sample and focusing lenses, both the polarizer and analyser were tilted to an incidence angle of 45° with respect to the optical axis, as shown in Fig. 2(a). Standing waves can create interference patterns that are not captured by the physical model equations and are therefore undesirable.

The bulk YTZP ceramic samples were the same as tested previously in transmission [22]. These specimens were sintered and fired into a dogbone shape of 5 mm thickness and then ground to a thickness of 656 ± 5 μm. The YTZP had a density of 6.02 g/cm³, an average crystal size of <1 μm, a Young’s modulus of 210 GPa and Poisson’s ratio of 0.3, values supplied by the manufacturer.

The thermal barrier coatings were manufactured and supplied by Rolls Royce Plc. using the APS manufacturing process. Mild steel substrates of two thicknesses (3 mm and 1 mm) in the same dogbone shape were used. The test region of each dogbone specimen was coated on one side with yttria-stabilized zirconia (YSZ). During coating, the ends of the dogbones were covered so that the coating did not interfere with specimen mounting. A typical specimen is shown in Fig. 3 and another can be seen in the tensile test rig in Fig. 2(b). The average coating thickness, measured using a micrometer, was 369 ± 17μm, which is typical for the APS coating process [1,6]. A cross-section of a coating produced in this way was shown in Fig. 1.

Fig. 3. Dogbone specimen with YSZ thermal barrier coating.
3. Measurement procedure

Before loading each sample, the tensile stage load cell was balanced by adjusting the tensile stage such that a load of 0 N was applied once the specimen had been clamped in place. The absolute load applied to each sample was then increased, and the refractive index was measured at the center of each specimen. The tensile stage maintained a constant load during each refractive index measurement.

![Normalized frequency spectrum for (a) bulk YTZP specimen and (b) a YSZ thermal barrier coated specimen loaded at 50 N. The black dots indicate the experimentally measured normalized power and the blue line is the fit of the Fresnel equation appropriate for each case.](image)

At each load, the refractive index was measured by tuning the source frequency between 260 and 400 GHz in 0.25 GHz steps, and measuring the reflected power at each frequency. Variations in the source emission and detector response were normalized by recording the frequency spectrum from a plane aluminum dogbone with no coating, i.e. a mirror, placed in
the tensile test rig prior to testing each specimen. Typical normalized spectra in reflection are shown in Fig. 4 for bulk YTZP and for a YSZ thermal barrier coating. The data were analyzed in the range between 280 and 380 GHz, where the response of the source and detector, combined with good transmission by the ceramic, yielded an acceptable intensity signal. The bulk YTZP specimens were thicker than the TBC and hence more cycles in the intensity variation are seen.

The shape of these spectra are described by the Fresnel equations for non-normal incidence of a single slab dielectric [22,29,30]. The reflection coefficient for a single slab dielectric, surrounded by air \((n_{\text{air}} = 1)\), at non-normal incidence is,

\[
R = \frac{\rho_1 + \rho_2 e^{-2\gamma d}}{1 + \rho_1 \rho_2 e^{-2\gamma d}} \tag{3}
\]

where \(\rho_1\), \(\rho_2\) are the Fresnel coefficients and \(L\) is the phase thickness. The phase thickness is defined to be,

\[
L = \frac{2\pi n \left(t_0 + t\right)}{\lambda} \tag{4}
\]

where, \(t_0\) is the actual substrate thickness, \(n\) is the complex refractive index, \(\lambda\) is the wavelength and,

\[
t = t_0 \left(1 - \cos \theta'\right) \cos \theta' \tag{5}
\]

is a thickness correction factor that depends on the refraction angle of the beam inside the material. The refraction angle is calculated using Snell’s law,

\[
\theta' = \sin^{-1}\left(\frac{\sin \theta n_{\text{air}}}{n}\right) \tag{6}
\]

The first Fresnel reflection coefficient for a transverse electric (TE) incident beam as used in our experimental arrangement is,

\[
\rho_1 = \frac{\cos \theta - \sqrt{(n_{\text{air}}/n)^2 - \sin \theta'^2}}{\cos \theta + \sqrt{(n_{\text{air}}/n)^2 - \sin \theta'^2}}. \tag{7}
\]

The second Fresnel reflection coefficient depends on whether the material is a dielectric slab in air as for the bulk YTZP samples, Fig. 5(a), or a dielectric coating on a reflective surface as for the YSZ coating samples, Fig. 5(b). For the single slab dielectric, bulk material,

\[
\rho_2 = \frac{\cos \theta' - \sqrt{(n_{\text{air}}/n)^2 - \sin \theta'^2}}{\cos \theta' + \sqrt{(n_{\text{air}}/n)^2 - \sin \theta'^2}}. \tag{8}
\]

For the dielectric coating applied to a substrate the Fresnel coefficients are,

\[
\rho_2 = \tau_1 = 1 + \rho_1, \quad \tau_2 = 0 \tag{9}
\]

where \(\tau_1\) and \(\tau_2\) are the Fresnel transmission coefficients.
Fig. 5. (a) Single slab dielectric and standard Fresnel coefficients for bulk material. (b) Single slab dielectric with revised Fresnel coefficients for a coating on a reflective surface.

The refractive index of the material was calculated from each normalized frequency spectrum by conducting a non-linear least squares fit to the measured frequency spectra, Fig. 4. Both the refractive index $\tilde{n}$ and the reflection coefficient $R$ [Eq. (3)], are complex quantities for a material with appreciable absorption such as ceramic. Hence the power measured by the detector was fitted to $R$ multiplied by its complex conjugate, which results in a real value. The thickness of each specimen was estimated from the measured 0 N frequency spectrum, by performing the non-linear least squares fit to the Fresnel equation and allowing both the refractive index and the thickness to vary, using the thickness measured with the micrometer as an initial estimate. The thickness determined from the fit to the specimen at 0 N was then used in the fits to subsequent spectra obtained at increased loads. In those subsequent fits, only the refractive index was allowed to vary.

4. Results

Results for the bulk YTZP and APS thermal barrier coatings are described in the following two sections.

4.1 Bulk YTZP ceramic

Three individual samples were tested, three times each. Each specimen was loaded between 0 and 600 N, in increments of 50 N and the refractive index, $\tilde{n}$, at each load measured as described in the previous section. The refractive index as a function of applied stress is shown in Fig. 6(a). Stress was calculated by dividing the applied load by the unloaded cross-sectional area of each specimen.

Excellent repeatability was achieved for each specimen, although there is an offset in the refractive index between each sample. This offset between samples was also observed in our previous transmission experiments. A sensitivity analysis showed it was due to uncertainty in the measured thickness of each specimen rather than differences in the manufactured material such as density, residual stress or grinding [22]. Furthermore, it is the change in refractive index with applied load that is important when calculating the stressoptic coefficient. Hence to remove the offset, the mean of all measurements for all specimens at a given stress was calculated, shown by the dashed line in Fig. 6(a). A constant offset was then applied to each data set for each specimen, based on a least-squares minimization to the mean of all specimens, as described in [22]. In this way, the refractive index offset due to measurement uncertainty in the specimen thickness was removed without affecting the gradient measured for a given experiment, Fig. 6(b). The same data are repeated in Fig. 6(c) with an expanded vertical scale. The points show the mean refractive index at each stress value from Fig. 6(b). Assuming a constant sample thickness under loading, i.e. the engineering stress, the direct stress optic coefficient was calculated from a least-squares linear fit, and is shown in Table 1.

Figure 6(c) and Table 1 also show the stress optic coefficient calculated using the reduction in cross-section under load, i.e. true stress. In this case, both the thickness at each load used in the fit to the Fresnel reflection equation, and the cross-sectional area used in
calculating stress from the applied load, were adjusted using Poisson’s ratio for the bulk YTZP material. Due to the high Young’s modulus of YTZP, there is only a small difference between the two values.

![Graphs showing measured refractive index versus applied stress for bulk YTZP, offset corrected refractive index versus applied stress, and stress optic coefficients for bulk YTZP using constant and variable sample thickness in the analysis.](image)

**Fig. 6.** (a) Measured refractive index versus applied stress for bulk YTZP (b) Offset corrected refractive index versus applied stress. (c) Stress optic coefficients for bulk YTZP using constant and variable sample thickness in the analysis.

| Table 1. Comparison of stress optic coefficient values for YTZP |
|---------------------------------------------------------------|
| **Direct stress optic coefficient (1/GPa)**                  |
| Reflection Constant thickness | Variable thickness |
| 0.1258 ± 0.0103 | 0.1335 ± 0.0102 |
| Transmission [22] Constant thickness | Variable thickness |
| 0.1285 ± 0.0155 | 0.1362 ± 0.0155 |
| Difference | 2.10% | 1.98% |

Table 1 includes the direct stress optic coefficient previously calculated for YTZP in transmission, also for constant and variable thickness. The percent difference using the transmission values as a baseline is shown. There is an excellent agreement between the two methods, resulting in a maximum difference of ~2% between the two approaches. Furthermore, the mean value for each method lies within the error of the other method, indicating that the measured variation is due to experimental error, which is principally intensity noise affecting the fit to the Fresnel equation. Having validated the reflection polariscope methodology, the measurements were extended to APS coatings.
4.2 APS thermal barrier coatings

Two samples of each substrate type were tested three times each. Each sample was loaded between 0 and 1,200 N in 100 N steps, while a frequency sweep of the GHz source was recorded at each load increment. Figure 7(a) and 7(b) show the measured refractive index as a function of applied strain obtained by the non-linear least squares fit to Eq. (3) from the frequency sweep at each load, for the thick and thin substrates. The applied load was similar for both substrates and hence the applied strain range for the thick substrate is smaller, Fig. 7(a). It is worth emphasizing that the coating thickness on each substrate thickness was the same.

The measured refractive index was plotted against strain, rather than stress, because it is not straightforward to calculate stress in the APS coating. Firstly, the load was not applied directly to the coating, but was transferred via strain in the substrate through the bond coat. Secondly, Young’s modulus for a TBC varies significantly from that of bulk zirconia (~200 GPa) and depends on its chemical composition, the substrate material and the manufacturing process. It is clear that the TBC carries a significantly smaller proportion of the total load than the substrate, because it is thinner and a typical Young’s modulus of a TBC (~50 GPa) is significantly smaller than that of mild steel (210 GPa). Calculating the strain in the substrate alone, ignoring the small reinforcement from the TBC, results in an overestimate of the strain in the TBC of only ~2% and ~6% for the thick and thin substrates, respectively. Hence, the strain in the TBC was assumed to be equal to the strain in the substrate, which was calculated by dividing the applied load by the unloaded cross-sectional area and Young’s modulus of the substrate. This small overestimate of strain results in a lower bound for the calculated strain optic coefficient.

Fig. 7. Measured refractive index versus applied strain for YSZ TBCs. (a) Thick substrate (3 mm) and (b) thin substrate (1 mm). Offset corrected refractive index versus applied strain for (c) thick and (d) thin substrate specimens. The coating thickness is the same for each specimen.
The measured refractive index, as a function of applied strain for the TBCs is shown in Figs. 7(a) and 7(b). Again there is an excellent repeatability between experiments on a given specimen but an offset in the refractive index between each sample. This offset between samples was most noticeable for the TBC deposited on the thin substrate, although with only two specimens per substrate thickness, it is not possible to draw a general conclusion from this observation. The dashed lines in Figs. 7(a) and 7(b) represent the mean of all measurements for all specimens at a given strain. The constant offset in refractive index was removed by the procedure described in the previous section, resulting in the scatter plots shown in Figs. 7(c) and 7(d). A linear least-square fit was applied to each sample in Figs. 7(a) and 7(b) to obtain the direct strain optic coefficients (dimensionless) of \((-0.0133 \pm 0.0102) \times 10^{-9}\) and \((-0.0190 \pm 0.0043) \times 10^{-9}\) for the thick and thin substrates, respectively. The measured strain optic coefficient may be converted to the stress optic coefficient if the Young’s modulus of the TBC is known.

In order to visualize more clearly the strain optic coefficient measured for the thick and thin substrates, Fig. 8 shows the mean refractive index at each applied strain from Figs. 7(c) and 7(d), with an expanded vertical scale. The maximum refractive index at zero strain for both specimen thicknesses has been normalized to unity, in order to remove the offset in the refractive index between the samples with thick and thin substrates. The change in refractive index with applied strain, i.e. the direct strain optic coefficient calculated above is shown by the straight line in each case, with appropriate scaling to the normalized refractive index. The strain response of the APS coatings on both substrate thicknesses is very similar.

5. Discussion

The refractive index measured for the YSZ coatings, Fig. 8, was subject to more variation than the bulk YTZP, Fig. 6. The main reason for this is the relatively rough surface of the APS coating and the bond coat interface, of the order of ± 25 μm in Fig. 1, compared to the ground surface of the bulk YTZP. Similarly the internal structure of the APS coating is less homogeneous than the bulk YTZP. The associated increase in scatter of the reflected beam in the TBC introduces noise into the measured reflected power and hence the accuracy of the non-linear least squares fit.

As discussed above, the relatively small difference in refractive index between specimens of the same type was attributed to uncertainty in the measured thickness. However, a much
more significant difference in refractive index was observed between the YSZ coatings (of the same nominal thickness) deposited on the thick and thin substrates, Fig. 7(a) and 7(b). The APS coatings were applied to the dogbone substrates at exactly the same time, so the variation in coating thickness between specimens was minimal. It indicates that the refractive index depends on the manufacturing process, in particular the thermal history of coating application and cooling which is different between the thick and thin substrates. However, the strain optic coefficient measured in each case was very similar, as emphasized by Fig. 8.

Another interesting feature of testing the YSZ coatings was a systematic reduction in the measured refractive index with each loading cycle. Figure 7(a) and 7(b) show three repeated tests for each of the two specimens. Within each grouping of three measurements, the small offset in refractive index decreased from the first to third loading, although the strain optic coefficient remained the same. This apparent systematic change in refractive index with load cycle, and possibly thermal cycles, could be due to a structural change that might be related to aging but clearly requires further investigation.

The results presented open the way for sub-surface strain measurements in structural ceramics and ceramic coating systems using GHz illumination. We are currently using the techniques described in this paper to investigate how variations in refractive index are related to intrinsic material properties, manufacturing process parameters and ageing effects. We are particularly interested in attempting to quantify the strain distributions in the TBC that lead to failure, for which the direct strain optic coefficient determined in this paper is the starting point. It is possible that strain distributions cannot be quantified by this approach, perhaps because the effect of strain on the refractive index cannot be isolated from local variations in coating thickness and thermal history during the manufacturing process. If that turns out to be the case, we have already begun to observe qualitative variations after manufacture that can be related to time to failure in accelerated ageing trials of TBCs. Ultimately, either a quantitative correlation between the in situ strain after manufacture and the expected remaining lifetime of the TBC, or an empirically determined birefringence distribution that is characteristic of TBCs that will fail quickest, would enable sub-optimal components to be discarded before they are put into service and address a major challenge to TBC development [3].

Conclusions

A reflection polariscope and GHz illumination was used to measure the stress optic coefficient of bulk YTZP. A value of 0.1335 ± 0.0102 /GPa in the range 260-380 GHz was measured in reflection, in agreement with our previously reported transmission measurements. The system was then used make the first observation of stress induced birefringence in YSZ thermal barrier coatings. Variations in the measured refractive index were observed between identical coatings deposited on substrates of different thickness and between repeated loading experiments, indicating the importance of both the thermal and mechanical history on the TBC properties. However, despite variations in the absolute refractive index, similar strain optic coefficients of (−0.0133 ± 0.0102) × 10⁻⁹ and (−0.0190 ± 0.0043) × 10⁻⁹ were measured for APS coatings of the same thickness deposited on substrates of 3 mm and 1 mm steel, respectively. The strain optic coefficient can be converted to stress using Young’s modulus, but it is not readily available for thermal barrier coatings. Research is currently underway to characterize variations in refractive index for TBCs after manufacture, and as they age, in order to develop improved TBC coating systems.

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