

DEVELOPMENT OF NONDESTRUCTIVE EVALUATION METHODS FOR CERAMIC COATINGS

J. G. Sun

Argonne National Laboratory, Nuclear Engineering Division
9700 S. Cass Ave., Argonne, IL 60439

E-mail: sun@anl.gov; Telephone (630)252-5169; FAX (630)252-2785

ABSTRACT

Nondestructive evaluation (NDE) methods are being developed at Argonne National Laboratory for thermal barrier coatings (TBCs) applied to components in the hot-gas path of advanced high-efficiency and low-emission gas turbines, including syn-gas fired turbines. TBCs are typically applied by electron beam-physical vapor deposition and air plasma spraying on metallic vanes, blades, and combustor liners to allow for increased temperature capabilities of these hot gas-path components. As TBCs become “prime reliant,” it becomes important to know their conditions to assure the reliability of these components. Six imaging NDE methods were developed/identified for TBC characterization. These NDE methods can be used to assess the reliability of new coating processes, identify defective components that could cause unscheduled outages, monitor degradation rates during engine service, and provide data for reaching rational decisions on replace/repair/re-use of components.

INTRODUCTION

Advances in thermal barrier coatings (TBCs), applied by two deposition methods, electron beam-physical vapor deposition (EB-PVD) and air plasma spraying (APS), are allowing higher temperatures in the hot-gas path of gas turbines, including syn-gas fired turbines.¹⁻³ However, as TBCs become “prime reliant” to the performance and reliability of the engine components such as vanes, blades, and combustor liners, it becomes important to know their condition after coating application and at scheduled or unscheduled outages.

Work at Argonne National Laboratory (ANL) is underway to develop NDE methods for TBCs. TBC failure normally starts from initiation of small cracks at the TBC topcoat/bond coat interface. These cracks then grow and link together to form delaminations which eventually cause TBC spallation. Effort at ANL has been focused on optical NDE methods including laser backscatter to detect TBC pre-spall and delamination⁴ and optical coherence tomography to measure TBC thickness.⁵ Optical NDE methods take advantage of the fact that TBCs are optically translucent. As a result, surfaces within and below the TBC can be examined using appropriate optical wavelengths. These methods are mainly used for semi-transparent EB-PVD and thin APS TBCs because of the limited optical penetration depth.

Effort at ANL in last year was also directed to develop thermal-imaging methods that are not limited to TBC thickness. Because large thermal-conductivity disparities exist between the TBC topcoat, the metallic substrate, and the air that fills TBC cracks when they present, thermal imaging is sensitive to detect TBC degradation (cracks and delaminations) because it involves

nondestructive measurement of TBC thermal properties. Two thermal imaging methods were developed. One is a multilayer thermal-imaging processing method that can simultaneously determine the 2D TBC thickness, conductivity, and optical absorptance distributions.^{6,7} This method directly accounts for the TBC translucency that has been a major problem for thermal-imaging application to TBCs. The other is a thermal tomography method which can directly image the 3D distribution of thermal effusivity in a TBC material system.⁸

NDE METHODS FOR TBC CHARACTERIZATION AND IMAGING

Although many NDE methods have been proposed for characterizing TBCs, few were capable or practical to image TBC condition on entire component surface. Because TBC degradation likely starts at locations with abnormal properties due to poor processing or severe service condition, imaging NDE methods, instead of spot-check methods, are especially valuable for monitoring TBC degradation and for predicting TBC lifetime. ANL has developed and identified six imaging NDE methods for TBCs. Four are optical methods: (1) cross-polarization laser backscatter developed by ANL; (2) mid-infrared-wavelength reflectance (MIRR) developed by NASA;⁹ (3) optical coherence tomography (OCT);¹⁰ and (4) cross-polarization confocal microscopy developed by ANL.¹¹ The other two are thermal imaging methods both were developed by ANL as described earlier: (5) multilayer thermal imaging and (6) 3D thermal tomography. Within these six methods, three are capable for 2D imaging [(1), (2), and (5)] and three for 3D imaging [(3), (4), and (6)]. ANL has developed four of these methods and has experience in using the other two methods (OCT and MIRR). All methods are being evaluated at ANL to determine their capability/limitation for quantitative characterization and life prediction for TBCs. In the following, these methods are briefly described and additional research needs to be carried out are identified.

Cross-Polarization Laser Backscatter

When a polarized laser beam is incident on a translucent material such as a TBC, the total backscattered light consists of surface reflection and subsurface backscatter. However, the surface reflection typically has no change in its polarization state while the subsurface scatter has a significant change. Based on this principle, ANL developed the cross-polarization backscatter detection method to selectively measure only the subsurface backscatter from translucent materials, while filtering out the strong surface reflection.¹² The method has been used to investigate TBCs, specifically for health monitoring during isothermal heat-treatment testing, and preliminary results for pre-spall prediction were determined for EB-PVD and APS TBCs.^{4,5,13} This method will be further studied to establish the quantitative correlation between backscatter intensity and TBC degradation (cracking) near the topcoat/bond-coat interface and as functions of TBC thickness. In addition, because optical penetration depth increases with wavelength, detection sensitivity at longer wavelengths (in near-infrared range) will be examined.

Mid-Infrared Reflectance (MIRR)

Mid-infrared reflectance (MIRR) imaging was developed specifically for health monitoring of TBCs by Eldridge at NASA.⁹ Because optical penetration for TBCs is at maximum in mid-infrared wavelengths (3-5 μm), MIRR may have higher sensitivity to detect TBC degradation near the topcoat/bond-coat interface. In this method, a steady-state infrared light source is used to

illuminate the TBC surface and the total reflection, including those from the TBC surface, TBC volume, and cracks near topcoat/bond coat interface, is imaged by an infrared camera in the 3-5 μm band pass. Correlations have been established between progression of delamination cracks, which is related to pre-spall condition, and the MIRR data. Because this method is simple and the instrumentation is available at ANL, it will be investigated for detecting delaminations and predicting pre-spall condition for relatively thick APS TBCs, and for comparison with other NDE methods.

Optical Coherence Tomography (OCT)

OCT is a 3D method originally developed for imaging biological materials.¹⁰ It is based on the Michelson interferometer between a reference and a detection beam to differentiate the reflection from different depths of a translucent material. Using a low-coherence diode laser, an average OCT system can achieve a spatial resolution of 5–15 μm . This method was explored at ANL because the cross-sectional scanning images from this method allow for a direct measurement of the thickness of ceramic coatings, including most environmental barrier coatings (EBCs) and TBCs. For these coatings, thickness uniformity is an important parameter because temperature drop across the coating is dependent upon the thickness, especially if the thermal conductivity is uniform.¹⁴ Details of the experimental setup for the ANL system and preliminary results for EBCs and TBCs have been presented previously.⁵ This method will be further studied to determine its detection depth and spatial resolution when imaging the microstructure and cracking within TBCs.

Cross-Polarization Confocal Microscopy

Cross-polarization confocal microscopy is a new 3D imaging method developed by ANL.¹¹ It combines two well-established optical methods, the cross-polarization backscatter detection and the scanning confocal microscopy, and can achieve 3D subsurface imaging with sub-micron spatial resolutions, which is about one order of magnitude smaller than other optical technologies. Similar to OCT, this method could be used to directly image the TBC thickness but with better spatial resolution. A schematic diagram of the system is shown in Fig. 1. Preliminary tests indicated that, with a moderate 40X objective lens at an optical wavelength of 633 nm, the system has an axial (depth) resolution of ~ 2 μm and a lateral resolution of ~ 0.6 μm . Figure 2 shows a typical cross-sectional scan image of the subsurface of a silicon-nitride specimen with two surface opening cracks induced by indentations at different loads;¹⁵ this is the first known result of a direct image of such fine cracks. The crack extension in the subsurface is clear visible up to ~ 40 μm . This method will be evaluated for imaging TBC microstructure and cracking during the next year.

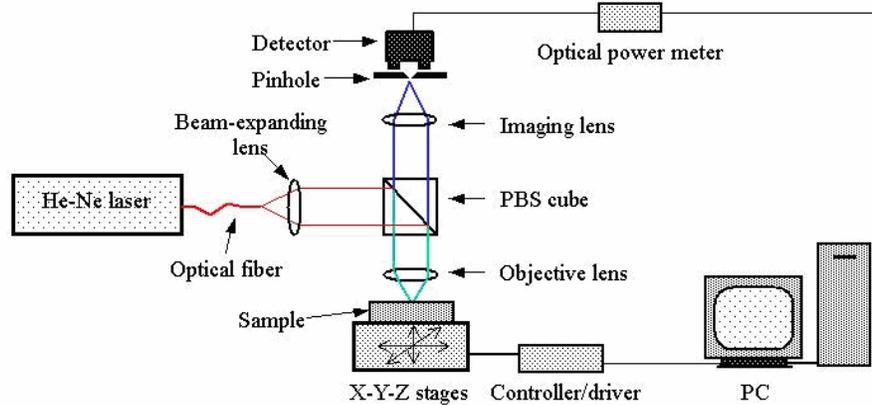


Fig. 1. Illustration of cross-polarization confocal microscopy system.

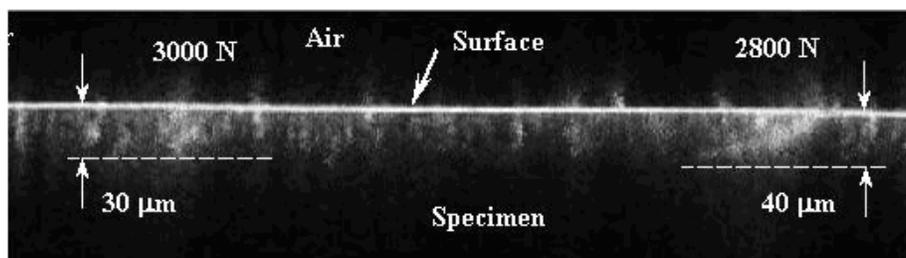


Fig. 2. Cross-sectional confocal-scan image showing cracks in a silicon-nitride ceramic subsurface.

Multilayer Thermal Imaging

In a three-layer TBC system consisting of a ceramic topcoat, a bond coat, and a metallic substrate, a large disparity in thermal conductivity exists between the topcoat and the substrate and, when the topcoat is delaminated with air filling the gap, between the topcoat and the air. For TBC system characterization, flash thermal imaging is effective because it involves nondestructive measurement of thermal properties. Based on pulsed thermal-imaging data, a new multilayer processing method was developed recently by ANL for simultaneously imaging the TBC thickness, conductivity, and optical absorptance.^{6,7}

Figure 3 shows a schematic diagram of a one-sided pulsed-thermal-imaging setup. After a pulsed thermal energy is applied onto the sample surface, the temperature decay on the surface is continuously monitored by an infrared camera. The premise is that the heat transfer from the surface (or surface temperature/time response) is affected by internal material structures and properties.¹⁶ In the new multilayer method, a TBC is modeled by a multilayer material system and the 1D heat-transfer equation under the pulsed thermal-imaging process is solved by numerical simulation. The numerical formulation also incorporates finite heat absorption depth effect due to the TBC translucency. The numerical solutions (of surface temperature decay) are then fitted with the experimental data by least-square minimization to determine unknown parameters in the multilayer material system. Multiple parameters in one or several layers can be determined simultaneously. For a TBC system, the most important parameters are the thickness, thermal conductivity, and absorption coefficient of the TBC in the first layer. This data fitting process is automated for all pixels within the thermal images and the final results are presented as images of TBC thickness, conductivity, and absorption coefficient.⁶

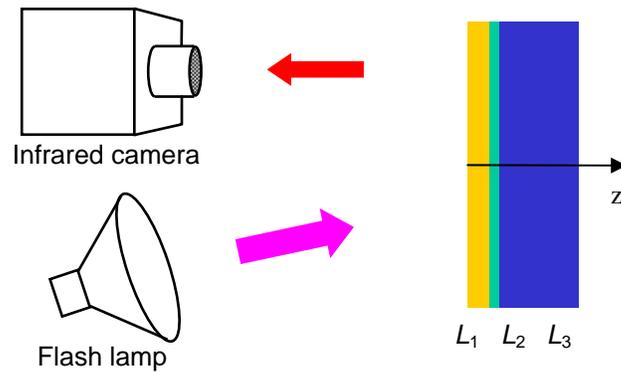


Fig. 3. Schematic of pulsed thermal imaging of a 3-layer material system.

Pulsed thermal imaging test was conducted for an as-processed APS TBC specimen shown in Fig. 4a. It consists of a nickel-based substrate of 2.5 mm thick and a TBC layer with its surface being divided into 4 sections having nominal thicknesses 0.33, 0.62, 0.95, and 1.2 mm. Because this TBC specimen is as-processed, its thermal conductivity and optical absorption coefficient are expected to be uniform. A typical thermal image is shown in Fig. 4b. Figure 5 shows surface-temperature slopes for the 4 thickness sections of this TBC specimen. Comparison of experimental data (Fig. 5a) with theoretical results (Fig. 5b), obtained using approximate material properties, clearly indicates the difference of TBC thickness in these 4 sections.

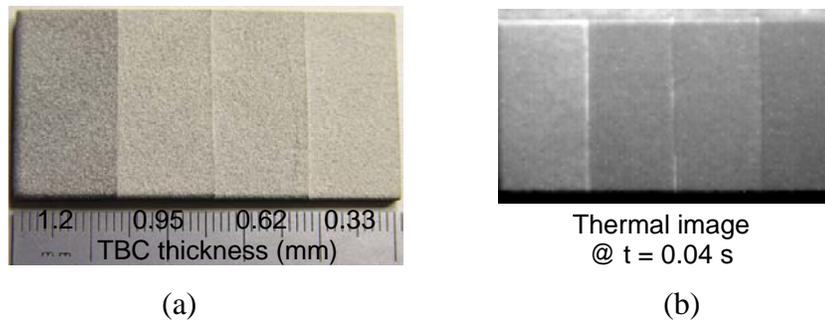


Fig. 4. (a) Photograph and (b) thermal image of a TBC specimen with 4 sections of thicknesses.

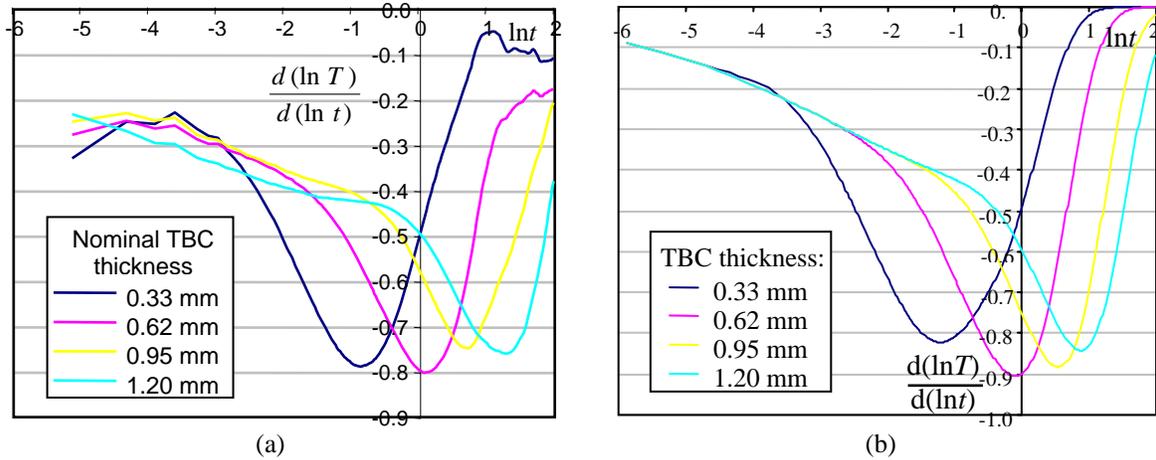


Fig. 5. (a) Measured and (b) predicted surface-temperature-slope data for TBCs of different thicknesses.

Thermal Tomography

All current thermal-imaging methods can only generate 2D images of different material parameters (including the multilayer method described above). A thermal tomography method, which for the first time can construct 3D images of the entire volume of a test specimen, has been developed recently at ANL.⁸ This method directly converts the pulsed thermal-imaging data into a 3D thermal effusivity data that can be viewed/sliced in any plane direction (similar to 3D data from x-ray CT); where thermal effusivity is an intrinsic material thermal property. This method can be directly used for imaging inhomogeneous and multilayer materials without prior knowledge of the material structure and can resolve all flaws, large or small, within the specimen volume; all are new capabilities not achievable by current methods. Thermal tomography has been evaluated for imaging delaminations in both EB-PVD and APS TBCs. Figure 6 shows a plane and a cross-section thermal effusivity image for a 0.3mm thick APS TBC specimen; delaminations (showing with darker grayscales) of various size and severity are detected in both images. In the cross-section image, the thickness of the topcoat and the substrate is also imaged. This method will be further investigated next year for determination of various properties of the TBC layer.

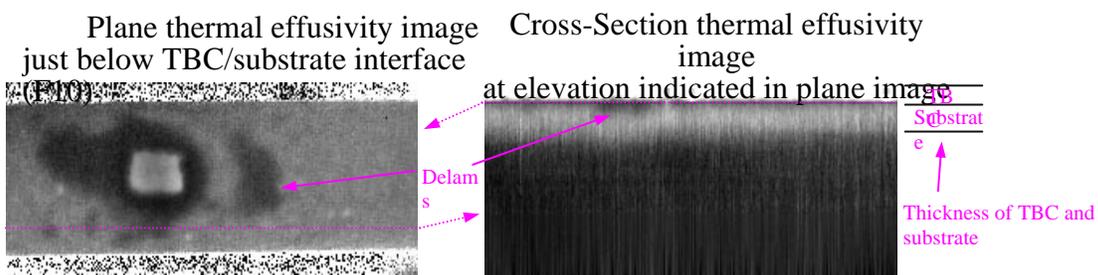


Fig. 6. Typical plane and cross-section thermal effusivity images constructed by thermal tomography method for a 0.3mm thick APS TBC specimen

CONCLUSION

Six NDE methods have been identified to be capable for characterization and imaging of various TBC parameters. Four methods are based on optical principles: (1) cross-polarization laser backscatter, (2) mid-infrared-wavelength reflectance (MIRR), (3) optical coherence tomography (OCT), and (4) cross-polarization confocal microscopy; and two on thermal imaging: (5) multilayer thermal imaging and (6) 3D thermal tomography. Within these methods, three can perform 2D imaging [(1), (2), and (5)] and three for 3D imaging [(3), (4), and (6)]. ANL has developed four of these methods [(1), (4)-(6)] and has experience in using the other two methods (OCT and MIRR). These methods have already been developed with various levels of maturity and have been preliminarily evaluated at ANL for TBC characterization. Effort in next year will be focused on quantitative determination of TBC parameters and on application for TBC life prediction.

REFERENCES

1. North Atlantic Treaty Organization, "Thermal Barrier Coatings," Advisory Group for Aerospace Research and Development Report, AGARD-R-823, Neuilly-Sur-Seine, France, April 1998.
2. US National Research Council, National Materials Advisory Board, "Coatings for High Temperature Structural Materials," National Academy Press, Washington, DC, 1996.
3. US National Aeronautics and Space Administration, "Thermal Barrier Coating Workshop," NASA Conference Publication 3312, 1995.
4. W.A. Ellingson, S. Naday, R. Visher, and C. Deemer, "Development of Nondestructive Evaluation Technology for High Temperature Ceramic Coatings," Proc. 19th Annual Conference on Fossil Energy Materials, Knoxville, TN, May 11-13, 2005.
5. W.A. Ellingson, S. Naday, R. Visher, R. Lipanovich, L. Gast and C. Deemer, "Development of Nondestructive Evaluation Technology for Ceramic Coatings," Proc. 20th Annual Conference on Fossil Energy Materials, Knoxville, TN, June 12-14, 2006.
6. J.G. Sun, "Method for Analyzing Multi-Layer Materials from One-Sided Pulsed Thermal Imaging," Argonne National Laboratory Invention Report, US patent pending, 2006.
7. J.G. Sun, "Thermal Imaging Characterization of Thermal Barrier Coatings," paper presented at the American Ceramic Society's 31st International Cocoa Beach Conference & Exposition on Advanced Ceramics & Composites, Daytona Beach, FL, Jan. 21-26, 2007.
8. J.G. Sun, "Method for Thermal Tomography of Thermal Effusivity from Pulsed Thermal Imaging," Argonne National Laboratory Invention Report, US patent pending, 2006.
9. J.I. Eldridge, C.M. Spuckler, and R.E. Martin, "Monitoring Delamination Progression in Thermal Barrier Coatings by Mid-Infrared Reflectance Imaging," Int. J. Appl. Ceram. Technol., Vol. 3, pp. 94-104, 2006.
10. B.E. Bouma and G.J. Tearney, Handbook of Optical Coherence Tomography, Marcel Dekker, New York (2002).
11. J.G. Sun, "Device and Nondestructive Method to Determine Subsurface Micro-structure in Dense Materials," US Patent No. 7,042,556, issued May 9, 2006.
12. J.G. Sun, W.A. Ellingson, J.S. Steckenrider, and S. Ahuja, "Application of Optical Scattering Methods to Detect Damage in Ceramics," in Machining of Ceramics and

- Composites, Part IV: Chapter 19, Eds. S. Jahanmir, M. Ramulu, and P. Koshy, Marcel Dekker, New York, pp. 669-699, 1999.
13. W.A. Ellingson, R.J. Visher, R.S. Lipanovich, and C.M. Deemer, "Optical NDT Methods for Ceramic Thermal Barrier Coatings," *Materials Evaluation*, Vol. 64, No. 1, pp 45-51, 2006.
 14. P. Bengtsson and J. Wingren, "Segmentation Cracks in Plasma Sprayed Thick Thermal Barrier Coatings," in. *Proc. Gas Turbine Materials Technology*, eds. P.J. Maziasz, I.G. Wright, W.J. Brindley, J. Stringer, and C. O'Brien, pp. 92-101 (1998). Published by ASM International, Materials Park, OH.
 15. W.K. Lu, Z.J. Pei, and J.G. Sun, "Subsurface Damage Measurement in Silicon Wafers with Cross-Polarization Confocal Microscopy," *Int. J. Nanomanufacturing*, Vol. 1, pp. 272-282, 2006.
 16. J.G. Sun, "Analysis of Pulsed Thermography Methods for Defect Depth Prediction," *J. Heat Transfer*, Vol. 128, pp. 329-338, 2006.