Application Note

High Speed Nanoindentation Mapping on Thermal Barrier Coatings

Introduction

Thermal Barrier Coatings (TBCs) are multi-layer, multi-material systems that are used to provide thermal insulation and oxidation resistance for underlying structural components[1]. TBCs typically consist of (1) an intermetallic bond coat, which is usually a MCrAlY alloy, where M = Ni, Co or both; (2) a porous ceramic top coat such as yttria stabilized zirconia (YSZ); and (3) a thermally grown oxide (TGO) layer that forms at the interface between bond coat and top coat during high temperature operation. To add to the complexity, different microstructural features such as the splat boundaries for thermal sprayed coatings, porosity, interface between layers, cracks, etc., also co-exist in TBCs. Moreover, because these coatings are usually subjected to extreme environments, material degradation such as aluminum depletion in the bond coat, growth of TGO at the bond coat-top coat interface, densification of the top coat, etc., occurs with time and temperature[2]. While in operation, these degradation mechanisms can operate simultaneously and cause changes in its microstructure and/or composition[3]. Figure 1 shows a cutaway view of an aircraft engine, showing the variation in temperature and pressure through the airflow path.

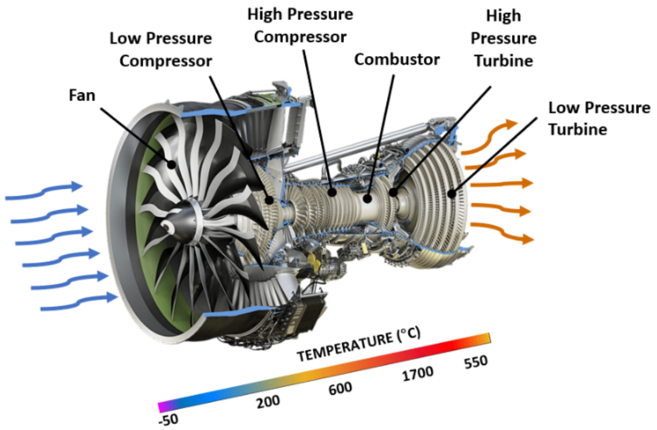

Figure 1. Cutaway view of a GE9X commercial aircraft engine[4] showing variation of temperature and pressure through the airflow path.

Given the complexities inherent to TBC systems and the advantages of emerging material characterization technique such as high-speed mechanical property mapping, there exists a strong case for applying these techniques to the study of TBCs. Doing so enables the study of the spatial and temporal variations in properties of the different TBC layers[5].

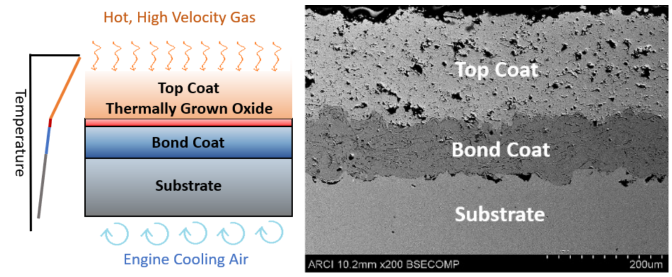

Figure 2. (a) Schematic illustration of the multilayer, multifunctional nature of the thermal-barrier coating system, and (b) cross-sectional scanning electron microscopy image of a TBC. *Schematic reprinted from MRS Bulletin Oct 2012.*

Experimental Method

In this work, the TBCs were tested with a KLA Nanoindenter utilizing NanoBlitz 3D. NanoBlitz 3D is a high-speed mapping technique where each indent typically takes less than a second. The measurement time includes the time taken for surface approach, surface detection, loading, unloading and positioning the sample for the next indentation. Maps of different sizes (typically containing more than 10,000 indents) were generated for the bond coat, top coat and bond coat-top coat interface regions of as-coated and thermally-cycled samples. After performing an area function calibration on fused silica samples and correcting for load-frame compliance, the hardness and elastic modulus for every indent was calculated using the standard Oliver-Pharr method[6].

Results and Discussion

The bond coat-top coat interface is one of the most critical regions of the TBC. During operation, the major microstructural variations and the corresponding mechanical property variations occur in this region, and ultimately have a bearing on the thermal cyclic life of the TBC. The most significant event at the bond coat-top coat interface is the formation of a thermally grown oxide (TGO) layer[7]. TGO forms due to a reaction between inter-diffused aluminum (from the β-NiAl of the bond coat) with the ingressive oxygen (through the top coat) at high temperatures. A dense, contiguous layer of TGO having a parabolic growth rate is known to prevent further oxidation of the bond coat and the underlying substrate. However, beyond a certain critical thickness, this layer causes severe strain incompatibilities and mismatch stresses, causing the TBCs to become progressively damaged and to ultimately delaminate[8,9]. Hence, determining the local elastic modulus in the TGO is extremely valuable. However, given that the optimal TGO thickness is typically on the order of several microns, there has been no reliable local mechanical property measurement when it is sandwiched between the bond coat and top coat. High speed mapping at the micron length scale demonstrates that in the case of the bond coat, it is now possible to measure the mechanical properties of the TGO.

Figure 3 shows the microstructure (top row), hardness map (middle row) and elastic modulus map (bottom row) at the bond coat-top coat interface in the as-coated state and after 5, 10 and 100 thermal cycles. The interface shows undulations that are typical of plasma spray coatings. A comparison of the microstructure of the as-coated and thermally cycled samples shows that the TGO (dark region at the interface) can be observed at five thermal cycles and that the thickness of the TGO increases with thermal cycling. The corresponding hardness and elastic modulus maps show a similar trend, where the TGO can be identified by the higher hardness and elastic modulus regions at the interface. Interestingly, the thickness of the TGO measured from the maps shows a parabolic behavior, which has been observed from microstructural studies in the past[10,11]. In addition to the growth of TGO with an increasing number of thermal cycles, the depletion of the β-NiAl phase, which acts as an aluminum source on the bond coat side, can be clearly observed from the hardness maps and the microstructure.

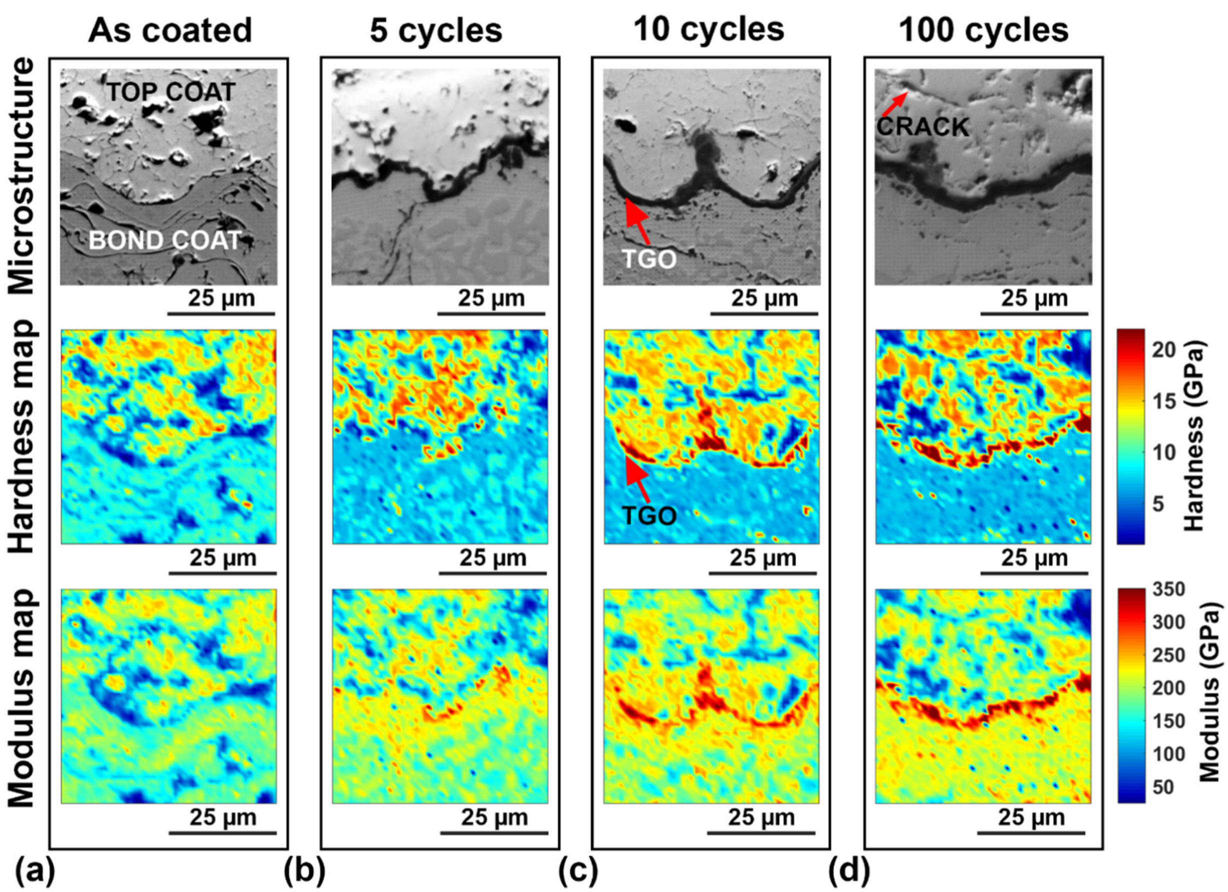

Figure 3. Cross-sectional SEM micrographs and the corresponding hardness and elastic modulus maps at the bond coat-top coat interface show TGO growth for (a, first column) the as-coated state; (b, second column) after 5 thermal cycles; (c, third column) after 10 thermal cycles; and (d, fourth column) after 100 thermal cycles.

The difference in elastic modulus due to the growth of the TGO is one of the primary driving forces for the development of mismatch stresses. These stresses lead to the generation of microcracks in the top coat just above the interface, as shown in Figure 3, micrograph (d), for the case of the sample subjected to 100 thermal cycles. The reduction in hardness and elastic modulus in the cracked regions is also captured by the maps. In summary, the NanoBlitz 3D high speed mapping technique can be used to measure the local mechanical properties at the interface of different layers of a coating and for the specific case of a bond coat-top coat interface of a TBC. This technique enables measurement of local elastic properties which can be readily used in finite element analysis (FEA) for simulating delamination of TBCs subjected to thermal cycling.

To determine phase-level information of the mechanical properties of the various layers in the TBC, the high speed mapping data must be deconvoluted. The clustering algorithm used in this work retains the spatial information about the phases after deconvolution, enabling the reconstruction of a phase map from the mechanical property map. Also, the clustering algorithm does not require an expected range of output, which is typically required for curve fitting procedures. The deconvoluted map obtained from the hardness map along with the microstructure for the case of a sample subjected to five thermal cycles is shown in Figure 4. It can be seen from the deconvoluted hardness map in Figure 4c that the property map in Figure 5b has been separated into three distinct clusters based on the hardness data, which in this case are (1) β-NiAl, (2) γ/γ'-Ni and (3) oxides due to internal oxidation. The mean and standard deviation of the data points in each cluster can be assumed to represent the mean and standard deviation of the corresponding phases. For more detailed information on data deconvolution, please refer to reference [5].

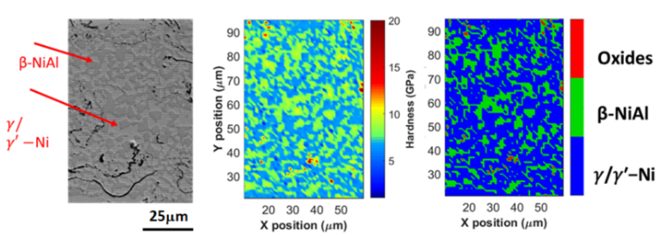

Figure 4. (a) Microstructure, (b) hardness map, and (c) deconvoluted hardness map of the bond coat after five thermal cycles.

Summary and Conclusions

A KLA nanoindenter in conjunction with the NanoBlitz 3D high speed mapping technique was used to investigate thermal barrier coatings, specifically, the bond coat-top coat interface. Excellent correlation was found between the microstructure and the local mechanical properties at the micrometer length scale, even at the interface between the various layers of TBC and in the porous top coat. The phase level properties obtained from this analysis can be readily used for microstructure-based finite element analysis and the large data sets obtained through extensive mapping can also be used to develop data-driven models for predicting residual life of TBCs.

References

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KLA Corporation
One Technology Drive
Milpitas, CA 95035
Printed in the USA
Rev 4 2020-08-20