JMEPEG (2004) 13:389-397
DOI: 10.1361/10599490419883

1059-9495/$19.00

Thermal Shock Testing of Thermal Barrier Coating/Bondcoat Systems

Ann Bolcavage, Albert Feuerstein, John Foster, and Peter Moore

(Submitted February 10, 2004)

Various methods of thermal shock testing are used by aircraft and industrial gas turbine engine (IGT) manufacturers to characterize new thermal barrier coating systems in the development stage as well as for quality control. The cyclic furnace oxidation test (FCT), widely used in aircraft applications, stresses the ceramic/bondcoat interface, predominantly through thermally grown oxide (TGO) growth stress. The jet engine thermal shock (JETS) test, derived from a burner rig test, creates a large thermal gradient across the thermal barrier coating (TBC), as well as thermomechanical stress at the interface. For IGT applications with long high-temperature exposure times, a combination of isothermal preoxidation and thermal shock testing in a fluidized bed reactor may better represent the actual engine conditions while both types of stress are present. A comparative evaluation of FCT, JETS, and a combined isothermal oxidation and fluidized bed thermal shock test has been conducted for selected ceramic/bondcoat systems. The results and the failure mechanisms as they relate to the TBC system are discussed. A recommendation on the test method of choice providing best discrimination between the thermal shock resistance of the ceramic layer, the ceramic/bondcoat interface, and even substrate related effects, is given.

Keywords MCrAlY, thermal barrier coating, thermal shock testing

1. Introduction

Thermal barrier coatings reduce the operating temperature of air-cooled, gas turbine engine components such as combustors, blades, and vanes. The thermal barrier protection system consists of an oxidation-resistant bondcoat and the ceramic thermal barrier layer with a low thermal conductivity (Fig. 1).

Bondcoat chemistries commonly used are either diffusion type coatings with nickel aluminide with additions of Pt-group elements, or MCrAlY-type overlay coatings. For ceramic barrier coatings, partially stabilized zirconia with 7-8 wt.% yttrium oxide is typically used. The ceramic thermal barrier layer is applied by thermal spray or by electron beam physical vapor deposition (EBPVD).

The life of the thermal barrier coating (TBC) system is essentially dependent on the thermal profile of the engine. There is not an insignificant mismatch of the thermal expansion coefficient between the ceramic and the bondcoat, which results in a stress accumulation at the TBC/bondcoat interface. This is the most critical area of the TBC/bondcoat system, due to the abrupt change in mechanical and physical properties. The adherence mechanism in the case of plasma sprayed TBC is to a great extent dependent on the mechanical anchoring of the TBC to the rough bondcoat surface, whereas in the case of EBPVD, the bond is essentially a chemical bond through the thermally grown oxide (TGO) of the bondcoat. Life limiting factors are bondcoat oxidation, i.e., excessive growth of the TGO, as well as in the case of plasma sprayed TBC, the disintegration of the ceramic at the splat interfaces. In reality, a combination of both is typically observed close to the TBC/bondcoat interface.

In present turbine applications, two ceramic coating morphologies are mainly used. The low-density air plasma spray (APS) coating is widely used to protect airfoils, vanes, and combustors (Fig. 2). For applications that need improved thermal fatigue life, vertically segmented coatings, either produced by thermal spray processes or by EBPVD, are successfully used. The dense vertically macrocracked Zircoat™ structure shown in Fig. 3 provides improved tolerance of the ceramic layer to the strain caused by the coefficient of thermal expansion (CTE) mismatch of ceramic and bondcoat.[2] Alternatively, EBPVD TBCs with a fine columnar microstructure (Fig. 4) are used in aircraft applications. For clearance control applications, ceramic multilayer systems or graded density ceramic layers are increasingly used.

To simulate the thermal conditions in an engine, two tests with their derivatives have been developed by the engine manufacturers: the burner rig test with its derivative, the jet engine thermal shock (JETS) test, and the cyclic oxidation, or furnace cycle test (FCT).

The burner rig test creates extreme temperature gradients in the ceramic and essentially stresses the ceramic coating integrity. Typically, pin or bar shaped samples are cyclically heated by a kerosene fueled torch and then cooled by forced air. The burner rig test is expensive to operate, so the JETS test has been developed as a more economic high thermal gradient test. Both the FCT and JETS tests are operated at Praxair Surface Technologies (PST) Technology Laboratories in Indianapolis, IN, and have proven to be effective tools in characterizing TBC systems.

For industrial gas turbine (IGT) applications with extremely

This paper was presented at the 2nd International Surface Engineering Congress sponsored by ASM International, on September 15-17, 2003, in Indianapolis, Indiana, and appeared on pp. 520-29.

Ann Bolcavage and **Albert Feuerstein,** Praxair Surface Technologies, Inc., Indianapolis, IN 46222; and **John Foster** and **Peter Moore,** Praxair Surface Technologies, Ltd., Oldmixon Crescent, Oldmixon Industrial Estate, Weston-super-Mare, No. Somerset BS24 9AX, UK. Contact e-mail: ann_bolcavage@praxair.com.

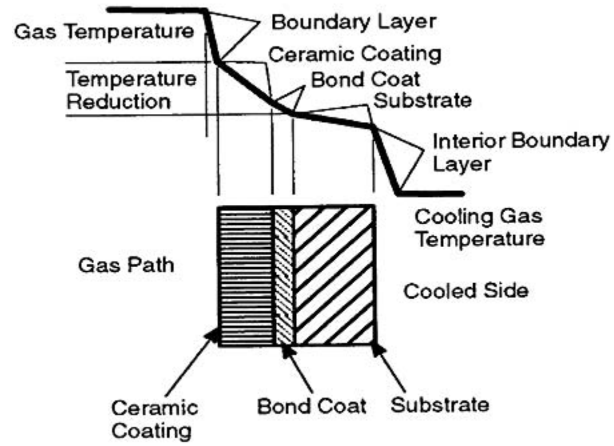


Fig. 1 Schematic illustration of a TBC on an air-cooled engine component[1]

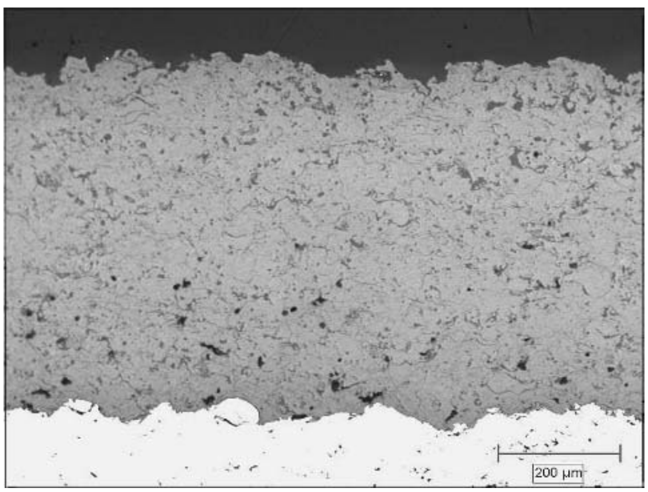


Fig. 2 Low-density YSZ TBC by APS, with a layered network of interconnected splats; typical density is 85%.

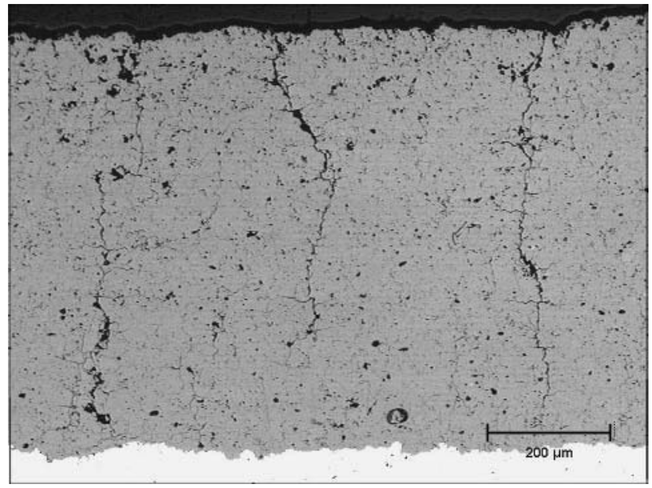


Fig. 3 Dense vertically macrocracked APS TBC-Zircoat™; three segmentation cracks per linear millimeter

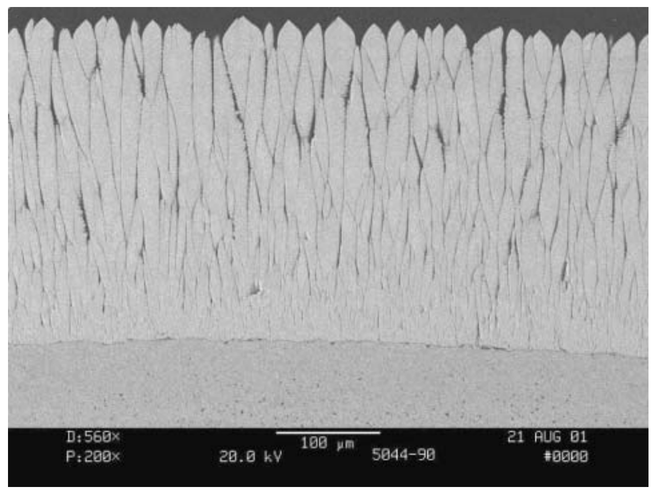


Fig. 4 EBPVD TBC with a fine columnar microstructure

long oxidation times at moderate temperatures, Praxair's Weston-super-Mare facility has built a thermal shock test that exposes isothermally preoxidized samples to a rapid cycle thermal shock test in a fluidized bed reactor.

In this paper, the JETS, FCT, and fluidized bed thermal shock tests are described and compared. The failure mechanism of selected TBC/bondcoat systems in each of these test environments is evaluated. To better understand and distinguish the TBC systems and test specific failure mechanisms, extreme TBC/bondcoat combinations have been chosen for this investigation. TBC morphologies include low-density yttrium-stabilized zirconia (YSZ), dense vertically macrocracked TBC by plasma and EBPVD, and dual layer plasma sprayed TBC. Bondcoat chemistry and morphologies include high-velocity oxy-fuel (HVOF) or plasma MCrAlY bondcoat and electro-deposited MCrAlY bondcoat applied with the Tribomet process, developed by Praxair Surface Technologies, Concord, NH.

2. Testing Methods

2.1 FCT Test

The FCT test creates a high-temperature isothermal environment for enhanced bondcoat oxidation with a moderate thermal shock to cyclically stress the ceramic/bondcoat interface.

The TBC system is oxidized and cycled between room temperature and temperatures ranging from 1100 to 1200 °C. Thermal gradients across the TBC are moderate during the heat-up phase and significant during the cool-down phase. The sample is exposed for a long time to elevated temperatures, which leads to significant bondcoat oxidation. However, the TBC temperature during soaking is too low to cause pronounced sintering in the ceramic. The FCT test is a long term one; typically several hundred hours are required to differentiate between coating systems.

Figure 5 shows a bottom-loading furnace and horizontal

Fig. 5 Bottom-loading furnace and horizontal tube furnaces for cyclic FCT testing of TBC/bondcoat systems

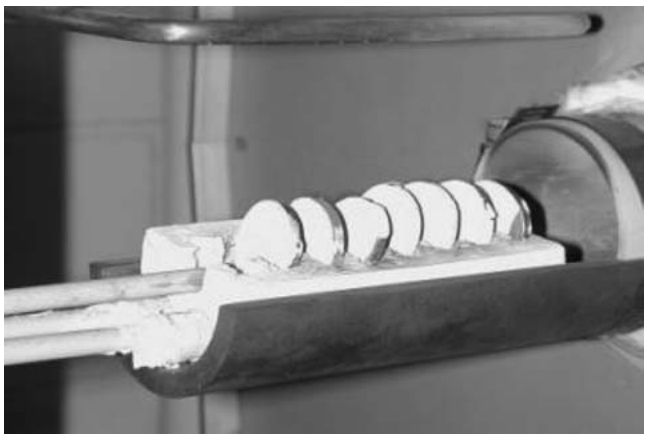


Fig. 6 FCT sample holder for button samples

tube furnaces used for testing. In the case of the horizontal tube furnace, the alumina tube is heated to the desired temperature, creating an isothermal heating zone of typically 15-20 cm in length. The samples are loaded onto a ceramic boat and introduced into the hot zone with a pneumatic manipulator (Fig. 6). The heating and cooling rates are several hundred degrees Celsius per minute (Fig. 7). The highest thermal gradients are achieved during cooling when forced air is used to cool the surface of the TBC. In the case of a bottom-loading furnace the hot zone is typically a cube with dimensions of 15 × 15 × 15 cm. The samples are loaded on a ceramic fixture attached to a retractable bottom lid. Forced air cooling is accomplished through the use of a fan.

Typical test cycles for aircraft engine parts are 1 h cycles (45-50 min at elevated temperature and 15-10 min of forced air cooling) between 1080 °C and room temperature (RT) or 1135 °C and RT. For IGT applications with their extended dwell at elevated temperatures, typically 24 h cycles (23 h at elevated temperature with 1 h of forced air cooling) at 1080 or 1121 °C are used. Failure criterion is typically 20% spallation of the TBC surface. In the FCT test, mostly 2.54 cm diameter button samples are used, although in some instances, pins are used to simulate the added stress due to surface curvature.

2.2 JETS Test

The JETS test is described in great detail in Ref 1. Figure 8 shows the JETS rig assembly at PST in Indianapolis. The JETS test creates a large thermal gradient (several hundred degrees Celsius) across the TBC. TBC front surface temperature can achieve 1400 °C and can initiate sintering. This test mainly stresses the ceramic layer and bondcoat interface thermomechanically. Due to the large thermal gradient through the ceramic, the TBC/bondcoat interface oxidizes only marginally. The JETS test is reasonably quick, and typically results are available within two days for a 2000 cycle test.

Four oxygen-propylene torches sequentially heat the TBC coated surface on 2.54 cm diameter buttons, which are attached to a carousel. A stepping motor advances the buttons through the positions. Immediately after being heated, the buttons are cooled by a nitrogen jet to provide the maximum possible temperature gradient during cool down. Typically, the coated surface temperature is in the range of 200-300 °C at the end of its ambient cooling time.

The carousel has 15 positions for coated buttons, which are secured with special Hastelloy X fixtures to allow for temperature measurement of both the front and back face of the button by pyrometery, as well as a position containing a fixed Hastelloy X reference panel with a thermocouple welded to the backside. This is used to ensure that the heat flux coming from each torch is balanced with respect to one another.

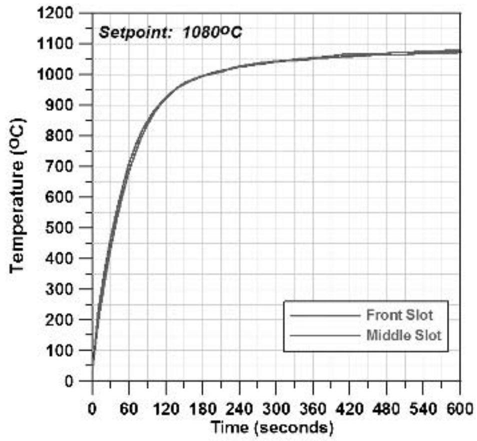
A typical test cycle consists of a 20 s heat up, a 20 s forced nitrogen cooling cycle, and a 40 s dwell cycle in ambient air. The gas flow and torch tip standoff distance are adjusted so as to achieve a standardized front surface temperature of approximately 1400 or 1232 °C at the end of the 20 s heat-up phase, dependent on the required severity of the test. The front and back surface temperature is measured with a two-color pyrometer at a wavelength of 1.2 μm.

The temperature gradient over the sample is dependent on the TBC thickness, composition, density, and microstructure. For example, a vertically macrocracked 40 mil 8YSZ coating typically gives a temperature difference between the front-face and back-face of approximately 400-500 °C. The temperature difference is also used to monitor the integrity of the TBC coating. A sudden significant increase in the temperature difference indicates a delamination within the TBC or at the bondcoat interface (Fig. 9).

Zirconium oxide has some translucency in the near infrared, and due to this, the temperature measured with the short wavelength two-color pyrometer can be somewhat on the low side, depending on the temperature gradient over the TBC system. Therefore, a single-color pyrometer at a wavelength of 7.9 μm is used in addition to measure the front surface temperature for special coating systems and test requirements.

Before testing, each coated button has its edges ground to achieve an overall diameter of 2.38 cm to remove any coating shouldering or overhang. The buttons are then inspected using a 30× magnification viewer to look for any machining damage or interfacial edge cracking in the coating.

After 2000 cycles, the edge of the button is inspected for



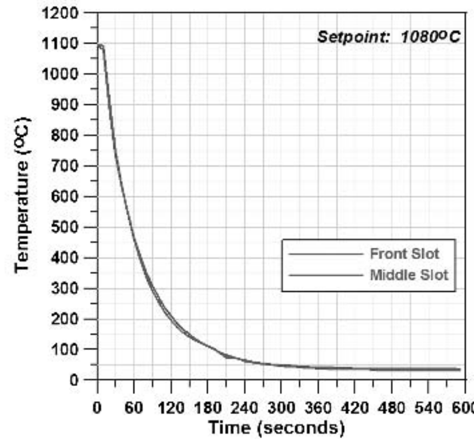


Fig. 7 Typical heat-up and cool-down rates in the FCT test measured with thermocouples attached to the sample surface. Furnace 5: temperature profile vs. boat position

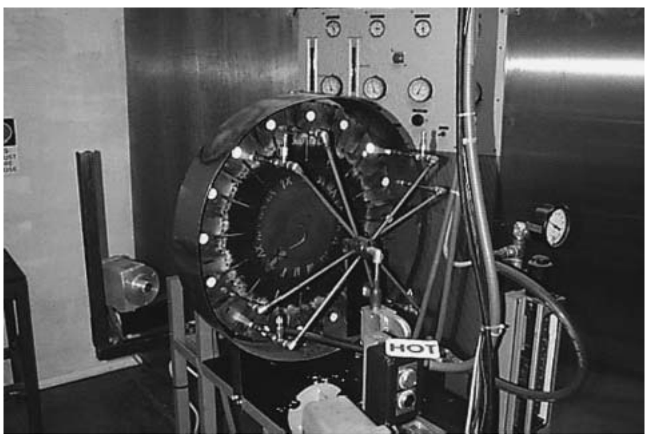


Fig. 8 Large JETS rig with four heating/cooling stations in front of samples

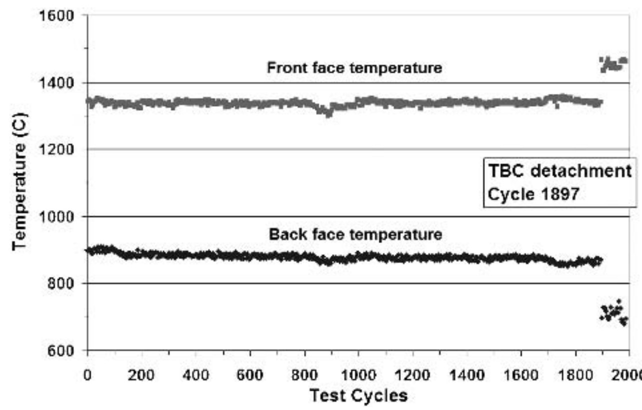


Fig. 9 Typical JETS temperature profile front- and back-face temperature over 2000 cycles with TBC detachment at 1897 cycles

cracks in the TBC layer or at the TBC/bondcoat interface (Fig. 10). A percentage of circumferential edge cracking is calculated using the formula:

$$\%\text{ edge cracking} = \frac{\begin{array}{c}\text{total length of cracks along}\\ \text{edge circumference} \times 100\end{array}}{\text{circumference of button}}$$

The length of the cracks and circumference must be in the same units. The percentage of circumferential crack length is used as a measure to characterize the coating system performance. For example, in well-performing, dense, vertically macrocracked TBC systems with a thickness of 1.27 mm, the increase of edge cracking should be less than 15% after 2000 test cycles with an average front surface temperature of 1400 °C.

Typically, a crack initiates at the edge and propagates radially to the center of the button until complete spallation of the coating occurs (Fig. 11).

2.3 Fluidized Bed Test

In the fluidized bed test, the hot zone of the test rig consists of a standard fluidized bed furnace using alumina. It is typically operated at 1000 °C. Test samples are transported via a pneumatic system into a cooling zone in which compressed air is directed onto the TBC surface. A photograph of the rig is shown in Fig. 12.

Heating and cooling rates, using thermocouples located in the center and on the surface of a nickel alloy block 60 × 40 × 9 mm, are equal to 15-20 °C per second over the range 950-350 °C (center of the block). Typically, samples are heated for 5 min in the fluidized bed and then cooled for 5 min. The temperature at the TBC surface follows much faster, with Fig. 13 showing a typical temperature cycle of a thermocouple immersed into the fluidized bed.

2.4 Comparison of JETS Test and FCT

To demonstrate the suitability of the FCT and JETS test to reveal the essential failure mechanism of TBC systems, three examples have been selected that vary significantly in the morphology of the ceramic structure and the bondcoat surface topography. Example 1 included systems consisting of a low-density ceramic layer produced with powders from two different vendors, deposited on either an HVOF or Tribomet applied bondcoat. This combination was chosen to determine which

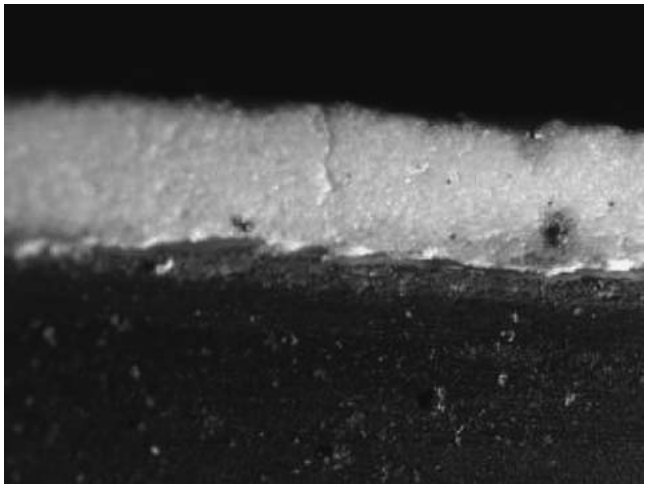


Fig. 10 Edge cracking between TBC and bondcoat after JETS testing

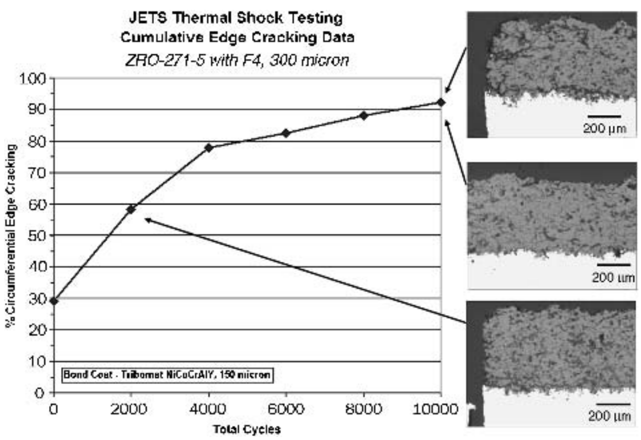


Fig. 11 Cumulative edge cracking and failure appearance mechanism at different stages of the JETS test; with low-density TBC, typically some initial edge damage is observed after outer diameter (OD) grinding due to the fragility of the coating

test is the better suited to discriminate between bondcoat induced TMF failure and ceramic induced TMF failure. Example 2, a two-layer ceramic system, was used to evaluate the capability of the FCT and JETS test to identify critical interfaces within the ceramic only. Example 3 was chosen to demonstrate the capability of the JETS test to assess the performance of long-life ceramic coatings. A comparison of a plasma torch-applied vertically macrocracked and EBPVD TBC was made.

2.4.1 Example 1. Spray dried and sintered 7-9YSZ powders from two different manufacturers were sprayed onto bondcoats with a Sulzer-Metco F4-MB (Westbury, NY) torch to yield an 85% dense TBC with a thickness of 300 μm. Two different bondcoats were investigated. The HVOF bondcoat was a NiCoCrAlY (bal Ni, 22Co, 22Cr, 12Al, 0.5Y; in wt.%) sprayed with a JP5000 system (Tafa Inc., Division of Praxair Surface Technologies, Concord, NH) to a thickness of approximately 150 μm. TM309, an electroplated NiCoCrAlY (bal Ni, 21Co, 20Cr, 8.5-10Al, 0.4Y; in wt.%) was applied with PST's

Fig. 12 Fluidized bed thermal shock rig in PST Weston-super-Mare, UK

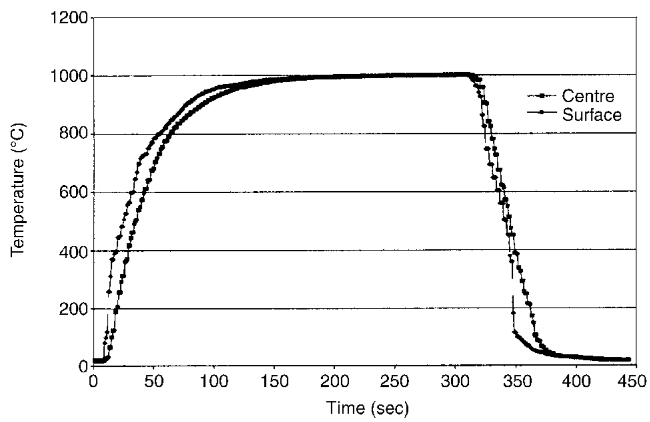


Fig. 13 Typical temperature profile of the fluidized bed thermal shock test

proprietary Tribomet process to a thickness of approximately 125 μm. Details of the Tribomet process are given in Ref 4. The TBC/bondcoat system of Example 1 is shown in Fig. 14(a) and (b).

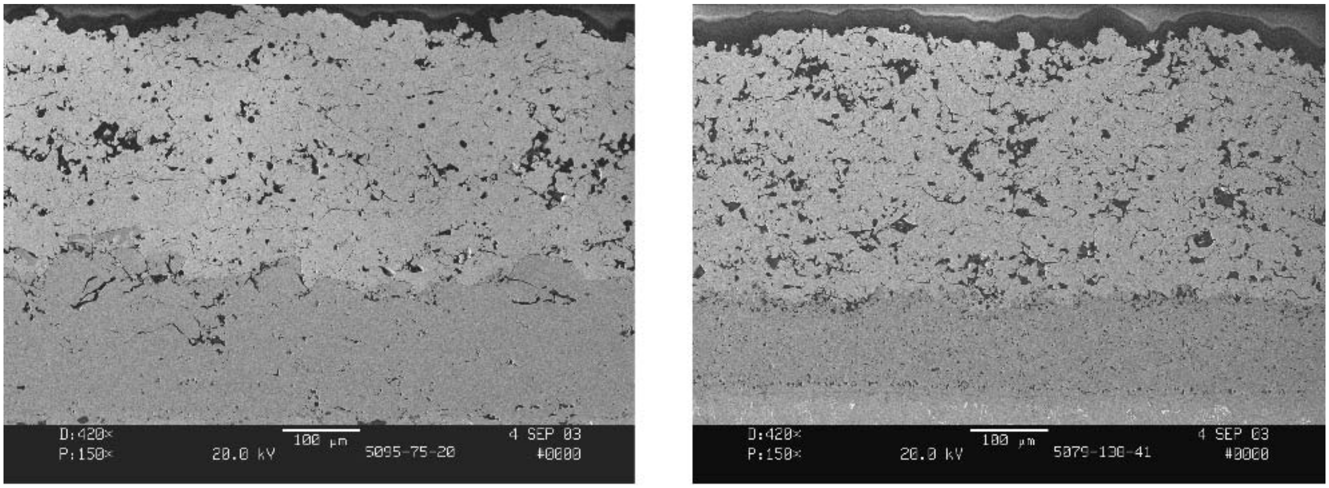
The surface morphology of the bondcoat is significantly different, with the Tribomet surface having a lower surface roughness (R_a). The R_a is typically 2-4 μm, but provides more exposed surface area and interlocking locations to the TBC. In contrast, the HVOF coating typically has an R_a of 8-10 μm.

FCT testing was performed with 24 h cycles at 1121 °C.

The results are shown in Fig. 15. The FCT data show a clear trend of a slightly higher life for the HVOF bondcoat for both YSZ powders. This may be caused by the higher aluminum content of the HVOF bondcoat, resulting in a longer time to deplete the beta, or aluminide phase, which supplies the aluminum for the TGO layer.

JETS testing of the samples from Example 1 was conducted for a total of 10 000 cycles. The results of the accumulated increase in the edge cracking are shown in Fig. 16 in 2000 cycle intervals.

For up to 10 000 cycles, there is no significant difference in



(a) (b)

Fig. 14 **(a)** Low-density TBC on HVOF NiCoCrAlY; **(b)** low-density TBC on Tribomet NiCoCrAlY

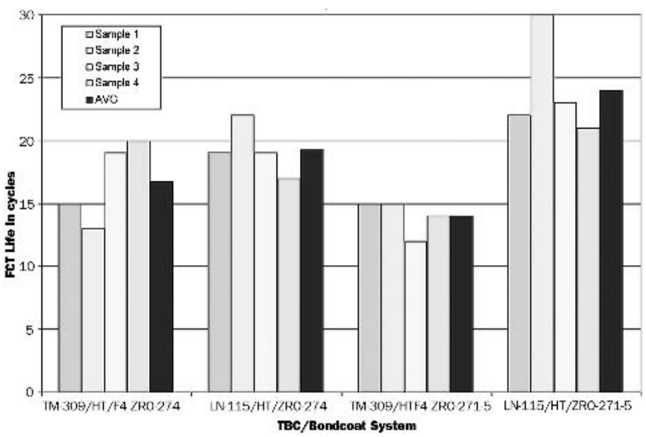


Fig. 15 FCT life of low-density TBC on HVOF and Tribomet bondcoat; 1121 °C, 24 h cycles

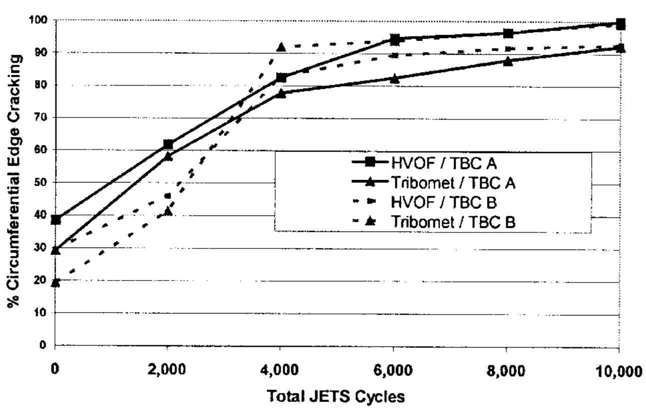


Fig. 16 Increase of cumulative edge cracking during JETS testing for TBC A and B on Tribomet and HVOF bondcoats

the edge crack accumulation, and there is no evidence for a bondcoat dependence. Despite the different bondcoat morphologies, the failure mechanism is comparable (Fig. 17).

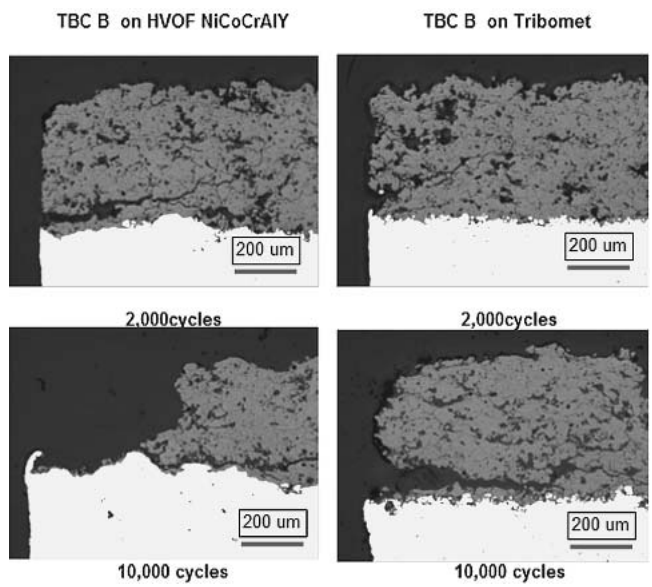
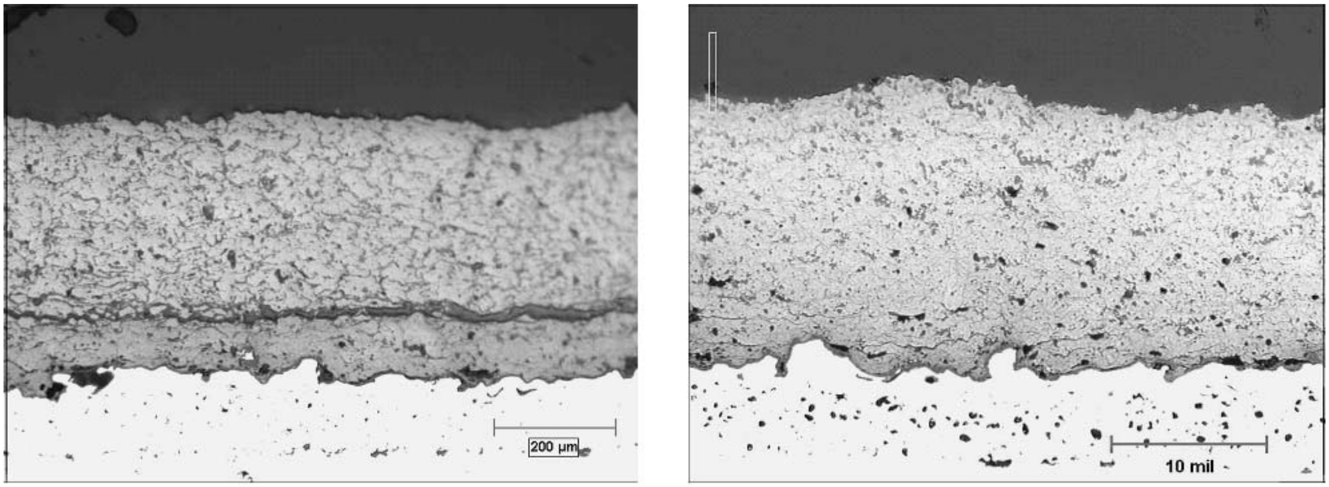


Fig. 17 Failure mode of the JETS samples at 2000 and 10 000 cycles

Both the FCT test and the JETS test show no significant distinction between the ceramics. Each powder produces a comparable TCF life when coupled with the same bondcoat. However, whereas the JETS test does not differentiate between the bondcoat, the FCT test shows a clear trend of a slightly higher number of cycles before failure with the HVOF bondcoat process. This can be explained by the larger aluminum reservoir of the HVOF bondcoat.

2.4.2 Example 2. A proprietary dual-layer TBC system with different ceramic compositions was applied on shrouded plasma sprayed CoNiCrAlY. Two variations of the multilayer system with matching and dissimilar density of the ceramic layers have been tested in the FCT (1121 °C in 24 h cycles) and JETS test.

In FCT testing, only the samples with the density mismatch between the two ceramic layers started to fail after four cycles



(a) (b)

Fig. 18 **(a)** Two-layer TBC system (dissimilar density) after 4 FCT cycles. Note the delamination at the ceramic interface between the two ceramic layers. The density difference is visible. **(b)** Improved two-layer TBC system (matching density) after 10 FCT cycles; no visible delamination at the ceramic interface

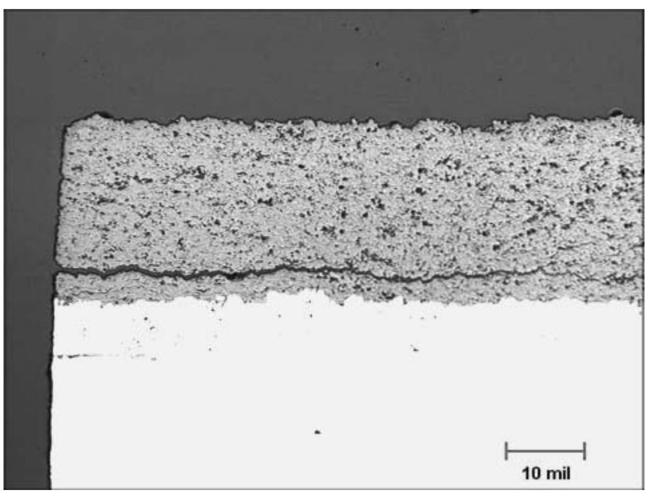


Fig. 19 Improved two-layer TBC system (matching density) after 2000 JETS cycles; delamination occurs at the ceramic interface.

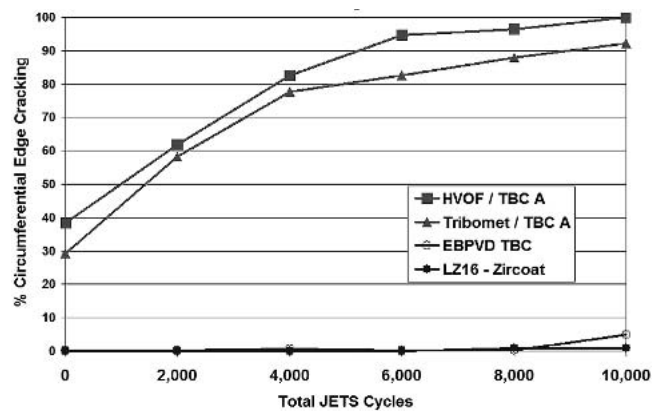


Fig. 20 JETS results for segmented TBC (1.1 mm thickness) and EBPVD TBC (0.5 mm thickness); low-density TBC is included for reference. The JETS heat flux corresponds to a front-face temperature of 1400 °C on the segmented TBC.

by complete spallation of the upper TBC layer. The samples with the optimized matching density, however, survived 10 cycles without any indication of interface separation (Fig. 18a and b).

In the JETS Test, both the samples with the density mismatch and the density match showed 100% edge spallation at the TBC interface after 2000 cycles. Obviously the JETS test stresses the ceramic system so as to reveal potential failure modes even in the optimized dual-layer system, which the FCT test does not do (Fig. 19).

2.4.3 Example 3: Dense Vertically Macrocracked APS and EBPVD TBC. In the case of a highly strain-resistant TBC, such as a dense vertically macrocracked APS TBC, or an EBPVD TBC, the JETS test does not lead to failure within a reasonable measuring time. In Fig. 20, the JETS results for these two coatings are shown for over 10 000 JETS test cycles. The APS TBC with a dense vertically macrocracked microstructure of approximately two cracks per linear mm, and the EBPVD TBC with a columnar growth microstructure, only develop minor edge cracking after 8000 JETS cycles. In the FCT test, however, the life of these coatings is typically several hundred hours at 1135 °C and is predominantly dependent on the bondcoat degradation.

3. Fluidized Bed Test

This test has been developed by one of the major IGT manufacturers as a quality control tool. See Ref 5 for details. Typical test conditions are 1000 cycles at 1000 °C. Each cycle consists of 5 min in the fluidized bed and 5 min of forced air cooling. To evaluate the discrimination capability of this test, TBC/bondcoat systems with different bondcoat processes were chosen. In this program, the influence of the base alloy on the FCT life has been investigated.

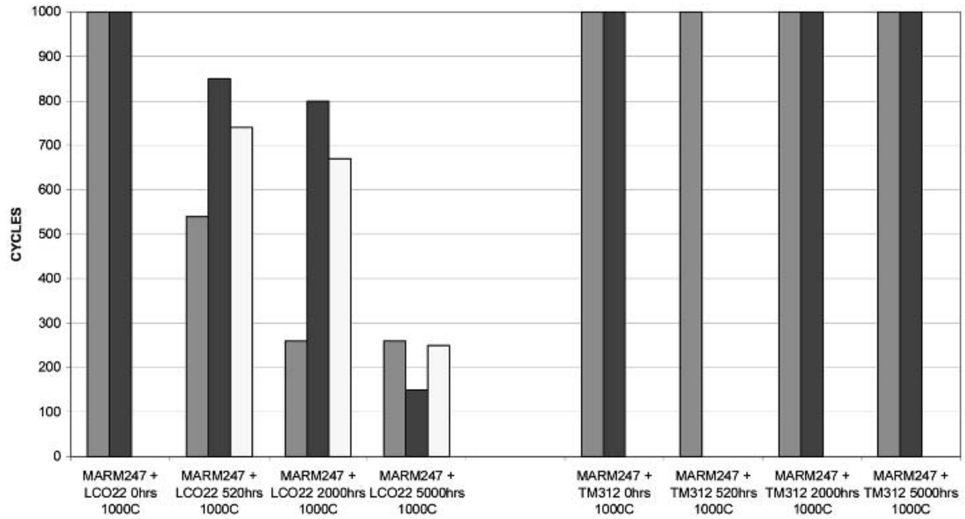


Fig. 21 FCT life of LCO22 and Tribomet TM312 in the fluidized bed test after 0-5000 h isothermal preoxidation at 1000 °C

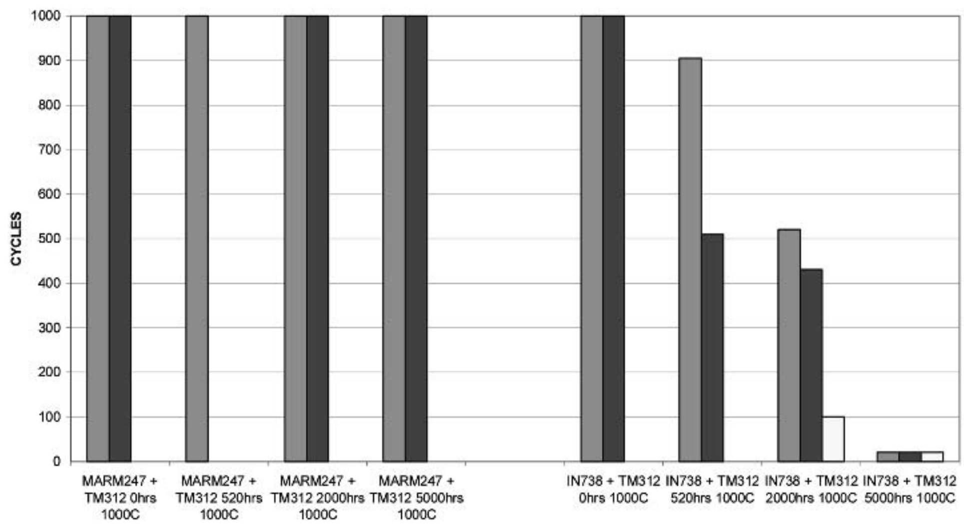


Fig. 22 FCT life of TM312 on MARM247 and IN738 after 0-5000 h isothermal preoxidation at 1000 °C

For the fluidized bed test, a low-density coating on plasma and Tribomet bondcoats was evaluated with and without isothermal preoxidation to demonstrate the capability of distinguishing between the bondcoat and substrate effect due to long term interdiffusion.

The TBC system used was a low-density TBC (85-90% density) on a CoNiCrAlY bondcoat applied by shrouded plasma (LCO22) and by Tribomet (TM312). The substrate alloy was either MARM247 or IN738. The test pieces were nominally 35 mm in diameter × 8 mm in thickness with an 8.5 mm diameter hole drilled for positioning onto the test jig.

Each of the coating combinations was subjected to the standard 1000 cycle thermal fatigue test without signs of failure. Therefore, to achieve some ranking between coatings, it was necessary to induce failure by promoting the formation of a thermally grown oxide at the interface between the bondcoat and TBC. A reduction in fatigue life was achieved by isothermally oxidizing samples at 1000 or 1065 °C for times of up to 5000 h. The resulting failure of the coating consequently occurred at this interface with a typical "black failure" appearance. In such cases the thickness and composition of the oxide will be critical in determining the performance of the coating combination.

In Fig. 21, the performance of LCO22 is compared with TM312 when applied to a substrate of MARM247. In Fig. 22 the performance of TM312 is compared for MARM247 and IN738 substrate.

The results from this test program suggest that the performance of a coating can also be significantly influenced by the substrate. For example, the TM312 bondcoat worked well in conjunction with MARM247, but the fatigue life was reduced when it was used with IN738. Microstructural studies suggest that this impairment in performance is related to the diffusion of harmful elements, such as titanium, from the substrate which effectively poison the TGO.

Table 1 Test Method Recommendation Matrix (+ Well Suited; − Not Suitable)

Test Method	Ceramic Layer	Ceramic Multilayer Interfaces/ Graded Ceramic	Bondcoat Interface (TGO)	Substrate-Induced Effects
Furnace cycle	(+)	(+)	++	+
JETS	++	++	+	−
Fluidized bed w/preoxidation	−	−	++	++

4. Summary

An attempt for a recommendation of the suitability of the three different tests investigated is given in Table 1, which is a matrix describing the suitability of the individual test methods in distinguishing between selected TBC system features.

The furnace cycle test reflects the actual engine condition well because it not only cyclically stresses the TBC but also degrades the bondcoat through severe oxidation. It also reveals performance and design limits such as bondcoat depletion. However, it does not really address TBC degradation caused by defects in the ceramic layer, such as crack propagation or sintering, which typically initiate above 1300 °C. It is suitable for performance ranking of the complete TBC system emphasizing the bondcoat/TBC interface.

The JETS test is well suited to provide performance ranking data on the ceramic itself. It does not discriminate very well between bondcoat induced failures because it does not degrade the bondcoat. Due to the high thermal gradients within the ceramic layer it can reveal weak points such as ceramic interfaces.

The fluidized bed, as used as a quality control tool with the standard 1000 cycle TCF test, does not discriminate differences well enough to provide quantitative data for a performance ranking. When combined with an isothermal exposure up to 5000 h at 1000 °C, however, this test is capable of doing so. The 5000 h isothermal preexposure is a time-consuming and expensive test. Attempts at reducing the time to failure using a higher preoxidation temperature of 1100 °C are in progress, but so far, the ranking has been the same as samples preoxidized at 1000 °C.

Acknowledgments

The authors would like to thank Anthony Stavros and Nick Walters for performing the FCT testing, Sergio Comarella for the metallographic preparation, and Carl Rice for providing the scanning electron microscope (SEM) images.

References

1. A. Maricocchi, A. Barz, and D. Wortman: "PVC TBC. Experience on GE Aircraft Engines," in *Proceedings Thermal Barrier Coating Workshop,* NASA Lewis Research Center, Cleveland, Ohio, March 27-29, 1995, p. 85.
2. T.A. Taylor: "Thermal Barrier Coating for Substrates and Process for Producing It," U.S. Patent No. 5,073,433 and 4,743,013, December 17, 1991.
3. T.A. Taylor and R.E. Taylor: "Testing of Stability and Thermal Properties of Thermal Barrier Coatings," *ASM Handbook Volume 5, Surface Engineering,* ASM International, Materials Park, OH, 1994, pp. 654-59.
4. J. Foster: "Protective Coating," U.S. Patent Nos. 5,558,758, 5,824,205, and 5,839,829, 20 October 1998.
5. A. Kahn, T. Duda, A. Taylor, P. Moore, P. Rutter, and J. Foster: "Oxidation Behavior of Electroplated MCrAlY Coatings," Proc. Thermal Spray Conference 2003, ASM International, Materials Park, OH, 2003, pp. 741-44.