National Aerospace Laboratory NLR



NLR-TP-2002-048

Thermal testing of low porosity microcracked thermal barrier coatings

M.F.J. Koolloos and M.J.W. Schouten*

* Eindhoven University of Technology

The investigation described in this report has been carried out at Eindhoven University of Technology, Department of Mechanical Engineering, Eindhoven, The Netherlands, as part of the first author's PhD-research.

This report is based on a paper submitted for publication in Surface and Coatings Technology.

This report may be cited on condition that full credit is given to NLR and the authors.						
Customer:	National Aerospace Laboratory NLR					
Working Plan number: S.1.B.4						
Owner:	National Aerospace Laboratory NLR					
Department:	Structures and Materials					
Distribution:	Unlimited					
Classification title: Unclassified						
	January 2002					



Summary

The mechanisms of damage and ultimate failure of TBCs are still not entirely understood. Many coating characteristics and thermal loading effects may affect the top coat durability. The number of cycles to failure and predominant failure mechanism depend on the TBC system, the thermal testing method and the severity of the test method. During the present investigation the effects of top coat thickness, bond coat preoxidation, thermal cycle lengths and test temperatures are investigated using three thermal test methods: furnace testing and burner rig thermal shock and thermal cycling. The results were described in terms of the number of thermal cycles to failure and a phenomenological description of failure. It appeared that failure of a TBC under realistic testing conditions must be due to an interaction between thermal stresses and bond coat oxidation. Good thermal shock resistance can be obtained from a high microcrack density in the top coat, resulting in better coating flexibility. For thick TBCs this flexibility is augmented by vertical cracking or segmentation cracking of the top coat. Thick TBCs failed by delamination of the top coat, starting at a free edge. This means that specimen (and also component) geometry play an important part in the susceptibility to failure.

Keywords

[C] Plasma spraying, [X] Thermal Barrier Coating, [B] Burner rig test

-4-NLR-TP-2002-048



Contents

1	Intro	5						
2	Coati	5						
3	Theri	6						
	3.1	Test conditions	6					
	3.2	Temperature profiles in burner rig tests	7					
4	Test 1	results	7					
	4.1	Furnace tests	7					
	4.2	Burner rig tests	8					
5	Failu	9						
	5.1	Furnace tests	9					
	5.2	Burner Rig Thermal Shock Tests	9					
	5.3	Burner Rig Thermal Cycling Tests	10					
	5.4	Microstructural Changes in the Top Coats	10					
6	Discu	ssion	11					
	6.1	Furnace Tests	11					
	6.2	Burner Rig Thermal Shock Tests	12					
	6.3	Burner Rig Thermal Cycling Tests	12					
	6.4	Comparison of the Test Methods	13					
	6.5	The Significance of Top Coat Microcracks	13					
7	Conc	lusions and Recommendation	14					
8	Ackn	owledgements	14					
9	9 References							
5 7	Tables							
9 F	Figures							

(23 pages in total)



1 Introduction

Thermal Barrier Coatings (TBCs) are applied to the hot components of gas turbines to allow higher turbine entry temperatures (which increase the thermal efficiency of the gas turbine) or to achieve lower base metal temperatures (Ref. 1). A TBC consists of a heat-resistant ceramic top coat and a metallic bond coat. The bond coat's main purpose is to attach the top coat to the substrate, but the bond coat also prevents or delays oxidation of the substrate. Unfortunately, a major life-limiting weakness of TBCs is oxidation of the bond coat itself; the other major problem is their susceptibility to damage by thermally-induced stresses (Ref. 2). The mechanisms of damage and ultimate failure of TBCs are still not entirely understood, because of the many possible effects that occur and may interact (Refs. 2-8):

- 1) Coating chracteristics like top coat thickness and density, top coat / bond coat interface roughness and as-deposited residual stresses.
- 2) Thermal loading effects like bond coat oxidation; thermal stresses owing to thermal expansion mismatch; thermal stresses owing to rapid temperature changes and steep temperature gradients; top coat phase transformations, creep and sintering; and bond coat creep and aluminium-depletion.

In practise there will be a predominant failure mechanism. This will depend on the TBC system, the thermal testing method and the severity of the test method.

The purpose of the present investigation is twofold: firstly to better describe and understand the failure mechanisms of plasma sprayed TBCs in general; and secondly, more specifically, to determine the thermal shock / thermal cycle resistance of a new, low porosity microcracked TBC developed at Eindhoven University of Technology (Refs. 9, 10). The experiments are designed to support and validate modelling activities (Refs. 10, 24). This approach requires an extensive test programme, in which three different kinds of thermal loads are applied (furnace testing, burner rig thermal shock and burner rig thermal cycling) to TBCs of various thicknesses and with or without bond coat preoxidation. The emphasis is on determining the TBC lives and providing a phenomenological description of the failure modes.

2 Coating preparation

The substrate to be coated was the nickel-base superalloy Hastelloy X, in the form of $125 \times 30 \times 3.25$ mm strips. The coating material and spraying parameters are given in table 1. The bond coat preoxidising conditions and spraying parameters were selected from optimisation studies (Refs. 10, 11). The top coat spraying parameters combined with water-cooling the



substrate gave a high deposition efficiency (about 60 %), low porosity (< 5 %) and a dense network of microcracks, and low tensile stresses (< 50 MPa) in the as-deposited state (Refs. 9, 10, 12).

3 Thermal test programme

Table 2 surveys the thermal test programme, which encompassed furnace tests at the National Aerospace Laboratory (NLR) and the Interturbine Coating Centre (ITC)¹ in Lomm, the Netherlands; and burner rig tests at Eindhoven University of Technology, the Netherlands. Details of the furnaces and burner rig are given in references 10 and 13.

3.1 Test conditions

During the ITC furnace tests 30×30 mm samples cut from the coated strips were heated to 1137 °C. The 1 hr cycle included 4 min for heating up and 6 min cooling down to 200 °C. Every 20 cycles the samples were cooled to room temperature and kept there for 4 hr while being thoroughly inspected for signs of failure. The failure criterion was 20 % delamination of the top coat.

During the NLR furnace tests 20×10 mm samples were heated to 1100 °C with cycle lengths of 1, 2, 8 and 24 hr, and 1000 °C with a cycle length of 1 hr. The heating cycles included 4 min for heating up and 3 min for cooling down to room temperature. Samples undergoing 1, 2 and 8 hr cycles were thoroughly inspected twice daily, while the 24 hr cycled samples were inspected once a day. Each inspection took about 10 min. The failure criterion was 25 % delamination of the top coat.

The burner rig tests were done on complete 125×30 mm strips. As shown in table 2, there were two types of tests, thermal shock and thermal cycling:

- (1) During the thermal shock tests the strips were locally flame-heated in 28 s to a maximum surface temperature, T_{surf} , which was varied from 1300 °C to 1475 °C. After this heating the entire strips were cooled for 28 s, reaching a substrate temperature, T_{sub} , of 400 °C.
- (2) During thermal cycling tests the strips were locally flame-heated for 59 min with 1 min cooling down to 200 °C. The steady state top coat surface temperature was deliberately increased with increasing coating thickness, see also table 2. The reason is as follows. In actual gas turbines the heat input is constant under constant operating conditions. For

¹ Now: Sulzer Metco Coatings.



TBCs this means that owing to their low thermal conductivity the surface temperatures of thicker top coats will be higher.

The burner rig tests were stopped if the top coat delaminated in the flame-heated area, if there was severe bond coat and/or substrate degradation, if there was severe strip bending, or if no damage had occurred after a large number of cycles.

After all tests the specimens were cross-sectioned and examined microscopically. To prevent any sectioning and polishing damage the coatings were impregnated with a resin adhesive, and in some cases a small coupon of stainless steel was glued onto the top coat to assist its retention.

3.2 Temperature profiles in burner rig tests

Figure 1 shows a measure of the through thickness temperature differences in the strips tested with the burner rig. As expected, the temperature difference between the top coat surface and the substrate rear side, $\Delta T = T_{surf} - T_{sub}$, increased with increasing top coat thickness and was larger during the thermal shock tests. Also, the data for the 1 mm thick top coats show that the thermal barrier effect was greater for greater heat input, i.e. ΔT increased with increasing T_{surf} for both types of test.

Figure 2 gives an example of radial temperature profiles measured with thermocouples on the substrate rear side during a burner rig test. The temperature decrease towards the top edge of the strip (c-f2-e2) is larger than that across the strip (c-f1-e1), namely 200 °C compared to 100 °C. This is explicable (generically, for all tests) because the distance between e2 and the top edge is larger than that between e1 and the side edge. This means there is more material between the flame area and the top edge to act as a heat sink.

4 Test results

4.1 Furnace tests

Figure 3 presents the 1137 °C and 1100 °C furnace test results. The ITC tests, figure 3a, showed a dramatic effect of top coat thickness. The coatings with 0.3 mm top coats failed after an average of 310 cycles (without bond coat preoxidation) and 440 cycles (with preoxidised bond coat), while the coatings with 0.6 mm and 1.0 mm top coats all failed at 20-40 cycles. The results for coatings with 0.3 mm top coats show clearly that bond coat preoxidation is beneficial. However, there was essentially no difference between the two samples with bond coats preoxidised for 5 hr (450 cycles) and 10 hr (430 cycles).

-8-NLR-TP-2002-048



The NLR furnace tests at 1100 °C, figure 3b, show that longer heating cycles reduce the number of cycles to failure but increase the time to failure, defined as greater than 25 % delamination of the top coat. Figure 3b also plots the cycles and times at which corner delamination first occurred. The trends are similar to those for greater than 25 % delamination, though at lesser number of cycles and times.

The NLR tests at 1000 °C showed top coat delamination at all four corners, beginning at about 1200 cycles and hours, which is much longer than the equivalent tests at 1100 °C (less than 100 cycles, see figure 3b). After 2000 cycles and hours the 1000 °C test sample top coats still had not reached 25 % delamination.

4.2 Burner rig tests

Figure 4 presents the burner rig test results. The thermal shock tests, figure 4a, showed a strong effect of maximum temperature on the number of cycles to failure for the 0.3 mm thick top coats. There was also a change in failure mode, discussed in section 5.2. Somewhat unexpectedly, these strips did not show a significant effect of bond coat preoxidation. The thicker 0.6 mm and 1.0 mm top coat strips without a preoxidised bond coat did not fail – or would not have failed – within 5000 cycles (testing of the 1.0 mm TBC at 1475 °C was stopped already after 3000 cycles because there was little external evidence of damage (Ref. 10)). However, the two strips with 0.6 mm top coats *and* preoxidised bond coats failed at about 2400 cycles.

Figure 4b shows the thermal cycling results. The thin top coats failed after about 100 cycles with a $T_{surf} = 1350$ °C, but the strips with 0.6 mm and 1.0 mm top coats survived 250 cycles with a $T_{surf} = 1400$ °C. However, the thickest top coats, 1.0 mm and 2.0 mm, failed after only 60 and 3 cycles, respectively, with a $T_{surf} = 1500$ °C. As in the case of thermal shock testing, there was a change of failure mode, but in this case it depended on the top coat thickness rather than the maximum temperature, see section 5.3.

In contrast to thermal shock testing, bond coat preoxidation was beneficial to the lives of the thermally cycled 0.3 mm top coats, with an increase in life of about 25 %. The difference in lives for 5 hr and 10 hr preoxidation was negligible: delamination failure occurred after 116 and 115 cycles respectively.



5 Failure modes

Figure 5 and table 3 classify and explain the observed failure modes of the test samples and strips. Before discussing the failure modes in detail, it is necessary two provide two further definitions: (a) the *remaining* coating is non-delaminated, e.g. the grey region in failure mode NO; and (b) the *residual* top coat is that still adhering to the bond coat after delamination, e.g. the grey regions in failure mode CD.

5.1 Furnace tests

The 0.3 mm top coats degraded similarly: first, delamination occurred at the sample corners and free edges, after which the top coat was gradually "nibbled off" (NO, see Fig. 5). The *remaining* coating showed interface–associated and vertical cracking (IC and VC). In some cases the top coat delaminated completely (CD) without first reaching the failure criterion of 25 % delamination. Figure 6 gives example cross-sections for the 1 hr cycle samples: (a) near the edge of the *remaining* top coat after 90 cycles; (b) after failure (300 cycles) by complete delamination. Figure 6a shows the interface-associated cracks (IC) in the *remaining* top coat, and figure 6b shows that these would have linked up to form a crack path just above the interface roughness peaks. Both micrographs also show severe bond coat degradation (BD). As stated previously, and listed in table 3, similar degradation and failure modes were observed for the other 0.3 mm top coats. This was irrespective of furnace cycle length and bond coat preoxidation or non-preoxidation.

Failure of the thicker (0.6 mm and 1.0 mm top coat) TBCs was essentially different. The top coat delaminated completely (CD) after a low number of cycles and without "nibbling off". However, the delamination was preceded by through-thickness segmentation cracking (SC) of the top coat. This feature is illustrated schematically in figure 5, and was found to correspond near the interface to "islands" of *residual* top coat separated by "canals" of bond coat.

5.2 Burner Rig Thermal Shock Tests

The failure modes of the 0.3 mm top coats depended on test temperature. For $T_{surf} = 1300$ °C the top coat delaminated partially (FD) or completely (CD) in the flame area after a large number of cycles. There was also considerable bond coat degradation (BD) and the strips were slightly bent (SB). Cross-sections made before complete delamination showed the crack path was similar to that observed for the furnace tested 0.3 mm top coats, figure 6, i.e. interface-associated cracks (IC) running in the top coat between bond coat roughness peaks. For $T_{surf} = 1350$ °C the strips underwent severe bending (SB) that resulted in wide vertical cracks (VC) within relatively few cycles (about 1500 cycles compared to more than 3500 cycles with



 $T_{surf} = 1300$ °C, see Fig. 4a). Outside the flame area the 0.3 mm top coats all showed vertical cracks (VC). An example is given in figure 7.

The 0.6 mm and 1.0 mm top coats with non-preoxidised bond coats did not fail during thermal shock testing, see figure 4a. However, they did show some vertical cracks (VC) both inside and outside the flame area, and also a little interface-associated cracking (IC) in the flame area. The bond coat degradation and strip bending were negligible. On the other hand, preoxidised bond coats led to 0.6 mm top coats completely delaminating from the strip side edges to the flame area (ED/CD, see Fig. 5). Even so, the bond coat degradation was much less than that observed for the 0.3 mm top coated strips.

5.3 Burner Rig Thermal Cycling Tests

The 0.3 mm top coated strips failed by complete delamation in the flame area (FD/CD). The crack path was similar to that for the failed 0.3 mm top coated samples and strips subjected to furnace and thermal shock testing, i.e. interface-associated cracks (IC) running in the top coat between bond coat roughness peaks. The bond coat degradation (BD) was intermediate to that from furnace testing (severe) and thermal shock testing.

The thicker 0.6 mm and 1.0 mm top coats tested with a $T_{surf} = 1400$ °C showed segmentation cracking (SC) in the flame area, but did not delaminate despite interface-associated cracks (IC). The 1.0 mm and 2.0 mm top coats tested with a $T_{surf} = 1500$ °C failed after relatively low numbers of cycles by complete delamination from the strip side edges to the flame area (ED/CD, see Fig. 5). There was also evidence of segmentation cracking (SC) in the flame area.

5.4 Microstructural Changes in the Top Coats

As reported previously (Ref. 13), XRD phase analysis showed that the monoclinic phase did not appear in top coats thermally shock tested with maximum T_{surf} values of 1300 °C and 1400 °C. Thus it is reasonable to conclude that failure during thermal shock testing was not due to the T \rightarrow M transformation (Refs. 10, 13).

On the other hand, optical microscopy on the sample and strip cross-sections showed that the number of microcracks and pores had decreased in the heated areas of the top coats owing to sintering. This is a well-known densification effect that can be followed indirectly by measurement of hardness and erosion resistance (Refs. 10, 14, 15).

Figures 8, 9 and table 4 present microhardness measurements on as-sprayed, isothermally heated and thermal shock tested TBCs (Ref. 10). The isothermal heating results in figure 8 agree with previous work, which indicates that below about 1350 °C some localised sintering

-11-NLR-TP-2002-048



of small defects occurs (Refs. 14, 16), and above 1350 °C bulk sintering takes place (Ref. 17). The average microhardnesses for thermal shock specimens, table 4, show dependence on T_{surf} , the number of cycles, and the location. Thus, in the flame area and after large numbers of cycles the hardnesses increased to the same level as at similar temperatures during isothermal testing, cf. table 4 and figure 8. This indicates that the amount of sintering during thermal shock testing depends on time rather than cycles. Figure 9 provides more insight by illustrating that though there are average trends, the local hardness variations in the top coat (and the bond coat) can be large.

6 Discussion

Most thermal tests are not standardised, making it difficult to compare results from different sources. For example, Haubold *et al.* (Ref. 18) compared the thermal shock resistance of six different TBCs tested with three different burner rigs, and obtained different rankings of the coatings. Also, the coatings in the present investigation had specific designed-in characteristics: low porosity, a dense network of microcracks, and low tensile stresses in the as-sprayed state (Refs. 9, 10). Thus the following discussion and ensuring conclusions are limited to the present coatings, though there appear to be generic trends.

6.1 Furnace Tests

Increased cycle length reduced the cycles to failure but increased the time to failure. The same general trend was found by McDonald and Hendricks (Ref. 19). This trend demonstrates the interaction of thermal fatigue and oxidation. For shorter cycles the amount of oxidation per cycle is small and the number of cycles to failure is larger. However, the time-based life is relatively short. This means that thermal fatigue has a larger contribution to failure than oxidation when the cycle length is short.

The thin top coat failure modes during furnace testing, "nibbling-off"(NO) and interfaceassociated cracking (IC), indicate that high shear or normal stresses developed near the free edges of the samples and just above the top coat / bond coat interface roughness peaks, and that these stresses had a primary contribution to failure (Refs. 20, 22). Severe bond coat degradation (BD) owing to long exposure could well enhance the observed failure modes.

The thicker 0.6 mm and 1.0 mm topcoats performed poorly. Failure occurred by segmentation cracking (SC) followed by complete delamination (CD) after only a few cycles. The segmentation cracking indicates that high in-plane tensile stresses most probably developed to



cause this failure mode. Subsequent delamination occurred owing to the additional contribution of the high shear or normal stresses mentioned above.

6.2 Burner Rig Thermal Shock Tests

The effects of test temperature and top coat thickness in thermal shock tests can be classified as follows:

- (1) For a maximum $T_{surf} = 1300$ °C the thicker coating (1.0 mm top coat) survived 5000 cycles, while the 0.3 mm top coats delaminated and failed, on average, at 3700 cycles. The 0.3 mm top coated strips also showed severe bond coat degradation, indicating that this played an important role in failure.
- (2) For a maximum $T_{surf} = 1350$ °C the test on 0.3 mm top coated strips had to be stopped because of severe strip bending, which is not regarded as coating failure per se.
- (3) For a maximum $T_{surf} = 1400$ °C the 0.6 mm top coated strip life depended greatly on whether the bond coat was preoxidised or not. Preoxidation reduced the life from greater than 5000 cycles to about 2400 cycles. However, this strong effect cannot be attributed to bond coat degradation.
- (4) For maximum $T_{surf} = 1475$ °C the 1.0 mm top coated strip life would have been much longer than 3000 cycles. This test was stopped because there was little external evidence of damage.

In the light of these results it would appear that the main effect of an increase in top coat thickness is to lengthen the thermal shock lives and temperature capabilities (T_{surf}) by decreasing the top coat / bond coat interface temperature and therefore reducing oxidation-induced degradation of the bond coat. This is evidently distinct from the slight $(T_{surf} = 1300, 1350 \text{ °C})$ and considerable $(T_{surf} = 1400 \text{ °C})$ detrimental effect of bond coat preoxidation.

The contribution of preoxidation involves competing factors. On the one hand, preoxidation prevents oxide growth stresses occurring at the top coat / bond coat interface during the early stages of thermal testing (Refs. 21, 22); but on the other hand, preoxidation means that (high) interface stresses are present from the beginning of thermal testing (Refs. 10, 24). Preoxidation also reduced the top coat / bond coat adhesion strength (Ref. 11), which could facilitate top coat delamination especially for thicker top coats.

6.3 Burner Rig Thermal Cycling Tests

The thermal cycling test results showed that only the 0.3 mm top coated strips underwent significant bond coat degradation, yet all top coats failed by delamination if the test temperature was high enough. Thus failure appears to be caused by oxidation-induced and thermal stresses. The failure mode of the thicker top coats (complete delamination from the

-13-NLR-TP-2002-048



strip side edges to the flame area) indicate that high shear or normal stresses developed near the free edges (Ref. 20). Based on the low number of cycles to failure, these stresses must be the predominant cause of failure.

6.4 Comparison of the Test Methods

Table 5 compares the test methods in terms of the failure times for 0.3 mm top coated samples and strips, whereby the top coat / bond coat interface temperatures were comparable. This comparison shows that failure during thermal shock tests is mainly cycle-dependent and failure during furnace tests is mainly time-dependent. In turn, this reflects different primary failure mechanisms, namely thermal fatigue stresses during thermal shock and oxidation-induced failure during furnace tests.

It is evident from this comparison that furnace tests are unlikely to be reliable indicators of coating performance in gas turbines. On the other hand, thermal shock tests may also be unrealistic because they can favour the survival of thick TBCs in comparative testing. Thus on balance it would seem that thermal cycling tests have the broadest significance, since the coating failure behaviour is intermediate to the types of failure observed from furnace and thermal shock tests.

6.5 The Significance of Top Coat Microcracks

The TBCs in the present investigation had good thermal shock resistance, especially the thicker top coats. This may be attributed to the dense network of microcracks already present in the as-sprayed top coats (Ref. 10). However, the results of Wigren and co-workers (e.g. Ref. 23) disagree: they found poor thermal shock resistance for microcracked top coats and good thermal shock resistance for segmentation cracked top coats. From these disparities one may conclude that good thermal shock resistance is not unambiguously correlatable to the microcrack density, but is obtained primarily via a low value of Young's modulus (high flexibility), which can be achieved by:

- 1. Omitting substrate cooling during top coat deposition, resulting in segmentation cracking (Ref. 23).
- 2. Forced substrate cooling during deposition, as in the present investigation, resulting in a high density of microcracks.

Moreover, segmentation cracking can occur in microcracked top coats after only a few thermal cycles, as observed for the thick top coats in the present tests, resulting in excellent thermal shock resistance.



- (1) Failure of TBCs in service environments is most probably due to a combination of thermal stresses and bond coat oxidation. Translating this to laboratory tests means that burner rig thermal cycling is likely to be the most appropriate testing method for TBC evaluation.
- (2) Good thermal shock resistance can be obtained from a high density of microcracks in the as-sprayed top coat, resulting in better coating flexibility. For thick TBCs this flexibility is augmented by vertical cracking or segmentation cracking of the top coat.
- (3) Thick TBCs fail by delamination of the top coat, starting at a free edge. This means that specimen (and also component) geometry play an important part in the susceptibility to failure.
- (4) Empirical testing, no matter how realistic, is insufficient for understanding the observed failure mechanisms, the effects of top coat thickness, and the effect of bond coat preoxidation. The microstructural changes that occur during thermal exposure, including sintering and bond coat degradation, need to be investigated in detail. Also, and most importantly, to better understand the failure mechanisms it is necessary to obtain quantitative information about the thermal stresses that develop. In the present work indications were found for high shear or normal stresses developing near the free edges of the samples and just above the top coat / bond coat interface roughness peaks. But one has also to consider oxide growth stresses during bond coat degradation and interface stresses owing to bond coat preoxidation. A paper describing the modelling of thermal stresses is currently in preparation (Ref. 24).

8 Acknowledgements

The authors would like to thank Giel Marijnissen at Sulzer Metco Coatings, Lomm, the Netherlands for the ITC furnace tests and R.J.H. Wanhill at the National Aerospace Laboratory NLR, Amsterdam, the Netherlands, for assisting with the manuscript. -15-NLR-TP-2002-048



9 References

- Stringer, J.; J. Proc. Materials Solutions '98, Eds. P.J. Maziasz et al., ASM International, Materials Park, OH, USA, 3-12, 1999.
- 2. Miller, R.A.; Lowell, C.E.; Thin Solid Films, 95, 265-273, 1982.
- 3. Watson, J.W.; Levine, S.R.; Thin Solid Films, 119, 185-193, 1984.
- 4. Wu, B.C.; Chang, E.; Chang, S.F.; Tu, D.; J. Am. Ceram. Soc., 72, 212-218, 1989.
- DeMasi-Marcin, J.T.; Sheffler, K.D.; Bose, S.; *J. Eng. Gas Turbines and Power*, 112, 521-526, 1990.
- 6. Nissley, D.M.; J. Thermal Spray Technol., 6, 91-98, 1997.
- 7. Haynes, J.A.; Ferber, M.K.; Porter.D., W.D.; J. Thermal Spray Technol., 9, 38-48, 2000.
- 8. Rabiei, A.; Evans, A.G.; Acta Mater., 48, 3963-3976, 2000.
- Verbeek, A.T.J.; *Plasma Sprayed Thermal Barrier Coatings: Production, Characterization and Testing*, Ph.D. Thesis, Eindhoven University of Technology, Eindhoven, The Netherlands, 1992.
- Koolloos, M.F.J.; Behaviour of Low Porosity Microcracked Thermal Barrier Coatings under Thermal Loading, Ph.D. Thesis, Eindhoven University of Technology, Eindhoven, The Netherlands, 2001.
- 11. Koolloos, M.F.J.; Houben, J.M.; J. Thermal Spray Technol., 9, 49-58, 2000.
- 12. Koolloos, M.F.J.; Houben, J.M.; Materials Science Forum, 347-349, 465-470, 2000.
- 13. Koolloos, M.F.J.; Liempd, G.G. van; Houben, J.M.; Surf. Eng., 14, 144-148, 1999.
- 14. Eaton, H.E.; Novak, R.C.; Surf. Coat. Technol., 30, 41-50, 1987.
- 15. Janos, B.Z.; Lugscheider, E.; Remer, P.; Surf. Coat. Technol., 113, 278-285, 1999.
- 16. Wesling, K.F.; Socie, D.F.; Beardsley, B.; J. Am. Ceram. Soc., 77, 1863-1868, 1994.
- 17. Sato, T.; Shimada, M.; J. Am. Ceram. Soc., 67, C212-C213, 1984.
- Haubold, T.; Wigren, J.; Gualco, C.; *Proc. 15th Int. Thermal Spraying Conference*, Ed. C. Coddet, ASM International, Materials Park, OH, USA, 1617-1622, 1998.
- 19. McDonald, G.; Hendricks, R.C.; Thin Solid Films, 73, 491-496, 1980.
- 20. Hu, S.Y.; Li, Y.L.; Munz, D.; Yang, Y.Y.; Surf. Coat. Technol., 99, 125-131, 1998.
- 21. Evans, A.G.; Crumley, G.B.; Demaray, R.E.; Oxid. Met., 20, 193-216, 1983.
- 22. Freborg, A.M.; B.L. Ferguson, B.L.; Brindley, W.J.; Petrus, G.J.; *Mat. Science Eng.*, A245, 182-190, 1998.
- 23. Bengtsson, P.; Ericsson, T.; Wigren, J.; J. Thermal Spray Technol., 7, 340-348, 1998.
- 24. Koolloos, M.F.J.; *Finite Element Modelling of Thermal Stresses in Plasma Sprayed Thermal Barrier Coatings*, in preparation.



Thickness(mm)0.10.5, 0.6, 1.0, 2.0Arc current(A)500500Voltage(V)6056	Spraying condit	ions	NiCrAlY bond coat, Amperit 413.6, as-sprayed and preoxidised (a)	ZrO ₂ -8wt.%Y ₂ O ₃ top coat, Amperit 825.1
Arc current (A) 500 500 Voltage (V) 60 56	Thickness	(mm)	0.1	0.5, 0.6, 1.0, 2.0
Voltage (V) 60 56	Arc current	(A)	500	500
	Voltage	(V)	60	56
Powder flow rate(g/min)4545	Powder flow rate	(g/min)	45	45
Argon flow rate(1/min)5545	Argon flow rate	(l/min)	55	45
% H ₂ 15 20	% H ₂		15	20
Plasma enthalpy (b)(MJ/kg)11.28.0	Plasma enthalpy (b)	(MJ/kg)	11.2	8.0
Spray distance (mm) 100 100	Spray distance	(mm)	100	100
Torch traverse speed(mm/s)315	Torch traverse speed	(mm/s)	3	15
Specimen holder rotation (c) (rpm)100225	Specimen holder rotation	n (c) (rpm)	100	225

Tabla 1	Conting	motoriala	and	oproving	conditions
Table I	Coating	materials	and	spraying	contantions

(a) Pre-oxidised bond coats were obtained by furnace heating at 1050 °C for 5 and 10 hours. The oxide layers were $1.5 \pm 0.25 \ \mu m$ and $2.0 \pm 0.5 \ \mu m$ thick, respectively: more details in references 10 and 11.

(b) The plasma enthalpy is defined as the electric power input $(A \times V)$ minus the cooling water losses and then divided by the gas mass flow.

(c) A hollow cylindrical specimen holder was used for rotationally spraying eight circumferentially mounted strips at one time: more details in reference 10.

Table 2Thermal test programme. The burning test temperatures are for the top coat surface,
and the cycle lengths include heating and cooling

Top coat	thickness		(mm)	0.3 0.6 1.0 2.0					2.0	
Bond coat pre-oxidation time (hr)					5	10	0	10	0	0
Test	type	T _{test} (°C)	Cycle length	Number of specimens						
	ITC	1137	1 hr	6	1	1	3	2	1	
Furnace	NLR	1100	1,2,8,24 hr	3	•••					
	NLR	1000	1 hr	3						
Burner Rig		1300	1 min	2	•••	1			1	
Thermal S	Shock	1350	1 min	2	1	1				
		1400	1 min				1	2		
		1475	1 min		•••				1	
Burner Rig		1350	1 hr	1	1	1				
Thermal Cycling		1400	1 hr		•••		1		1	
		1500	1 hr						1	1



	Top cost	т	Top coat						Bond	Strip	
Test type	thickness (mm)	(°C)	CD	IC	VC	SC	FD	ED	NO	coat	specimen
						~ ~				BD	SB
Furnaça	0.3*	1137/1100	•	•	•				•	•	
Turnace	0.6/1.0*	1137/1100	•			٠					
	0.3*	1300	•	•	•		•			•	•
		1350			٠						•
Burner Rig	0.6	1400		•	•						
Shool	0.6 pre-oxi	1400	٠					•			
SHOCK	1.0	1300		•	•						
		1475		٠	٠						
Burner Rig	0.3*	1350	•	٠	٠		٠			•	
Thermal	0.6/1.0	1400		•	•	•					
Cycling	1.0/2.0	1500	•			•		•			

Table 3Classification of failure mode. Figure 5 illustrates and explains the two-letter codes.Asterisks (*) signify both preoxidised and non-preoxidised bond coats

Table 4 Microhardness (HV 200) for thermal shock tested top coats (Ref. 10)

Top coat thickness	T _{surf}	Cualas	Location of hard	lness indentation
(mm)	(°C)	Cycles	Flame area	Edge of specimen
0.3	1300	4220	968 ± 127	808 ± 117
1.0	1300	5000	976 ± 138	686 ± 36
0.3	1400	750	918 ± 83	
0.6	1410	2690	935 ± 34	
0.6	1425	4870	1046 ± 114	

Table 5Times-at-temperature for 0.3 mm top coated TBCs in furnace tests and burner rig
thermal shock and thermal cycling tests

Test tures	T _{test}	Top coat/bond coat	Average cycles to	Heating	Total time
Test type	(°C)	interface temperature (°C)*	failure	period	(hr)
Thermal Shock 1300		1050	3800	28 s	30
Thermal Cycling	1350	1170	100	1 hr	100
{	1100	1100	220	1 hr	220
NLR Furnace	1100	1100	210	2 hr	420
	1100	1100	110	8 hr	880

* Calculated for the burner rig test using finite element analysis (Refs. 10, 24).



Fig. 1 Temperature difference ($\Delta T = T_{surf} - T_{sub}$) during the beginning of each burner rig test, plotted against topcoat thickness. The temperature values are the corresponding surface temperatures of the topcoats. For thermal shock tests ΔT is at 30 s from starting a cycle, while for thermal cycling ΔT represents steady-state conditions



Fig. 2 Example of radial temperature profiles at the substrate rear side during a burner rig test. This example is for a topcoat thickness of 0.3 mm, heating time 30 s and T_{surf} =1300°C The locations f_1 and f_2 and e_1 and e_2 , are equidistant from the centre of the topcoat surface flame area, c



D340-01N

Fig. 3 1137°C and 1100°C furnace test results



Fig. 4 Burner rig test results

R -

top coat Complete Delamination	CD	
top coat Interface-associated Cracking	IC	~~~~~
top coat Vertical Cracking	VC	
top coat through-thickness Segmentation Cracking (furnace test or burner rig)	SC	or
top coat Flame area Delamination	FD	
top coat Edge-to-flame-area Delamination	ED	
top coat Nibbling-Off	NO	
severe Bond coat Degradation (interface oxide layer, internal oxidation, substrate/ bond coat interface carbides and oxides)	BD	
Strip Bending (deformation rather than coating failure per se)	SB	

Fig. 5 Schematic illustrations and explanations of observed failure modes (see also Tab. 3). The grey-shaded regions represent the top coat



Fig. 6 Furnace tested 0.3 mm coat (a) after 90 cycles near edge of remaining topcoat, (b) after failure (300 cycles) by complete delamination



Fig. 7 Vertical cracks outside the flame area in a 0.3 mm topcoat after burner rig thermal shock testing with $T_{surf} = 1300^{\circ}C$



Fig. 8 Microhardness (HV 200) for as-sprayed and isothermally heated 0.3 mm topcoats



Fig. 9 Microhardness (HV 200) variations in a thermal shock strip undergoing topcoat Flame area Delamination (FD): topcoat thickness 0.3 mm, maximum T_{surf} = 1300°C, 4220 cycles, see table 4