2007

Time-Economic Lifetime Assessment for High Performance Thermal Barrier Coating Systems

Marion Bartsch

Linder Hoehe, amarion.bartsch@dlr.de

Bernd Baufeld

Katholieke Universiteit Leuven

S. Dalkilic

Anadolu University

Iulian Mircea

German Aerospace Center

K. Lambrinou

Katholieke Universiteit Leuven

See next page for additional authors

Follow this and additional works at: https://engagedscholarship.csuohio.edu/enme_facpub

Part of the [Engineering Science and Materials Commons](#), and the [Mechanical Engineering Commons](#)

[How does access to this work benefit you? Let us know!](#)

Original Citation

Bartsch, M., Baufeld, B., Dalkili, S., Mircea, I., Lambrinou, K., Leist, T., Yan, J., Karlsson, A. M., 2007, "Time-Economic Lifetime Assessment for High Performance Thermal Barrier Coating Systems," *Key Engineering Materials*, 333, pp. 147-154.

This Article is brought to you for free and open access by the Mechanical Engineering Department at EngagedScholarship@CSU. It has been accepted for inclusion in Mechanical Engineering Faculty Publications by an authorized administrator of EngagedScholarship@CSU. For more information, please contact library.es@csuohio.edu.

Authors

Marion Bartsch, Bernd Baufeld, S. Dalkilic, Iulian Mircea, K. Lambrinou, T. Leist, J. Yan, and Anette M. Karlsson

Time-Economic Lifetime Assessment for high Performance Thermal Barrier Coating Systems

M. Bartsch^{1,a}, B. Baufeld^{2,b}, S. Dalkilic³, I. Mircea¹, K. Lambrinou², T. Leist⁴, J. Yan⁵, A.M. Karlsson^{5,c}

¹Institute of Materials Research, German Aerospace Center, Linder Hoehe, D-51147 Cologne, Germany

²Department of Metallurgy and Materials Engineering, Katholieke Universiteit Leuven, Kasteelpark Arenberg 44, 3001 Heverlee, Belgium

³College of Aviation, Anadolu University, 26470 Eskisehir, Turkey

⁴Material- and Geo-Sciences, Darmstadt University of Technology, Petersenstr. 23, D-64287 Darmstadt, Germany

⁵Department of Mechanical Engineering, University of Delaware, 19716 Newark, DE, USA

^amarion.bartsch@dlr.de, ^bbernd.baufeld@mtm.kuleuven.be, ^ckarlsson@udel.edu

Keywords: Lifetime Assessment, Thermal Barrier Coating, Accelerated Testing, Damage Parameter.

Abstract. Strategies for time-economic lifetime assessment of thermal barrier coatings (TBC) in service are described and discussed on the basis of experimental results, achieved on material systems with coatings applied by electron beam physical vapour deposition. Service cycles for gas turbine blades have been simulated on specimens in thermo-mechanical fatigue tests, accelerating the fatigue processes by an increase of load frequency. Time dependent changes in the material system were imposed by a separate ageing, where the samples were pre-oxidized prior to the fatigue test. Results of thermo-mechanical fatigue tests on pre-aged and as-coated specimens gave evidence of interaction between fatigue and ageing processes. An alternative approach is used, which is focused on the evolution of a failure relevant damage parameter in the TBC system. The interfacial fracture toughness was selected as a damage parameter, since one important failure mode of TBCs is the spallation near the interface between the metal and the ceramic. Fracture mechanical experiments based on indentation methods have been evaluated for monitoring the evolution of the interfacial fracture toughness as a function of ageing time. It was found that the test results were influenced by both changes of the interface (which is critical in service) and changes in the surrounding material.

Introduction

Thermal barrier coating (TBC) systems are used in the hot gas path of gas turbines, e.g. on turbine blades. Components like turbine blades, especially for aero engines, have to sustain thermal and mechanical cycling but also up to 10,000 h at high temperature. Thus, a lifetime assessment of TBC systems has to consider changes of the material properties during service due to time and temperature depending processes, along with damage accumulation due to fatigue. Considering the long time of service, realistic ‘real time’ testing is not economical. In the case of pure (mechanical) fatigue, tests can be accelerated to cover many cycles by increasing the load frequency. Moreover, it is an established method to accelerate processes depending on time-at-high-temperature by increasing the test temperature. However, at higher temperatures different mechanisms may be triggered, e.g. other oxidation products may form. Nevertheless, limited but reasonable reduction of test time can be achieved. For the case when the fatigue and time dependent processes are dependent on each other (i.e. formation and growth of fatigue damages are influenced by the status achieved by time dependent changes in the material), acceleration of the laboratory tests may give

misleading results. An alternative strategy to obtain accelerated testing is to monitor a failure-relevant damage parameter in realistic cyclic experiments and extrapolating the evolution of the damage parameter from interrupted experiments long time before failure occurs.

We will here review two promising test methods for developing time-economic lifetime assessments for high performance TBC systems.

Accelerated close to reality testing - Thermal Gradient Mechanical Fatigue

Specimens. Hollow, dog bone shaped, coated specimens with an inner diameter of 4 mm and an outer diameter of 8.6 mm were used for the thermo-mechanical fatigue tests. The substrate was a nickel-based super-alloy IN 100 DS, which was directionally solidified (DS) with the $\langle 100 \rangle$ direction approximately in the axial specimen direction, in order to simulate the elastic behaviour of single crystal materials used in turbine blades. The elastic modulus in the axial direction of IN100 DS was measured under tensile load with a high temperature extensometer and displays a distribution with values between 117 and 138 GPa at room temperature and between 72 and 83 GPa at 950°C. The coating system comprises a metallic oxidation protection layer, the so-called bond coat (BC), and a ceramic top coat. Both coatings were applied by electron beam physical vapour deposition (EB-PVD). The BC is a NiCoCrAlY with the standard composition in wt %: 20Co, 21Cr, 12Al, 0.15Y, balance Ni, and the top coat is partially stabilized zirconia with 7-8 wt % yttria (PYSZ). The thickness of the BC is about 120 μm and of the ceramic top coat about 220 μm . During the coating procedure, a 0.3 μm thick alumina scale, the so-called thermally grown oxide (TGO), forms between the BC and the ceramic top coat. The TGO growth entails the formation of an Al-depleted zone in the adjacent BC. All materials were processed at the German Aerospace Center in Cologne. Part of the specimens was pre-aged in a separate furnace in air before thermo-mechanical testing, in order to economically accumulate time at high temperature. The heat treatment was cyclic, with each cycle for about 24 h at high temperature, accumulating 250 and 500 h at high temperature, respectively. Cooling down to ambient temperature was achieved by removing the specimen from the furnace.

Test procedure. The specimens were subjected to simultaneous thermal and mechanical cycling. The mechanical load was applied by a servo-hydraulic testing machine, which allowed very fast changes of the load level during the cycling. The thermal load was imposed using a radiation furnace powered by quartz lamps. High cooling rates were achieved with an active air cooling from vents in a shutter, which was introduced into the furnace by a pneumatic device. The high heating and cooling rates made it possible to simulate the thermo-mechanical fatigue load of an entire flight of a turbine blade in an aero engine within 3 minutes. A detailed description of the testing set up is given in [1]. Thermal cycles were between 100 and 1000°C, the mechanical load was tensile and in phase. A typical test cycle is displayed in Fig. 1.

During fatigue testing, the specimen was internally cooled by a constant airflow of 45 norm litres per minute. The inlet temperature of the internal cooling air was about 270°C. Internal cooling and external heating and cooling, respectively, generated thermal gradients over the cross section of the specimen. The temperature difference between the outer and the inner surface was measured at a calibration specimen with sheet thermocouples. Under the quasi-stationary conditions during the high temperature sequence of the test cycle, a temperature difference between the inner and outer surface of about 170°C was measured. Because of the thermal gradient, the test is called thermal gradient mechanical fatigue (TGMF), in contrast to conventional thermo-mechanical fatigue (TMF).

Fig. 1. Typical TGMF cycle with the temperature and the nominal mechanical load profile related to the cross section

Results. Coated specimens, which were pre-aged for 250 h or longer at 1000°C, showed final failure of the TBC system by spallation of the top coat after about 1000 cycles with a nominal maximal mechanical stress of 100 MPa during TGMF. Specimens in the “as-coated” condition have been cycled for even longer times and with higher mechanical loads, but did not show spallation of the ceramic coating [2]. The spallation has been associated to fatigue cracks, which propagated underneath the TGO, parallel to the surface in the axial and circumferential directions [3]. The cracks, which resemble in length sections in their mature state a ‘smiley face’, always display a crack in the TGO perpendicular to the mechanical load. Fig. 2 gives a schematic of the ‘smiley crack’ feature and Fig. 3 shows the scanning electron microscope (SEM) image of the crack, which motivated the name ‘smiley crack’.

Fig. 2. Sketch, showing the main features of the observed ‘smiley cracks’ in relation to the specimen geometry

Fig. 3. SEM image of a typical ‘smiley crack’ prepared by length sectioning

Discussion. Key to the understanding of the ‘smiley crack’ evolution is the stress situation in the TGO during TGMF-cycling. The SEM images indicate that (in at least one sequence of the TGMF cycle) the tensile stresses in the TGO must have exceeded the TGO strength otherwise a crack through the TGO perpendicular to the mechanical load would not appear. Once the TGO is cracked, oxygen can access the metallic BC and weaken the material by local oxidation and an oxidation assisted fatigue crack is likely to grow. However, linear elastic calculations of the stress distribution across the wall of the coated specimen indicate compressive stresses in the TGO for the case of a nominal maximal mechanical stress of 100 MPa [2]. Looking for inelastic phenomena, which may entail tensile stresses in the TGO, we found (through SEM analysis) that the IN 100 DS substrate shows significant rafting [3]. However, assuming linear elastic TGO in the numerical simulations, the accumulation of inelastic strain in the substrate would develop tensile stresses, not only in the pre-aged but in the as-coated specimens as well. Since the as-coated specimen was loaded during part of the experiment with even higher maximum tensile forces (2182 cycles with maximum nominal tensile stress of 280 MPa), TGO cracks should be more likely to occur in these specimens but were not observed. Thus, the pre-ageing, which results in top coat sintering, TGO growth, and diffusion processes, changes the response of the system. As described by several authors [4, 5] and observed here (Fig. 5), the TGO in the as-coated condition has an intermixed (mainly Al_2O_3 and ZrO_2), very fine grained morphology with grain sizes of less than 100 nm. During high temperature exposure the TGO grows and forms a dense zone (mainly $\alpha\text{-Al}_2\text{O}_3$) with grain sizes of more than 1 μm . Following calculations by Rösler et al. [6], the TGO can relax its stresses at high temperature due to creep processes that depend on the TGO grain size, resulting in relaxation times for the as-coated TGO at 1000°C of less than 1 second and for the dense TGO of more than 10 seconds. In the investigated TGMF cycle, both the ‘mixed zone’ and the dense TGO, should be able to relax most of the compressive stresses (induced due to the combination of thermal gradient, growth stress and thermal property mismatch) during the first 2 minutes of the cycle (before a fast mechanical load step follows). The incremental mechanical load step imposed in the end of the load cycle takes about 5 seconds. Therefore, there may be sufficient time for the fine-grained ‘mixed zone’ TGO to relax but not for the evolved ‘dense zone’ TGO, introducing higher tensile stress in the aged samples. This is currently being investigated and will be published at a later state [7].

Extrapolation of damage parameters - determination of interfacial fracture toughness

Test method. Among several proposed methods for determining the interfacial fracture toughness of EB-PVD TBC systems, the Rockwell indentation test with a conical brale C indenter has been selected. This test method has some advantages: in principle it can be applied on specimens and components of any geometry, requiring only a small quantity of material. Indentation of the coating perpendicular to the interface using a brale C Rockwell indenter was analyzed by Drory and

Hutchinson [8] and applied on EB-PVD TBCs by Vasilev and Beuth [9]. In this test method, the indenter penetrates the coating perpendicular to the surface and generates plastic and elastic deformations in the substrate, which are a driving force for the formation of delamination cracks at the interface between coating and substrate. Experiments on systems with a 100 μm thick TBC resulted in circular, concentric debonding and buckling of the coating behind the crack front [9]. The interfacial fracture toughness is estimated from the calculated in-plane deformation of the substrate and the measured radii of both the indenter and the delamination crack. So far, the mechanics of the Rockwell indentation test is analyzed for flat specimens, presuming isotropic material properties and neglecting the interaction between ceramic topcoat and the indenter during the indentation process.

Experimental Procedure. Rockwell indentation tests were performed on flat specimens comprising a 4 mm thick isotropic IN 625 substrate, coated by EB-PVD first with a 100 μm NiCoCrAlY bond coat and subsequently with a 280 μm thick zirconia layer with 7-8 wt% yttria (YSZ). The chemical composition of the coating layers was the same as for the fatigue specimens described above. The material system was investigated in as-coated condition and after heat treatments of 50, 100, 200, and 400 h at 1000°C in air. The indentation was performed using an electromechanical testing machine. The displacement of the indenter was recorded by means of an inductive displacement transducer; the load was recorded by means of a load cell. Test series with indentation loads from 50 to 1000 N were conducted on the specimens. After the indentation, cross sections of the tested specimens at the imprint of the indenter were prepared and investigated by optical microscopy and SEM. The length of the generated crack system at the interface or parallel to the interface was measured, and the crack paths have been evaluated.

Results. The length of the crack systems is a function of the applied load, i.e. higher loads give longer cracks. Comparing as-coated and pre-aged specimens, the same load resulted in longer crack systems for the as-coated specimens (Fig. 4).

Fig. 4. Crack length as function of indentation load for TBC systems in both as-coated and aged condition

Specimens aged for 400 h showed delayed but spontaneous spallation of the ceramic top coat after removing from the furnace. This phenomenon has been often observed, and it is identified as

stress corrosion cracking [10]; it is also nicknamed ‘desk-top effect’ since it often happens when the specimen is laid on the ‘desk top’ after the completion of thermal exposure experiments. Thus, it was not possible to perform indentation tests on 400 h aged specimens. Moreover, after indentation of the 200 h aged sample, delayed spallation of the top coat occurred starting from the free edges of the flat specimens. However, it was possible to prepare cross sections of some of these indented specimens for further microscopic investigations.

Analysis of the crack path revealed that in the as-coated condition the crack system propagated mainly at the interface between TGO and BC. After 50 h of thermal treatment the crack path was partially along the interface between TGO and BC, but mainly parallel to this interface within the TGO and near to this interface within the top coat. With increasing ageing time, the crack systems remained within both the top coat and the TGO, never penetrating the dense zone of the TGO. The characteristics of the crack path are illustrated in Fig. 5.

Fig. 5. SEM images displaying the changes of the interfacial region due to ageing, especially the growth of the TGO with a light grey intermixed zone next to the top coat and a darker dense zone, separated from the intermixed zone by a line of pores. The schematics show the respective crack path after indentation.

Discussion. Rockwell indentation tests performed on the TBC surface resulted in crack systems propagating at or parallel to the interface between the TGO and the BC. Given a constant indentation load, the length of these crack systems is shorter after ageing compared to the as-coated condition. Applying the equations given in [9], the experiments would result in an *increase* of the interfacial fracture toughness of the coating system due to ageing at high temperatures, which is in contradiction to the observed coating behaviour in thermal and thermo-mechanical tests as well as in service. Usually, failure of the TBC occurs by spallation of the top coat at the interface between the BC and the TBC. Long-term aged specimens of this test series showed delayed, spontaneous spallation at the TGO-BC interface as well. Examination of the crack paths reveals that the crack

systems propagate along different paths in the as-coated and the aged condition. In the aged condition the indentation-induced cracks did not penetrate the TGO but were either trapped in the ceramic top coat or within the TGO at a porous line that forms between the mixed zone and the dense zone during long-term ageing. Thus, in aged specimens the indentation-initiated cracks do not reach the weakest interface. In particular, it appears that the dense zone of the TGO is strong enough, compared to the mixed zone and the top coat, and that it shields the weaker interface between the BC and the dense zone of the TGO. Since the top coat material has gained in hardness and strength during the time at high temperature due to sintering processes, the cracks needed more energy for propagation. Thus, the crack systems in aged specimens are shorter than in the as-coated condition, and they are not a measure of the fracture toughness of the decisive interface.

Concluding remarks

Strategies are needed to reduce the test time for reliable lifetime assessment of components that have to survive long term service. For example, it is impractical to perform realistic thermo-mechanical fatigue tests for turbine blade materials in real time until failure, if the blade or the TBC system on the blade is supposed to function 5,000 to 10,000 flights of 1 to 10 h. In this paper, we discuss two test methods designed to achieve this goal.

Strategies for test-acceleration have been applied on EB-PVD TBC systems, i.e. thermo-mechanical fatigue has been accelerated by high loading rates as well as high heating and cooling rates. Operating time in the fatigue testing facility has been reduced by pre-ageing the specimens separately. The resulting damage features in the pre-aged specimens were significantly different to those in the as-coated specimens. A specific type of fatigue cracks, the 'smiley cracks,' with a TGO crack perpendicular to the mechanical tensile load, evolved only in pre-aged specimens. The pre-aged samples had formed a coarse-grained, dense TGO, which was not present in the 'as-coated' samples. The analysis of the results suggests that the loading rate was too high to allow relaxation processes in the dense TGO of pre-aged specimens but allowed relaxation in the thin fine-grained TGO of the as-coated specimens. Thus, tensile stresses develop only in the pre-aged TGO, entailing fracture of the TGO. For lifetime assessment of the TBC system in service, this result showed how important it is to keep the acceleration of tests within limits, which ensure that damage mechanisms in testing are the same as in service.

Experiments have been performed pursuing an alternative acceleration-strategy, monitoring a failure-relevant damage parameter and extrapolating its evolution from interrupted experiments, long time before failure occurs. In this study, the interfacial fracture toughness of the TBC system was selected and the experiment involved the Rockwell C indentation of the surface of both as-coated and aged specimens. It was found that the results were not governed by changes of the interface between BC and TGO (which is critical in service) but by the sintering of the top coat and the growth of a dense TGO, which shielded the weakest interface from indentation-induced cracks. This example shows how important it is to capture with the selected experiment the evolution of the critical feature, which determines the lifetime of the material system in service.

Acknowledgement

The work is supported in part by the European Community's Human Potential Program under contract HPRN-CT-2002-00203, [SICMAC]. I. Mircea and K. Lambrinou acknowledge the financial support provided through the European Community's Human Potential Program under contract HPRN-CT-2002-00203, [SICMAC]. J. Yan and A.M. Karlsson thank the National Science Foundation for financial support under contract DMR-0346664

References

- [1] M. Bartsch, G. Marci, K. Mull and C. Sick: Advanced Engineering Materials 2 (1999), p. 127.
- [2] B. Baufeld, M. Bartsch, S. Dalkilic and M. Heinzelmann: Surface & Coatings Technology. 200 (2005), p. 1282.
- [3] M. Bartsch, B. Baufeld, S. Dalkilic and M. Heinzelmann: International Journal of Fatigue (2006) in press.
- [4] U. Schulz, M. Menzebach, C. Leyens and Y. Q. Yang: Surface & Coatings Technology 146-147 (2001), p. 117.
- [5] A.M. Karlsson, J.W. Hutchinson and A.G. Evans: Materials Science & Engineering. A351 (2003), p. 244.
- [6] J. Rösler, M. Bäker and M. Volgmann: Acta materialia 49 (2001), p. 3659
- [7] M. Hernandez, M. Bartsch and A.M. Karlsson, in progress
- [8] M.D. Drory and J.W. Hutchinson: Proceeding Of the Royal Society London A 452 (1996), p. 2319.
- [9] A. Vasinonta and J. Beuth: Engineering Fracture Mechanics 68 (2001), p. 843.
- [10] V. Sergo and D. Clarke, Journal of the American Ceramic Society 81 [12] (1998), p. 3237.