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Thermal and Mechanical Properties of Thermal Barrier Coatings

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Abstract

The material property measurements of thermal barrier coatings from cyclic furnace, thermal rig, thermal expansion, acoustic emission and tensile adhesion test methods are critically examined. Some basic engineering properties of coatings such as the elastic modulus have not been measured without ambiguity. Data of this nature is essential to the success of modeling studies.

Insights into the mechanical properties of coatings have been gained by carrying out instrumented tensile adhesion tests. The general view of the coating deformation process is that the individual lamellae slide over each other and this promotes a "pseudo-ductility" response in the coatings. Monitoring of the acoustic emission response of coatings during thermal cycling experiments suggests that there are two distinct cracking processes. The macro-cracking behavior, indicated by a change in the acoustic emission count rate, is the predominant mechanism which leads to coating failure.

It is further shown that the acceptance tests used by industry, although useful in ranking coatings in terms of a particular property, present no fundamental knowledge concerning the material properties of coatings. It is only when the phenomenological characteristics of the thermo-mechanical response of coatings is understood that coating development will substantially progress.

1. Introduction

This work will focus on the material property measurements that are carried out on thermal barrier coatings (TBCs). Some of the industrial and research tests will be examined. The industrial manufacturers follow an acceptance standard /1/ which characterizes the mechanical properties of the coating and which also examines the coating microstructure. The bond strength of the coating is determined by employing a tensile adhesion test and the strain tolerance is established from a cupping test where the coating is indented with a 22 mm diameter ball.

These quality control tests are carried out on test panels which are sprayed at the same time as the turbine engine components. The acceptance of a coating relies on a qualitative assessment; for example, a minimum adhesion strength must be attained; a maximum degree of cracking must not be exceeded for the cup test; and the cleanliness (i.e., the amount and distribution of any porosity and .unwanted inclusions) of the coating must meet certain visual standards. A further requirement for some coatings is that the dimensional accuracy of the component and, indeed, individual layers within the coating system and at precise locations on the component (e.g., a turbine blade) must be ensured.

The basis for choosing a specific application of a coating is drawn from these mechanical and microstructural tests; from the considerable practical experience of engine manufacturers and thermal spray contractors and; from some of the limited engine tests that have been performed. However, the quality control tests are far removed from the physical and chemical interactions that thermally sprayed coatings experience during usage; especially when it is considered that the tests are carried out under ambient conditions whereas the coating application may call for high temperatures and pressures under corrosive atmospheres. The following sections address some of the high temperature test methods which have been used to characterize the thermal performance of coatings.

2. Thermal Cycling Tests

The driving force for TBC development 12–61 is either an economic advantage through increased efficiency in the domestic market place (with either increased fuel efficiency or increased component life) or improved performance for military applications. Cyclic furnace tests have been extensively used during TBC development /7/. Small coupons of the required base material are coated, inserted into a rapid heating furnace and then thermally cycled to temperatures in the vicinity of 1000–1200°C. The specimens are inspected at intervals of about 12 cycles to ascertain whether failure has occurred.

Other thermal tests have been carried out under a variety of heat-source conditions and specimen shapes and sizes /8–13/. Table 1 compares these conditions to those experienced within the environment of an operating engine /14/. Much work

TABLE 1. Comparison of thermal test conditions

Method	No.	Specimen size (cm)	Heating time (min)[1]	Maximum temp. (°C)	Hold time (min)[1]	Cooling time (min)[1]	Minimum temp. (°C)	References
Cyclic Furnace	1	2.5 x 1.3 x 0.25	6	990–1095	60	60	280	8, 9, 10
	2	2.5 x 2.5 x 0.5	6	990–1095	60	60	280	8, 9, 10
	3	?	4	975	60	60	280	8, 9, 10
	4	2.5 x 2.5 x 0.5	13	1100	60, 360, 1200	90	?[2]	8, 11
Natural gas-oxygen torch	5	7.5 x 1.3 x 0.5[3]	3	1185–1250	60	5	100	9, 10
	6	J-75 blades[4]	3	1185–1250	60	5	100	9, 10
Burner rig	7[5]	1.3 cm diameter cylinders	4	1040	0, 57	3	?	12
	8[6]	5-75 blades	0.5	1450–1570[7]	60	0.33	<75	9
Plasma torch	9	1.3 cm diameter cylinders	0.5s	1100[8]	0.5s, 2.5s, 5.0s	30s, 75s, 120s	?[2]	13
Engine	10	JT9D–7F first stage blades	2s 14s	870 1095		3s 12s	760 650	14

1. Times are expressed in minutes; unless specified otherwise.
2. Probably less than 50°C.
3. A hot zone of 2.5 x 1.3 x 0.6 cm is mentioned but this thickness is greater than the original specimen /10/. Forced air cooling is at a rate of 14–17 gs^{-1} air flow.
4. 6.5 cm^2 test area on the leading edge.
5. Test 7 was carried out at 0.3 Mach.
6. Test 8 was carried out at 1.0 Mach.
7. The surface temperature range of the blade is given. The corresponding substrate temperatures are 1450–1570°C.
8. Surface temperature.
9. The times to reach specific temperatures are given.

of a similar experimental nature has been carried out with regard to the development of TBCs. However it is difficult to compare these results because of the various specimen geometries,' different substrate materials, different coating compositions and spray deposition parameters, as well as the different experimental procedures and failure criterion that have been observed.

For example consider Fig. 1 which presents some of the thermal test results for the well-documented Ni-17Cr-5,4Al-0.35Y/ZrO_2-(4-24wt.%)Y_2O_3 system. The most attention has been paid to the ZrO_2-8wt.%Y_2O_3 ceramic composition where results from cyclic furnace, natural gas-oxygen rig, and burner rig tests are available. The lines drawn on the figure are used to indicate the trend of cyclic life time with respect to the Y_2O_3 percent and it is seen that the optimum ceramic composition is probably between 6–8wt.%Y_2O_3. There is more than one decade in difference between the lifetimes of specimens tested at approximately 990°C and 1100°C. There may also be a large distribution in the cyclic lifetime data; as is indicated by, for example, the 12 wt.%Y_2O_3 coating where similar coatings exhibited lifetimes of 35 and 60 cycles.

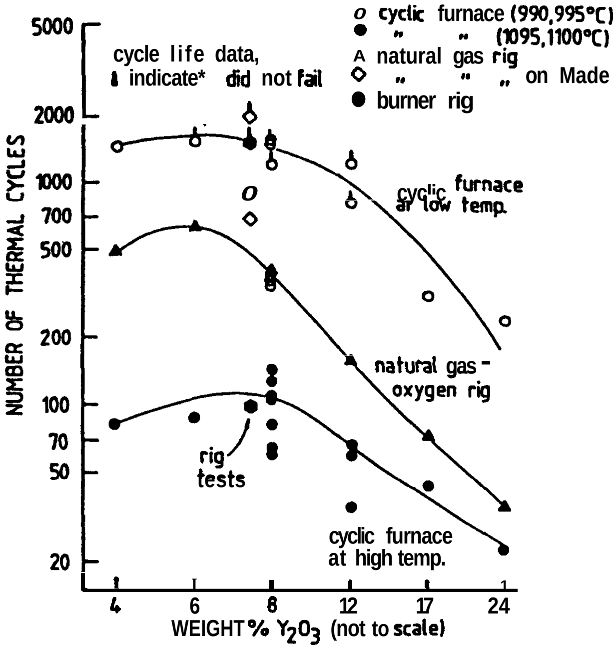

Fig. 1. **Collation of some thermal testing-cyclic life data. The data comes from references /18—241.**

Some of the variance in the data may be attributable to the different substrates used /10/, the relatively poor control in the production of identical coatings, and the different experimental methods for classifying failure. Another observation from Fig. 1 is that turbine blades have been used as specimens for the two rig tests. These tests generally exhibit greater lifetimes than the rig tests on cylindrical specimens or the cyclic furnace tests which have been carried out at equivalent temperatures (i.e., 1100°C).

These results raise at least two important questions. First, it is important to carry out an accelerated test so that advanced material development can proceed rapidly and relatively inexpensively. If this was the only consideration then cyclic furnace tests would seem to have a clear advantage over other methods, Fig. 1. However, a major shortcoming of cyclic furnace tests is that they are qualitative and they do not predict the ultimate service life of a coating. Burner rig tests, on the other hand, are more suited to establishing the service life of the coated blade; but they are more time consuming and expensive to run.

A second important consideration is that the accelerated test must also reflect the operative mechanism of failure. Failure of the coating/substrate system is often ascribed to thermal fatigue, low temperature (650–750°C) or high temperature (750–950°C) hot corrosion, or to oxidation (> 950°C) /3, 15/. The engine designers and materials engineers wish to understand the reasons for coating spallation from the substrate. This has led to studies on the factors which determine coating adhesion to the substrate, the operative failure mechanisms within coatings and fundamental investigations into the thermo-physical properties of TBCs. The lifetimes of coated blades which the engine manufacturers would like to achieve for a range of turbines is shown in Table 2 /3, 4/.

3. Thermal Expansion Measurements

A crucial requirement for TBCs is good adhesion to the substrate at high temperatures under thermal cycling conditions. It is thought that strain incompatabilities,` brought about by thermal expansion mismatches between the substrate and coating, is one of the important mechanisms whereby coatings may fail. Some work has been reported /16, 17/

TABLE 2. Design specifications for coated turbine blades/3,4/

Application	Maximum Thermal strain	Operating Temperature (°C)	Percent of time used	Overall Time (h)
Land-based military aircraft	0.42	1180	10	1,000
Commercial transport aeroengine	0.20	1093	5	5,000
Base-load power generation gas turbine	0.08	820	90	25,000 to 30,000

where the average thermal expansion coefficient (*a*) over the temperature range of interest has been used to calculate the strain difference between the substrate and coating. This data is used in conjunction with the elastic modulus and Poisson's ratio of the coating to find the stresses which are imposed on the coating. The coating will fail when these stresses are greater than the ultimate tensile stress of the material.

The assumption of a constant α over the temperature range of interest is an over simplification in most cases; as is shown by the data presented in Fig. 2 /18–20/. The figure shows that α may vary in the longitudinal (in plane or parallel to the substrate) and transverse (across the coating thickness) directions for a ceramic overlay of ZrO_2-8wt.%Y_2O_3 material. It is also important to remember that the material properties of the coating should not be assumed to be the same as those of the corresponding bulk ceramic or bond coat materials. The microstructural characteristics of thermally sprayed coatings are very different from those of the bulk materials; and thus their thermomechanical properties are also expected to be different.

4. Mechanical Properties

The adhesion property of thermally sprayed coatings to the substrate is an important property to assess since this will limit the utility of the TBC. There are standard methods /21/ of determining this adhesion by adhering a plug (or pull-off bar) to the coating and this mechanical attachment allows the coating to be pulled off in tension. There are a number of criticisms with regard to methods of this nature /22/; and, for example, a major shortcoming is that this test method does not reproduce the stress state which the coatings experience during service.

Tensile adhesion test measurements also exhibit a large variability in strength values for any particular coating /23/. For example the Weibull modulus of stabilized zirconia coatings is about 5; whereas the bulk materials have values of, typically, in excess of 30. The large spread in results does not affect routine quality control procedures; since the acceptance requirement is normally that a batch of 5 samples exhibit a certain minimum tensile adhesion strength. However, another viewpoint when applied research is being carried out, is the necessity for understanding the material characteristics which give rise to this variability in strength.

An 'alternative approach of establishing a measurement for the adhesion of the coatings has been to carry out fracture mechanics tests /22, 24/. However; these tests have not found favour as a routine testing method and are at present more suitable as a tool for the development of coatings. Indentation

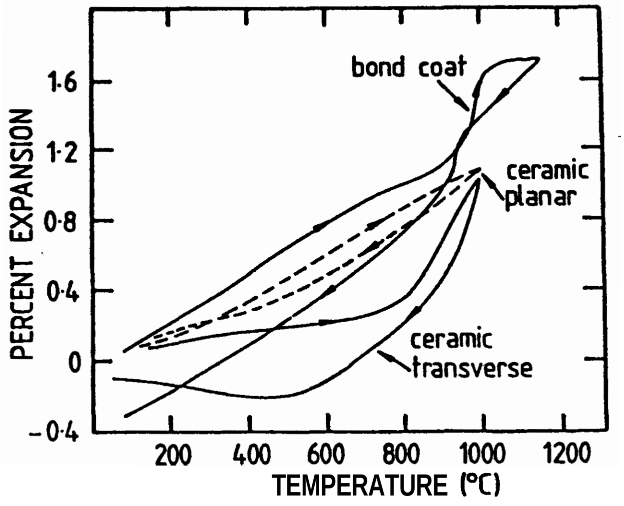

Fig. 2. Thermal expansion data for bond coat and ceramic thermal barrier coating components. From references /18/ and 1191. The data has been redrawn to the same axes. The ceramic is ZrO_2-8wt.%Y_2O_3 and the bond coat is of Ni-22Cr-1Al-1Y.

tests are not suited to finding the fracture toughness of coatings because the required Boussinesq stress field is not attained /25/. In fact, indentation tests have very limited application for the testing of TBCs since the hardness value seems to be more **determined** by the lamellar and inhomogeneous structure of the coating rather than to any unique material property of the coating.

Some mechanical property measurements have been reported for yttria stabilized **zirconia** TBCs /18, 26–28/ of similar compositions. The elastic modulus (E) has been measured as 7–35 **GPa** (reported in 26), 46.2 **GPa** /18/, 4.7 **GPa** at low stress levels /27/, 0.032–0.115 **GPa** over the entire stress range prior to failure /27/, and approximately 0.125–1.56 **GPa** (calculated from the data in reference 28). Although there is **disagreement** with regard to the precise value of E, there is a **consensus** that TBCs exhibit pseudo-ductility because the lamellae slide over each other. This ductility of the coating leads to a large net extension of TAT specimens which are loaded to high stress values /28, 29/.

A major concern of the above referenced data is that any tests which require the adhesion of a test fixture is limited to near ambient conditions and the measurements so-determined may have 'no relevance to the eventual high temperature application of TBCs. It can also be seen from Fig. 3 that the nature of the stress-extension plot of the coating alone (at room temperature) is dependent on **the eventual failure morphology and that it is** non-linear. Thus E is highly variable and depends on both the failure mode and the stress level.

Tensile adhesion test methods have also been used to study the affects of bond coat oxidation on the adhesion of the **TBC** /29/, Fig. 4. The failure stress of the **NiCrAlY** coated materials changes from about 16.3 **MPa** to 12 **MPa** after a specific weight gain of 2.9 mg cm^{-2}; and to 8 **MPa** after a weight gain of about 4.6 mg cm^{-2}. The failure morphology of these materials changed from exhibiting a cohesive character (i.e. within the ceramic overlay) for the as-received TBC to adhesive-cohesive (i.e., at the substrate surface rather than within the ceramic) after the cyclic furnace treatments. Fig. 4 shows that the pre-treatment of specimens by thermal cycling has reduced the TBC failure stress. Other work /8/ has shown that coatings which exhibited a high specific weight gain would also show a lower cycle life than coatings with a lower specific weight gain. It can **therefore** be speculated that coatings which show the greatest tensile adhesion strength, after thermal cycling tests, would. also **exhibit** the greatest lifetime if the thermal cycling tests were continued until failure was observed.

5. **Acoustic Emission** Tests

The previous section on strength measurements emphasized the difficulty in carrying out mechanical tests on coatings. It is important to note that failure

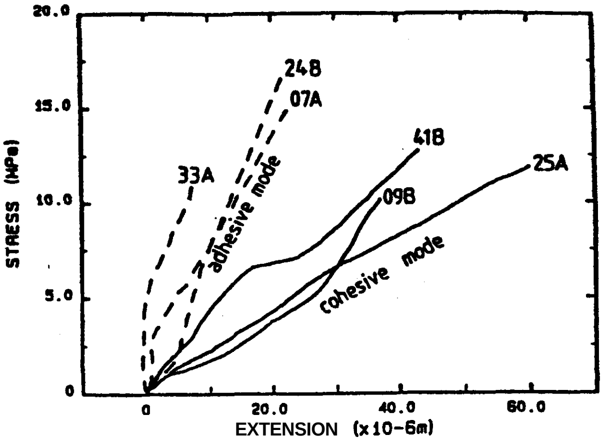

Fig. 3. **Stress/extension** plots of tensile adhesion tests to illustrate adhesive and cohesive modes of failure, from reference 1271.

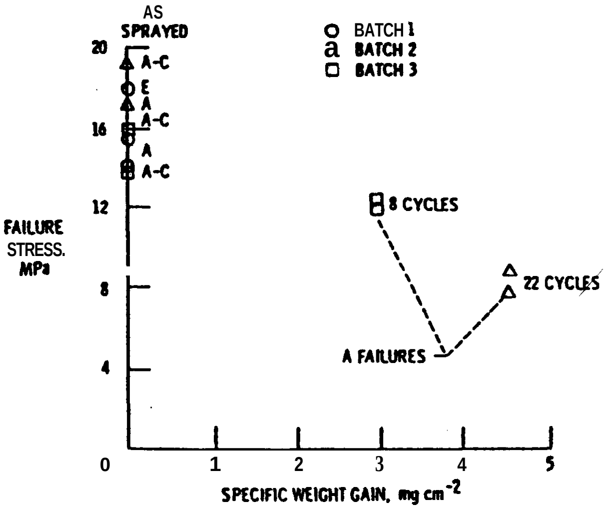

Fig. 4. Specific weight gain versus failure stress for Ni-16Cr-6Al-0.3Y bond coat and ZrO_2-8wt.%Y_2O_3 ceramic overlay and TBC. U-700 substrate material. From reference 1291.

mechanism(s) have not been well-characterized; especially the cracking behaviour of the TBC during thermal fatigue studies.

The "mode" and "mechanism" of TBC failure should be distinguished. The mode is a general term and refers to the physical description of failure; for example coating failure has been described as adhesive, cohesive, mixed mode of spallation. This description, by itself, does not give much insight into the failure process; that is, how microcracks initiate then grow, coalesce and interact to form macrocracks; and the additional effects of oxidation, residual stresses, thermal fatigue and mechanical stresses on these processes. This understanding of the fracture process is called the mechanism of failure. Acoustic emission (AE) tests /28, 30–34/ have been carried out to qualitatively assess these material properties.

The tensile adhesion test has been used in conjunction with AE methods. These tests, carried out at room temperature /28, 30/, discriminated between two different failure processes within coatings and these were distinguished in some tests by at least two count rate regimes which dominated at various times of the test. The count rate is directly proportional to the crack activity and thus the high count rate that was observed during the initial loading of the TAT specimen is indicative of a large number of cracks. The crack interaction and growth during the test allowed some of these microcracks to evolve into several larger cracks and thus the observed AE count rate decreased. These macrocracks lead to coating failure. A major problem with any AE count rate analysis is to obtain a quantitative measurement such as the crack growth rate and the exact number of cracks and their 'size. When this information becomes available then theoretical solutions to the TBC failure and thermal fatigue lifetime may also become available.

Several AE tests have also been performed under thermal cycling conditions /32–34/. Different AE count rate distributions were observed when the TBC coated superalloy specimens were cooled from 1200°C. An apparently random count rate phenomenon was superimposed on a systematic response. These "large random count rates were presumed to evolve from macrocracking processes" /34/ and this cracking process limits the TBC structural integrity.

The AE studies are fraught with difficulties. For example, the experiments are difficult to carry out and it is common for them to have a high variability. Another factor is the difficulty in analysing the AE data in terms of the phenomenological behaviour of the TBC. Regardless of these criticisms it is felt that AE methodology is a useful method of comparing the thermal cyclic response and mechanical behaviour of TBCs.

6. Concluding Remarks

The present work has detailed some thermal and mechanical testing methods which have been applied to TBCs in order to assess their adhesion to the substrate. These tests allow coatings to be ranked in an order which is presumably the same order as that of coatings performing under service conditions.

Cyclic furnace tests are used as an accelerated test method to develop suitable TBC compositions and to study the influence of substrate and coating deposition variables on the coating life. These tests are the best at hand; however, it is important to ensure that exactly the same failure mechanism is operative during these tests as that of coatings in service. Otherwise the results from these tests will be misleading.

The mechanical testing of coatings is important because the material property data soderived is essential for the modeling studies which are in progress. At present there is no agreement concerning the precise value of the elastic modulus. It has been reported as ranging from 0.032–46.2 GPa; and in fact all these values may be correct for the particular technique by which the modulus was determined on the variety of differently prepared coatings. It can also be pointed out that the modulus may be a difficult property to define for a material which exhibits pseudoductility. Thus further studies are necessary to establish the precise manner of deformation for TBCs. It is usual to think that the material properties of these coat-ings are isotropic, but this assumption does not seem to be valid in the light of the anisotropic microstructure of coatings.

Some fundamental work on the mechanical behaviour of coatings is necessary. This work should be focussed on the failure processes which occur within TBCs. The application of these micro-mechanical behaviour models to the thermal environment will promote a basic understanding of coating spal-

lation from the substrate. Such knowledge will enhance the development of future **TBCs.**

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