

Acoustic Emission Evaluation of Plasma-Sprayed Thermal Barrier Coatings

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Acoustic emission techniques have recently been used in a number of studies to investigate the performance and failure behavior of plasma-sprayed thermal barrier coatings. Failure of the coating is a complex phenomena, especially when the composite nature of the coating is considered in the light of possible failure mechanisms. Thus it can be expected that both the metal and ceramic components (i.e., the bond coat and ceramic overlay) of a composite thermal protection system influence the macroscopic behavior and performance of the coating. The aim of the present work is to summarize the 'state-of-the-art' in terms of this initial work and indicate where future progress may be made.

Introduction

Plasma-sprayed thermal barrier coatings may be used to improve the efficiency and utility of gas turbine engines [1-4]. Their main advantage lies with a low thermal diffusivity and conductivity [5, 6] which is representative of a ceramic material. Usual methods of characterization for thermal barrier coatings include burner rig tests which simulate the high heat flux conditions that these coatings experience during their service life. These tests are destructive and indicate the relative number of thermal cycles that **substrate/coating** systems endure. However, it is expected that failure of the thermal protection system is progressive, since cracking and crack growth have been observed prior to ultimate failure, and thus catastrophic **failure** occurs at some stage when there is a transformation from the microcrack to a macrocrack network. Acoustic emission (AE) techniques have recently been used to examine failure characteristics of **plasma-sprayed thermal barrier coatings**.

Comprehensive reviews of AE methodology already exist [7-10]. The essential thrust in materials characterization has been to relate an AE response—either rise time [11], amplitude distribution [11-20], count rate magnitude [21, 22], events [19], accumulative counts [10, 22-24], or energy [25, 26]—to a materials parameter such as crack growth or interaction, oxidation, and other measurements of materials durability and performance. The most elementary analysis examines qualitative trends drawn out by the accumulative AE counts that occur above a specific threshold voltage.

Acoustic emission methods have been used, with respect to plasma spray coating technology, in two distinct research areas. The major assumption is that the AE response is related to the cracking behavior. It is well known that elastic energy is

emitted during cracking processes, and this is expected to be fundamentally related to cracking mechanisms. However, it is very difficult to analyze an extremely large volume of data that may be generated in a few microseconds. Therefore, the data are usually averaged in some manner during the time frame of experimentation. The "count rate" parameter is one such measurement. It is derived from the frequency that a signal exceeds a particular threshold voltage during a specific time interval. It should be remembered that the initial studies were carried out on thermal barrier coatings of interest at that time and were based on Al_2O_3 and TiO_2 ceramics.

Experimental Details

The AE investigations which have been related to properties of plasma-sprayed coatings are summarized in Table 1 [27-37]. Very brief comments about the test methods and AE results are included in this table. Two areas of applied research have been investigated. These are the determination of mechanical properties (i.e., indentation and tensile adhesion tests) and the coating response to thermal environments. These methods may be used with X-ray diffraction and scanning electron microscopy to characterize the chemical composition and failure processes. A major aim has been establishing a data base for mechanistic studies, and much exploratory work has evolved. An important thrust has been to understand how the original powder formulation and substrate processing parameters change the coating integrity and AE response. For example, powders may be produced by fusing, **sintering**, spray drying, or composite methods to give a variety of particle morphologies and size distributions. Also the substrate surface may be prepared by various methods, such as preheat conditioning or grit blasting, prior to coating deposition. Examples from both research areas are detailed below.

Mechanical Testing. This area includes indentation and tensile adhesion tests. The early experiments of Safai et al. [27] were carried out on plasma-sprayed and flame-sprayed

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Table 1 Acoustic emission studies on thermal barrier coatings

| Reference and Coatings Studied ⁽¹⁾ | Type of Test | AE Parameter Examined |
|--|---|---|
| (27) Al ₂ O ₃ (f), Al ₂ O ₃ -13% TiO ₂ Al ₂ O ₃ , Al ₂ O ₃ -40% TiO ₂ | - indentation | - total AE counts |
| (28) Al ₂ O ₃ , Al ₂ O ₃ -2.5% TiO ₂ TiO ₂ , Al ₂ O ₃ -13% TiO ₂ Al ₂ O ₃ -40% TiO ₂ | - furnace testing to 475-520°C | - accumulative AE counts |
| (29) unknown | - cooling of specimen - bending of coating attached to substrate | - accumulative AE counts - effect of deposition parameters i.e., current, air pressure, preheating |
| (30) stainless steel, 7% YSZ NiCrAlY (ht) | - tensile adhesion tests | - accumulative AE counts - sensitive strain tests of coatings |
| (31) 8% YSZ (ht) | - tensile adhesion tests | - accumulative AE counts - count rate distributions |
| (32) 20% MSZ (ht) | - tensile adhesion tests | - accumulative AE counts - average AE count |
| (33) NiAl-20% Al ₂ O ₃ , NiAl-40% Al ₂ O ₃ NiAl-40% ZrO ₂ , NiAl-50% ZrO ₂ | - bending tests | - count rate observation - examination of RMS |
| (34) 8% YSZ | - tensile adhesion tests | - analysis of count rate distributions |
| (35) Al ₂ O ₃ , 20% YSZ, TiO ₂ , 7% CSZ, NiCrAl | - thermal tests | - accumulative AE counts |
| (36) 12% YSZ, NiCrAlZr (ht) | - thermal tests | - accumulative AE counts - processing effects - count rate analysis - bond coat effects |
| (37) 12% YSZ (ht) | - thermal tests | - accumulative AE counts - count rate analysis - sequential failure of coatings |

(1) (ht) indicates that some coatings were heat treated. (f) indicates that the coating was flame sprayed. All the other coatings were plasma-sprayed. YSZ indicates yttria stabilized zirconia. MSZ is magnesia stabilized and CSZ is calcia stabilized. Weight percentages are used.

ceramic coatings and are pertinent to the study of thermal barrier coatings. A summary of their results is shown in Table 2 and reveals an inverse correlation between total AE counts measured during hardness testing and the tensile adhesion strength. The AE data have the additional advantage of conveying information about fracture mechanisms within the coating. Thus the flame-sprayed coating displayed noise levels almost one order of magnitude in excess of the most adherent coating. It was suggested that the coating porosity played a significant role as centers for noise activity. It is clear then, from this initial study, that a propensity for crack evolution and mobility as measured by AE during indentation tests, also correlates to a lower adhesion strength. This research has not been actively pursued with respect to the field of thermal barrier coatings. However, a modified technique, which incorporates attaching a transducer to a scribe and then scratch-testing coatings, has been successful [41-43] in the routine assessment of adhesion characteristics of coatings.

Monitoring of AE during tensile adhesion tests permits direct comparison of the mechanical properties to the noise level and intrinsic character of the coating. A summary of AE and bond strength data is presented in Table 3 and shows that the total AE count is proportionately related to the tensile adhesion bond strength of the coating. In other words, coatings which released a high amount of elastic energy prior to ultimate tensile failure also exhibited a high bond strength. This relation holds even if the different experimental arrangements of these studies are considered. It should be further noted that the bond strengths and the accumulative AE count data show a wide scatter for nominally replicate coatings. This variation may be related to either real changes in actual processing conditions (i.e., the plasma deposition parameters or changes in the substrate) or to factors associated with the preparation of the tensile adhesion test specimen and testing methods. For example, a low measurement of bond strength may result from imperfect specimen preparation where fine peripheral cracks have been

introduced into the specimen. It has also been demonstrated, from the mechanical testing of coatings, that high count rates may be associated with cracking around fine topographic features on the surfaces of individual particles while low count rates can be related to interlamellar failure [31,341].

Thermal Tests. It is difficult to correlate the various thermal cycling experiments due to the different AE equipments used in these investigations. In one case, an "Acoustic Emission Technology Corporation" system was used [27, 28, 30-33, 35], whereas the other workers used a "Dunegan/Endevco 3000 series" system [36, 37].

The first area [28] of investigation concerns the high-temperature response of plasma-sprayed Al₂O₃, Al₂O₃ with either 2.5, 13, or 40wt percent TiO₂ and TiO₂ coatings which were heated up to 520°C. This initial work revealed how the accumulative AE changed for the various coatings and qualitatively related the response to the cracking morphology that was observed by scanning electron microscopy. An important result was the relative effect of the weight percentage of TiO₂ in an Al₂O₃-TiO₂ powder mix. It was found that a coating manufactured from a two-component system displayed less noise than coatings formed from each individual component. This was interpreted as less cracking within the coating and therefore enhanced durability. However a major shortcoming of this preliminary work, and one that is common with all AE analyses, is in quantitatively assessing the AE information to give crack densities and distributions.

Another AE study has been carried out during the thermal cycling of Al₂O₃, TiO₂, ZrO₂-7wt percent CaO, and ZrO₂-20wt percent Y₂O₃ coatings [35]. In some cases, a NiCrAl bond coat was also used. It was found that the total AE count (count rate was not examined) changed with respect to temperature and that these trends could be correlated to the coating integrity. The extent of cracking within the coating

during thermal cycling was reflected by the character of AE activity in the temperature regime of experimentation. These tests showed that the bond coat acted as a compliant layer between the substrate and the ceramic coating. For example, the initial thermal cycles of a bondcoated specimen may show more counts than a specimen coated only by a ceramic. However, it was demonstrated that the total count of the bond-coated sample decreased on repeated thermal cycling, and this indicated that the bond coat may arrest cracks and thereby improve coating integrity.

A more recent study differed from the previous work by examining the count rate distribution as well as the accumulative counts. The count rate was considered as distinguishing between different cracking phenomena. Coatings of ZrO_2 -12wt percent Y_2O_3 were tested by heating to $1200^\circ C$ and cooling to room temperature over a cycle period of 10 hrs [36, 37]. The magnitude of the count rates were then compared to establish the nature of the major failure mechanism. Thus the most predominant count rate that contributed to the total accumulative count could be discerned; and this was termed the "peak count rate." However, the temperature or time sequence nature of the data is lost in using this method to analyze the AE data. One way of finding the influence of temperature on the count rate is to calculate an average temperature from the measured count rate. This method is an approximation because it is based on establishing an "average independent parameter" from the "measured dependent value." On the other hand these results gave insights into: (i) the relative distribution of the count rate phenomena; and (ii) the influence of temperature with regard to crack growth. The main emphasis is that the effect of temperature on the AE response from the coating can be studied. Other more rigorous analyses are currently being developed.

Discussion

Cracking Processes. Several considerations and assumptions of the AE method are detailed below. A basic premise is that the AE is related to cracking processes within the specimen. The AE generated from other processes, such as phase transformations, is negligible since the heat treatment times are very short. However, noise may be generated from oxidation processes and, more importantly, from the effects of oxide growth on the coating structure.

It is argued that the count rate parameter may have significance in regards to cracking processes. It is certain that many different cracking events are integrated for any specific count rate value and each of these events may generate their own characteristic AE count rate. On the other hand, there are many variables associated with cracking; for example, the number of cracks, the size of cracks, the distribution of cracks, and how all of these relate to crack growth. High count rates arise from an increase in crack initiation and/or growth, and they were not randomly distributed with respect to the temperature. It has been shown that a particular count rate interval contributes a significant proportion of the total counts and that the count rate value and its share is dependent on the coating formulation and deposition technique. It is possible to relate the peak count rate to the temperature at which AE events occur but no rigorous analysis presently exists.

It has been pointed out previously that acoustic emission during any sampling period is dependent on both the number of cracks and growth of cracks. Thus it is conceivable that two sets of contrasting conditions (large growth of a few cracks or small growth of many cracks) may give rise to the same count rate. This is a crucial problem to reconcile because it relates to the mechanism of cracking. Therefore the "count rate" does not indicate the kinetic processes of crack in-

teraction. It is reasonable to assume that identical deformation processes are occurring throughout the coating and therefore the relative effects can be examined at different temperatures. In the same manner, different coatings may be compared by their different AE responses. With regard to these assumptions, the experimental data can be related in a semiquantitative manner to cracking behavior.

It is important to distinguish between the terms "extent of cracking" and "cracking mechanisms." The extent of cracking refers to the crack growth, and this is quantified directly by the total number of AE counts. However, the rate at which these counts occur identifies whether cracking occurs via lamellar, translamellar or through bond coat and substrate interactions (or any other processes which generate noise) and this is termed the cracking mechanism.

Mechanical Tests. Tensile adhesion tests are primarily carried out to ascertain materials properties of coatings [44, 45]. Several critiques have already pointed out the limitations of this test technique with respect to the important material properties of the coating [46, 47]. The modes of failure are broadly classified into two types: (i) cohesive when failure occurs entirely through the coating, and (ii) substrate-interfacial where failure occurs at the interface between the coating and the substrate. Failure may also occur near the bond coat/ceramic coating interface and this is often termed adhesive failure. Mixed modes of failure may occur in the same specimen and therefore characterization of the bond strength by a single number is too simplistic. Also, such data do not contribute to an understanding of fundamental coating properties and do not give insight into failure mechanisms.

However, the count rate parameter may present further insights into cracking phenomenon and rate activated processes. For example, the count rate distributions of samples subjected to tensile adhesion tests, in some instances, displayed bimodal distributions [31, 34]. The nature of this distribution could be directly correlated to the fine topographic detail on the fracture surface. This, in turn, resulted from a powder consisting of the coalescence of many fine particles (produced by a sintering process) instead of using the ball-milled product of the fused powder. Therefore, in the foregoing example, the count rate was a more sensitive measurement than the accumulative counts and could be related to the cracking mechanism.

Any detailed analysis of the failure mechanisms of coatings must correlate the count rate distribution to the coating microstructure and bond strength data. Other workers [22, 25] have used AE methods to characterize the bond strength of organic adhesives that were tested by the lap shear technique. An interesting result was that faulty preparation of joints that resulted in low bond strengths could also be detected by greater amounts of emission at relatively low loads. This is analogous to work carried out on plasma-sprayed coatings, and thus very high count rates observed during the initial loading may represent a different cracking mechanism. The magnitude represents either: (i) the nucleation of new cracks, or (ii) the growth and interaction of the existing cracks. Therefore, the physical processes which account for the monomodal distributions of increasing count rate are crack formation and growth.

It should be pointed out that the AE response from indentation and tensile adhesion tests show inverse behavior. For example, the AE counts are a maximum for the most adherent coatings, as measured by bond strength determinations (Table 3), but are a minimum for the same coating during indentation tests (Table 2). The change in AE response occurs due to the nature of both test methods. The tensile adhesion test is an endurance test ending with catastrophic failure of the specimen. On the other hand, indentation tests yield a result when the fracture resistance of the test material

Table 2 Acoustic emission and mechanical tests for plasma-sprayed coatings(1)

| Coating(2) | AE counts (10 ³) | Strength (MPa) | Hardness (R _c) |
|--|---------------------------------|-------------------|-------------------------------|
| Al ₂ O ₃ (f) | 308 | 4.4 (3) | 40 |
| Al ₂ O ₃ (p) | 139 | 6.9 (4) | 60 |
| Al ₂ O ₃ -13% TiO ₂ (p) | 85 | 15.5 | 63 |
| Al ₂ O ₃ -40% TiO ₂ (p) | 39 | 27.6 | 61 |

(1) Strength data from sources as indicated, otherwise from reference 38. AE data from reference 27.

(2) (f) is flame sprayed, (p) is plasma sprayed.

(3) reference 39.

(4) reference 40.

Table 3 AE studies and tensile adhesion tests of plasma-sprayed coatings(1)

| Reference and Coating (2) | Bond strength (MPa) | | Total AE | |
|---------------------------|---------------------|----------|----------|--------|
| | mean | s.d. (3) | mean | s.d. |
| (30) Stainless steel | 54.2 | 8.5 (5) | 61,300 | 42,636 |
| 7% YSZ | 25.8 | 6.9 (5) | 10,670 | 5,630 |
| 7% YSZ (ht) | 19.2 | 2.8 (5) | 8,530 | 2,856 |
| 7% YSZ + NiCrAlY | 54.8 | --- | 40,575 | ---- |
| 7% YSZ + NiCrAlY (ht) | 59.5 | 9.4 (3) | 36,025 | 16,743 |
| (31) 8% YSZ | 34.5 | 3.3 (6) | 51,270 | 26,851 |
| 8% YSZ (ht) | 12.9 | 6.9 (4) | 6,528 | 4,232 |
| (32) 20% MZ | 18.2 | 2.3 (4) | 7,320 | 3,326 |
| 20% MZ (ht) | 22.3 | 3.4 (3) | 14,073 | 7,112 |

(1) Different AE equipment has been used for these experiments.

(2) YSZ indicates yttria stabilized zirconia and MZ is magnesia stabilized. Weight percentages are used. (ht) indicates that coatings were heat treated in argon at 1200°C.

Note that the original powder morphologies differ.

(3) Standard deviation. Values in brackets indicate the number of tests.

balances a predetermined force. Therefore, in the latter case, minimum AE response indicates the more durable material.

Thermal Cycling. The AE data of thermal cycling experiments can be regarded in the same fashion as the bond strength measurements. The thermal tests examined the influence of the bond coat and substrate heat treatment on the AE character of the coating, whereas the strength measurements concentrated on effects arising from powder morphology and heat treatment of the coating. Incorporation of a bond coat, in the case of thermal cycling, decreases the absolute value of the peak count rate, while the number of counts which occur over these intervals, increases. This relationship is observed regardless of the substrate conditioning treatments. The inference is that many small cracking events occur (rather than fewer large cracks) when the bond coat is used. Such failure mechanisms as microcracks have a beneficial effect of dissipating energy and increasing the fracture toughness [48] of materials.

Heat treatment of the substrate immediately prior to coating deposition also changed the AE response during

thermal cycling. Coatings that were deposited onto preheated substrates showed evidence of cracking at higher temperatures during cooling than coatings sprayed under optimal conditions. This clearly demonstrates the application of AE methods to follow, in situ, thermally activated cracking processes. It was also possible to examine the sequence of coating failure during thermal cycling. In this case, the analysis of the AE data is quite complex, since the prior cracked structure of the coating influences the AE response. There is also the matter of dissociating new crack activity with cracking phenomenon that result from crack edges rubbing against each other. This will to a certain extent mask newly occurring fracture processes from detection.

Future Studies. Initial work on coating evaluation via AE studies has shown promise as a characterization technique for the fracture behavior of plasma-sprayed thermal barrier coatings. Further work is aimed at precisely quantifying the count rate and crack growth (or kinetic) aspects of coating failure. It will be necessary to characterize the different failure modes of the coating/substrate system, that is, the interlamellar and translamellar failures of the ceramic and bond coat components. A more fundamental analysis of coating behavior is to examine the unprocessed AE signal. An analysis of this nature is difficult to carry out but is expected to lead to a detailed understanding of cracking mechanisms.

Conclusions

The present work reviews experiments carried out during thermal cycling and tensile adhesion tests of plasma-sprayed coatings which were simultaneously monitored for acoustic emission. In this way the progressive failure of coatings can be ascertained.

The mechanical studies have been based on tensile adhesion tests and show that adhesion strength is related to the accumulated AE counts. Thus coatings that fail at a high tensile stress also exhibit a high AE count. More detailed analysis related the count rate distribution, with respect to time, to the fracture morphology.

The AE data indicate that the thermal cycling performance of plasma-sprayed coatings that have been prepared under different conditions can be distinguished by their AE spectra. The spectrum can be described in terms of either accumulative counts or total counts for a specific count rate interval. The latter method enables dissociation of the peak count rate, which is expected to be related to the cracking mechanism. It was also established how these phenomena changed with respect to temperature.

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