

**DISCRIMINATION OF MICRO- AND MACROCRACKING PROCESSES
IN PLASMA SPRAYED CERAMIC COATINGS**

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ABSTRACT

The progressive failure of **NiCrAlY + ZrO₂-12wt.% Y₂O₃** coatings has been followed by acoustic emission (**AE**) methods during **thermal** cycling tests. Several methods of analysis are presented. **AE** was acquired at two thresholds and this enabled discrimination of the number of **AE** events and qualitative appraisal of the failure mechanisms.

The accumulative **AE** count versus temperature curve was approximately linear and the gradient of this line is proportional to the crack activity. The crack activity was observed to change during thermal cycling from specimen-to-specimen and this also reflects a change in the failure mechanism. The degree of cracking may be assessed from the magnitude of the **AE**. Thus it is proposed that **AE** levels of 30 to 50 counts per event separate microcracking from macrocracking processes.

Finally, the **AE** count data was found to fit the Weibull distribution function. The characteristic count and Weibull parameter for each specimen at every thermal cycle was determined and can be used to propose mechanisms for the cracking behaviour of plasma sprayed coatings.

KEYWORDS

Acoustic emission; ceramics; cracking; distribution functions; failure mechanisms; material properties; plasma sprayed coatings; Weibull parameter; zirconia.

INTRODUCTION

The industrial usage of thermal spray coatings has in many instances surpassed the engineering science which describes the mechanical properties of the coatings. These empirical developments have lead to significant technological advancements in powder processing, thermal spray equipment, deposition techniques and pre- or post-conditioning of the substrate/coating system. However, regardless of these advances there is still much discussion in the literature concerning the adhesion mechanisms to the substrate and failure mechanisms of the

The natural heating and **cooling** rates of the furnace were used and the specimens, hanging down into the furnace, were thermally cycled to **1150°C** for up to 7 cycles. Some **AE** was observed during the heating cycles but more significant **AE** response occurred **during** cooling. Only the **AE** which was generated during cooling of the specimen will be mentioned in the present **work** and the results from two specimens with different ceramic overlays will be examined. In one instance **5.19 g_m** of **ceramic** was deposited (specimen 1) whereas the other coating was produced with an overlay of **3.97 g_m** of ceramic (specimen 2).

A major feature of these tests, not addressed by previous work in this area (**Shankar et al., 1983; Berndt et al., 1983**), was that the coatings covered 100 percent of the specimen surface. Therefore **AE** effects associated with oxidation of the uncoated substrate and from preferential cracking at corners and edges are not expected to bias the **AE** data with respect to **AE** events which are associated solely with the bulk of the coating.

RESULTS

The method of analysis should allow some correlation of the variables to **the** experimental outcome. Specifically, for this work, a major aim is to relate cracking processes (**i.e.**, the **AE distribution**) to the temperature. It is also important to follow the cracking process from thermal cycle-to-thermal cycle and to compare and contrast both of the coatings to the different physical characteristics of the overlay. Several different analyses of the **AE** data are given in this section.

Burst Emission Analysis

A simple analysis used only the **AE data** from the 91 dB channel. Previous work in the area of plasma sprayed coatings (**Berndt et al., 1984**) has distinguished that the overall **AE** response is composed of at least two **AE** distributions. These have been termed as systematic and stochastic responses and are related to **micro-cracking** and **macrocracking** processes respectively.

A common method of displaying **AE** data is to plot the accumulative count with respect to the variable under consideration; which in this case is temperature. The gradient of this plot will be negative since the accumulative number of counts increases as the temperature decreases. Usually more **AE** counts were observed on the first thermal cycle than the second and this is thought to arise from the initial **coating/substrate** relaxation. The **AE** activity increases after the first thermal cycle up to the failure point.

An arbitrary count threshold was set for microcracking and all counts above this were presumed to result from macrocracking. These high counts may also be termed as resulting from "burst emission". It was observed how the slope of the cumulative distribution of **AE** counts versus temperature (**i.e.**; $\Delta N/T$) varied with respect to; (i) the sequence of thermal cycling, (ii) the bound level for transition from micro-to-macrocracking, and (iii) for each coating (Fig. 1). The gradients generally fitted a linear equation with regression coefficients (R^2) greater than 0.9. The greatest effect of bound level on the slope is observed at values greater than 30 counts. Specimen 2 exhibited slopes which gradually become more negative with the thermal cycle number; even for **thermal** cycles beyond the observed failure point. There is a distinct difference between the upper bound levels only from the fourth thermal cycle. Specimen 1, on the other hand, reveals a significant effect of bound level on cycle three and exhibits a **minimum** slope for most of the bound levels at cycle **four**.

ADVANCES IN THERMAL SPRAYING

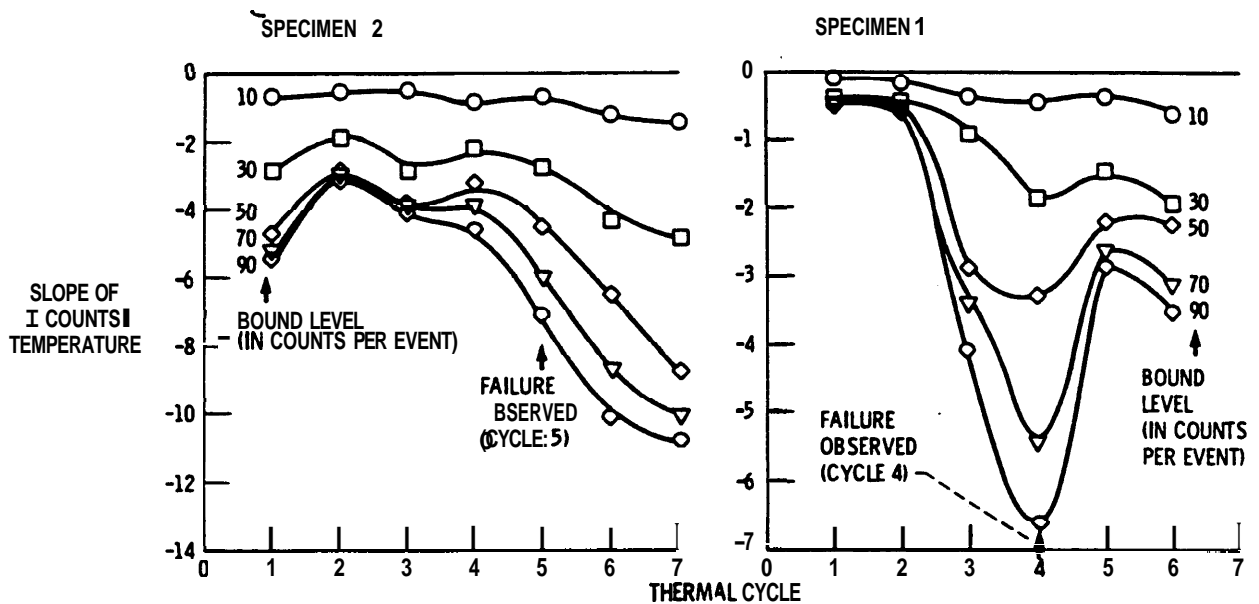


Fig. 1, Effect of burst emission bound on the accumulative count versus temperature data,

A wide range of cracking events (from 10 to 90 counts) occurs over the entire temperature range of experimentation in the case of specimen 2 since the bound levels only serve to scale the gradient. At the same time microcracking events which gave rise to counts of less than 50 also occur to a large extent. On the other hand, specimen 1 reveals a large dependence on the bound level and, as well, the slope changes markedly on the fifth cycle; that is directly after the observed failure cycle. In this case the macroscopic cracking is the more predominant failure mode and after failure there is a tendency for continued microcracking.

The **burst/continuous** AE analysis qualitatively indicates the relative effects of cracking magnitude but can not be used to ascertain the crack population (i.e.; the size and number of cracks). In fact large counts may arise from either large growth of a few cracks or small growth of many cracks. Both of these factors are confounded in the above type of analysis and the following section concentrates on this problem.

AE Event Analysis

The total accumulative counts per thermal cycle are shown for each gain level and specimen in Fig. 2. The count difference between the two AE gain levels is proportional to the total number of AE events (Berndt, 1985b). Both coatings exhibited a decrease in total AE on the second cycle; most probably due to "relaxation effects" of the coating/substrate system after its initial high temperature deposition. The relaxation phenomenon is quite important since it is at this stage that the initial microcracks form and thereby establish a cracking network for the coating,

Several features of the accumulative curves should be pointed out. Specimen 2 exhibited more AE counts immediately after failure whereas specimen 1 showed a large decrease in AE counts. The count difference for specimen 1 is greater than for specimen 2 for thermal cycles 2, 3 and 4 but the count difference parameter increases up to the 6th cycle for specimen 2. It can be inferred that specimen 2

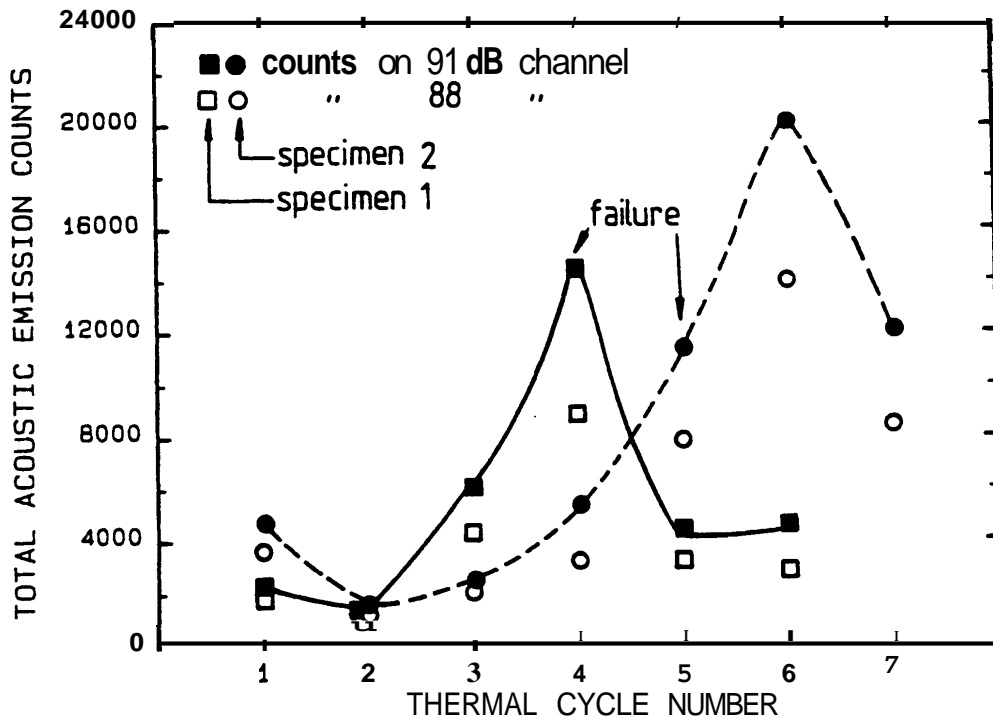


Fig. 2 Accumulative acoustic emission counts at each thermal cycle.

exhibits an increase in the number of cracks as thermal cycling progresses up to the sixth cycle. Coating 1, on the other hand, reaches a maximum in the number of events on the failure cycle and thereafter the number of events is smaller. Both coatings exhibit a gradual increase in the number of events up to the thermal cycle where the accumulative AE is a maximum and then, immediately after this stage, the number of AE events significantly decreases.

The significance of these observations is that the thin coating fails, at least initially, by a relatively low number of cracking processes but that these processes continue after failure. The thick coating, on the other hand, initially fails from a large number of smaller cracks; some of these develop into major cracks which localize any further cracks during thermal cycling.

Distribution Analysis

A technique of examining grouped data is to find the nature of the distribution which it forms. In this way it is possible to assign a unique "signature" to the AE response of each specimen which enables direct comparison of the thermal cycling behaviour. The Weibull distribution has wide applicability in the physical world and its form is (Batdorf, 1978) -

$$F(x) = 1 - \exp \left[- \left(\frac{x-x_u}{x_o} \right)^m \right] \tag{1}$$

- where **F(x)** - the median rank value of the data (see Bergman, 1984)
- x** - AE count of each data point
- x_u** - minimum AE count value
- x_o** - characteristic value below which 63.2% of the data lie
- m** - Weibull parameter (or Weibull modulus)

The distribution is **completely** described by the three parameters of m , x_u and x_0 . The parameter x_u is set to zero since this is the least AE count which is possible in the case of this work. The values of m and x_0 can be found by reducing the Weibull function into its linear form -

$$\ln \ln \left(\frac{1}{1-F(x)} \right) = m \ln x - m \ln x_0 \quad (2)$$

Therefore m is found from the gradient of the $\ln \ln(1/(1-F(x)))$ versus $\ln x$ graph. Once m is calculated then the y axis intercept is used to find x_0 - i.e.,

$$x_0 = \exp(\text{intercept}/-m) \quad (3)$$

The characteristic AE count value can be thought of in the same way as an "average AE count value" except that it applies to the 63.2 percentile rank of a non-normal distribution. The count data for each thermal cycle were treated to the above Weibull analysis.

The form of the Weibull plot is shown in Fig. 3. for the first two cycles of specimen 2. A change in each distribution can be observed at values of 50 and

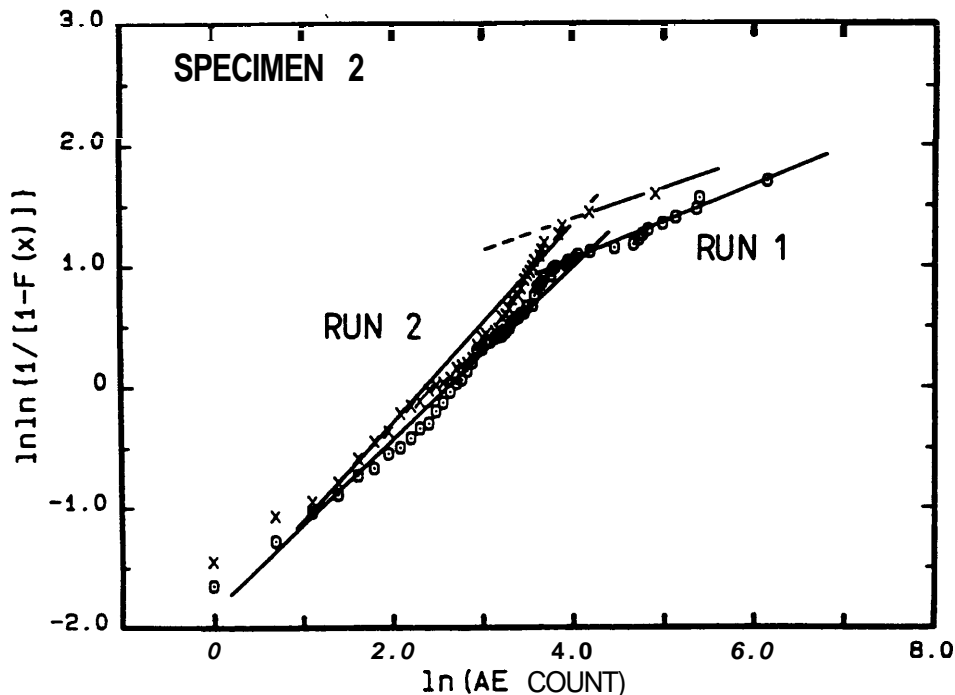


Fig. 3. Weibull plot for acoustic emission count data.

35 counts respectively for cycles 1 and 2. However, the occurrence of **monomodal** distributions was a more typical result and this assumption was used to find m and x_u values. The regression coefficients (R^2) to equation 2 were all greater than 0.90 and were typically 0.97. The trend of the characteristic AE count parameter with respect to the **thermal** cycle for each specimen is shown in Fig. 4. The general observations for each specimen are that the characteristic AE count was high on the first cycle, decreased on the second cycle and then increased up to the failure cycle. The characteristic AE count value decreased after the failure cycle. Specimen 1 which had the greater weight of ceramic

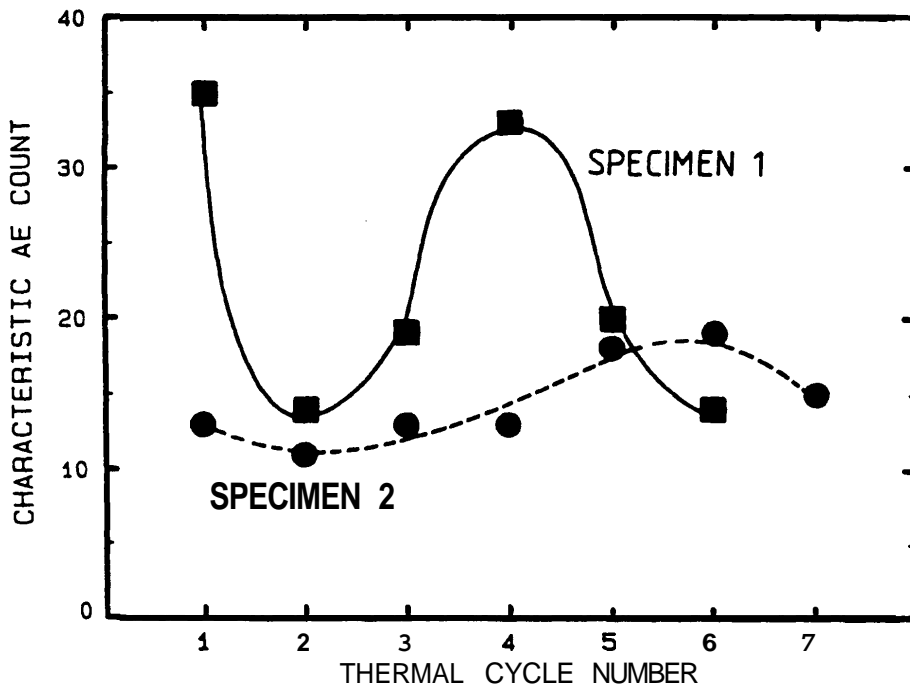


Fig. 4. The characteristic acoustic emission count of plasma sprayed coatings subjected to thermal cycling.

deposit also exhibited the highest characteristic AE count values and the greatest change in these values from cycle-to-cycle.

The final way of examining the AE data is by looking at the Weibull parameter ("m") and this is essentially a measure of the distribution shape. A high m value infers that the AE count values are not widely dispersed but grouped about a central value (i.e., leptokurtic in nature). Both specimens exhibited similar trends in their Weibull parameter behaviour (Fig. 5). The count values occur over a wide range of values during the first thermal cycle but become more centrally located for the second and third thermal cycles. This could be expressed as a transition to AE count data which is more leptokurtic. Prior to ~~observed failure the distribution of specimen-2 is again altered to a~~ widely distributed range of AE counts whereas the m parameter of specimen 1 does not change significantly. The physical inference is that specimen 2 cracks at cycle 3 from events which are all very similar in terms of AE count (i.e., microcracks); however, on cycle 4 there is a large variety of cracking processes (i.e., including macrocracks) and this is reflected by a wide range in the AE count data. Specimen 1 on the other hand always exhibits a large variability in AE count range and never any AE processes which are concentrated within a narrow band of activity.

DISCUSSION

The prime aim of this work has been to assess the cracking activity of plasma spray coated specimens which have been subjected to thermal cycling. Acoustic emission (AE) methods have been used to acquire information concerning cracking processes and this has been treated in a number of ways to reveal trends about the degree of cracking.

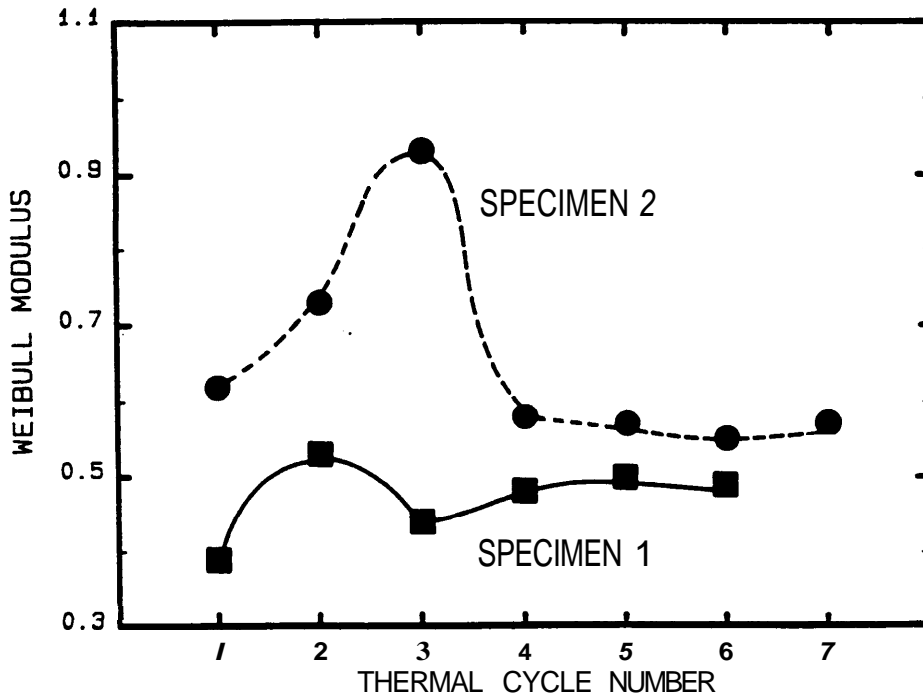


Fig. 5. Weibull parameters for plasma sprayed coatings subjected to **thermal** cycling.

Significant **AE** was only observed during cooling of the specimens. An initial analysis examines the accumulative count function with respect to temperature and this function was found to be linear. The effect of burst emission on the accumulative **AE** trend was measured by subtracting specific **AE** count values from the total distribution. Thus by testing many different **AE** levels it was established that **AE** counts of 30 to 50 per event influence the relative shape of the accumulative distribution with respect to the **thermal** cycle. These count levels correspond closely to the bimodal limits which were observed for specimen 2 at cycles 1 and 2 (Fig. 3). Thus there is some evidence that count values of this order demark a transition from microcracking to macrocracking processes.

The regime of macrocracking has been related to the **AE** phenomenon of burst emission. However, large counts may arise from the simultaneous activity of many small cracks. Thus there is some error introduced when the absolute **AE** count value is used to **ascertain** the type of cracking without regard to the physical processes which occur during failure. One method of solving this problem is to examine the cracking **behaviour** at two levels of amplification and then use the count difference as a measure for the number of **AE** events (Fig. 2).

Another method of studying coating behaviour is by classifying the **AE** distributions. The **AE** data for each thermal cycle was fitted to the Weibull distribution and the **parameters** of m and x_0 were used to characterize the coating. The "characteristic **AE** count" shows that specimen 2 behaves at a lower level of **AE** activity than specimen 1 and this low activity (about 12 counts per event) is probably related to microcracking rather than macrocracking processes. The first thermal cycle of specimen 1, on the other hand, exhibited large **AE** counts (characteristic value of 35 counts per event) and this represents a far greater degree of cracking. Another feature of both distributions is that observable failure of the specimen occurred at a local maximum of the

characteristic stress and on subsequent thermal cycles the characteristic AE count decreased. One shortcoming of the analysis presented above is that the total **number** of accumulative counts is not considered and therefore no assessment of the number of **AE** events can be made.

The shape of the distribution is given by the Weibull parameter. A high value corresponds to a distribution where the values are centrally located whereas "**m**" is low if the AE counts are dispersed over the range. Specimen 2 exhibited distributions with a greater **m** than specimen 1 (Fig. 5). Cracking mechanisms can be postulated which are based on these AE observations. A crack network is developed during the thermal processing of the coatings such that AE events of a similar nature (**i.e.**, microcracking) are produced and therefore **m** increases during the initial thermal cycles. Prior to failure the AE range of events increases and thus the Weibull parameter decreases (cycle 3 for specimen 1 and cycle 4 for specimen 2). This crack structure acts as a network for further cracking of specimen 2 and therefore **m** does not change significantly during further cycling but remains constant at about 0.57. At the failure cycle **m** is low for specimen 2 and indicates that a large range of cracking **events** occur - and the physical picture to represent this is macrocracking. However further crack interaction occurs for specimen 1 and the degree of cracking becomes more uniform so that **m** increases **slightly**.

CONCLUSIONS

The purpose of **this** research has been to examine cracking processes within coatings. Specimens, plasma sprayed coated with **NiCrAlY + ZrO₂-12wt.%Y₂O₃**, have been **thermally** cycled to **1150°C** and simultaneously monitored for acoustic emission.

No single method of analysis is capable of providing a complete description of cracking behaviour. Several methods of **AE** analysis have been detailed in this work. These examine (**i**) the accumulative AE count versus temperature distributions, (**ii**) the accumulative AE count per thermal cycle with respect to two gain levels, and (**iii**) a description of the **AE** count distribution by the Weibull function.

The coatings revealed different AE behaviour in terms of the absolute number of counts **and** in the statistical description of the AE count distribution. The AE from the specimen can be correlated to cracking processes. Thus measures for the total degree of cracking and the similarity of cracking processes during a thermal cycle can be obtained. Information of this nature is used to characterize each specimen and allow the failure of plasma sprayed specimens to be described by microcracking and macrocracking processes.

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