

Acoustic emission studies on thermal spray materials

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Abstract

Acoustic emission (AE) has been used to assess the integrity of ceramic coatings. The early studies enabled cracking mechanisms to be qualitatively assessed so that comparative studies of coating behavior could be formulated. The present work examines a quantitative assessment of crack populations and crack sizes so that more detailed analyses of "crack density functions" can evolve for mechanistic studies of thermal spray materials. Coatings and solid deposits of thermally sprayed ceramic have been tested with the stress oriented in directions perpendicular and parallel to the spray direction. It has been determined that macrocracking with catastrophic failure occurs in the perpendicular orientation; whereas several distinct crack populations, consisting of micro- and macrocracking events, are observed for the parallel orientation. © 1998 Elsevier Science S.A.

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1. Introduction

"Acoustic emission (AE) is a term describing a class of phenomena whereby transient elastic waves are generated by the rapid release of energy from localized sources within a material." The energy usually arises from one or a combination of sources [1], including phase transformations, plastic deformation, corrosion and crack initiation and growth [2]. An AE event is detected by a transducer, amplified, and then AE features such as the ring down count, amplitude, and energy can be analyzed. The term ring down count refers to the physical process whereby an acoustic emission signal decays with time due to natural attenuation. Therefore, the AE signal is said to "ring" and the number of times that the signal crosses an arbitrary threshold results in a "count" that is directly related to strength of the active source that generated the original signal.

A thermal spray coating has a very rich microstructure and both macro- and microcracking, among other sources, can release energy during coating service. AE technology has been combined with fracture mechanics measurements [3] or thermal tests [4,5] to better understand failure mechanisms of thermal spray coatings. In conjunction with mechanical property measurements,

indentation with in situ AE has been used to monitor the cracking [6]. By use of AE technology, the overall cracking responses during the indentation can be recorded to provide more detailed information. It has been established that the number of AE counts emitted during a hardness test increased as the density of plasma sprayed ceramics decreased [6]. Similar correlations have been proposed on the basis of AE measurements performed during tensile adhesion tests (TATs). The AE count accumulated during a TAT can be graphed with respect to the so-determined bond strength, and coatings which incorporate metallic constituents exhibit a lower activity than nonmetallic coatings (at equivalent bond strengths).

Studies for AE responses during the thermal cycling of plasma sprayed TBCs have shown that the type of cracking and the AE responses may be influenced by the porosity, the presence of metastable phases, the thermal expansion mismatch between substrate and coating, and the anisotropy of thermal expansion among the phases [7-9]. A stable microcracking network can be generated for thermal barrier coatings with good adhesion strength. These coatings remain effective even after the formation of a microcrack network. In contrast, catastrophic failure occurred because of unstable crack growth. A "crack density function" (CDF) [10], considering both the number of cracks and size of cracks,

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has been proposed and it is found that macrocracking events tend to occur at low values of the CDF.

AE has been used to in situ monitor the cracking behavior of plasma sprayed free standing ceramics during four point bend tests [11,12]. The AE responses were assessed with respect to time and displacement in cumulative and/or normalized forms. Two basic types of cracking mechanisms, i.e. catastrophic failure and microcracking before failure, have been determined. It was also reported that micro-, transitional, and macrocracks can be better discriminated by the energy than the amplitude of the event. In the present study, the energy distributions will be further examined to allow an improved CDF analysis. In addition, the AE characteristics of individual events were assessed to better understand the failure mechanisms.

2. Experimental

Since detailed descriptions concerning the process, four point bend tests, and AE analyses are available elsewhere [12,13], only brief procedures are outlined below.

The alumina-13 wt.% titania free standing specimens were sprayed using a water-stabilized plasma process. Free standing plates were obtained by etching away a thin aluminum layer, presprayed by wire-arc spraying, using hydrochloric acid, and then cut to a width of ~5 mm by a diamond saw. Samples (~5 x 5 x 50 mm³) were then sintered at 1450 °C for 86,400 s, slowly cooled to 1100 °C (at 100 °C/h), and aged for a further 86,400 s. After heat treatment, the furnace was cooled at a rate of 100 °C/h to 700 °C, then furnace cooled to room temperature.

A universal test machine (ATS, model 1101, Bulter, PA) was used to perform the four point bend tests with an AET 5500 instrument (Hartford Steam Boiler Inspection Technologies, Sacramento, CA; now Babcock and Wilcox, Barberton, OH) to in situ monitor the cracking behavior. The lengths of the inner and outer spans were 20 and 40 mm respectively, and the crosshead speed was 0.06 m/s. Detailed procedures concerning the four point bending and AE analyses are described elsewhere [13]. The sample identifications used in the current study are listed in Table 1.

3. Results and discussion

3.1. Preliminary AE analyses

It has been shown that there is an increase of the modulus of rupture (MOR) and elastic modulus (E) after heat treatment [11]. However, the results of tests performed in the in-plane and cross-section orientations were not significantly different (within one standard deviation range). For AE responses, two different types of cracking mechanisms were also distinguished, i.e. catastrophic failure and microcracking before failure. The distinction of "catastrophic" and "microcracking" features was based on the AE response prior to failure.

The energy and amplitude distributions of AE responses for tests performed in either cross-section or in-plane directions did not change significantly, however, they were altered after heat treatment. In addition, the total number of events was smaller than the value for "as-sprayed" samples. The decrease in total events was due to the change in microstructure. The energy distributions can be used to better discriminate micro-, transitional and macrocracks by the energy levels of 45 and 100. The numerical value of the energy level corresponds to a response from the piezoelectric transducer and, therefore, has engineering dimensions which incorporate the voltage and time of each AE emission.

3.2. Population analyses

It is postulated that micro-, transitional, and macrocracks have different characteristics, so histogram plots and corresponding Weibull plots of the distribution from corresponding test conditions were investigated. However, due to the similarity of analyzing methods, only the results from "as-sprayed" samples tested in the cross-section direction are shown in Fig. 1. At least three distributions can be noticed, i.e. a distribution centered around a high energy (~115), a mid-range energy (~70), and a low energy (~40).

The energy distributions with respect to micro-, transitional and macrocracks for "as-sprayed" and heat-treated samples are summarized in Table 2. It was found that the proportion of micro-, transitional and macrocracks did not change significantly. The microcracks were ~10% for "as-sprayed" samples and 6% for heat-

Table 1
Sample identifications for four point bend tests with *in situ* AE

"As-sprayed" conditions		Heat-treated conditions	
In-plane	Cross-section	In-plane	Cross-section
AS-BI to AS-BIO	AS-CI to AS-CIO	AH-BI to AH-BIZ	AH-CI to AH-C13

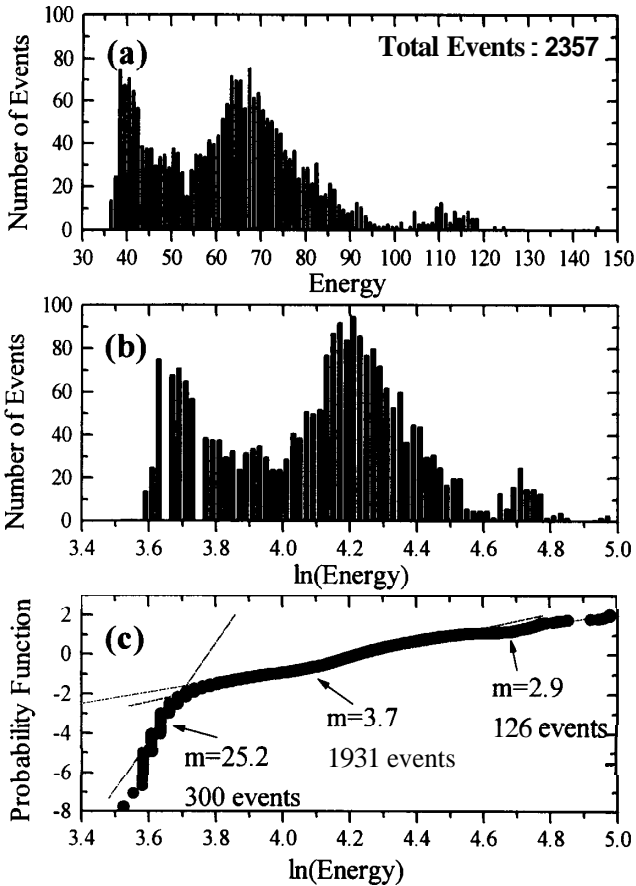


Fig. 1. (a, b) Energy distributions; (c) Weibull plot for "as-sprayed" samples (AS-C3 to 10) tested in the cross-section direction.

treated samples and the distributions showed a high central tendency; that is, the grouping of data was highly localized within a relatively narrow band of energy. The transitional cracks were the majority (>80%) of the overall AE response. The macrocracks were <10% for "as-sprayed" samples and 12% for heat-treated samples. Both transitional and macrocracks were widely distributed. It is postulated that the same thresholds may exist for all specimens.

The AE feature of either catastrophic failure or microcracking could be distinguished by the total number of events from the tests. It was found that the average event activity per sample decreased after heat treatment. It was also noticed from Table 2 that the average events per sample for specimens where catastrophic failure occurred were lower than the value for specimens where microcracking could be observed. The AE analysis showed the same trends such that macrocracking occurred at "a low average events per sample"; which was similar to the AE response of thermal barrier coatings subjected to thermal cycling [10].

3.3. AE responses vs. normalized displacement and quartile analysis

A quartile analysis of the normalized AE responses for all specimens is illustrated in Fig. 2, where 25%, 50% and 75% of the cumulative AE responses are indicated by different symbols with respect to normalized displacement. It is important to keep in mind that there is a large difference in MOR and E between the "as-sprayed" and heat-treated specimens. Therefore, the same normal-

Table 2
Summary of energy distribution for "as-sprayed" and heat-treated samples with respect to micro-, transitional, and macrocracks

		Energy description			No. of samples (Total events)	Events per sample
		Micro <45	Transitional 45-100	Macro >100		
AS-IP (C)"	<i>m</i>	20.0	3.5	2.7	4 (760)	190
	% ^b	12	80	8		
	events	90	609	61		
AS-IP (M)'	<i>m</i>	27.1	3.7	3.0	5 (1887)	377
	%	8	86	6		
	events	150	1631	106		
AS-CS	<i>m</i>	25.2	3.7	2.9	8 (2357)	295
	%	13	82	5		
	events	300	1931	126		
AH-IP	<i>m</i>	30.8	3.7	3.3	10 (1091)	109
	%	6	82	12		
	events	68	897	126		
AH-CS	<i>m</i>	29.6	3.8	3.3	11 (1073)	98
	%	6	82	12		
	events	69	877	127		

^a Samples which showed catastrophic failure.
^b Percentage calculated by events over the total number of events.
^c Samples which showed microcracking before failure.

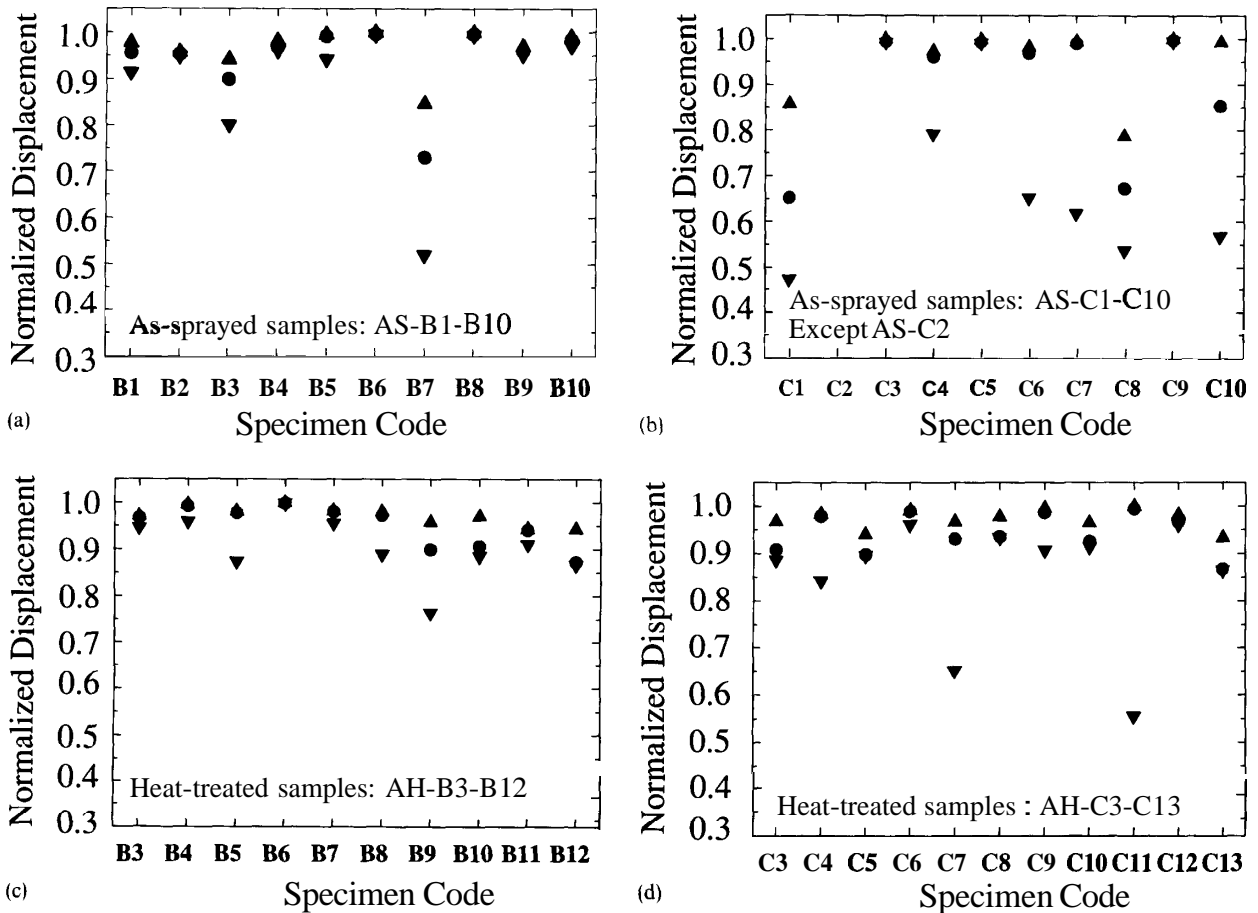


Fig. 2. Quartile analysis of normalized AE responses with respect to normalized displacement for (a) "as-sprayed" samples tested in the in-plane direction, (b) "as-sprayed" samples tested in the cross-section direction, (c) heat-treated samples tested in the in-plane direction, and (d) heat-treated samples tested in the cross-section direction: ∇ , first quartile analysis; \circ , second quartile analysis; \blacktriangle , third quartile analysis.

ized displacement for different specimens may be referred to different stress levels at which cracks propagate.

It is noted from Figs. 2(a) and (b) that for the "as-sprayed" specimens which showed catastrophic failure (AS-B1, -B2, -B6, -B8, -C3 and -C9), the normalized displacement of the first quartile (25%) of the cumulative energy was larger than 0.90. In addition, the energy released was more widely distributed for specimens tested in the cross-section direction. For the heat-treated specimens [Fig. 2(c) and (d)], although microcracking was commonly observed, most of the AE activity was detected close to the final failure. For example, the normalized displacement for the first quartile was larger than 0.85 in most of these cases.

3.4. Total AE responses vs. normalized displacement

In order to avoid being misled by an individual test result, the AE responses from four test conditions (i.e. "as-sprayed" and heat-treated samples tested in the in-plane and cross-section orientations) were pooled together. The total AE responses (presented by histo-

grams) versus normalized displacement are illustrated in Fig. 3. It was noticed that the "as-sprayed" samples tested in the cross-section direction exhibited more events before final failure, say between a normalized displacement of 0.2 to 0.8. No significant difference was distinguished for heat-treated samples.

Fig. 4 shows the normalized total AE responses (total AE responses normalized with respect to their total events) versus the normalized displacement (normalized with respect to their total displacements). The difference for "as-sprayed" samples for tests performed in the in-plane and cross-section orientations could be better distinguished. The normalized AE responses are noticed before a normalized displacement of 0.1 for tests performed in the cross-section direction. On the other hand, this response occurred after a normalized displacement of 0.2 for the in-plane direction. In addition, the normalized AE responses for samples tested in the cross-section direction were higher than the value for the cross-section samples from a normalized displacement of 0.1 to 0.98, i.e. most of the energy release occurred at failure for tests performed in the in-plane direction.

Slight differences were found for heat-treated samples

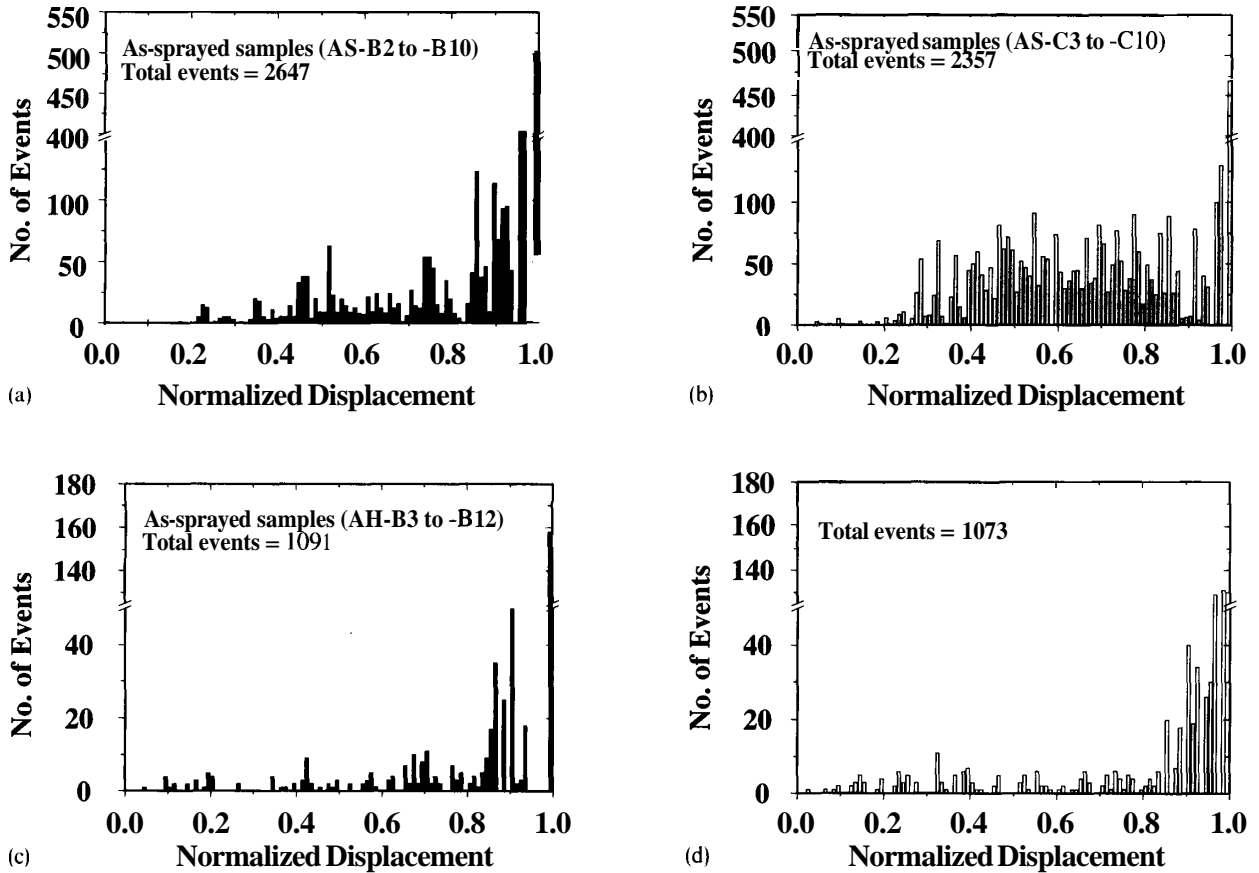


Fig. 3. Histogram plots of total AE events for "as-sprayed" samples tested in the (a) in-plane and (b) cross-section orientations, and heat-treated samples tested in the (c) in-plane and (d) cross-section orientations with respect to normalized displacement.

tested in different orientations. The value of normalized AE response was higher for tests performed in the cross-section orientation before the final failure, which implies that the probability for microcracks to propagate was slightly larger for samples tested in the cross-section direction. The normalized AE energy releases at a normalized displacement of 0.8 were 0.59 and 0.24 for "as-sprayed" samples tested in the cross-section and in-plane

orientations, and 0.14 and 0.12 for heat-treated samples tested in the cross-section and in-plane orientations respectively.

3.5. AE characteristics of individual events

The AE characteristics of an individual event are more complex than the results discussed above because several thousand events, with many variables, must be interpreted. The energy of individual events versus normalized displacement has been found to provide some physical understanding of cracking processes and will be discussed.

The AE response from individual crack events for "as-sprayed" and heat-treated samples tested in the in-plane orientation is illustrated in Figs. 5 and 6 respectively. AE responses from different specimens are indicated by different symbols and each symbol represents an individual event. For "as-sprayed" samples (Fig. 5), it is noted that most of the high energy events (i.e. macrocracks) were detected close to the final failure (say after a normalized displacement of 0.90) and only several macrocracks were observed prior to failure. After heat treatment, the high energy events (i.e. macrocracks)

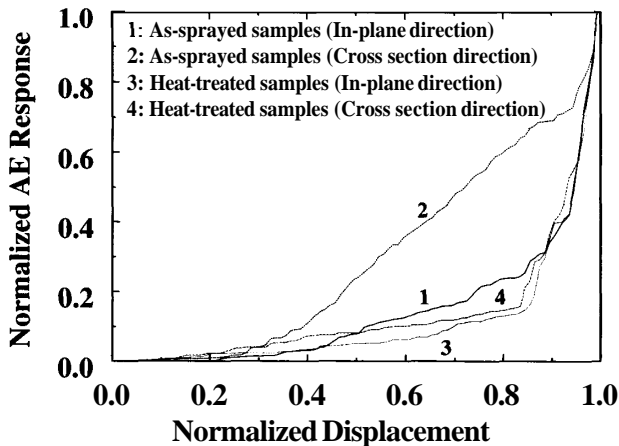


Fig. 4. Normalized total AE responses versus normalized displacement.

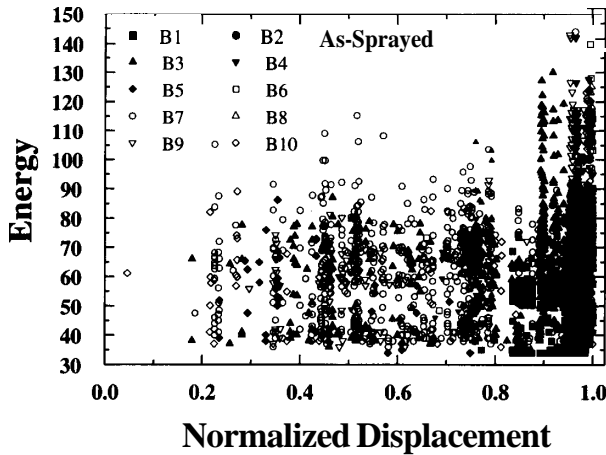


Fig. 5. AE responses from individual crack event for "as-sprayed" samples tested in the in-plane direction.

could only be detected close to the final failure (say after a normalized displacement of 0.82) and no such macrocracks were detected before that displacement. The majority of the events (say 99%) detected before a normalized displacement of 0.82 exhibited an energy less than 80. Meanwhile, most of the events occurred in the final step, which corresponded to the same conclusions as discussed by the quartile analysis (Fig. 2). The AE responses for the "as-sprayed" and heat-treated samples differ not only from the percentage of macrocracks (Table 2) but where they arose during the tests; for example, the absence of macrocracks in the early stage of loading for heat-treated samples.

In summary, AE technology provides an opportunity to collect information concerning material responses to stress and interpretation of these results enables a better understanding of cracking mechanisms of these materials.

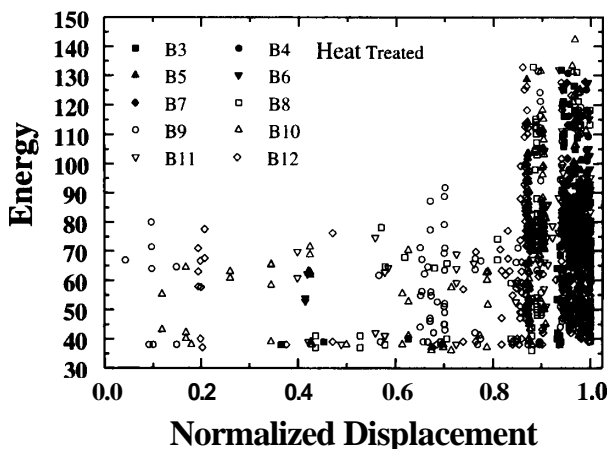


Fig. 6. AE responses from individual crack event for "as-sprayed" samples tested in the in-plane direction.

4. Concluding remarks

The energy distributions of AE responses for tests performed in either cross-section or in-plane directions did not change significantly; however, the distributions altered after heat treatment. AE events with high energy (>100) could be characterized as macrocracks and were widely distributed (with a Weibull modulus $m < 4$). Events with lower energy (i.e. energy < 100) could be separated into micro- and transitional cracks by the energy level of 45 according to the energy distribution. Microcracks formed an energy distribution with a high central tendency ($m > 20$) and transitional cracking behavior was the major constituent of the crack density function ($>80\%$).

For the "as-sprayed" samples which showed catastrophic failure, the first quartile (25%) of the cumulative energy was found after a normalized displacement of 0.9. The released energy was distributed more widely for samples tested in the cross-section direction. The quartile analysis of heat-treated specimens confirmed the finding by AE responses versus displacement, i.e. most of the AE activities occurred close to the final failure. It was noticed that the "as-sprayed" samples tested in the cross-section direction exhibited more events before final failure and no significant differences could be distinguished for heat-treated samples.

The difference for "as-sprayed" samples for tests performed in the in-plane and cross-section orientations can be better distinguished after normalization. The normalized AE responses for samples tested in the cross-section direction are higher than the value for the cross-section samples even after heat treatment. This implies that the probability for microcracks to propagate is larger for samples tested in the cross-section direction. The normalized AE energy releases at a normalized displacement of 0.8 are 0.59 and 0.24 for "as-sprayed" samples tested in the cross-section and in-plane orientations, and 0.14 and 0.12 for heat-treated samples tested in the cross-section and in-plane orientations respectively.

The influence of AE characteristics of individual events was investigated and it is noted that most of the macrocracks (energy > 100) were detected close to the final failure for the "as-sprayed" samples. For heat-treated samples, macrocracks were only detected after a normalized displacement of 0.82, and the majority of the events detected before this point exhibited an energy < 80 .

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