# ACOUSTIC EMISSION FEATURES IN TEXTILE REINFORCED THERMOSET AND THERMOPLASTIC COMPOSITES

A. D'Ettorre<sup>1</sup>, V. Carvelli<sup>1</sup> and S.V. Lomov<sup>2</sup>

<sup>1</sup> Department ABC, Politecnico di Milano, Piazza Leonardo Da Vinci 32, 20133 Milan, Italy Email: alessandrodettorre91@gmail.com; valter.carvelli@polimi.it

<sup>2</sup> Department of Materials Engineering, K.U. Leuven, Belgium, Kasteelpark Arenberg 44, B-3001 Leuven, Belgium Email: Stepan.Lomov@mtm.kuleuven.be

Keywords: thermoset; thermoplastic; textile composites; acoustic emission; cluster analysis.

### Abstract

In this work, the AE cluster analysis methodology was applied to investigate the peculiarities of the AE evolution and clustering in thermoset and thermoplastic reinforced 2D carbon and glass textiles. One thermoset (Epoxy) and two thermoplastic (Polyphenylene sulphide (PPS) and Polyether ether ketone (PEEK)) resins were considered. The epoxy was reinforced with balanced twill 2x2 carbon fabric and balanced twill 2x2 glass fabric. The PPS and the PEEK were reinforced with carbon plain weave textile. The comparison of the cumulative energy of AE events show that the thermoplastics reinforced composites have an early beginning of the damage and rapid saturation while a later beginning and more continuous growth of the damage was monitored in the textiles reinforced epoxy materials. The cluster analyses highlighted that the thermoset and thermoplastic reinforced composites have a net separation of the clusters. The crack monitoring in the thickness allowed the preliminary correlation of the transverse cracks in the PPS reinforced composite to the cluster containing AE events of low amplitude and low frequency.

### 1. Introduction

Acoustic emissions (AE) registration allows detection of micro damage events in materials and structures and potentially identification of the damage nature [1]. Damage characterization using AE is already a well-established procedure to monitor, in real time, damage growth in conventional materials such as metals. In the case of fibre reinforced composite materials several microscopic types of failure exist, which result in complex macroscopic failure. The microscopic failure mechanisms of interest are: matrix cracking, interfacial failure occurring between fibre and matrix and fibre breakage. Each of these microscopic failure mechanisms is accompanied by a rapid microscopic grow of the crack surface and, as consequence, an excitation of an ultrasonic elastic wave occurs inside the material. The detection of the signals (acoustic emissions) at the surface of the solid and their analysis is thus a powerful tool to investigate the damage in composites. The evolution of cumulative energy of AE events was adopted for textile reinforced polymers allowing the identification of damage initiation and propagation during loading [2]. But methods for the identification of the damage modes based on the features of the AE events are still under investigation.

Cluster analysis is a powerful methodology to analyse multi-parametrical AE signals. The analysis classifies events based on clustering of their multi parametrical descriptors. This creates a framework for subsequent identification of the links between the established cluster event classification and the physical nature of the damage.

In this work, the AE cluster analysis methodology, previously used for 2D and 3D glass and carbon/epoxy woven composites [3] and [4], is applied to investigate the peculiarities of the AE evolution and clustering in thermoset and thermoplastic reinforced 2D carbon and glass textiles. One thermoset (Epoxy) and two thermoplastic (Polyphenylene sulphide (PPS) and Polyether ether ketone (PEEK)) resins were considered. The epoxy was reinforced with balanced twill 2x2 carbon fabric and balanced twill 2x2 glass fabric. The PPS and the PEEK were reinforced with carbon plain weave textile. Tensile tests were assisted with two acoustic sensors. Moreover, two high resolution cameras were adopted: the first for the evaluation of the full filed strain by digital image correlation techniques, the second for the local damage observation in the thickness during loading.

The comparison of the cumulative energy of AE events show that the thermoplastics reinforced composites have an early beginning of the damage and rapid saturation while a later beginning and more continuous growth of the damage was monitored in the textiles reinforced epoxy materials.

From the primary nine AE features, two were selected for the cluster analysis: peak amplitude (PA) and peak frequency (PF) of the signal. The optimal number of cluster was computed considering the highest value of the Silhouette coefficient (SC) and the lowest value of the Davies-Bouldin coefficient (DB). Three clusters were selected for the analyses.

The cluster analyses highlighted that the thermoset reinforced composites have a net separation of the clusters. This is valid for PPS/carbon composite, as well, while the PEEK/carbon textile does not have a clear distinction probably due to the inherent plastic behaviour of the matrix.

The three different types of clusters can be subdivides as: CL1 low amplitude and low frequency; CL2 high amplitude and low frequency; CL3 high frequency. The recorded limit of the three clusters in term of amplitude and frequency are similar to the previously found in literature ([3], [4]).

The crack monitoring in the thickness allowed the preliminary correlation of the transverse cracks in the PPS reinforced composite to the CL1 cluster containing AE events of low amplitude and low frequency.

## 2. Composite materials and experimental procedure

The two thermoset composites were: a twill 2x2 carbon textile reinforced epoxy resin (CF/epoxy) and a twill 2x2 glass textile reinforced epoxy resin (GF/epoxy).

The two thermoplastic materials were: a five-harness satin weave carbon textile reinforced PPS matrix (CF/PPS) and a five-harness satin weave carbon textile reinforced PEEK matrix (CF/PEEK).

The main features of the thermoset composites are listed in Table 1, while those of the thermoplastic composites are in Table 2.

Specimens had the dimensions: total length 260 mm, gage length 160 mm, width 25 mm.

For all the materials tensile tests were performed using an Instron 4505 with a crosshead speed of 1 mm/min.

Specimens equipped with two AE sensors were loaded up to 80% of the ultimate tensile strength to avoid damage of the equipment. Details on the software and sensors used for the AE recording are listed in Table 3.

**Table 1.** The main characteristics of the thermoset composites.

	CF/epoxy	GF/epoxy
Fibres	Carbon HS	E-glass
Yarns	3K	300tex
Fabric Density [g/m <sup>2</sup> ]	245	375
Plies	10	10
Fibre volume fraction [%]	49.5±0.6	57.8±0.8
Thickness [mm]	2.75±0.04	$2.52{\pm}0.04$

During loading images were acquired (frequency 2 Hz) using a LIMESS system. The images postprocessing allowed the measurement of the full field strain on the external surface of the specimen by the digital image correlation technique adopting the Vic-2D software. For this purpose the surface of the specimen for a length of about 30 mm was speckled with white and black acrylic paints.

AE sensors were placed in the un-speckled surface near the end tabs at a distance of 13 cm. Before starting the proper test, pencil test was carried out on each specimen.

The set up for the cracks development observation in the thickness consisted of a second Limess camera acquiring pictures at 2 Hz in the centre of the specimen length.

	CF/PPS	CF/PEEK
Fibres	Toray T300J	Toray T300J
Yarns	3K	3K
Fabric Density[g/m <sup>2</sup> ]	285	285
Plies	6	6
Fibre volume fraction [%]	57.6±1.8	50.8±1.7
Thickness [mm]	1.65±0.09	1.88±0.17

**Table 2.** The main characteristics of the thermoplastic composites.

**Table 3.** Software specification and sensors features for the acoustic emission recording.

Software	Vallen AMSY-5	
Amplifers	Vallen AEP4	
Amplification [dB]	34	
Discrimination time [ms]	0.4	
Rearm time [ms]	3.2	
Range [Mhz]	0.0025-1.6	
Sample rate [Mhz]	5	
Sensors	Vallen VS375S-M	
Sensor diameter [mm]	20	
Threshold [dB]	40	

## 3. Theoretical background of the AE clustering

The AE clustering technique adopted is detailed in [3] and [4]. Here a brief description is summarized for symbols and concepts.

The acoustic emission features considered here are: peak amplitude (A); duration (D); energy (E), i.e. the area under the voltage-time envelope; counts (CNTS), i.e. the comparator output pulses corresponding to the threshold crossings; rise time (R), i.e. the time interval from the first threshold crossing to the maximum amplitude; frequency centroid (FCOG); peak frequency (FMAX); weighted frequency value (WF); RA value, i.e. rise time divided by amplitude.

The statistically representative features for further analysis are selected considering two parameters: the Laplacian Score (LS), from an advanced variance analysis; the Correlation Coefficient (CC), it ranges from 0 to 1 and shows how features are correlated and dependent one with the other.

The features with the best LS and CC are used for the principal component analysis (PCA). PCA is an orthogonal linear transformation of multidimensional AE data into lower dimension (a new coordinate system) set of uncorrelated features that are the principal components.

The proper number of clusters was evaluated considering:

- the Silhouette coefficient (SC): it has a value between 0 and 1, the score is higher when clusters are dense and well separated
- the Davies-Bouldin index (DB): based on a ratio of within-cluster and between-cluster distances and it relates to the cluster centroids.

The best cluster quality has the lower DB index and the higher SC coefficient.

The clusters generation algorithm used in this work is the k-means++. This is a modified release of the k-means algorithm, based on an iterative algorithm in which a predefined number 'k' of centroids is spread throughout the data and the data samples are allocated to the closest centroid.

### 4. Results and comparison

Tensile failure tests were conducted for all type of composite materials to get the main mechanical properties in the load direction and the complete stress vs. strain, where strain was measured by DIC analysis as average in the centre of the speckled surface.

The average elastic modulus and failure stresses are compared in in Figure 1. Considering the different fibre volume fraction of the composites (see Table 1 and Table 2), properties in Figure 1 are normalized to the same fibre volume fraction which was arbitrarily taken as 50% ( $\overline{E} = E 50/V_f$ ,  $\overline{\sigma}_u = \sigma_u 50/V_f$ ).

Cumulative energy curves of the acoustic emissions showed that the thermosets materials get a plateau close to the maximum stress, in particular the GF/epoxy, implying that damage continuously develops in the complete loading history (Figure 2a,b).

On the contrary cumulative energy curves of the thermoplastic reinforced composites revealed a plateau reached very quickly during loading meaning that the major part of damage occurs in the low strain range, see in Figure 2c,d.

Same understand is processing the AE results to determine: the threshold strain  $\varepsilon$ min, for which relatively low energy acoustic events start to occur; the first damage threshold strain  $\varepsilon$ 1 at the first increase of the slope of the cumulative AE energy curve; the second damage threshold strain  $\varepsilon$ 2 at the second "knee" on the AE cumulative energy curve. The average values of the thresholds are compared in Figure 3.

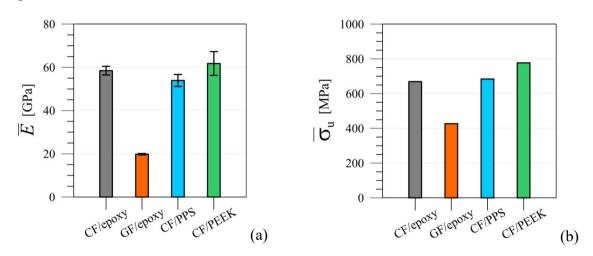


Figure 1. Normalized (a) average elastic modulus and (b) average failure stress. Bars give the standard deviation of 3 tests.

Cluster analysis revealed similar results for all materials. From the Laplacian score and the correlation coefficients, the features selected for the principal components analysis were A, RA, FMAX and FCOG. Two principal components were chosen having 3 as the ideal number of clusters.

The adopted  $k^{++}$  mean algorithm generates three clusters represented in the A vs. FMAX and A vs. FCOG space, being A the main component of the first principal component and FMAX and FCOG the main components of the second principal component. Those diagrams showed that the best cluster division is in the A vs. FCOG diagram, as also reported in literature [5].

For the thermoset resin reinforced composites (GF/epoxy and CF/epoxy), three separated cluster were observed (Figure 4a,b): CL1, at low amplitudes and low frequencies, CL2, at high amplitudes and low frequencies, and CL3 at high frequencies. Their separation along A axis is similar to those reported in literature for the similar materials assuming the same cluster analysis [5].

For the thermoplastic resin reinforced composites, similar AE monitoring and investigations were not found in the literature, therefore comparable results are not available.

Diagrams for CF/PPS and CF/PEEK composites show a clear division of three clusters in the domain of A – FCOG (Figure 4c,d), as for the thermoset materials.

The clusters separation occurred at slightly higher amplitudes for CF/PPS and CF/PEEK compared to CF/epoxy and GF/epoxy, but higher frequency boundaries were recorded for CF/PPS (Figure 4d).

Similar values of separation of A and FCOG for CF/epoxy and GF/epoxy revealed that the matrix is mainly responsible for distinction levels of the clusters.

Thus, according to literature CL1 was assigned to matrix transverse cracking, CL2 to matrix and fibres deboning and CL3 to fibres breakage. Having this scheme in mind, the next step was to assess the correlation of cluster CL1 with the actual transverse cracks observed on the lateral surface of specimens. Unfortunately due to the very sensible experimental setup and to the intrinsic nature of the specimens themselves, the only clear and useful observations of cracks were for CF/PPS specimens (see Figure 5).

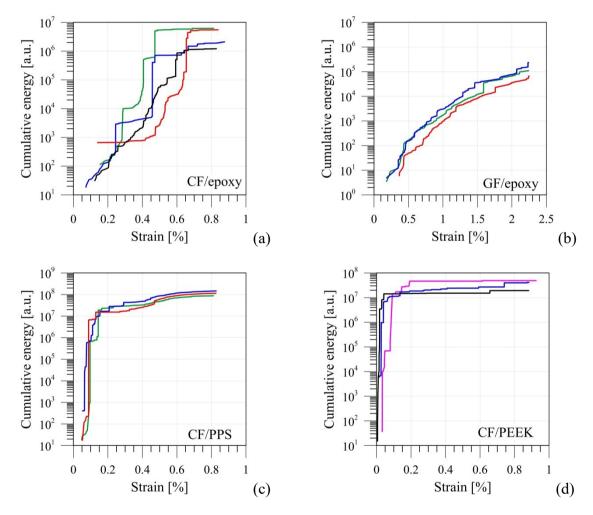


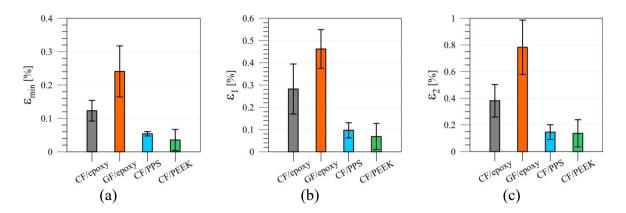
Figure 2. Some representative cumulative energy of the acoustic emission events vs. strain for: (a) CF/epoxy; (b) GF/epoxy; (c) CF/PPS; (d) CF/PEEK.

For the sake of correlation, cluster analysis was carried out for three specimens of CF/PPS only for acoustic emissions coming from the analysed lateral surface portion according to pencil tests and considering an uncertainty of 2 mm on both sides as literature suggests [5].

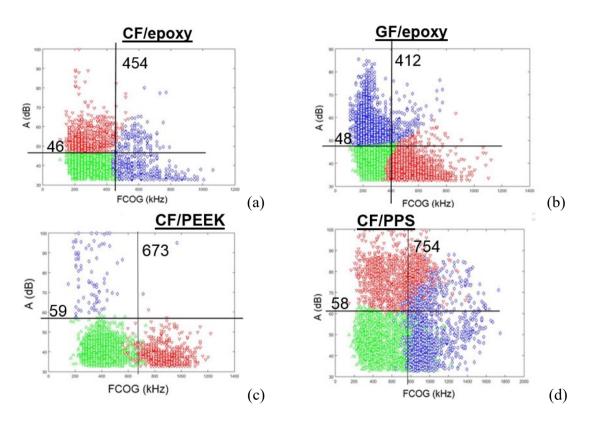
The results of cluster analysis on the small region under observation (Figure 6a,b) were similar to those including all signals presented above (see Figure 4). The only difference with the latter was that the clear three clusters division was in the A - FMAX domain.

Excerpt from ISBN 978-3-00-053387-7

ECCM17 - 17<sup>th</sup> European Conference on Composite Materials Munich, Germany, 26-30<sup>th</sup> June 2016



**Figure 3.** Average strain thresholds: (a)  $\varepsilon_{\min}$ , (b)  $\varepsilon_1$ , (c)  $\varepsilon_2$ . Bars give the standard deviation.



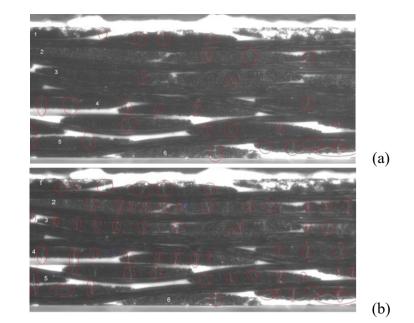
**Figure 4.** Representative cluster diagrams in the A vs. FCOG space for: (a) CF/epoxy; (b) GF/epoxy; (c) CF/PPS; (d) CF/PEEK. The values highlighted are the average value of divisions among clusters.

Cumulative curves were computed considering the cumulative number of acoustic emissions belonging to CL1 and the cumulative number of new cracks counted every strain increment of 0.1%. Being the observation region smaller than the one used for pencil tests, the number of new cracks counted was always smaller than the number of AE events.

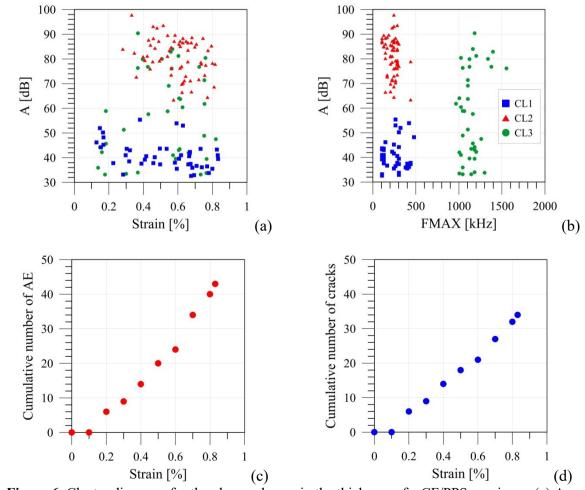
Results for CF/PPS specimens showed a good correlation between the cumulative AE in CL1 and the cumulative number of new cracks at the same strain levels, having both curves similar shapes and being proportional to each other (see Figure 6c,d).

The analysis also revealed that CL2 and CL3 clusters cannot be correlated to the cumulative number of new cracks having in some strain intervals more new cracks than AE events belonging to the two clusters. This is physically meaningless.

Thus it is concluded that for CF/PPS specimens CL1 is surely correlated to transverse matrix cracking.



**Figure 5.** Images of cracks on the thickness of a representative CF/PPS specimen. Plies are numbered and cracks are highlighted on the first (a) and the last (b) image.



**Figure 6.** Cluster diagrams for the observed zone in the thickness of a CF/PPS specimen: (a) A vs. strain and (b) A vs. FMAX. (c) AE events in the CL1 vs. strain and (d) the cumulative number of new cracks vs. strain.

A. D'Ettorre, V. Carvelli and S.V. Lomov

## 6. Conclusions

In this work, the AE cluster analysis methodology, previously used in the literature, was adopted to investigate the peculiarities of the AE evolution and clustering in thermoset and thermoplastic reinforced 2D carbon and glass textiles.

One thermoset (epoxy) and two thermoplastic (Polyphenylenesulphide, PPS, and Polyetheretherketone, PEEK) resins were considered.

The epoxy was reinforced with balanced twill 2x2 carbon fabric and balanced twill 2x2 glass fabric. The PPS and the PEEK were reinforced with carbon plain weave textile.

The comparison of the cumulative energy of AE events show that the thermoplastics reinforced composites have an early beginning of the damage and rapid saturation while a later beginning and more continuous growth of the damage was monitored in the textiles reinforced epoxy materials.

The cluster analyses highlighted that the thermoset and thermoplastic reinforced composites have a clear separation of the clusters.

The three different types of clusters can be subdivides as: CL1 low amplitude and low frequency; CL2 high amplitude and low frequency; CL3 high frequency.

The recorded limits of the three clusters are similar to the previously found in literature.

The crack monitoring in the thickness allowed the preliminary correlation of the transverse cracks in the PPS reinforced composite to the CL1 cluster containing AE events of low amplitude and low frequency.

## Acknowledgments

Lamiflex (Italy) and TenCate (The Netherlands) are acknowledged for manufacturing and supplying the thermoset and thermoplastic, respectively, composite plates adopted in this investigation.

## References

- [1] R. Gutkin, C. J. Green, A. Vangrattanachai, S. T. Pinho, P. Robinson and P. T. Curtis, "On acoustic emission for failure investigation in CFRP: Pattern recognition and peak frequency analyses," *Mechanical Systems and Signal Processing*, vol. 25, pp. 1393-1407, 2011.
- [2] S. V. Lomov, M. Karahan, A. Bogdanovich and I. Verpoest, "Monitoring of acoustic emission damage during tensile loading of 3D woven carbon/epoxy composites," *Textile Research Journal*, vol. 84, p. 1373–1384, 2014.
- [3] L. Li, S. V. Lomov and V. Carvelli, "Cluster analysis of acoustic emission signals for 2D and 3D woven glass/epoxy composites," *Composite Structures*, vol. 116, pp. 286-299, 2014.
- [4] L. Li, Y. Swolfs, I. Straumit, X. Yan and S. V. Lomov, "Cluster analysis of acoustic emission signals for 2D and 3D woven carbon fiber/epoxy composites," *Journal of Composite Materials*, 2015. Doi: 10.1177/0021998315597742.
- [5] L. Li, S. V. Lomov and X. Yan, "Correlation of acoustic emission with optically observed damage in a glass/epoxy woven laminate under tensile loading," *Composite Structures*, vol. 123, pp. 45-53, 2015.