AE MONITORING OF MECHANICAL TESTS ON PLANT FIBER COMPOSITES

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Abstract

Recently, composites including plant fibers are increasingly used for large-volume applications, aimed at obtaining a more bio-degradable material than fiberglass. However, serious concerns are still present on the level of mechanical performance that can be achieved with these materials. In this regard, applying acoustic emission (AE) to monitor monotonic mechanical loading, such as during tensile and flexural tests, can provide useful information.

In this work, three case studies on AE monitoring of mechanical tests of plant-fiber composites, performed over the last couple of years in our laboratory, are discussed. The fibers used for composites reinforcement are jute (case 1), celery (case 2) and phormium (case 3). As a whole, AE data analysis can yield some indications about the effectiveness of reinforcement, the prevalent mode of failure and the selection of different configurations, to obtain the best mechanical performance. In particular, an extensive use of 3-D data plotting including events localization and amplitude or duration allowed clarifying all the aforementioned aspects. This contributes to the selection of fibers and laminates for the development of plant-fiber composites as an alternative to fiberglass.

Introduction

Plant-fiber composites and hybrid laminates including plant fibers are increasingly used for semi-structural applications, as they show a better end-of-life profile, being intrinsically carbon-dioxide neutral [1]. However, the mechanical behavior of plant fibers as reinforcement is not easily predicted. In particular, plant fibers are inhomogeneous, being cellular structures assembled in nature through a hierarchical procedure, and present a hollow, or lumen, of variable dimensions [2]. In addition, their introduction in a polymer matrix may generate compatibility issues, whose consequence may be a large scattering of properties in the final laminate [3].

Real-time monitoring of mechanical tests is a classical application of AE, which has been hardly ever applied on plant fiber composites, e.g., to contribute in material characterization during post-impact loading [4] or to offer further indications on failure mode during tensile or flexural testing [5]. The results were able, e.g., to confirm concerns about scarce impact resistance of these materials [6] and the difficulty in obtaining predictive indications about materials performance, which suggest that specific tools of analysis of AE data would need to be disposed for these materials [7].

In this paper, three case studies of the application of AE monitoring to the characterization of plant-fiber composites are presented as suggestive examples indicating a wide range of problems encountered in applying these materials in engineering practice.

Materials and Test Methods

The experimental characterization was carried out on three different natural-fiber-reinforced composites. Tests were referred respectively as Case 1, 2 and 3. Laminates used in Case 1 consisted of hybrid composites reinforced with glass and jute fibers. In particular, jute/glass fiber hybrid laminates were manufactured using an RTM procedure. Plain-woven jute fabric (300 g/m^2) and E-glass (Vetrotex VR38, 290 g/ m^2) were used as reinforcement. The resin was unsaturated polyester (1629 NT from Lonza). Two different configurations (labelled as Q and T) were obtained, whose stacking sequences and characteristics are summarized in Table 1. From the plates, four-point bend specimens were obtained with 150-mm length, 30-mm width, and 5(±0.2)-mm thickness. Specimens from T and Q laminates were impacted and then subjected to post-impact four-point bending tests. The impact point was located at the centre of the specimens. The impact energy was changed varying the mass of the hemispherical drop-weight striker (OD: 12.7 mm), which has a constant velocity of 2.5 m/s. Impact tests were performed on an instrumented impact tower fitted with an anti-rebound device. Four different impact energies were considered: 5, 10, 12.5 and 15 J. The flexural tests were carried out in accordance with ASTM D-790 using quarter-point loading configuration. These tests were performed in a universal testing machine (Zwick Roell Z010) with a support span length of 140 mm and a crosshead speed of 5 mm/min. The strain at the mid-span was determined by means of strain gauges. Five specimens were tested for each configuration (T and Q) and for each impact energy value, as well as for non-impacted specimens, which served as reference materials. Post-impact flexural tests were monitored by AE until final fracture occurred using an AMSY-5 AE system by Vallen Systeme GmbH. The AE acquisition settings used throughout were as follow: threshold = 35 dB, RT (re-arm time) = 0.4 ms, DDT (duration discrimination time) = 0.2 ms and total gain = 34 dB. Four PZT AE sensors resonant at 150 kHz were used: two sensors were placed on the surface of the specimens at both ends to allow linear localization, while the other two sensors were used as guard sensors in order to discriminate between AE signals and noise. Post-impact flexural and AE test results are summarized in Table 2.

Table 1. Summary of laminate configuration (case 1).

Sample name	Stacking Sequence (G = Glass; J = Jute)	Number of Jute Layers	Number of Glass Layers	Overall Fibre Content [% vol]
Q	4G/1J/2G/1J/2G/1J/2G/1J/4G	4	14	50±2
T	7G/4J/7G	4	14	50±2

Table 2. Post-impact flexural strength and AE cumulative counts "knee" stress for Q and T glass/jute hybrids impacted at different energies.

Specimens	*Flexural Strength (MPa)*	*σ_c (MPa)*
Q_{0J}	209.91±0.37	93.66±3.12
Q_{5J}	172.89±7.18	84.92±9.95
Q_{10J}	160.42±8.98	75.40±7.45
$Q_{12.5J}$	157.08±10.42	71.07±0.99
Q_{15J}	154.57±3.31	72.50±10.34
T_{0J}	269.34±15.47	90.33±6.43
T_{5J}	244.13±0.69	82.64±4.73
T_{10J}	227.43±14.87	79.85±3.11
$T_{12.5J}$	212.76±14.20	80.61±2.56
T_{15J}	195.04±18.49	54.50±10.13

In Case 2, celery-fiber reinforced composites were tested. Natural untreated fibers from local celery (*Apium graveolens*) and P25 epoxy system produced by REA Industries were used. The average length of the fibers was 10 cm and they were randomly arranged like a mat. The composites were manufactured using the hand lay-up process in a closed aluminum mould. The curing cycle was 7 days at room temperature and under slight pressurization followed by 48 h at 40°C. The fiber volume fraction was $V_f \sim 0.17 \pm 0.01$, corresponding to about 10 wt%. Five tensile samples having a length of 230 mm, a width of 20 mm and a thickness of 3.5 (±0.2) mm were obtained and tested. In addition, five tensile samples of neat epoxy resin were manufactured to evaluate the effect of fiber introduction on the composite properties. The mechanical characterization of the composites was conducted by longitudinal tension tests (ASTM D-3039) using an Instron 5584 test machine. The crosshead speed for tensile tests was 0.5 mm/min. All the specimens tested were equipped with strain gauges to determine the Young's modulus. Tensile tests were monitored by AE using the same equipment, set-up and parameters previously described.

In Case 3, untreated *Phormium tenax* (New Zealand flax, harakeke)-reinforced epoxy composites were tested. The matrix was an epoxy resin (Ampreg 26) by SP systems. The fibers were cut to a length of approximately 2 cm and were randomly arranged in the final composite. The specimens were manufactured using the hand lay-up process in a closed aluminum mould. The curing cycle was 20 days at room temperature. The final fiber content was 20 wt%. Five specimens were tested in flexure (200 mm x 30 mm x 4.5±0.2 mm). For comparison purposes, five specimens of neat resin were tested. Flexural tests were performed in accordance with ASTM D-790 using a three-point loading configuration. These tests were performed using an Instron 5584 test machine at a constant crosshead speed of 2.5 mm/min. Span-to-thickness ratio in these three-point bending tests was 20:1. The strain at midpoint was determined by means of strain gauges. Flexural tests were monitored by AE using the same equipment, set-up and parameters previously described.

Results

In Case 1, the idea was to demonstrate which one of the hybrid configurations, T (sandwich hybrid) or Q (intercalated hybrid), both with the same amount of plant fibers and therefore with a similar reduction in weight, would prove more suitable to sustain the application of load. Flexural tests carried out on both configurations demonstrated that T hybrids are superior to Q hybrids at all impact energies by more than 20% both in strength and modulus [8]. AE analysis was focused on the measurement of the cumulative counts curve "knee": the stress value associated with the change in slope of the above curve was referred to as σ_c and was supposed to be an acceptable indication of the upper limit for materials service. Values of σ_c were comparable both for T and Q configuration, representing in general about 30-40% of the maximum strength. However, in the specific case of flexural tests after 15-J impact, which was the highest impact energy applied, T configuration showed a much lower value of σ_c than Q configuration (54.5±10.1 MPa vs. 72.5±10.3 MPa). It is suggested that the intercalated layers of glass-fiber composite in Q hybrids offer more gradual degradation to the material with increasing impact energy. In contrast, on T hybrids, the mechanism of failure is dominated by the breakage of jute-fiber composite core, which takes place by abrupt tearing off of the fibers from the matrix, as noticed elsewhere [9].

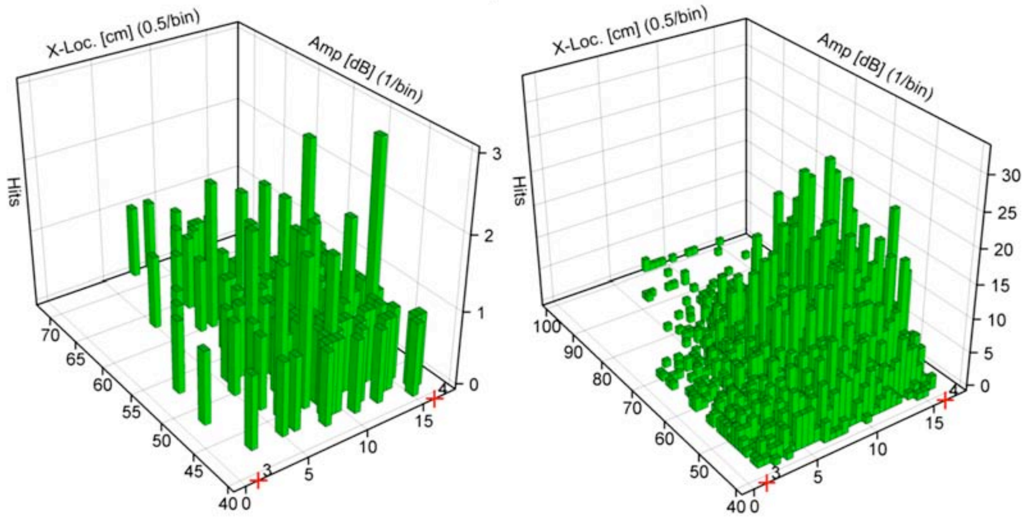

Fig. 1. AE localization plots vs. amplitude for Q hybrids taken at σ_c (left) and at failure (right).

Further characterization on laminates impacted at 15 J, shown in Fig. 1 for Q hybrids and in Fig. 2 for T hybrids, did indicate that AE events are more concentrated in T hybrids than in Q hybrids, although damage appears to be more precocious in Q hybrids, as shown by localization plots taken at σ_c. AE data confirm therefore the effectiveness of intercalated hybrids in dispersing impact damage close to the penetration site.

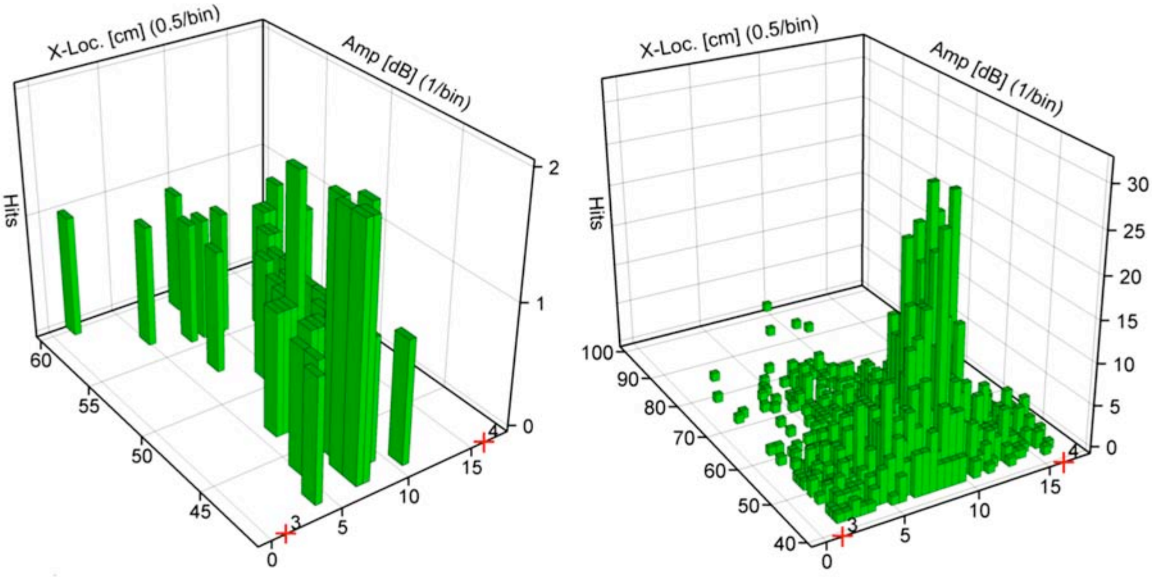

Fig. 2. AE localization plots vs. amplitude for T hybrids taken at σ_c (left) and at failure (right).

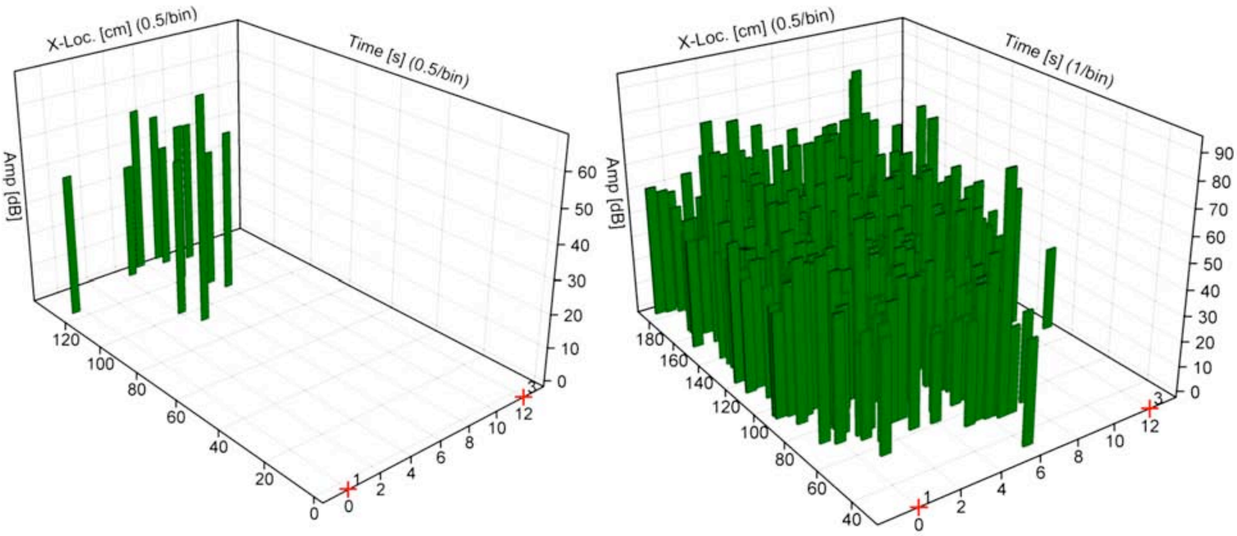

Fig. 3. AE localization plots vs. amplitude at failure for neat epoxy resin (a, left) and for 10% wt. celery/epoxy (b, right).

In Case 2, the introduction of celery fibers in a polymer matrix was first attempted. In this case, it could be observed that the largest part of localized events is connected with the presence of fibers. This can be shown by comparison between Fig. 3a, showing AE events for the neat resin and Fig. 3b, showing AE events for the resin with 10% wt. celery fibers. An issue. which was encountered in fiber introduction due to their variable diameter, is that it was not possible to obtain a balanced random orientation of fibers in the matrix. As a consequence, resin-rich areas and conversely areas with higher fiber content are heavily present, which affected the mechanical properties of the laminate [10]. In particular, the tensile strength and modulus of the neat-resin samples were 29.1±3.9 MPa and 3.45±0.1 GPa, respectively, whilst the tensile strength and modulus of the celery/epoxy laminates were 18.67±0.60 MPa and 3.3±0.17 GPa, respectively.

The poor mechanical performance of the composite indicates that the introduction of fibers does not result in effective reinforcement. This would cause the composite to break because of concentration and accumulation of damage in a local region of the sample, ideally situated at the center of the gauge length [11]. In contrast, no preferential localization of AE events and hardly any high amplitude event are revealed from Fig. 3b, even at stress close to failure: it may be suggested that fibers tend to pull out and break prematurely.

In Case 3, phormium, which proved to be a particularly strong and elastic fiber with constant geometrical properties [12], was introduced in a polymer matrix. However, this fiber showed an elastic behavior up to a stress close to failure. In this sense, the measurement of σ_c, as suggested above, did not allow obtaining a suitable method to calculate a value for the maximum load in material service. In particular, this happened because the value obtained was hardly predictive, being too close to the ultimate load: this is likely to be due to the scarce deviation from elasticity of the behavior of phormium fibers. However, alternative parameters to cumulative counts, which could provide indications of critical damage development, were searched for, in order to confirm σ_c as the limit for material use, or suggest a lower value for it.

A different parameter, mean-amplitude hit frequency or MAHF, defined as the *average amplitude in a time interval multiplied by the fraction of hits in that interval with respect to the total hits number*, plotted against time, gives in a repeatable way on phormium-fiber composites a limit for material use which is consistently inferior to that given by $\sigma_{c,}$ as described in Fig. 4. The perceived advantage of MAHF with respect to σ_c is that it is sensitive to the increase in average amplitude of the hits that may precede the cumulative counts "knee".

For the particular sample selected in Fig. 4, the ultimate flexural strength was 71.85 MPa, σ_c was 67 MPa, whilst MAHF was only 59.26 MPa, as indicated by the two circles on the curve in Fig. 4. This indicated that, in spite of the perceived macroscopic elasticity of the composite almost up to failure, increasing average amplitude of detected hits appears to indicate the presence of significant damage at a lower stress than σ_c. In our opinion, this suggests that MAHF offers an additional opportunity for AE analysis to characterize the behavior during quasi-static tests in composites not showing appreciable plastic deformation, because of the nature of the reinforcement used.

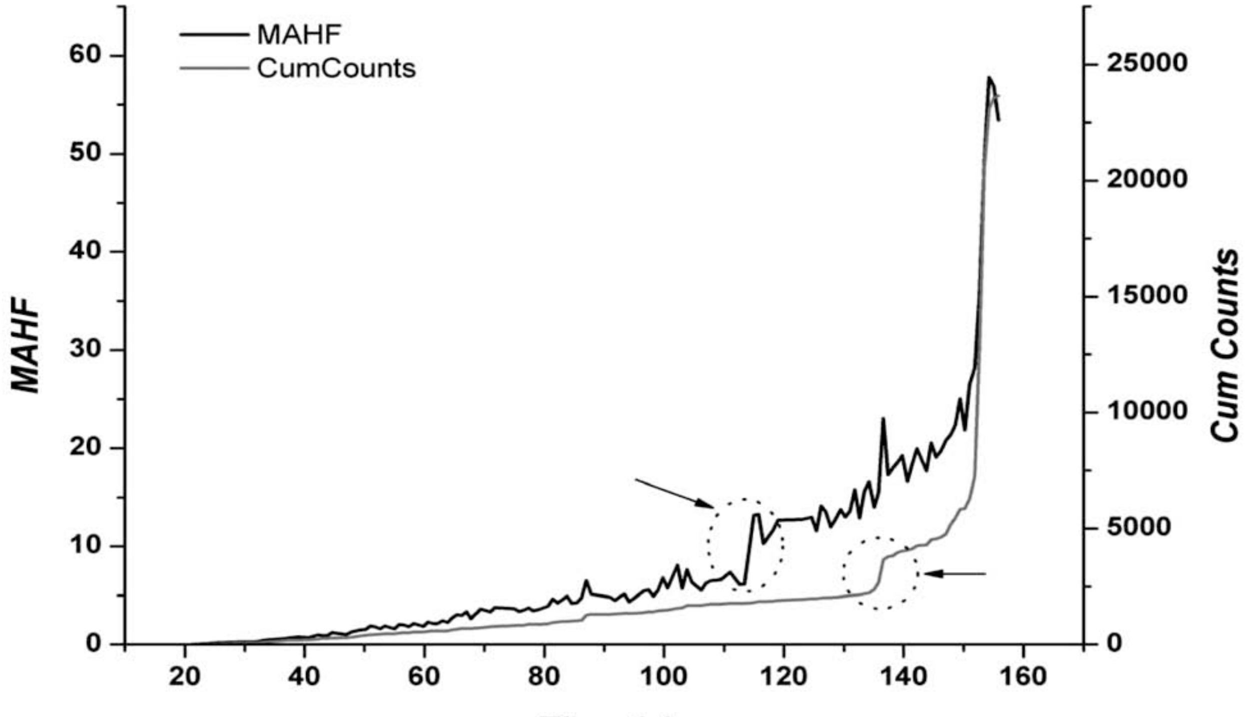

Fig. 4. MAHF and cumulative counts vs. time for phormium-reinforced epoxy composites.

Conclusions

AE proved suitable to analyze the mechanical behavior of plant-fiber-reinforced composites. These materials, which are increasingly used for environmental reasons, are not easily characterized by other methods, because of the inherent variability in fiber properties, and the not-always-effective fiber/matrix compatibility, which leads to an absence of warnings as regards materials failure. The three case studies reported, focused on jute, celery and phormium fiber composites, respectively, indicate that AE analysis centered on amplitude distribution can offer some indications on the mode of failure and the maximum service load for these materials.

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