ACOUSTIC EMISSION ANALYSIS FOR QUANTITATIVE CHARACTERISATION OF THE DAMAGE BEHAVIOUR IN FIBRE REINFORCED COMPOSITES UNDER STATIC AND DYNAMIC LOADING

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Abstract

In this study the damage behaviour of glass and carbon fibre reinforced composites are investigated. Epoxy and polyurethane matrix based unidirectional fibre reinforced laminates, manufactured by VARTM-process, are loaded with static and dynamic tensile forces. Acoustic emission (AE) analysis is used to investigate the microscopic damage mechanisms and damage progress in these FRPs taking into account the whole sample volume. In case of static loading, the influence of fibre orientation on damage initiation and propagation is determined as well. Results show that the use of a novel polyurethane matrix system significantly enhances the materials performance in terms of load levels of crack initiation, crack growth, damage tolerance and off-axis tensile strength. Hysteresis measurements during stepwise increasing dynamic load tests reflect the overall damage situation in the material. It is shown that the beginning of fibre breakage correlates with a significant increase of loss work per cycle. Different damage behaviours observed by AE analysis are correlated with scanning electron micrographs of the fracture surfaces.

1 Introduction

In the last decades the use of fibre reinforced plastics (FRP) in engineering applications has dramatically increased. The biggest advantage of FRPs is their superior specific strength and stiffness. Starting in space and aeronautic industries, FRPs are nowadays used for lightweight structures in automotive, as well as marine and wind turbine industry.

For the design of FRP structures, the knowledge of the damage behaviour is essential to prevent failure during service life. Generally, mechanical testing methods are used to investigate the performance and failure characteristics of FRP. Unfortunately most of the static testing methods only provide information about the final failure without giving an insight in the initiation and the propagation of damage. To overcome this limitation, acoustic emission (AE) combined with frequency analysis and pattern recognition techniques is a promising approach. By the use of AE analysis crack initiation and propagation can be detected online during mechanical testing. Based on the frequency composition of acoustic signals, different damage mechanisms as matrix cracking, interface failure and fibre breakage are distinguishable even under dynamic loading.

First AE analysis in the field of fibre reinforced composites was done in the 1970s [1-4]. Activities were expanded in the 1980s but analysis was focused on the detection of damage onset, fracture activity and intensity. Correlations between the acoustic signals and fracture mechanisms as matrix cracking, fibre breakage and interphase failure were not possible due to insufficient knowledge about the physical backgrounds and inapplicable analysis techniques. The identification of the different microscopic damage mechanisms succeeded in the mid 1990 by means of the determination of the maximum in the frequency spectrum of the AE signals. Matrix cracks show the lowest, interphase failure a higher and fibre breakage the highest peak frequency [5, 6]. Further investigations showed that in general - in addition to the peak frequency - the entire frequency composition of an AE signal is characteristic for the respective failure mechanisms. The characteristic frequency spectra can be attributed to density and stiffness of the materials involved [7, 8]. Furthermore, the application of pattern recognition techniques helps to improve the validity of AE analysis. It is useful to combine several frequency-based features for the identification of various failure mechanisms. Fig. 1 shows typical frequency spectra for the respective damage mechanisms according to the classification results of their frequency-based features.

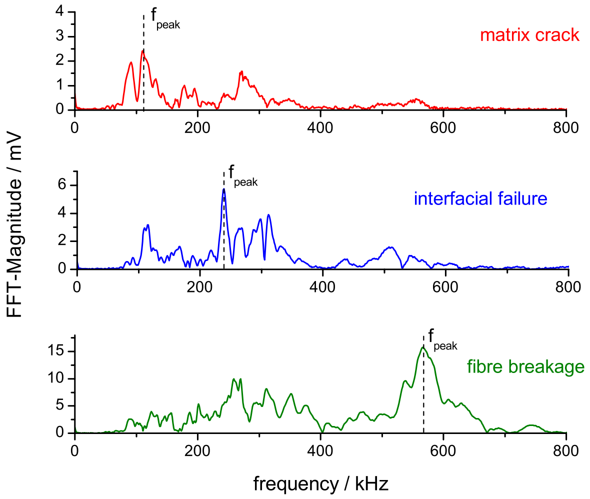

Figure 1. Typical frequency spectra of matrix cracking, interfacial failure and fibre breakage

2 Experimental

This study focuses on the microscopic failure mechanisms of glass and carbon fibre reinforced composites under quasi-static as well as under dynamic loading. State of the art mechanical testing of composites has only limited explanatory power regarding the damage evolution until final failure. In particular it is not possible to determine the load levels at which first micro cracking occurs within the material. Furthermore, it is not clear which components are getting damaged and how crack propagation develops. The investigation of different fibre and matrix combinations with acoustic emission analysis during mechanical testing allows to reveal basic structure-properties-relationships concerning fibre-matrix interaction in composite materials. The combination of AE analysis with static and dynamic testing of fibre reinforced composites allows to establish a fundamental understanding of their failure behaviour by detailed analysis of the microscopic damage mechanisms.

2.1 Materials

Matrix systems used were a two part standard epoxy/amine infusion resin EPR L 1100 + EPH 294 (‘*EP*‘) from MOMENTIVE and a novel thermosetting polyurethane formulation provided by Henkel AG & Co. KGaA (‘*PU*‘). Glass fibre (‘*GF*‘) reinforced laminates were made of unidirectional SAERTEX Non Crimp Fabric (NCF) (E-Glass) with an areal weight of 701 g/m^2. The carbon fibre reinforcement ('*CF*') was a SAERTEX unidirectional HTS carbon fibre NCF with 244 g/m^2.

2.2 Processing and sample preparation

The unidirectional glass and carbon fibre reinforced laminates were manufactured by VARTM-process. Laminate thickness of 2 mm corresponds in both cases - GFRP and CRFP laminates - to fibre volume contents of about 55 %. The pre-cut dry textiles were placed in an aluminium RTM-tool, which is afterwards clamped together and heated in a hydraulic hot press. Before injection, the two-part resin systems were stirred in a laboratory mixer and degassed after being homogenously mixed. A curing cycle of four hours at 90 °C was chosen for both resin systems. Quality assurance was done by visual inspection for the GFRP laminates and with ultrasonic C-scans for the CFRP laminates. The tensile testing samples were prepared with end tabs according to DIN EN 527-5 [9] and cut out from the laminates with a circular diamond saw. The sample width of 20 mm was chosen for proper attachment of the piezo AE sensors. From the unidirectional reinforced laminates samples were prepared with fibre orientations between 0° and 90° to the loading direction.

2.3 Static testing

Static tensile tests were conducted in a Zwick 1475 universal testing machine with hydraulic clamping fixtures. Crosshead speed of 0.5 mm/min was chosen for a better differentiation of the single AE signals. Strain measurement was done with an extensometer (Fig. 2). To collect acoustic signals during testing two AE sensors were clamped to the specimens with silicon grease as coupling medium. Testing conditions were 23 °C and 50 % relative humidity.

Figure 2. Static testing setup. Two AE sensors are clamped to the sample on defined positions. Strain is measured by an extensometer.

2.4 Dynamic testing

Dynamic testing was carried out in an Instron Schenk IPLH50K servo hydraulic testing machine under laboratory conditions (23 °C and 50 % r.h.). The tension-tension fatigue tests were performed as stepwise increasing load tests at 1 Hz testing frequency with stress controlled sinusoidal loading and a stress ratio of $R = 0.1$. Strain was measured by means of piston displacement of the servo hydraulic testing machine. First load level was at maximum stress per cycle of 100 MPa. For the following load levels the maximum stress was increased by 100 MPa each level. Recovery levels at 100 MPa maximum stress after each stepwise increasing load level allow comparing the specimens condition in terms of dynamic modulus and loss work with their initial state at 100 MPa maximum stress. Cycle number of the load levels was 5000, of the recovery levels 1250.

2.5 Acoustic emission setup

Acoustic emission signals were detected with a PCI-2 AE-System and AEWin software from Physical Acoustics. Two AE sensors are used in case of static loading (Fig. 2) for filtering noise signals from outside of the sample volume. Only signals, which could be localized between the two AE sensors, were recorded. For static testing the sensitivity threshold was 36 dB, sampling rate 10 MHz and frequency range was restricted to 100 kHz – 1000 kHz.
For dynamic testing no AE signal localisation was carried out and the sensitivity threshold was increased to 60 dB due to continuous noise signals from hydraulic actuation of the dynamic testing machine. Noesis Software was used for post processing and classification of the recorded AE signals. Pattern recognition techniques were applied for the AE signal classification in terms of damage mechanisms. The features which the classification was based on are listed in table 1.

Feature	**Definition**
Peak Frequency	Maximum of the frequency spectrum
Weighted Peak Frequency	$f_{WPF} = \sqrt{f_{peak} \cdot f_{centroid}}$
Partial Power 1	0 – 250 kHz (fraction of frequency spectrum)
Partial Power 2	250 – 450 kHz
Partial Power 3	450 – 800 kHz

Table 1. Frequency based signal classification features

3 Results and Discussion

3.1 Static testing

Under quasi-static loading parallel to the fibre orientation the composites properties are dominated mainly by the fibre properties. The stress-strain diagrams of *glass fibre reinforced EP* and *PU* matrices show almost equal course (Fig. 3). In both cases the first damage mechanism detected by AE analysis is interfacial failure followed by fibre breakage. In the epoxy based composite first matrix cracking is detected earlier than in its *PU* counterpart. Due to the lower fracture toughness of the *EP* system (K_{Ic} = 0.75 MPa√m) cracks starting from the interphase grow faster in the resin rich regions. In case of the *PU* system with its high fracture toughness of around 1.2 MPa√m matrix cracking starts later and less signals are detected until final failure. Matrix cracks can also induce fibre breakage [10], which are not immediately critical in terms of total failure of the UD ply [11]. This under-critical fibre breakage until around 700 – 800 MPa tensile stress is more pronounced in the brittle epoxy composite.

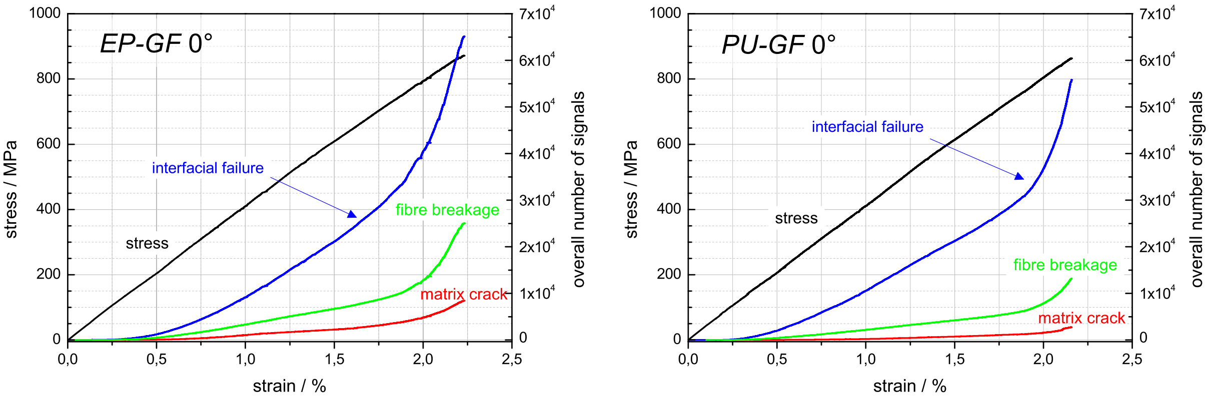

Figure 3. Quasi-static 0° tensile testing stress-strain diagrams with corresponding AE signals for glass fibre reinforced *EP* (left) and *PU* (right).

The beginning of the final failure can be observed in both systems at around 800 MPa when the rate of detected interphase and fibre failure signals significantly increases.
Off-axis tensile tests were performed to emphasise the characteristics and impact of the matrix system used on the composites` overall properties. Fig. 4 shows representative stress-strain diagrams and the corresponding AE signals obtained from tensile testing under a 20° angle between load direction and UD fibre orientation. Here the differences between both composites – *EP* and *PU* resin reinforced with the same glass fibre – are clearly visible. The 20° off-axis ultimate tensile strength of the epoxy based composite is 134 ± 1 MPa. First damages within the material are detected already at around 84 MPa tensile stress. Remarkably, the first significant acoustic signals of *PU-GF* are detected at around 135 MPa. At this load level the *EP-GF* composite already failed (Fig. 4).

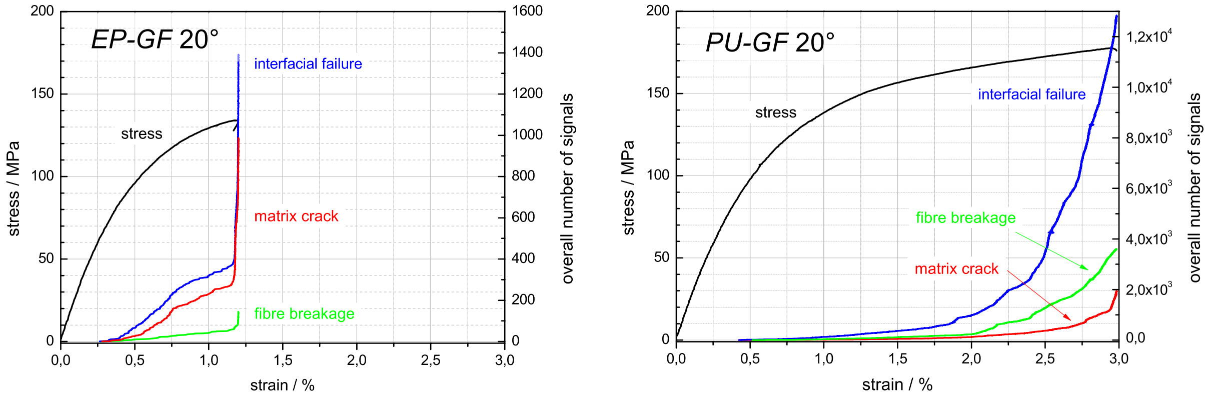

Figure 4. 20° off axis tensile testing. The *PU* matrix (right) significantly enhances the composites damage resistance.

The low amount of fibre breakage until final failure in *EP-GF* indicates minor load transfer from the matrix into the fibres. The small overall number of acoustic signals detected before final failure is another hint for weak interphase. This is confirmed by SEM pictures, which show very smooth and even interphase fracture surface of *EP-GF*. The *PU-GF* fracture surfaces are structured and show more plastic matrix deformation (Fig. 5) Random fibre matrix debonding in *EP-GF* leads to unstable propagation of few cracks and localized sudden specimen failure (Fig. 4). In contrast, *PU-GF* emitted an around one order of magnitude higher overall number of interfacial failure signals than *EP-GF*. This multiple interphase debonding and micro cracking (Fig. 6) indicates local stress relaxation and stable crack growth. Moreover, a considerable load transfer is still maintained to the fibres, which leads to pronounced fibre failure signals even though the 20° off-axis loading state.

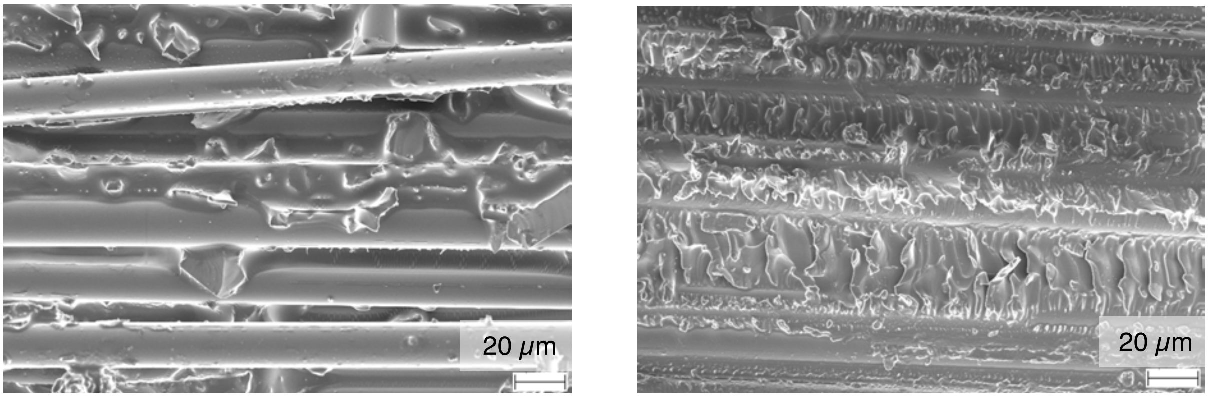

Figure 5. SEM fracture surface micrographs of *EP-GF* (left) and *PU-GF* (right)

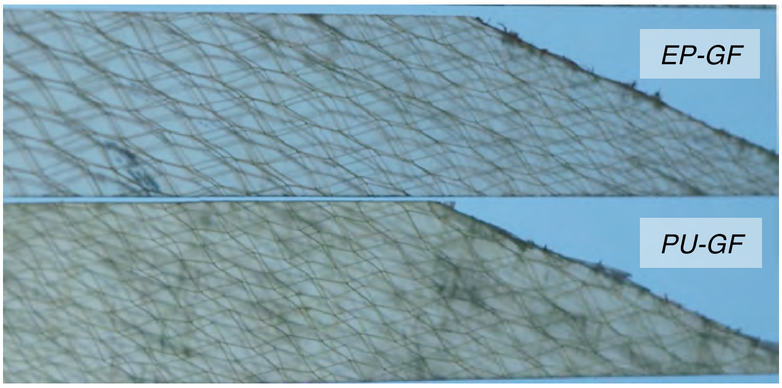

Figure 6. Visualization of the fracture behaviour from 20° of axis tensile testing samples. *PU-GF* shows opaque spots indicating multiple micro cracking before final failure.

The off-axis tensile performances of *carbon fibre reinforced EP* and *PU* are quite similar to the ones of their analogous composites with glass fibre reinforcement. Fibre-matrix adhesion is again stronger for *PU* in combination with carbon fibres. Therefore, the interfacial failure, which is again the first damage mechanism detected, begins much earlier in *EP-CF* and is followed immediately by fibre and matrix breakage. The overall number of acoustic signals until final breakdown is relatively low in *EP-CF*. Whereas *PU-CF* shows much higher damage tolerance and still a good load transfer into the carbon fibres, since more fibre breakage than matrix cracking is observed (Fig. 7).

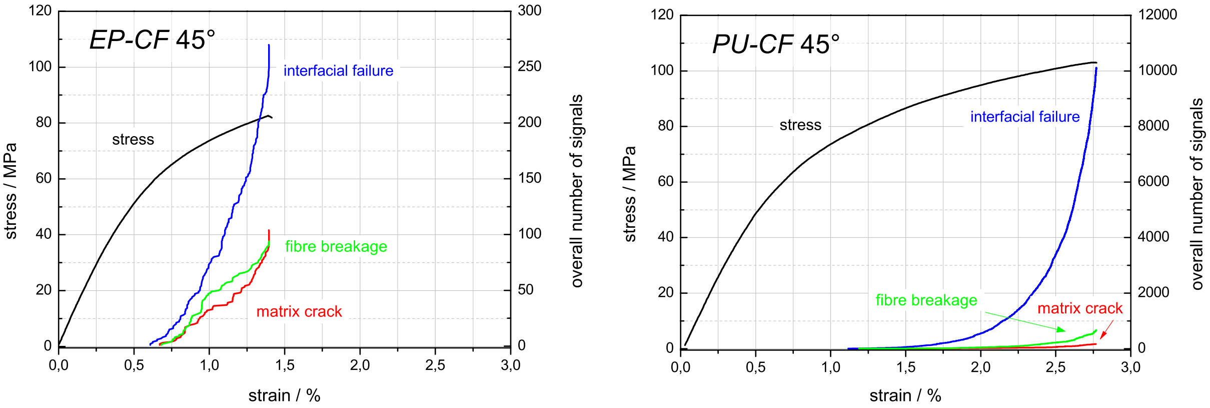

Figure 7: 45° off axis tensile testing with carbon fibre reinforcement. Early unstable interfacial failure initiates subsequent fibre and matrix cracks in *EP-CF*. The damage resistance of *PU-CF* leads to high numbers of recorded AE signals before failure.

3.2 Dynamic testing

The behaviour of *EP-GF* and *PU-GF* in 0° quasi-static testing is rather similar while the off axis tensile performance is significantly dependent on the interphase and matrix properties. Therefore dynamic 0° tensile-tensile testing was chosen to determine the influence of matrix properties on composite performance under cyclic loading. Hysteresis measurements were conducted to determine loss work during a single loading cycle, which corresponds to the energy dissipated in the material. Increasing loss work indicates proceeding material damage, but reveals no information about the involved damage mechanisms. Acoustic emission analysis has very sensitive detection capabilities for the onset of material damage. Moreover it allows differentiation between damage mechanisms in the material. For *EP-GF* significant damage onset is detected during the 300 MPa load level. In this case all three components of the composite are getting damaged. Most signals are matrix cracks, mainly recorded in the second half of this load level. As observed during static testing the first interfacial failures may induce the matrix crack propagation. The material already fails during the next (400 MPa) load level, which is around 46 % of the static tensile strength. The analysis of the acoustic signals shows steep increase of interfacial failure. On the one hand, interfacial failure cracks along the fibres do not reduce significantly composite strength, since the reinforcing fibres carry the load. But, on the other hand, these interfacial cracks prevent load transfer to other fibres when local fibre breakage occurs. In case of *EP-GF* interfacial failure is accompanied immediately by a strong increase in fibre breakage. Until final breakdown the global weakening of the structure due to failure of its reinforcing elements, the fibres, is observed by a dramatic increase in loss work. As can be seen from Fig. 8 the loss work is in good correlation to the increasing number of fibre breakage signals.

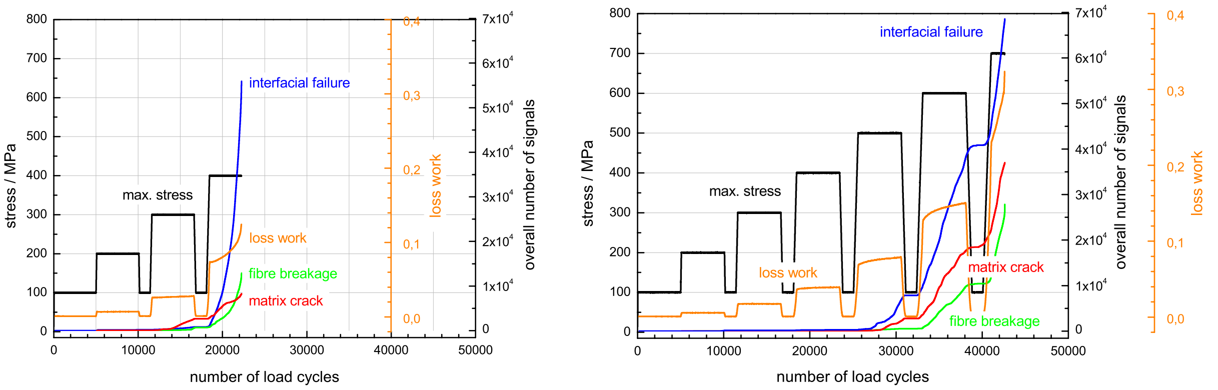

Figure 8. Stepwise increasing dynamic load tests. Black: maximum stress per cycle. Orange: Loss work.

The use of the novel *PU* matrix system significantly enhances the dynamic damage resistance of the glass fibre reinforced composites. First noteworthy acoustic damage signals are recorded at the 500 MPa load level. The *EP* based composite did not even reach this stage. Multiple interfacial failure is detected in *PU-GF* under quasi-static as well as under dynamic loading conditions. In contrast to *EP-GF* the interphase signal number increases more linearly. This again indicates stable crack growth. In addition to the clearly visible interphase and matrix signals also a small amount of fibre breakage is observed at the 500 MPa load level and induces a slight increase in loss work. More heavy fibre failure starts from the beginning of the 600 MPa stage. The material becomes more and more damaged but does not fail within 5000 load cycles thanks to good fibre matrix adhesion and high toughness. Local damage, in particular fibre failure is effectively bridged by the *PU-GF* composite and the stresses are transferred again into intact fibre parts. Material damage also shows up in the following recovery stage as the material has lower dynamic modulus. Damage accumulation continues at the 700 MPa load level and final failure is announced by a high amount of fibre breakage signals with corresponding increase in loss work.

4 Conclusion

Acoustic emission analysis was performed during static and dynamic tensile testing of fibre reinforced composites. Acoustic signals are emitted from the materials due to fibre-, matrix- and interphase-failure. The different interactions and damage behaviour of glass and carbon fibre in combination with epoxy and polyurethane matrices were investigated. It is shown that material damage always starts in the interphase. Dependent on interphase and matrix properties multiple stable and slow micro cracking is generated. This leads to damage resistant material behaviour. In case of unstable crack growth, initiated by interfacial failure, materials fail quite early under off-axis or dynamic loading. In high performance composite applications the materials always have to bear off-axis or dynamic loading. Therefore interphase and matrix properties are crucial for the composites` overall performance. Interphase quality plays a minor role only under 0° quasi-static loading. For other loading conditions interphase and matrix properties are at least as much as important as those of the fibres. The combination of mechanical and dynamic testing with online acoustic emission provides a powerful tool for the understanding and optimization of these basic structure-property-relationships and microscopic damage growth mechanisms in composite materials.

Acknowledgments

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References

[1] Fitz-Randolph J., Phillips D.C., Beaumont P.W.R., Tetelman A.S. The fracture energy and acoustic emission of a boron-epoxy composite. *Journal of Materials Science,* **7**, pp. 289-294 (1972).

[2] Rotem A., Baruch J. Determining the load-time history of fibre composite materials by acoustic emission. *Journal of Materials Science,* **9**, pp. 1789-1796 (1974).

[3] Carlyle J. Imminent fracture detection in graphite/epoxy using acoustic emission. *Experimental Mechanics*, **18**, pp. 191-195 (1978).

[4] Baram J., Rosen M. Fatigue life prediction by distribution analysis of acoustic emission signals. *Materials Science and Engineering*, **41**, pp. 25-30 (1979).

[5] Gutkin R., Green C.J., Vangrattanachai S., Pinho S.T., Robinson P., Curtis P.T. On acoustic emission for failure investigation in CFRP: Pattern recognition and peak frequency analyses. *Mechanical Systems and Signal Processing,* **25**, pp. 1393-1407 (2011).

[6] Ramirez-Jimenez C.R., Papadakis N., Reynolds N., Gan T.H., Purnell P., Pharaoh M. Identification of failure modes in glass/polypropylene composites by means of the primary frequency content of the acoustic emission event. *Composites Science and Technology*, **64**, pp. 1819-1827 (2004).

[7] Sause M., Horn S. Simulation of acoustic emission in planar carbon fiber reinforced plastic specimens. *Journal of Nondestructive Evaluation*, **29**, pp. 123-142 (2010).

[8] Giordano M., Condelli L., Nicolais L. Acoustic emission wave propagation in a viscoelastic plate. *Composites Science and Technology*, **59**, pp. 1735-1743 (1999).

[9] DIN EN ISO 527-5. *Plastics – Determination of tensile properties* (2009)

[10] Ehrenstein G. *Faserverbund-Kunststoffe: Werkstoffe, Verarbeitung, Eigenschaften*. Hanser, München (2006)

[11] Puck A. *Festigkeitsanalyse von Faser-Matrix-Laminaten*. Hanser, München, Wien (1996)