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Abstract

Fibre reinforced polymers (FRPs) are an important group of materials in lightweight constructions. Most of the parts produced from FRPs, like aircraft wings or wind energy turbine blades are designed for high load levels and a lifetime of 30 years or more, leading to an extremely high number of load cycles to sustain. Consequently, the fatigue life and the degradation of the mechanical properties are aspects to be considered. Therefore, in the last years condition monitoring of FRP-structures has gained importance and different types of sensors for load and damage sensing have been developed.

In this work a new approach for condition monitoring was investigated, which, unlike other attempts, does not require additional sensors, but instead is performed directly by the measurement of a material property of the FRP. An epoxy resin was modified with two different types of carbon nanotubes and with carbon black, in order to achieve an electrical conductivity. Glass fibre reinforced composites (GFRP) were produced with these modified epoxies by resin transfer moulding (RTM). Specimens were cut from the produced materials and tested by incremental tensile tests and fatigue tests and the interlaminar shear strength (ILSS) was measured. During the mechanical tests the electrical conductivity of all specimens was monitored simultaneously, to assess the potential for stress/strain and damage monitoring.

The results presented in this work, show a high potential for both, damage and load detection of FRP structures via electrical conductivity methods, involving a nanocomposite matrix.

1. Introduction

Fibre reinforced polymers (FRPs) have become the most important material class in the field of lightweight constructions, especially in the aircraft and wind energy industry. Due to their high specific stiffness and strength as well as their outstanding fatigue performance, FRPs are nowadays an irreplaceable material in structural component design. However, their specific discrete build-up makes maintenance as well as damage analysis of these materials a challenging task. As FRPs are especially sensitive to intrinsic damage, such as delamination or matrix cracking, a reliable method for the detection and assessment of such failures is desirable. Furthermore, an in situ monitoring of damage development and/or apparent stresses and strains could be a useful tool to increase the reliability and lifetime as well as to ease the maintenance of structural components exhibited to permanent cyclic loading conditions.

In this context, electrical conductivity methods have been widely investigated for the detection of failure in carbon fibre reinforced polymers (CFRPs). The electrical conductivity of the carbon fibres was first used by Schulte et al. [1] to monitor damage in CFRPs, which could be related to fibre breakage. AC and DC electrical methods have been extensively studied since then and have been used to study a variety of damage mechanisms, e.g. delamination or matrix cracking, under static and dynamic loading conditions [2-11]. The "piezoresistive" effect of carbon fibres also allows a sensing of the applied stress/strain [8,12-15]. Although these methods can offer quite some insight into matrix related failure mechanisms, e.g. delamination, the application of an electrically conductive matrix can provide a much more detailed insight into these mechanisms. Furthermore, the application of an electrically conductive matrix allows the sensing via electrical methods, also in composites reinforced with non conductive fibres, e.g. glass or polymer fibres.

The effects of external strain on the conductivity of insulator/conductor composites based on polymers have been investigated by Pramanik et al. [16] and Radhakrishnan [17] et al. These tests were performed above the glass transition temperature (T_g) of the polymer matrix and give a basic insight into the electrical behaviour of polymers containing a conductive phase under external load. Flandin et al. [18,19] first used nanoscopic conductive fillers with different aspect ratios in a thermoplastic matrix to monitor the applied macroscopic mechanical strain and the damage evolution during loading. Nanoscaled carbon black particles [8] as well as microscaled carbon black particles [20] have also been used to modify the matrix of glass-fibre reinforced thermosets. In these works, it was shown that external stress as well as apparent mechanical damage can be detected in these multiphase composites via electrical conductivity methods.

The advances in nanotechnology and the increasing commercial availability make carbon nanotubes (CNTs) a very interesting nanofiller. Due to their electrical properties, their high aspect ratio and their tendency to form percolated networks in a viscous matrix, they are excellent candidates for the modification of thermosetting matrix systems. A good review about carbon nanotubes and their composites can be found in [21,22]. Electrical conductivity can be achieved at very low filler contents [23,24]. Typical dimensions of multi-walled carbon nanotubes are diameters in the range of 10 – 30 nm and lengths of up to several micrometers. Because of their dimensions in the nanometre regime, they are excellent candidates for the modification of FRP matrix systems, since the small particles do not show a filtering effect in liquid composite moulding processes, the particle-doped matrix penetrates the fibre bundles and electrical conductivity in the composite can be provided.. We recently reported about the mechanical and electrical properties of a nanoparticle modified glass fibre reinforced epoxy composite, produced via a vacuum assisted resin transfer moulding process (VARTM) [25,26]. Especially the carbon nanotube modified matrix systems provide improved mechanical properties as well as an electrical conductivity in the order of 10^{-2} S/m

[26], which should be sufficient for stress/strain and damage sensing via electrical conductivity methods as the resulting absolute resistance values merasured in the composites are in a measurable range (e.g. several kΩ) [22,27]. Recently, Thostenson and Chou reported about carbon nanotube modified glass fibre reinforced epoxy composites and their potential for strain and damage sensing. It could be shown that with a weight fraction of 0.5% CNTs in the epoxy matrix, mechanical stresses/strains, as well as matrix failure can be detected via electrical conductivity measurements [28].

Damage sensing in larger GFRP parts is usually done with fibre-optical sensors. The optical glass fibres are embedded in the fabrics used for the fabrication. In case of damage in the part the strain in the fibre is changed locally, which results in changes in the coefficient of refraction. This leads to a characteristic light pattern that can be detected [29-33].

The reliability of fibre-optical sensors for damage detection in large composite parts is insufficient for different reasons. With respect to the production costs it is not possible to apply a dense network of optical glass fibres to large parts and if a crack is propagating without crossing one of the fibre sensors this damage will not be detected. The resolution of the sensors is also limited and is connected with a minimum size of the defect, because a small defect will not cause a change in fibre strain that can be detected.

Not to be neglected is the fact that fibre-optical sensors may also induce damage to composite parts. Due to their diameter, which is 5-10 times the diameter of the reinforcement fibres, they can act as a notch in the composite material and therefore reduce the threshold for crack initiation [34,35].

The detection of damage in composite parts by measuring variations in an intrinsic material property, as the matrix conductivity, therefore offers benefits compared to fibre-optical sensors.

In this paper, we report about glass fibre reinforced epoxy laminates, modified with very small amounts (0.1 and 0.3 wt.%) of conductive carbon nanofillers (carbon black and

MWCNTs). Combined mechanical/electrical tests were performed in order to evaluate the sensing potential of these multiphase composites. Static tests (interlaminar shear strength tests, stepped tensile tests) as well as dynamic fatigue tests (cyclic tensile tests) were performed and the electrical conductivity was measured in situ. It could be shown that all conductive nanocomposite matrix systems exhibit a potential for stress/strain and damage sensing, however exhibiting significantly different sensor characteristics.

2. Materials

The epoxy matrix in this study consists of the epoxy resin Araldite LY 556, the anhydride hardener Aradur 917 and the imidazole accelerator DY070, supplied by Huntsman Advanced Materials, Bergkamen / Germany. The LY556 system is a hot-curing epoxy system used in filament winding, pultrusion or pressure-moulding processes, for example.

To achieve an electrical conductivity, the epoxy matrix was modified by the addition of 0.3 wt.% of nanoscaled carbon particles. Three different types of nanoparticles were used in this study: Double Wall Carbon Nanotubes (DWCNT) from Nanocyl, Belgium, Multi Wall Carbon Nanotubes (MWCNT) from Arkema, France, and carbon black XE2 from Degussa, Germany. The modified epoxy systems were used to manufacture glass-fibre laminates, using the VARTM technique.

In order to disperse the nanoparticles in the epoxy matrix, the particles were manually mixed with the epoxy resin (without hardener and accelerator). This pre-mixture of nanoparticles and resin was then added batch-wise to a three roller mill. Details of the dispersion process are described elsewhere [24]. The blended epoxy resin was then collected, bottled and stored at low temperatures to avoid the re-formation of nanoparticle-agglomerates.

Prior to the VARTM manufacturing of the material, the suspension of epoxy and nanoparticles was mixed with the hardener and accelerator, using a laboratory vacuum stirrer to avoid mixing in air bubbles and to apply high shear rates to the compound. The mixing pot was then removed from the vacuum stirrer and then inserted in the resin-source container of the VARTM-system. The epoxy matrix was finally degassed and slightly heated up to 45°C to compensate the increased viscosity, resulting from the addition of the nanoparticles. As fibre-reinforcement, two layers of glass-fibre non-crimp fabrics (Saertex Wagener GmbH, Germany) were used to finally achieve a laminate with a [0°, +45°, 90°, -45°, +45°,90°,-45,0°] stacking sequence. The materials produced had a fibre-volume-content of 35%. The mould was slightly heated up to 45°C to keep the viscosity of the epoxy matrix low during the whole injection process.

For each material, additionally, a second laminate was produced. During its processing, an electrical field with the field strength of 333V/cm was applied to the RTM-mould in z-direction, in order to induce a preferred orientation of the nanoscaled carbon particles in z-direction and to stimulate the formation of a conductive network [26].

After 4 h curing at 80°C, the material was removed from the mould. Specimens according to DIN EN ISO 527-4 with the dimensions 25 x 3 x 250 mm and modified specimens acc. to ISO 14130 with the dimensions 21 x 3 x 10 mm were cut and edge-polished. The tensile-test-specimens acc. to DIN 527-4 were furnished with tabs of 2 mm thickness. Each tab consists of two 1 mm thick layers of [+/-45°] GFRP on the specimens side, aluminium on the fixtures side, respectively. The specimens were post-cured at 140°C for 8 hours and contacted with conductive silver paint. For resistance-measurements in 0°-direction of the specimens the two opposite front and back sides of the specimens were painted with conductive silver paint. For the measurements in z-direction of the ILSS-specimen the upper and lower surface was fully painted with conductive silver and the remaining edges were edge polished to prevent short-circuiting the resistance measurement. The contacting for the tensile test-specimen was carried out similar, however here only a 15mm wide electrode was applied to both sides in the middle of the specimen. The specimens and the experimental setup for both ILSS and tensile tests are shown in figure 1. “z” and “0°” indicate the direction of the in-situ resistance measurement during the mechanical testing.

3. Experimental

Three different mechanical testing methods were conducted in our investigations and the potential for electrical conductivity methods for condition monitoring was evaluated: the interlaminar shear strength (ILSS) test, incremental tensile tests and dynamic tensile tests. The interlaminar shear strength of the composites was measured according to ASTM D-2344 with the short beam method. A Zwick Z010 universal testing machine was used to determine the ILSS of at least 15 specimens per material.

The stepped and the dynamic tensile tests were performed on a servo-hydraulic Instron/Schenck testing machine with a maximum load capacity of 100 kN.

The electrical resistance of the specimens under mechanical loading was measured in situ using a Keithley 2602 sourcemeter instrument. A constant voltage was applied to the specimens, the current was measured and the resistance was calculated from these values. The sourcemeter-instrument provides sampling rates up to $50 s^{-1}$ for electrical measurements.

4. Results and discussion

4.1 Interlaminar shear strength

The ILSS test was performed to introduce a well-defined, spontaneously occurring damage (delamination) to the specimens while the electrical conductivity was continuously measured. In an earlier work, the interlaminar shear strength of a GFRP system was enhanced by addition of nanoparticles [26]. Therefore, the mechanical results of the tests were also investigated.

Fig. 2 shows the interlaminar shear strength of the nanoparticle-modified composites. It can be seen that the ILSS values are slightly increased by the addition of carbon nanoparticles, although the changes are not significant. It also seems that the application of an electrical field leads to a reduction in improvement. All in all, the influence of the nanoparticles on the ILSS is not comparable to the results found in [26].

Under mechanical load, the specimens showed a typical progression of force and resistance change with testing time. Figure 3, as an examplary testing result, shows that the through-thickness-(Z-direction)-resistance of the specimens is fairly unaffected by the introduction of shear forces. A similar course of resistance over time can be observed for the resistance in 0° direction of the specimen. At the time, when the sudden delamination of the laminates occurs, the resistance is rising abruptly. This behaviour is representative for all specimens investigated in this study. For each specimen, the variation of resistance was measured, and normalised to the initial resistance. Figure 4a shows the normalised values for the resistance changes at break of the specimens, measured in longitudinal (0°) direction of the specimens. It can be clearly seen that for all kinds of matrix modification a significant resistance change of at least 10% was obtained. The carbon-black modified epoxy laminates exhibited values around 25% to 45%. The values for the resistance change measured in z-direction (Fig. 4b) are about 10%-15% for the nanotube-modified laminates and around 20%-40% for the carbon black modified laminates. This abrupt increase in the electrical resistance can be easily explained with the interruption of conductive pathways in the materials by the delamination. In this context it is important to point out, that the delamination, which occurs in the fibre plane orthogonal to the z-direction, can be electrically detected in both measurement directions (0°-direction and z-direction). Two reasons can be figured out for this phenomenon. First, the delamination is never a purely two-dimensional failure, but is also propagating in thickness direction (translaminar crack). Second, the electrical pathways of the nanoparticles are randomly three-dimensionally oriented in the laminates and therefore, a resistivity measurement in 0°-direction is also affected by a delamination in the fibre plane, since the electrical pathways are also partly oriented in z-direction. This is confirmed by the results shown in Fig. 4a. It can be seen that the orientation of the nanoparticles in z-direction, introduced by the electrical field during curing, leads to an increase in failure sensitivity when measuring in 0°-direction. The conductive pathways in the laminates which were subjected to the electrical field are statistically preferentially oriented in the z-direction and thus an interlaminar failure will have a stronger impact on 0°-conductivity.

4.2 Incremental tensile tests

Incremental tensile tests proved to be very suitable to identify the potential of the nanocomposite matrix systems for the sensing of stresses/strains and occurring damage. Figure 5a shows exemplary courses of stress and strain over time for the chosen testing procedure. The example shows the measurement on a specimen containing 0.3 wt.% MWCNTs.

The sample was subjected to tensile loading cycles, with increasing maximum strain per cycle. At the end of each cycle, the sample was unloaded to $\sigma = 0$. In Figure 5a, it can be seen that after the fifth loading cycle, some residual strain occurs when the specimen is unloaded to zero stress. This residual strain is increasing with each subsequent loading cycle and can be attributed to plastic matrix deformation and damage occurring, such as matrix cracking in the 90°-layers [36]. Figure 5b shows a corresponding resistance change curve, in this case measured in z-direction. It can be clearly seen that the change in resistance can be directly correlated with the applied tensile load. The resistance increases and decreases monotonically with the cyclic stress/strain. Furthermore, it can be observed that after the fifth loading cycle an irreversible resistance change occurs when the specimen is unloaded. This directly correlates with the emerging residual strain which could be observed after the fifth loading cycle (Figure 5a). This result also underlines the high capability of conductive polymer matrix systems for damage sensing applications. Figure 6 shows the correlation of the emerging residual strain after each cycle with the corresponding irreversible resistance change.

It can be seen that already very small residual strains in the order of 0.05 % can be detected with the electrical resistivity measurements. The corresponding irreversible resistance change was 0.02 % in this case. In the subsequent loading cycles, the residual strain increases gradually to a value of 0.52% after the 11th cycle, where the corresponding irreversible resistance change was found to be 1.4%.

We found a very similar behaviour for all the nanocomposite matrix laminates. The occurring residual strains could always be correlated to irreversible resistance changes. For the carbon nanotube modified matrix systems, a final residual strain of ~ 0.5% was found to correlate with irreversible resistance changes between 1 and 5%. As already observed in the ILSS measurements, the carbon black modified epoxy matrix system exhibited a higher sensitivity, with irreversible resistance changes between 10 and 20%. However, the absolute resistance of these specimens was measured in the range of several tens of MΩ and was two orders of magnitude higher than the resistance of the carbon nanotube modified laminate specimens, which confirms the results measured on the ILSS specimens.

The setup of the incremental tensile test is also especially suited for the evaluation of the stress/strain sensing capabilities of the tested materials. As shown in Figure 5, the resistance changes reversible with the applied load. In order to evaluate the strain sensing properties and to compare the behaviour of the different nanocomposites, the resistance-strain behaviour was investigated. In this paper we just give a first introduction into the stress/strain sensing characteristics. A detailed analysis of this very complex behaviour is in preparation and will be published soon.

Figure 7 shows the resistance change vs. strain curves of the 10th loading cycle from the stepped tensile tests. In Figure 7a, the curve for the 0.3 wt.% MWCNT composite is displayed; in Figure 7b, the same curve is shown for the 0.3 wt.% carbon black composite. The resistivity was measured in z-direction in both cases and the maximum strain applied was 2.5%.

The different characteristics of these exemplary curves are representative for the behaviour of the respective nanocomposite matrix. The general resistance-strain behaviour was found to follow an exponential power law in the form of

$$\frac{\Delta R}{R_0} = r_n + \alpha \cdot e^{\beta \cdot \varepsilon} ,$$

where ΔR is the absolute resistance change, R_0 the initial resistivity of the specimen, r_n the residual resistance change at the beginning of the load cycle and ε is the applied strain. However, the resistance-strain behaviour of the CNT modified laminates can be approximated very well with a linear approximation. In contrast, the carbon black modified laminates exhibited as pronounced exponential resistance-strain behaviour. Here again, the higher sensitivity of the resistance versus mechanical loading can be observed for the carbon black. A value of 2.5% mechanical strain corresponds to a resistance change of 6.3% for the MWCNT modified laminates, while for the carbon black modified laminates a resistance change of 36.2% was observed. Furthermore, an initial gauge factor (IGF) was evaluated by calculating the slope of the tangent, applied between 0.05 and 0.25% of reversible strain. The IGF was found to be 1.54 for the MWCNT containing laminate and 3.39 for the carbon black containing laminate.

It is obvious that the different behaviour of the carbon nanotube modified matrix system and the carbon black modified matrix system has to be attributed to the intrinsic structure of the percolated conductive paths in the composite. Two main factors will influence the sensitivity for stresses/strains and occurring damage. The first factor is the redundancy of the network structure. In this context, the higher sensitivity of the carbon black modified composites can be explained by the lower density of conductive pathways, as the concentration of 0.3 wt.%

used in our experiments is quite low, regarding the percolation threshold of this material [24]. Concerning the measured specific conductivity of the carbon black composites, a value of around 10^{-4} S/m is a hint for the concentration being close to the percolation region. For the MWCNT modified laminates a specific conductivity of some 10^{-2} S/m was measured. The percolation threshold of this MWCNT/epoxy system was found to be below 0.1 wt.%. In summary, the carbon black modified laminate with a concentration of 0.3 wt.% is close to the percolation region and therefore much more sensitive to applied stress/strain and occurring damage. The laminates containing 0.3 wt.% of MWCNTs are far above the percolation threshold and therefore exhibit a much more redundant conductive network structure. Thus, the sensitivity of this system versus damage is lower, compared to the carbon black modified matrix.

Regarding the resistance-strain behaviour, the different characteristics can furthermore be explained by the different filler geometries. As the carbon black particles are of spherical nature, the conductive pathways through the composites consist of spherical particles, which are connected by point-point contacts. In the case of carbon nanotubes, the network structure is very different as the aspect ratio of the CNTs is very high. The CNTs can be up to several micrometers long. Each CNT can possess multiple contacts to other CNTs and the CNTs are three-dimensionally randomly oriented within the matrix and can be geometrically entangled. Such a network will behave completely different, when exhibited to mechanical strain. Because of the higher amount of contacts and the different orientation with respect to the loading direction, the CNT networks are electrically less sensitive to mechanical strain than networks consisting of spherical carbon black particles.

Furthermore, it is noteworthy that the sensing of strain applied in the 0° direction can be well detected via electrical resistance measurements performed in through-thickness-direction, meaning perpendicular to the loading direction. This can be explained by the three dimensional network structures of the conductive nanoparticles. As the glass-fibre fabrics are inhibiting a direct electrical path between the two specimen surfaces, the resulting electrical pathways will be partly oriented in 0°-direction.

4.3 Dynamic tensile tests

Dynamic measurements were performed to show the resistance change during fatigue loading of the materials. It could be observed that a decrease of the stiffness of the specimens is accompanied by an increasing electrical resistance. In Figure 8, the graphs for the stiffness of the material, expressed over the dynamic modulus, which is measured in terms of the slope between the maxima and the minima in the hysteresis loop of each cycle, are shown.

Basically, three facts are remarkable concerning the resistance development during fatigue life: *i.* a stiffness decrease is accompanied by a resistance increase, *ii*. the difference between the resistance at maximum and minimum load is increasing during fatigue life, *iii*. major defects, such as delamination of single 0° rovings on the specimen surface, for example, could be detected by an abrupt increase of the electrical resistance.

These effects could be observed for both, resistance measured in length- and thickness direction of the specimen, whereas the detection of major defects was easier by resistance measurements in thickness direction as shown in Figure 9. A 100% change in resistance was observed when a delamination of a major part of the specimen occurred.

Gagel [36] showed the correlation between stiffness degradation and matrix crack density during loading of a GFRP with the same lay-up. In this work a correlation between resistance change and stiffness was observed for a similar material. Hence, it can be concluded that the change in resistance correlates with the crack density. To sum up, electrical resistivity measurements seem to be a promising tool for the detection of damage in nanoparticle modified GFRPs. Both matrix crack density and single major defects show special effects on the electrical resistance of the materials. A detailed investigation of the electrical conductivity development in GFRP laminates during fatigue life is in preparation.

5. Conclusions

The results of the experiments presented here demonstrate the high potential of electrical conductivity methods for stress/strain and damage sensing in FRPs with carbon nanoparticle-modified matrix systems. This method works only by the measurement of the electrical resistance and without additional sensors that have to be applied to the materials and is therefore a good example for “self-sensing” materials.

The sensitivity of this new method seems to be much higher compared to other damage sensing methods. During incremental tensile tests and fatigue tests it was possible to clearly measure resistance changes in the materials that were related to microscale damage, such as inter-fibre failure. This kind of damage can not be detected by other damage sensing methods. Later in the fatigue tests, when macroscopic damage (delamination or rupture of fibre bundles) occurred, these events were leading to explicit signals in the electrical conductivity measurement of the materials. Therefore, this new method offers the possibility for an in-situ measurement of the damage accumulation during the entire fatigue life of large composite structures, beginning from microscale damage and ending in rupture of fibre bundles or final failure.

Interestingly, also a dependency of the electrical resistance on the load applied to the composite was found. Therefore, not only the accumulation of damage can be detected by these measurements. It is also possible to directly measure the strain state of a composite structure.

The interlaminar shear strength of the laminates was not significantly improved as shown in earlier studies [26]. It can be concluded that the significance of the effect of nanoparticles on the mechanical properties of an epoxy resin is different depending on the type of resin. However, in the context of functional matrix properties, such as health monitoring capabilities, it is of high importance to demonstrate that the mechanical properties are not affected negatively by the nanoparticle modification.

Future studies will deal with a detailed investigation of the stress/strain – conductivity characteristics and the development of conductivity during fatigue life.

5. Acknowledgements

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References

1 Schulte K, Baron Ch. Load and failure analyses of CFRP laminates by means of electrical resistivity measurements. Compos Sci Technol 1989;36:349-56.

2 Kaddour AS, Al-Salehi FAR, Al-Hassani STS, Hinton MJ. Electrical resistance measurement technique for detecting failure in CFRP materials at high strain rate. Compos Sci Technol 1994;51(3):377-85.

3 Todoroki A, Kobayashi H, Matuura K. Application of electric potential method to smart composite structures for detecting delamination. JSME Int J A 1995;38(4):524-30.

4 Irving PE, Thiagarajan C. Fatigue damage characterization in carbon fibre composite materials using an electrical potential technique. Smart Mater Struct 1998;7(4):456-66.

5 Seo DC, Lee JJ. Damage detection of CFRP laminates using electrical resistance measurement and neural network. Compos Struct 1999;47:525-30.

6 Abry JC, Bochard S, Chateauminois A, Salvia M, Giraud G. In situ detection of damage in CFRP laminates by electrical resistance measurements. Compos Sci Technol 1999;59(6):925-35.

7 Abry JC, Choi YK, Chateauminois A, Dalloz B, Giraud G, Salvia M. In situ monitoring of damage in CFRP laminates by means of AC and DC measurements. Compos Sci Technol 2001;61:855-64.

8 Kupke M, Schulte K, Schüler R. Non-destructive testing of FRP by d.c and a.c. electrical methods. Compos Sci Technol 2001;61:837-47.

9 Todoroki A, Tanaka Y. Delamination identification of cross-ply graphite/epoxy composite beams using electrical resistance change method. Compos Sci Technol 2002;62:629-39.

10 Todoroki A, Tanaka M, Shimamura Y, Kobayashi H. Effects of Delamination shape with a matrix crack on monitoring by electrical resistance method. Adv Compos Mater 2004;13(2):107-20.

11 Todoroki A, Omagari K, Shimamura Y, Kobayashi H. Matrix crack detection of CFRP using electrical resistance change with integrated surface probes. Compos Sci Technol 2006;66:1539-45.

12 Wang S, Chung DDL. Piezoresitivity in continuous carbon fibre polymer-matrix composite. Polym Compos 2000;21(1):13-19.

13 Todoroki A, Electric resistance change method for cure/strain/damage monitoring of CFRP laminates. Key Eng Mat 2004;270-279:1812-20.

14 Todoroki A, Yoshida J, Electrical resistance change of unidirectional CFRP due to apllied load. JSME Int J A 2004;47(3):357-64.

15 Ogi K, Takao Y. Characterization of piezoresitance behaviour in a CFRP unidirectional laminate. Compos Sci Technol 2005;65:231-39.

16 Pramanik PK, Khastagir D, Saha TN. Effect of extensional strain on the resistivity of electrically conductive nitrile-rubber composites filled with carbon filler. J Mat Sci 1993;28:3539-46.

17 Radhakrishnan S, Saini DR. Structure and electrical properties of polypyrrole – thermoplastic elastomer blends. Polym Int 1994;34:111-17.

18 Flandin L, Cavaillé JY, Bréchet Y, Dendievel R. Characterization of the damage in nanocomposite materials by a.c. electrical properties: experiment and simulation. J Mat Sci 1999;34:1753-59.

19 Flandin L, Bréchet Y, Canova GR, Cavaillé JY. AC electrical properties as a sensor of the microstructural evolution in nanocomposite materials : experiment and simulation. Mod Simul Mater Sci 1999;7:865-74.

20 Muto N, Arai Y, Shin SG, Matsubara H, Yanagida H, Sugita M, Nakatsuji T. Hybrid composites with self-diagnostic function for preventing fatal failure. Compos Sci Technol 2001;61:875-83.

21 Thostenson ET, Li C, Chou TW. Nanocomposites in context. Compos Sci Technol 2005;65:491-516.

22 Fiedler B, Gojny FH, Wichmann MHG, Nolte MCM, Schulte K. Fundamental aspects of nano-reinforced composites. Compos Sci Technol 2006;66:3115-25.

23 Sandler JKW, Kirk JE, Kinloch IA, Shaffer MSP, Windle AH. Ultra-low electrical percolation threshold in carbon-nanotube-epoxy composites. Polymer 2003;44:5893-99.

24 Gojny FH, Wichmann MHG, Fiedler B, Kinloch I, Windle AH, Bauhofer W, Evaluation and identification of electrical and thermal conduction mechanisms in carbon nanotube epoxy composites. Polymer 2006;47:2036–45.

25 Gojny FH, Wichmann MHG, Fiedler B, Bauhofer W, Schulte K. Influence of nano-modification on the mechanical and electrical properties of conventional fibre-reinforced composites. Composites A 2005;36:1525-35.

26 Wichmann MHG, Sumfleth J, Gojny FH, Quaresimin M, Fiedler B, Schulte K. Glass-fibre-reinforced composites with enhanced mechanical and electrical properties – Benefits and limitations of a nanoparticle modified matrix. Eng. Frac. Mech. 2006;73:2346-59.

27 B. Fiedler, F.H. Gojny, M.H.G. Wichmann, W. Bauhofer and K. Schulte. Can carbon nanotubes be used to sense damage in composites? Ann. Chim. Sci. Mat. 29 (6) (2004), 81-94

28 Thostenson ET, Chou TW. Carbon nanotube networks: Sensing of distributed strain and damage for life prediction and self healing. Adv Mater 2006;18:2837-41.

29 Takeda S, Okabe Y, Takeda N. Delamination detection in CFRP laminates with embedded small-diameter fibre Bragg grating sensors. Composites A 2002;33:971-980.

30 Park JM, Lee SI, Kwon OY, Choi HS, Lee JH. Comparison of nondestructive microfailure evaluation of fibre-optic Bragg grating and acoustic emission piezoelectric sensors using fragmentation test. Composites Part A 2003;34:203-216.

31 Leng J, Asundi A. Structural health monitoring of smart composite materials by using EFPI and FBG sensors. Sensors and Actuators A 2003;103:330-340.

32 Hofer B. Fibre optic damage detection in composite structures. Composites 1987;18:309-316.

33 Waite SR, Tatam RP, Jackson A. Use of optical fibre for damage and strain detection in composite materials. Composite 1988; 19:435-442.

34 Lee DC, Lee JJ, Yun SJ. The mechanical characteristics of smart composite structures with embedded optical fibre sensors. Composite Structures 1995;32:39-50.

35 Seo DC, Lee JJ. Effect of embedded optical fibre sensors on transverse crack spacing of smart composite structures. Composite Structures 1995;32:51-58.

36 Gagel A, Lange D, Schulte K. On the relation between crack densities, stiffness degradation, and surface temperature distribution of tensile fatigue loaded glass-fibre non-crimp-fabric reinforced epoxy. Composites A 2006;37:222–228.

Figures:

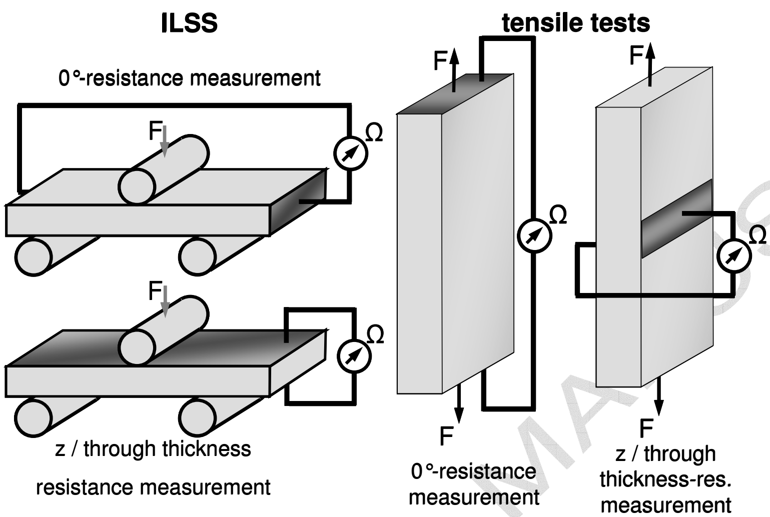


Figure 1: schematic visualization of specimen for in-situ resistance measurements during ILSS test (left) and tensile-test (right).

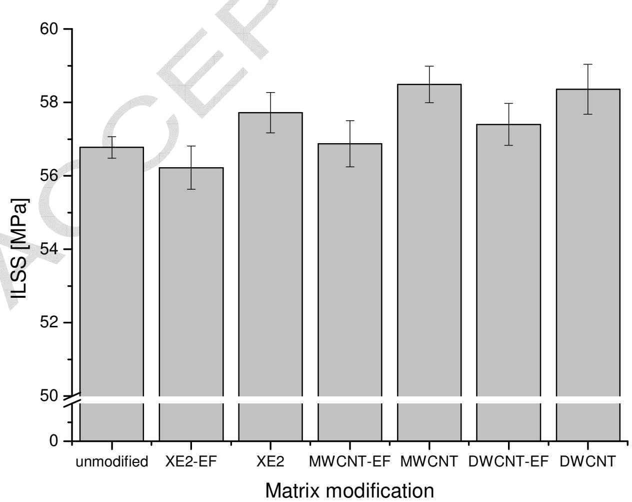


Figure 2: Interlaminar shear stresses of the modified GF-NCF-EP materials (EF = electrical field applied during curing).

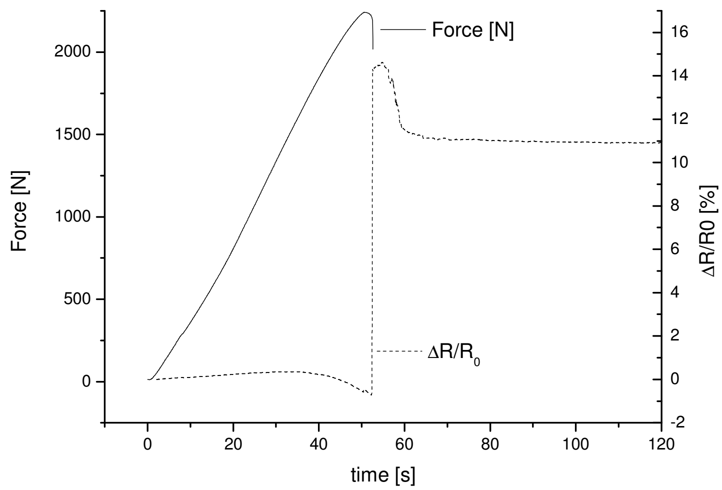


Figure 3: Progression of force and resistance change during ILSS test.

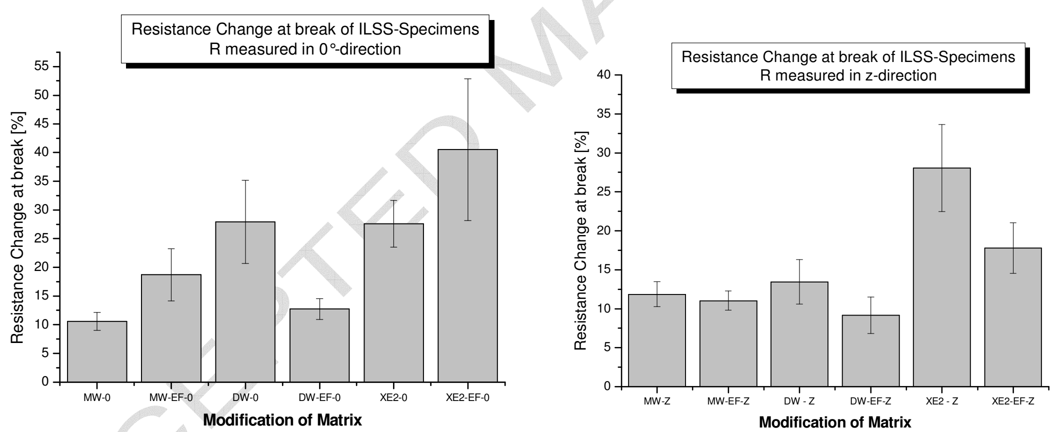
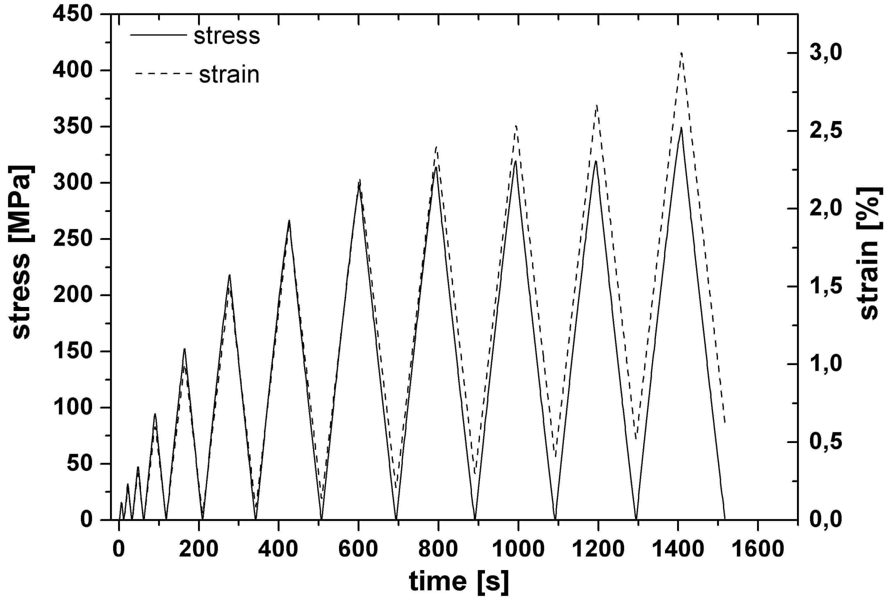


Figure 4: Resistance change at break of all tested materials; a) measured in length direction of the specimens, b) measured in thickness direction of the specimens (EF = electrical field applied during curing).



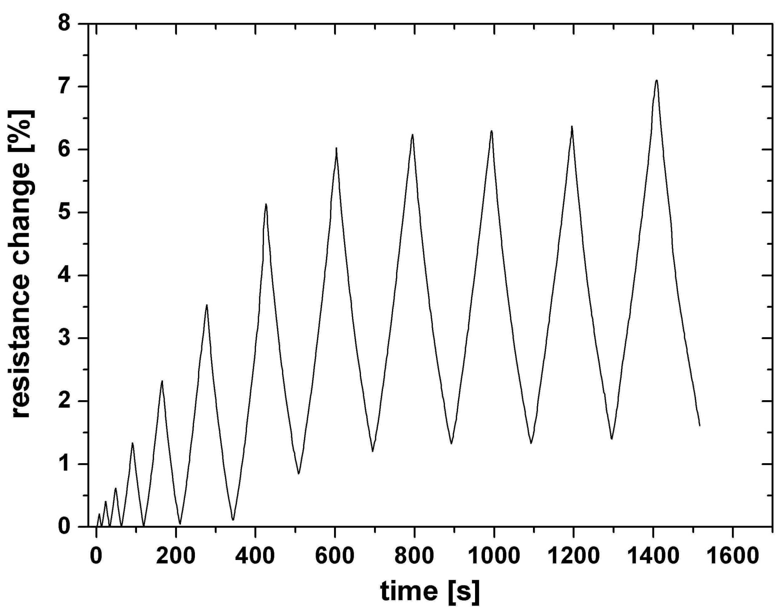


Figure 5.: **a)** Measured stress and strain and **b)** electrical resistance change during a stepped tensile test. The specimen contained 0.3 wt.% MWCNTs in the matrix and the resistivity was measured in z-direction.

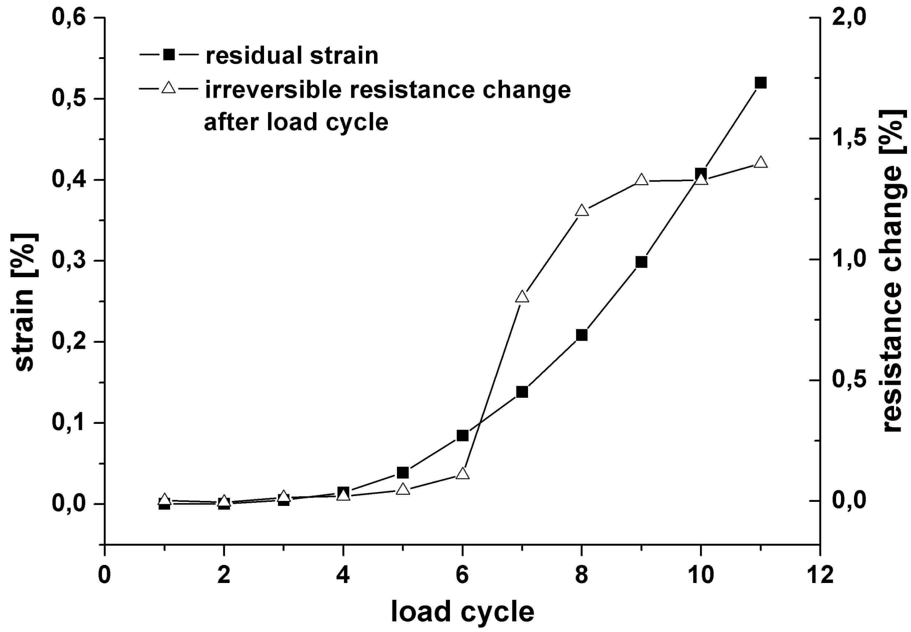
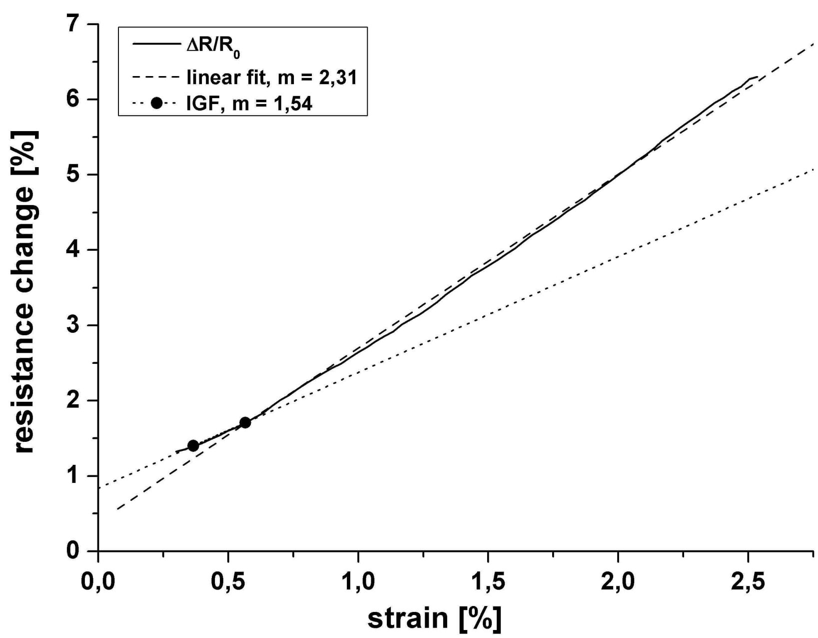


Figure 6: Apparent residual strain at the end of each loading cycle and corresponding irreversible resistance change, measured during the cyclic tensile test.



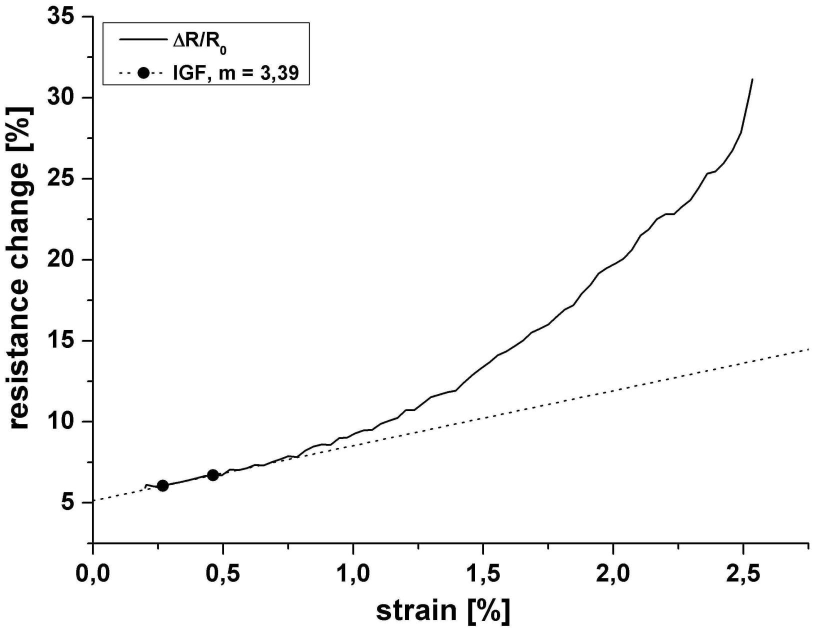


Figure 7: Electrical resistance change vs. mechanical strain, measured during the 10th loading cycle of a cyclic tensile test (resistivity measured in z-direction) for **a)** the composite containing 0.3 wt.% of MWCNTs and **b)** the composite containing 0.3 wt.% of carbon black.

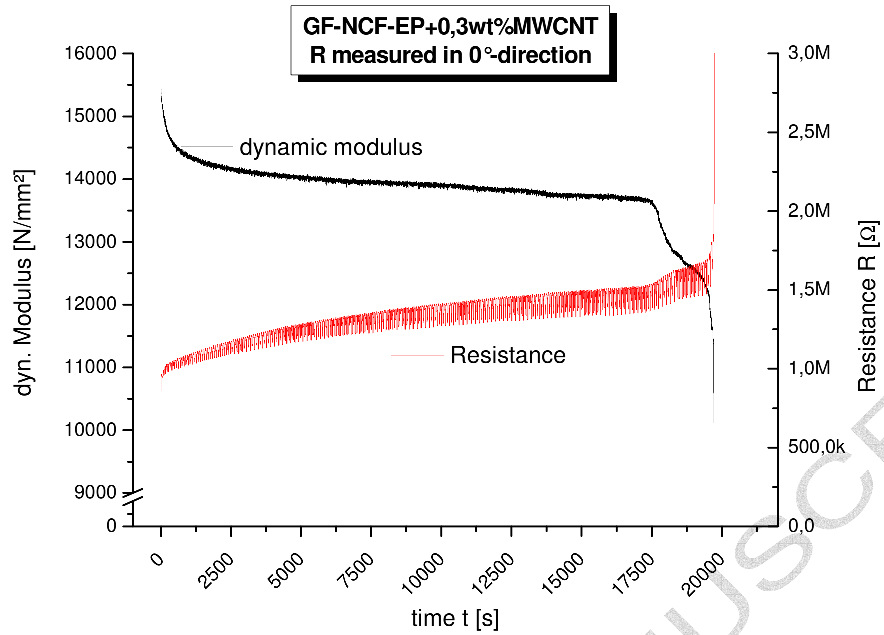


Figure 8: Stiffness and resistance change for a specimen under dynamic tensile load. Resistance measured in longitudinal (0°)-direction.

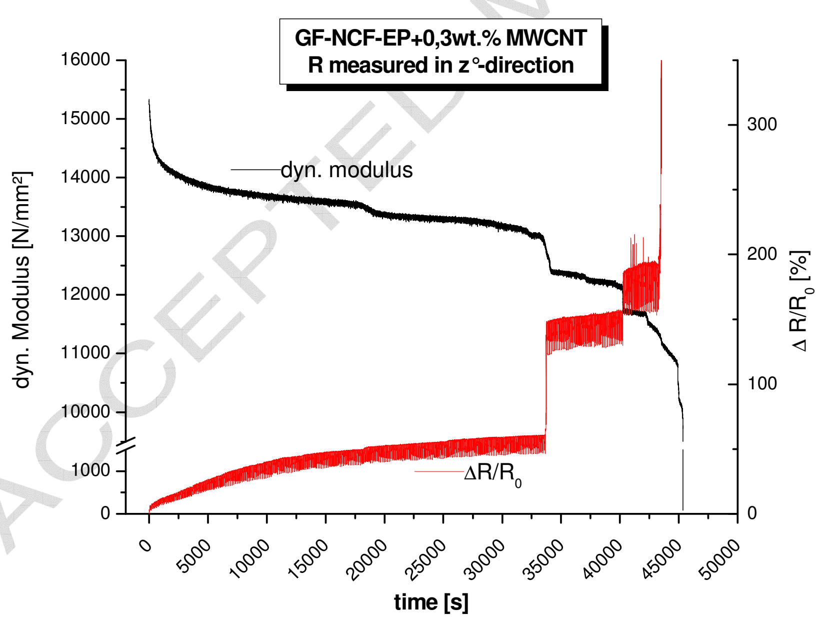


Figure 9: Stiffness and resistance change for a specimen under dynamic tensile load. Resistance measured in thickness (Z)-direction.