Development of high performance ductile hybrid composites

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1. Introduction

The failure of conventional structural materials (steel and other metals, alloys) has a favourable ductile character. They can undergo large deformation before their final failure, and a given part of this deformation is permanent, potentially providing a warning of overload after unloading. High performance composites have been in the spotlight for the last few decades because of their outstanding strength/density ratio, but their fundamental limitation is sudden brittle failure. Composites only withstand a few percent of strain before the usually catastrophic failure and they do not provide a detectable sign prior to final rupture. This usually leads to significant over-design and hinders the full exploitation of the outstanding properties of this material family especially in safety-critical fields. Ductile or pseudo-ductile behaviour, overload indication capability and the resulting safe operation could open new, high volume applications e.g. in the transportation and construction industries for composite components. This way, the materials of high-tech applications may finally penetrate into everyday products after more than half century.

Hybridisation of reinforcement materials (i.e. fibre types) is capable of improving the damage tolerance of the constituent composites. Thin pre-impregnated layers (prepregs) of different fibres with lower and higher failure strains were combined to mimic the ductile character of high strength metals. This so-called pseudo-ductility refers to the stress-strain response of the new material, not the mechanisms behind, as no yielding and plastic deformation is present in composites made of fibres and thermoset resins, both being brittle. Damage tolerance can be introduced by designing the architecture to promote fragmentation and stable delamination of the layers before final failure. Two types of unidirectional (UD) pseudo-ductile interlayer hybrid composites were developed recently: one with continuous fibres, where pseudo-ductility is provided by the mismatch in the failure strain of the constituent fibres [1],[2]. The other type material consisting of a discontinuous carbon fibre layer sandwiched between continuous glass fibre/epoxy layers have a uniquely designed structure with cuts in the carbon layer at pre-defined positions promoting the controlled and stable delamination of the carbon and glass layers before any fibre fracture [3]. Figure 1 shows the pseudo-ductile stress-strain curve of a thin-ply continuous interlayer hybrid composite featuring a pseudo-yield point, a wide plateau at around 1000 MPa stress and a second rising part before final failure. The stain between the pseudo-yield point and the final failure is called the pseudo ductile strain. This is up to 2% in the example and provides a significant safety margin against catastrophic failure which clearly indicates the potential for excellent damage tolerance in practice. Analytical and numerical models were also developed to predict the mechanical behaviour of thin-ply hybrid composites and initiate the preparation of a comprehensive design framework for the new materials [4]-[6].

The overall aim of the research programme is to introduce ductility and predictable, progressive failure as new key features for high performance composites along with structural health monitoring approaches to increase safety.

More specific key challenges of the research programme are summarised below:

- Development of new, advanced pseudo-ductile hybrid composites
- Investigating the compression performance of the previously developed (UD) pseudo-ductile hybrid composites
- Development and tensile characterisation of pseudo-ductile hybrid composites providing high strength in more than one directions

- Investigation of the effect of layer interface modification approaches on the performance of damage tolerant hybrid composites
- Studying the damage sensing functionality of thin-ply hybrid composites
- Attempt for small scale feasibility studies of the developed and characterised new materials for suitable applications potentially in collaboration with industrial partners

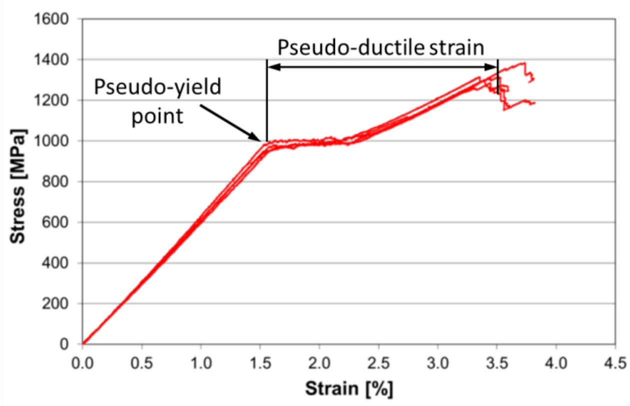

Figure 1. Thin-ply glass/carbon fibre hybrid composite specimens under tension showing pseudo-ductile stress-strain curves [2]

2. Results

2.1. Development of new, improved pseudo-ductile composites

After a literature survey and search for new raw materials, a variety of UD thin-ply carbon/glass-epoxy hybrid laminates made of different grade carbon and high strain S-glass fibres were fabricated and characterised in tension. The key trade-off in the properties of ductile hybrids was found between their pseudo-yield stress and pseudo-ductile strain (see figure 1). Strong correlation was found between the initial modulus and the pseudo-ductile strain of the tested hybrid materials. The results of this study were published in an international scientific journal paper [2].

As the next step, higher performance UD hybrids comprising ultra-high modulus (UHM), high modulus (HM), and intermediate modulus (IM) carbon/epoxy layers were developed and characterised in preparation for the more complicated multi-directional carbon/carbon hybrid laminate design. Favourable pseudo-ductile failure behaviour with linear-plateau-linear style stress-strain responses was demonstrated with progressive damage accumulation due to low strain material fragmentation and stable delamination instead of sudden, catastrophic fracture typical of conventional UD composites. All five material combinations exhibited exceptionally high initial moduli and a wide stress plateau with further increase in stress both of which can be exploited as a warning of accumulating damage before final failure. Our findings were published in an international scientific journal paper [7].

Multi-directional interlayer hybrid composites of ultra-high modulus (UHM) and intermediate modulus (IM) carbon/epoxy were designed and manufactured based on the previous studies. The new materials were characterised both under un-notched and notched conditions in tension. The motivation for this key task was to trigger a break-through in the exploitation of the ductility mechanisms demonstrated in our UD hybrid composites, as multi-directional pseudo-ductile laminates can be suitable for a wide range of applications not being limited to uniaxial load scenarios. The selected material pairs for this study had the highest initial modulus and the widest plateau of all the previously tested UD carbon/carbon hybrids. The design concept of the multi-directional laminates was that a UD three layer hybrid unit can be used as a sub-laminate and rotated to various directions according to a quasi-isotropic (QI) lay-up sequence. Both tested QI un-notched configurations exhibited favourable linear-plateau style pseudo-ductile failure mode due to local fragmentation of the low strain material in the 0° sub-laminate. Reduced notch sensitivity similar to the ductile net-section behaviour of metals was achieved in both hybrid laminates due to local damage (see figure 2) and induced load re-distribution around the notches, for both open holes and sharp notches. The detailed results can be found in an international scientific journal paper [8].

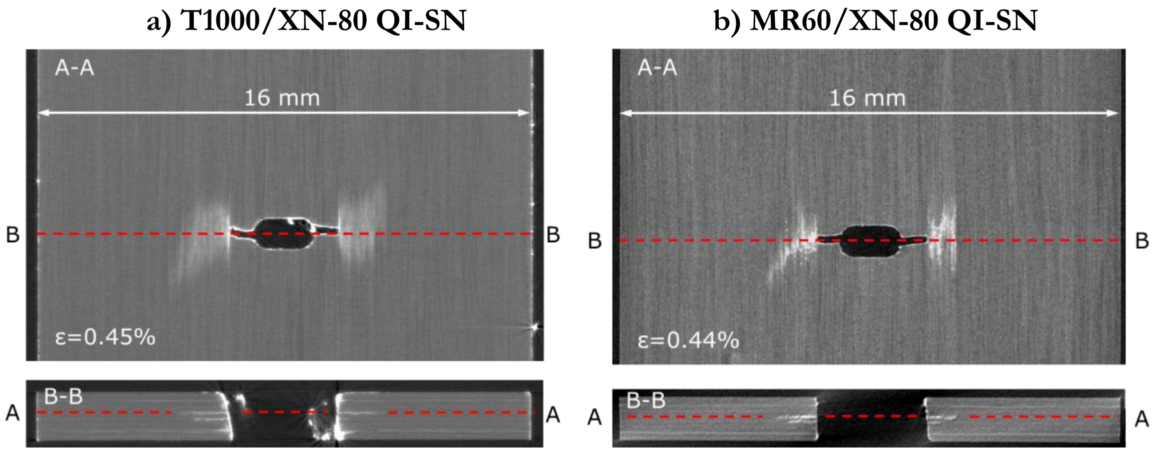

Figure 2. Full-width X-ray CT scans of different type notched specimens showing local damage near the notch tips in one of the 0° sublaminates [8]

During the test programme of several UD ply-by-ply hybrid configurations comprising a variety of glass/carbon-epoxy prepregs a deep insight was taken into the factors and mechanisms affecting the so-called hybrid effect in tension. The key strength of the study is the high quality test data generated under consistent conditions using several different materials and configurations. A valid baseline for carbon/epoxy failure strain was determined using the previously developed new hybrid tensile specimen type [9] to allow for accurate assessment of the increase in carbon failure strain in glass/carbon hybrids. The hybrid effect was also separated into a delay in carbon layer fracture initiation due to constraint on critical broken fibre cluster formation and an additional effect corresponding to the extent of multiple fragmentation required for a significant change in the stiffness of the hybrid laminate. Both effects were modelled successfully and the key issues of a four decade long debate around the hybrid effect mainly due to incorrect carbon composite failure strain baselines were addressed. The results of this study were published in an international scientific journal paper [10].

2.2. Characterisation of the developed pseudo-ductile composites

Compression

As a first approach 3 mm thick UD asymmetric glass/carbon-epoxy hybrid specimens were designed and the compressive damage and failure mechanisms of three layer glass/thin carbon hybrid blocks were investigated in four-point bending test setup. This new test setup was developed to allow for damage and failure type observations, which are extremely challenging in case of the conventional direct compression tests where the specimens typically fail catastrophically. The strains were monitored by a novel optical measurement procedure based on the tracked positions of five markers on the edge of the bending specimens. A new failure mechanism was observed which has not been reported in the international scientific literature before: Some of the investigated thin carbon/epoxy prepreg layers (i.e. the high and ultra-high modulus ones) have shown stable fragmentation distributed along the length of the stressed section. This type of failure has only been shown under tensile loading so far. Compressive failure strains significantly higher than those on the manufacturer's datasheets were obtained for all tested carbon/epoxy prepregs. The new observations suggested, that it may be possible to exploit the fragmentation as a key ductility mechanism in compression as well as tension dominated load scenarios and in the variety of corresponding applications. The results were published in an international scientific journal paper [9].

In order to understand the damage and failure mechanisms of pseudo-ductile carbon/glass hybrid composites, the mode II (shear) interfacial damage development of such materials was investigated on the micro-scale in situ in a scanning electron microscope (SEM). As a first step tensile load was applied on a special type specimen with a cut in the centre of the carbon layer to promote shear stresses between the layers. The full strain field of the interface was resolved with digital image correlation (DIC) with 4 μm spatial resolution [11]. Extensive analysis of the obtained strain fields revealed, that the fracture energy is not only dissipated at the layer interfaces but also in the composite plies through non-linear deformation. The shape of the mode II cohesive law of ply-by-ply carbon/glass-epoxy hybrid composites was found to be trapezoidal instead of the usually assumed bilinear type. The results of this study were published in an international scientific journal paper [12].

As a further step, the damage development and mechanisms in the carbon layer and at the glass/carbon layer interface was analysed at the micro-scale under compressive stress. Four-point bending specimens with carbon layers close to their top surface were deformed in a purpose-designed steel frame (see figure 3).

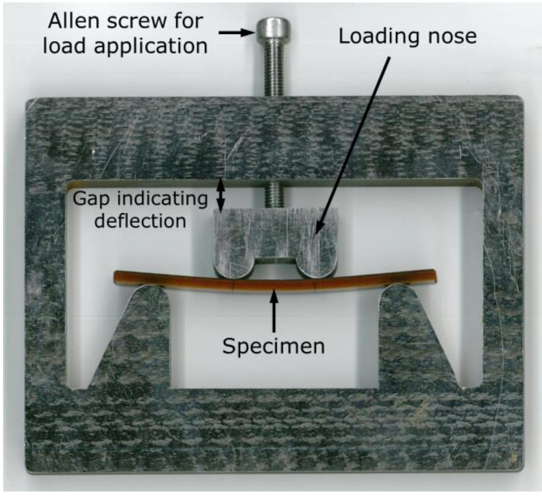

Figure 3. Four-point bending frame for microscopy under deformation [13]

The frame was put under optical and scanning electron microscope to analyse the damage modes and the micro-deformations in the hybrid material under compressive deformation. It was confirmed from edge-view mosaic scanning electron micrographs covering an approx. 4 mm long section of the specimen that the high modulus carbon layer was fragmented (see figure 4). Furthermore, the active micro-scale damage mechanisms were identified and summarised on an explanatory schematic. The results were presented at the 21st International Conference on Composite Materials (ICCM 21) in Xian, China, the leading scientific forum of the field. A full paper was also published in the conference proceeding [13].

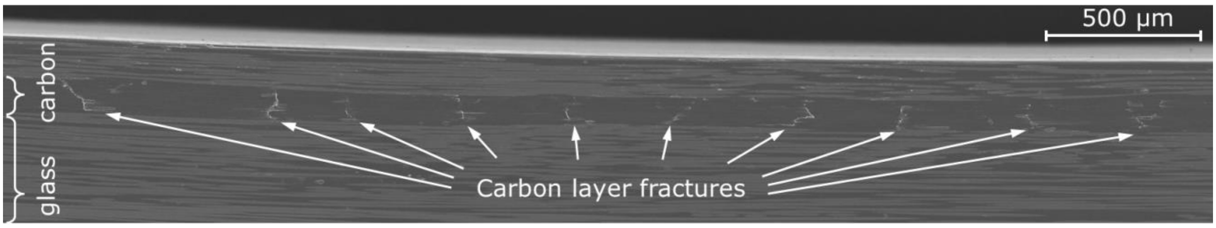

Figure 4. Carbon layer fractures on an edge view scanning electron micrograph [13]

The efforts to characterise the new pseudo-ductile interlayer hybrid materials under compressive loading was continued with direct compression tests of glass-carbon hybrid specimens with different carbon fibres and various carbon-to-glass ratios. Favourable pseudo-ductility was obtained in compression for the lower carbon-glass ratios with high modulus carbon fibres. This very important finding demonstrates that the new materials are suitable for reversed loading, not only tension. The results were presented at the prestigious Technical Conference of the American Society for Composites in 2018 September. A full paper was published in the proceeding of the conference [14] and an international journal paper is in preparation.

Fatigue

A separate project to assess the fatigue response of pseudo-ductile UD glass/carbon hybrids was completed by testing up to 20 specimens in tension with a stress ratio of 0.1. The key conclusions were the following: The non-damaged new materials can be loaded up to 80% of their pseudo-yield stress without fatigue damage accumulation. The pre-damaged specimens also fail gradually after delamination growth during thousands of cycles. The damage accumulation in the specimens can be monitored by the optical assessment of the delamination area between the carbon and glass layers (see figure 5). The results were published in an international journal paper [15].

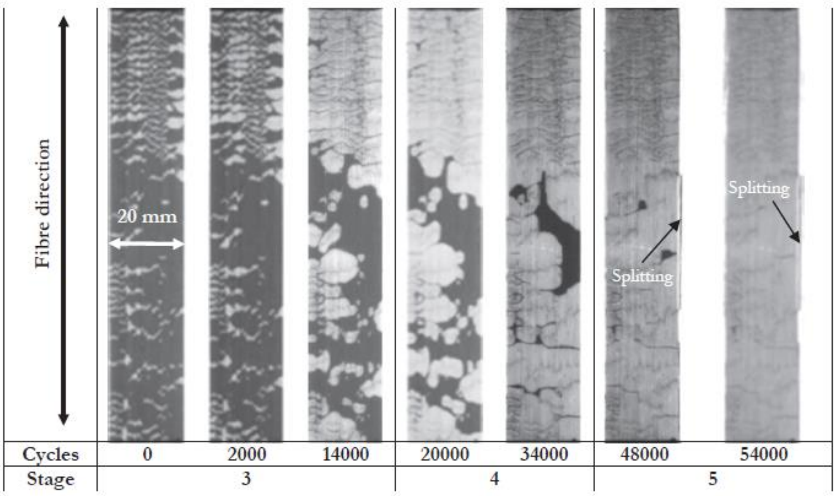

Figure 5. Typical fatigue damage development of an overloaded UD thin-ply hybrid composite cycled at 70% of the damage initiation stress [15]

Environmental effects

The effect of different environmental conditions on the mechanical properties is a key topic when it comes to introducing new materials to safety-critical applications. Therefore a comprehensive test campaign was executed to characterise the new pseudo-ductile glass/carbon interlayer hybrid composites at different temperatures (-50, 25 and 80°C) and to explore the effect of moisture on the failure strain and ductility of the materials. It was found that the temperature has minor effect on the fibre fragmentation dominated pseudo-yield strain of the hybrids, however up to 60% difference was observed in the mode II interlaminar fracture toughness values measured at the two extreme temperatures. This proves that the effect of temperature on the ply fragmentation mechanism is negligible, but the material is sensitive to the change of interlaminar toughness with temperature, which has to be considered during the component design phase. The results of the moisture sensitivity study indicated that long-term immersion of the specimens in 60°C deionised water has detrimental effect to the strength of the specimens, therefore a less aggressive ageing procedure was implemented at 60°C with 90% relative humidity. It was concluded, that wet conditioning changed the damage mode of the hybrid specimens from pseudo-ductile to catastrophic failure, because the glass/epoxy layers lost strength due to exposure to humidity. Wet conditioning decreased the mode II fracture toughness at all test temperatures. The results of the environmental effect study were presented at the prestigious ECCM18 and ICCM 22 conferences [16].

2.3. Special techniques to improve performance and exploit additional functions of pseudo-ductile composites

Interleaving with electrospun nanofibres

After testing several interlayer carbon/glass hybrid composite configurations the key role of the glass/epoxy and carbon/epoxy layer interfaces in the damage process was identified. Higher mode II interfacial fracture toughness could enable the use of thicker carbon layers in the hybrids leading to higher added stiffness and better overall performance. It was shown previously, that electrospun polymer nanofibre mats can improve the fracture toughness of monolithic composite layer interfaces [17], therefore we applied this approach in our hybrid materials.

The first activity in this field was the further development of the electrospinning method to scale the fibre production up by a new type spinneret operating with a circular orifice (see figure 4a). The new spinning head induces shear stresses in the polymer solution by mechanical ways. This makes it possible to set the viscosity of the non-Newtonian liquid during electrospinning, enabling a new level of process control finally leading to higher throughput and adjustable fibre diameters (figure 6b). The team demonstrated that the fibre diameters can be set in a wide range by simply adjusting the rotation speed of the spinneret. As a result, we managed to make continuous nanofibre mat strips with a useful width of 200 mm. A US patent application [18] was submitted and four international journal papers were published in the topic [19]-[22].

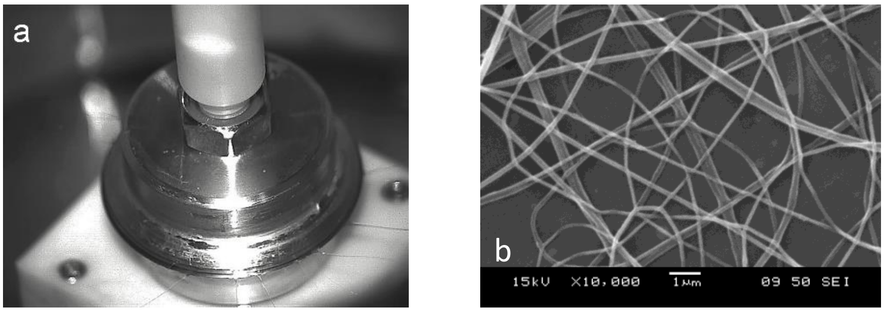

Figure 6. a) New type spinneret, b) nanofibers generated with the new method

The nanofibre mats were applied at the interfaces between glass/epoxy and carbon/epoxy layers of hybrid composites with no additional matrix (epoxy) added. Up to 30% improvement in the mode II interfacial fracture toughness was achieved with no significant mass increase. This massive improvement allows for higher carbon/glass ratios in the hybrids resulting in increased stiffness without losing the pseudo-ductile failure character. A BSc thesis was written on the topic and two PhD students were involved in the project.

Visual overload indication

An experimental campaign was completed to assess the visual strain overload indicating capability of carbon/glass hybrid composites due to the translucent glass layer which can reveal carbon layer fractures and interfacial damage. The results demonstrated that it is possible to use a retrofitted thin sensor patch of about 10x50 mm to indicate if a component (eg. a flat carbon/epoxy composite rod) was deformed beyond a pre-defined strain in tension. The key design parameters and the most important factors affecting the accuracy of the sensors were identified. The technology was demonstrated on a bike handlebar (see figure 7). The results were published in an international journal paper [23].

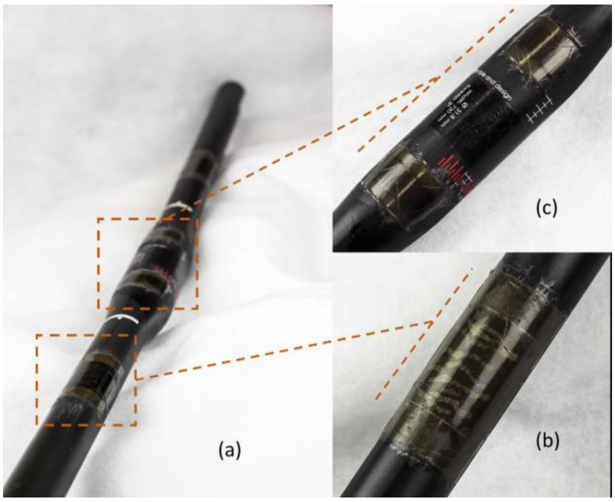

Figure 7. Bike handlebar fitted with hybrid composite overload sensors: (a) MTB Racing flat handlebar, retrofitted with (b) long single layer (c) short single layer sensors [23]

The previously demonstrated hybrid composite visual overload sensor was also applied at a larger scale not only as a retrofitted patch on tensile test specimens but as a multifunctional structural sensing layer on a 600x300 mm pseudo-ductile sandwich panel as well as on a full-scale longboard (up to 1 m long). These components are large enough to represent small-scale feasibility studies with realistic load scenarios. The structural sensing layer of the sandwich panel successfully gave warning of bending overload (see figure 8) at the beginning of a benign, progressive damage accumulation process well before the final failure of the sandwich beam specimens. The feasibility studies demonstrated that the developed materials and overload sensing technology can be applied to safety-critical components of medium size. An MSc thesis was written based on the results of this project.

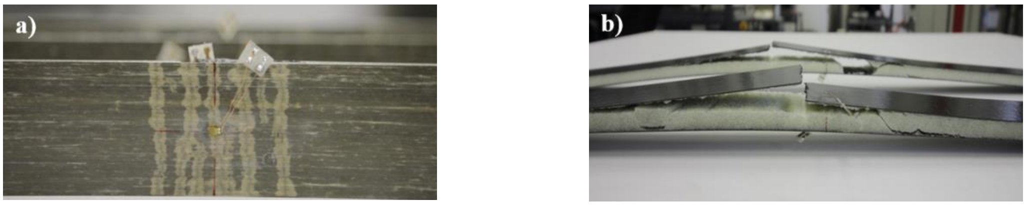

Figure 8. a) activated overload sensor, b) post mortem image of the sandwich beam specimens

Structural health monitoring through light transmission of glass fibre bundles

New structural health monitoring approaches based on the light transmission ability of glass fibre bundles and the attenuation of the light intensity was developed. The usability of reinforcing fibre bundles for deformation monitoring was studied extensively. It was proved experimentally, that the light intensity emitted by an arbitrarily chosen illuminated fibre bundle of the reinforcing glass fabric changes even when the load does not cause damage to the composite, and after unloading, the power of the emitted light is restored to its original level (see figure 9). This effect can be exploited for deformation monitoring of composite structures.

The usability of reinforcing fibre bundles for damage detection was also studied. It was demonstrated experimentally, that an arbitrary fibre bundle of a glass fabric reinforced composite can be used for damage accumulation monitoring under mechanical loading. If the illuminated fibre bundle of the composite structure fully or partially breaks and light exits the elementary fibres at that location, therefore the intensity of the light emitted at the end of the fibre bundle decreases to zero or a permanently reduced value. Delamination also results in a permanent decrease in the light intensity emitted at the end of the illuminated fibre bundle. This feature was utilised in a feasibility study, where the rear panel of a composite bus chassis (Modulo) was fitted with a fibre optic system to monitor the state of adhesive bonding points between the glass/polyester panel and the metallic fitting.

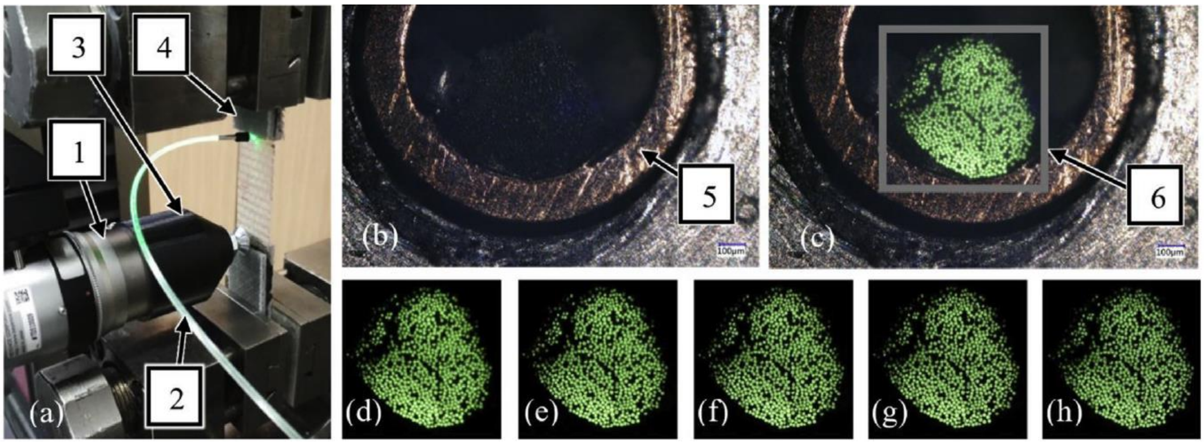

Figure 9. Layout for the observation of the light transmitted from fibres of the illuminated fibre bundle a), and the microscopy images of the end of the bundle without illumination b), illuminated with green laser light when not loaded c) and at a strain of 0.36% d), 0.72% e), 1.08% f), 1.44% g), and 1.8% h) (brightness−32%; contrast +100%) (1-digital microscope, 2-polymer optical cable, 3-the tailor-made cap of the digital microscope, 4-specimen, 5-cord-end terminal, 6-illuminated fibre bundle). [26]

Cheap, standard fibre optic components were also developed further and adapted to structural health monitoring purposes using reinforcing fibre bundles. The results were published in four international scientific journal papers [24]-[27].

Structural health monitoring based on electrical conductivity

Another approach to structural health monitoring is to observe the changes in electrical properties of carbon fibre bundles in a composite component. These properties are not only affected by the stress state of the material, but may be affected by environmental changes, for instance shifts in temperature or humidity. We investigated how temperature or relative humidity affects the electrical resistance of reinforcing carbon fibres in polymer composites. We found a linear relationship between temperature or relative humidity and resistance. This enables the use of carbon fibre bundles as sensors in different applications (for example in cure process monitoring and thermal fatigue monitoring). On the other hand, the environmental effects must be compensated for when using the fibres for monitoring stress in a component.

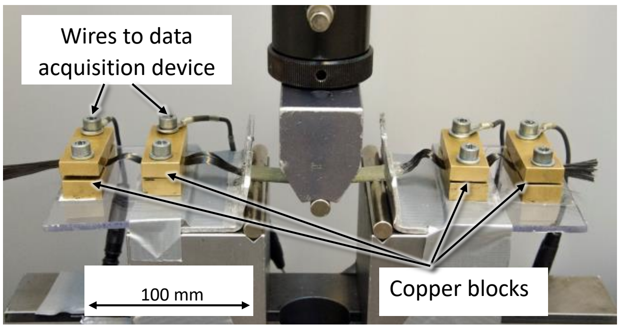

Figure 10. Experimental setup to measure the change in electric properties due to mechanical load in a carbon/epoxy composite sample

The electric current that is used for resistance measurement and damage monitoring in the carbon fibres may affect the mechanical properties of the composite structure itself. In order to study this effect, three-point bending tests (see figure 10.) and bending impact tests were executed under different electric currents. The exposure time before the impact was systematically varied as well. It was concluded, that the electric current has a negative effect on the elastic modulus and the impact strength of the investigated glass/carbon hybrid composite materials. The results were published in an international journal paper [28] and another one is in press [29].

Self-healing and reparability

The first approach to add self-healing functionality to epoxy was based on micro-capsules which were successfully produced and broken during the failure process of the dual cantilever beam (DCB) specimens. The healing system was effective, the healing agent was found to be well distributed on the crack surface. The results were published in an international journal paper [30].

An exploratory study was initiated recently to assess the possibility to add reparability as a new function to pseudo-ductile hybrid composites. So far only one configuration was tested with a 35 μm ABS film between the glass/epoxy and the periodically cut carbon/epoxy layer, but the bonding of the interlayer film was insufficient for good load transfer between the glass and carbon fibre reinforced composite layers. However the repairing cycle when the ABS film was re-melted and bonded again to the composite layers seemed to be feasible as the delaminations were no longer visible. These promising preliminary results provide a solid basis for further investigation with more suitable thermoplastic films. A BSc thesis was written and a PhD student is involved in this project.

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(The entries marked with * were produced with the support of the research programme.)

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