Adaptive, tolerant and efficient composite structures

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Chapter 2
Nano-Micro-Macro

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Abstract New materials with superior properties are the basis to exceed existing technological barriers and to explore new fields of application. Especially composites as multiphase materials offer the possibility to influence their properties or to add even new functionalities by a proper choice and combination of the different phases. In this context it is of particular importance to understand the interactions between the different material phases. This includes for example the effect of nanoscale additives in resins as well as the effect of microscopic manufacturing defects, like pores, on the macroscopic material properties. A systematic material design is only possible if cause and effect on the different material scales is well understood. Nanotechnology gives the opportunity to manipulate the structure of materials on a level, allowing to realize properties and functionalities that can't be achieved with conventional methods. Beside the improvement of mechanical, thermal, optical and electrical properties, the incorporation of new "smart materials" on a technical relevant scale is in the focus of our research.

2.1 Looking at Composite Materials at Different Scales

In literature it is common to distinguish four characteristics scales to describe the hierarchical constitution of materials [1]:

- The nanoscopic scale with a characteristic length of 10^{-9} m or a few nanometers,
- the microscopic scale with a characteristic length of 10^{-6} m or a few micrometers,

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- the mesoscopic scale with a characteristic length of 10^{-4} m or hundreds of micrometers,
- the macroscopic scale with a characteristic length of 10^{-2} m or centimetres and more.

An engineer, designing a fiber composite structure, is interested in properties describing the macroscopic behaviour of a certain material. Especially mechanical properties like stiffness and strength are required for the sizing of structures. Typically these properties are experimentally determined on a coupon level. A fiber composite material is build up by several layers of fiber materials oriented in different directions to carry the occurring loads. This mesoscopic scale describes the fiber architecture of the composite material. Also larger defects, like fiber undulations and pores, are in this order of magnitude. On a microscopic scale we look at a single fiber and the surrounding resin. A carbon fiber for example has a typical diameter of 7 μm. The behaviour of materials is finally determined by their smallest constituents, the atoms and molecules, and the way they interact among each other. On this scale also nano-sized particles and their interactions with the polymer are addressed.

2.2 Improving Fiber Composite Materials with Nanoscaled Particles

The modification of polymers with particles is a well known technology to adjust their properties. Depending on the particle size, shape, mechanical properties etc. attributes like viscosity, toughness, hardness, flammability can be affected to name a few. In the last decade the use of nano scaled particles came more and more into focus. At least one spatial direction of the filler material has to be in the order of nanometers to be designated as nano filler material. Although nano fillers have been used before, the current research aims to understand the effects that are related by adding particles of this scale to allow a systematic material development. There are several reasons why it is interesting to look at nanoscaled particles.

Due to the very small size of the individual particles the interphases between the particles and the surrounding polymer are extreme high. As an example the interphase area in a cubical volume of 1 mm^3 that is filled with 10% spherical particles, having a diameter of 1 μm, is 600 mm^2. In comparison to that the interphase area of particles with a diameter of 1 nm in the same volume with the same particle volume content is 600.000 mm^2. As a result a large portion of the polymer accumulates in the polymer/particle interphase. Assuming a good bonding between the particles and the polymer a relatively small fraction of particles can have a great effect on the properties of the polymer.

For fiber composite materials, where not only the neat resin is considered, nanoscaled particles have another important advantage, which is related to the

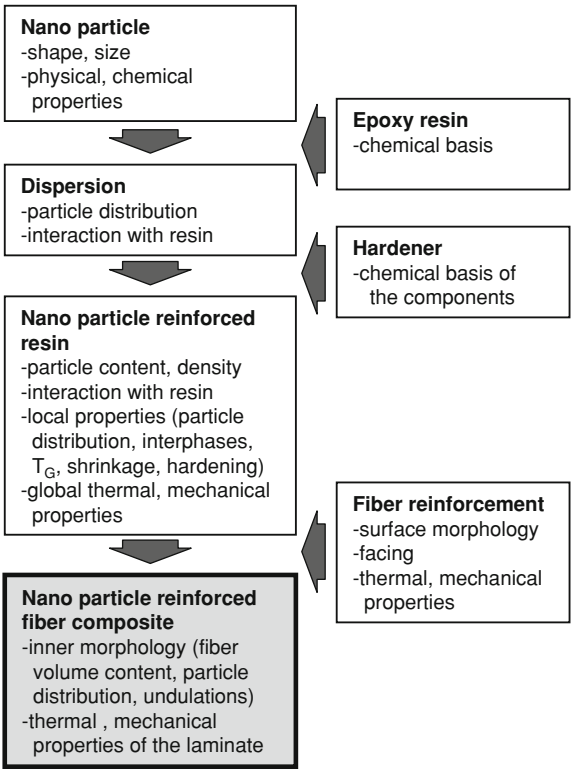

Fig. 2.1 Schematic drawing of parameters and influencing variables for nano particle modified fiber composites [3]

manufacturing process of fiber composites. Currently most high quality continuous fiber reinforced composites (fiber volume content around 60%) are manufactured with the prepreg method. Due to rising production costs, liquid resin infusion (LRI) methods are a promising cost effective alternative [2]. Quite a few different LRI processes have been developed so far. In all processes the liquid resin is infiltrated into the dry fiber material at process specific temperature- and pressure profiles. If a particle modified resin is used, filter effects can occur during the infiltration process, leading to particle density gradients in the composite material. With nanoscaled particles these effects can be minimized enabling a homogeneous particle distribution even in the consolidated fiber composite material.

Another issue in this context is the viscosity of the modified resin. LRI processes require a low viscosity of the resin. Otherwise the flow resistance during the infiltration process is to high leading to not infiltrated areas. Also in this case nanoscaled particles offer the chance to avoid this problem since only small particle volume contents are needed to get an effect.

Figure 2.1 illustrates the principle way from the nano particle to the reinforced fiber composite material and lists the most important parameters [3]. It is obvious that a great number of variables influence the fabrication process of the nano particle reinforced fiber composite material.

2.3 Smart Material Systems

The ideal basic material for "smart structures" possesses both actuator and sensor characteristics as well as load-bearing capabilities. Unfortunately, there is yet no existing material known that unites all of these characteristics in a sufficient manner. The assembly of a multifunctional material system is therefore realized by combining actuator and sensor materials with a primarily load-bearing component. Fiber composites are well-suited for the purpose of assembling a smart structure, since the active components can be inserted during the production process and, thus, become an inherent part of the structure. Additionally, the specific requirements and characteristics of active materials can be accounted for by the several production options for composites such as selection of the fiber/matrix material and the layup of the structure. Hence, the smart structure is designed and manufactured as a whole with embedded smart components including its supporting infrastructure of, e.g., lead wires, electrodes and terminals for power supply.

Sensors and actuators based on multifunctional materials are a substantial component of smart composite structures. Such multifunctional materials also called "smart" or "intelligent", are energy converters or transducers that respond in a technically usable manner to an external stimulus. The most widely employed types respond to an electric, thermal or magnetic field with a change in their mechanical properties. Well-known representatives are piezoelectric materials (load/deformation response to an electric field), shape memory alloys (temperature dependent load/deformation) as well as electro- and magnetorheological fluids (influence of shear transmission in an electrical or magnetic field respectively). Typically, the underlying actuation mechanism is caused by a microscopic reconfiguration in the material and operates in both directions. A change in the mechanical characteristics due to external loads can be detected and, thus, allows for sensor use also.

The reliable subsequent treatment and structural integration of the usually very sensitive materials is however, connected with some complexity and risk. The goal of our research activities is therefore to develop multifunctional material systems to enable the setup of reliable and reproducible smart structures that can be successfully applied in industrial production.

2.4 Integration of Smart Materials on a Macroscopic Level

Piezoceramic materials are very often used to fabricate smart composite structures. Figure 2.2 shows typical piezoceramic materials used for this purpose. The main reasons for the popularity of piezoceramics are their ability to operate at high frequencies and their stiffness, typically 60 GPa, matching the stiffness of many composite materials. The latter is of special importance for the usage as actuator. In the past quite a few investigations have been made to study the integration of

Fig. 2.2 Macroscopic piezoceramic fibers (∅200 µm) and plates (50 × 30 × 0.2 mm^3) that are typically used for the fabrication of smart composite structures

piezoceramic components into composite materials [4, 5]. In the following an exemplary case will be presented to discuss some issues related to the integration of components of this size into composite materials [6].

Here a piezocomposite with a monolithic piezoceramic plate with dimensions of 50 × 25 × 0.2 mm is considered (also referred to as transducer). The external dimensions of the piezocomposite transducer, including insulation and electrical contacts, were 58 × 29 × 0.47 mm^3. At this point the detailed transducer design is not of special interest. The transducers were integrated into a 16 layer laminate with quasi isotropic ply lay up $[(0/+45/90/-45)_2]_S$. The specimens were manufactured using the Differential Pressure Resin Transfer Moulding Process (DP-RTM) with unidirectional fiber material having an area weight of 125 g/m^2 (type UD-CST 125/300 from SGL). With a fiber volume content of 60% this result in a theoretical thickness of 0.12 mm for each ply. The fiber material was infiltrated with the three component resin system LY556/HY917/DY070 at 6 bar and 120°C. To enable a symmetrical specimen design two transducers were embedded in opposite positions.

Three different integration configurations were investigated as depicted in Fig. 2.3 According to the thickness of the transducers, in configuration (a) four layers of the prepreg material were cut out to accommodate the transducer. In configuration (b) no plies were cut out resulting in a thickened specimen and resin rich pockets at the edges of the transducer. A partial cut out leaving the plies in 0° direction intact was realized in configuration (c). As a reference additional specimen with bonded transducers were manufactured.

Figure 2.4 shows the final specimen design. The specimens were loaded in a tensile testing machine until failure. To measure the specimen deformation, strain gauges were attached inside (section 1) and outside (section 2) the integration area. An exemplary test result with a specimen configuration with no cut out (configuration b) is shown in Fig. 2.5. The stress was calculated with respect to the cross section area in each section and the test load. The gradient of the stress/strain curve describes the Young's modulus in the considered cross section. In addition to that the sensor signal of the piezoelectric transducer was measured during the test (red curve). Discontinuities in the sensor signal would indicate a crack in the

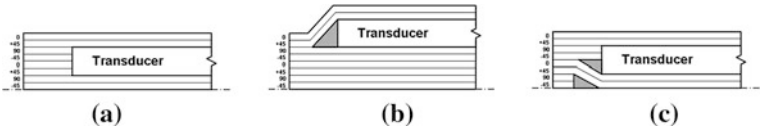

Fig. 2.3 Different integration configurations for macroscopic piezoelectric transducers. **a** Complete cut out. **b** No cut out. **c** Partial cut out

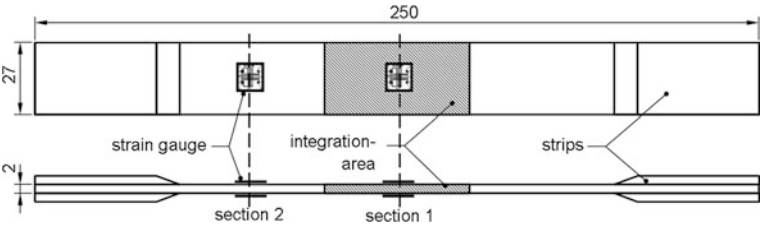

Fig. 2.4 Specimen design with embedded piezoelectric transducers

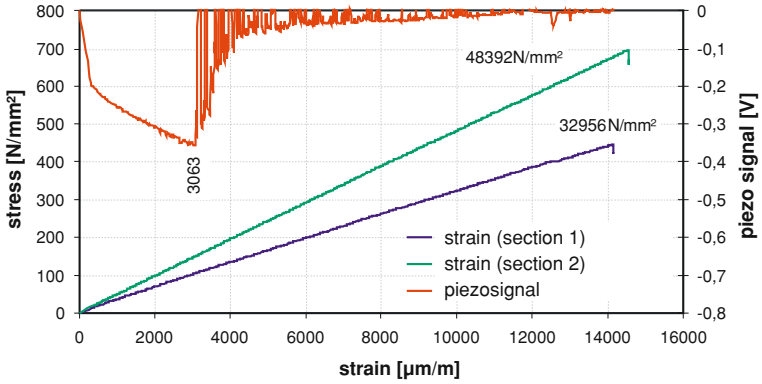

Fig. 2.5 Exemplary result of a tensile test with a specimen of configuration (b) no cut out

ceramic. Looking at the sensor signal in Fig. 2.5, first cracks are initiated at a strain of approximately 0.3% whereas the failure of the complete structure occurs at much higher strain levels. The piezoceramic fragments still generate signals but the performance is decreased. Nevertheless in comparison to very brittle plain ceramics, the strength of the embedded ceramic material is improved. Reasons for the higher strength are a mechanical pre compression in the ceramic due to higher thermal shrinkage of the composite material when cooled down from the curing temperature and the stoppage of crack propagation due to the embedding in a more ductile material.

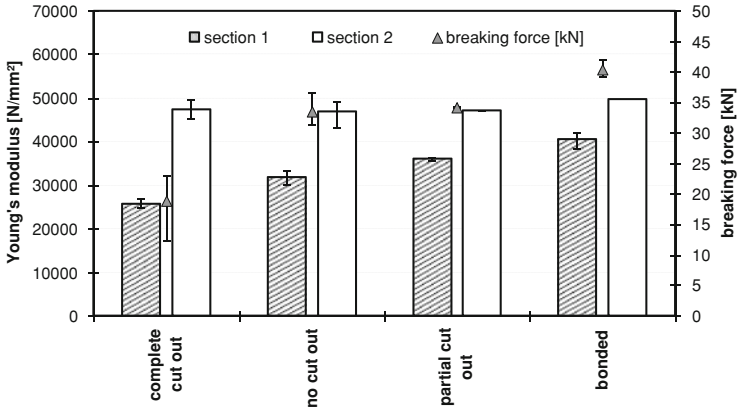

Fig. 2.6 Results of the tensile test with integrated piezoceramic transducer

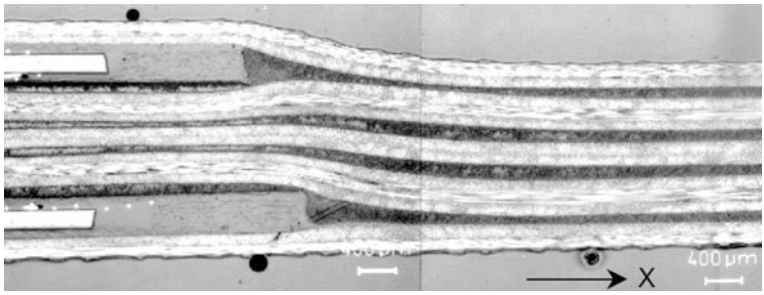

Fig. 2.7 Micrograph of a specimen cross section an embedded piezoelectric transducers (configuration with no cut out)

A summary of all test results is given in Fig. 2.6. At least 6 specimens were investigated for each configuration. The diagram depicts the breaking force and the Young's Modulus for each cross section. With respect to the reference specimen, for all configurations a clear reduction of the breaking force and Young's Modulus is observed for the cross section with the integrated transducer. As expected configuration (a) with a complete cut out shows the worst results. Best results are achieved with configuration (c) with a partial cut out. Even the configuration with no cut out is worse. Due to the large increase in thickness of the laminate in the integration area, large resin rich pockets are formed, resulting in high stress peaks in this area (Fig. 2.7). During loading the initiation of cracks was observed in the resin pockets actually.

The results show that an integration of smart materials of this macroscopic size in a monolithic composite material is most likely to have a negative effect on the strength of the material. In this example only the tension properties have been considered. It can be expected, that the impact on compression and fatigue

properties is even worse. A structural conformable, and in most cases applied configuration, is to bond transducers of this size onto the surface of the material to form the actual multifunctional material system. A good example for a structural conformable integration in this context is to bond the active components on the inside of the face sheets of a sandwich material.

2.5 Integration of Smart Materials on a Micro- and Nanoscopic Level

In the example described before the integration of smart materials into fiber composites was done by relatively large devices on a macroscopic level. A much tighter and more structural conformable way to integrate new functionalities into fiber composite materials can be expected, if the functional components have a similar or even smaller size than the constituent parts of the composite. One possible solution is the use of micro- or even nanoscopic particles. First steps in this direction have been made by using magnetostrictive materials. Some preliminary results of this research will be used to highlight the potential of this approach [7, 8].

Its outstanding magnetostrictive properties make Terfenol-D particles suitable for the use as sensor particles in smart composite materials. In this study the Terfenol particles were purchased from ETREMA (USA) and were received in batches with very different particle sizes ranging from a few micrometers up to 300 μm. To investigate the effect of particle size it is necessary to fractionate Terfenol-D particles. Figure 2.8 shows the particle composition of the original batch and after a sieving process to select particles with a size below 20 μm.

A homogeneous dispersion of the particles in the epoxy resin is fundamental to fabricate specimen with a reproducible quality. Master batches of epoxy resin with Terfenol-D particles were produced with a triple roll mill and with a basket mill. The resin used was the three component system LY556, HY917, HY070. To investigate the influence of particle size and particle content several master batches with different compositions were produced.

Since Terfenol-D has a very high density of 9.25 g/cm^3, especially in comparison to epoxy resins (1.2 g/cm^3), the manufacturing of specimen with a uniform particle distribution is a very challenging task. To avoid sedimentation a suitable solution is to use a magnetic field during the curing procedure. By this measure the Terfenol-D particle form chain structures oriented along the magnetic flux lines (Fig. 2.9).

The epoxy-Terfenol-D composite coupons were produced using a small casting mould. During casting two hard ferrite magnets were placed at the outer surfaces of the mould to generate a magnetic field (Fig. 2.10). To get the magnetic field as homogeneous as possible, all parts of the casting mould, including assembly screws, were made of aluminium. During casting the mould was heated to 80°C. The resin plate was pre-cured for 4 h at 80°C and post-cured for 4 h at 120°C.

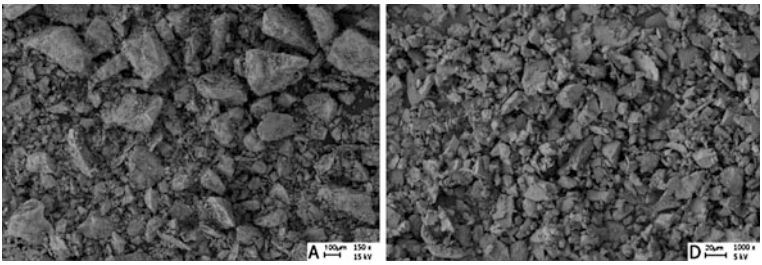

Fig. 2.8 SEM images of Terfonal particles (*left* original batch; *right* particles after sieving with a size <20 μm)

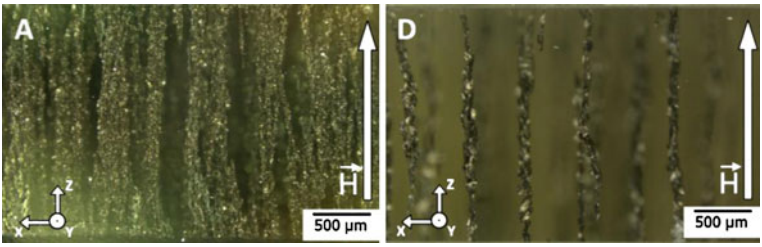

Fig. 2.9 Micrographs of specimen cross sections with Terfenol-D particles of different size and particle content aligned along magnetic flux lines (*left* particle size 15 μm and 20 wt%; *right* particle size <20)

After curing coupons with dimensions of 90 × 10 × 2 mm^3 were cut to size from the plate.

To verify the sensory effect of the Terfenol-D/epoxy composite the coupons were loaded in a universal testing machine. The change of the magnetic flux density against the mechanical stress was measured using a hall-effect sensor, which was applied above the center of the coupon. First measurements showed only little changes in the magnetic flux density. This was significantly improved after performing a post magnetization process with 1.5 T for 30 s using a customized electromagnet. A summary of the test results is shown in Fig. 2.11.

All specimens showed reproducible changes of the magnetic flux density depending on the applied mechanical stress. Besides the particle distribution the particle size as well as the particle concentration had an influence on the magnetic flux density change. Maximum changes were measured with higher particle concentration and lower particle size.

Tough a lot of open question remain the first results are very promising, offering numerous possibilities for future research activities. Up to now only modified neat resins have been investigated. The challenges to integrate them into fiber composites still have to be explored. It is also very interesting to look at alternative smart materials exploiting other physical effects.

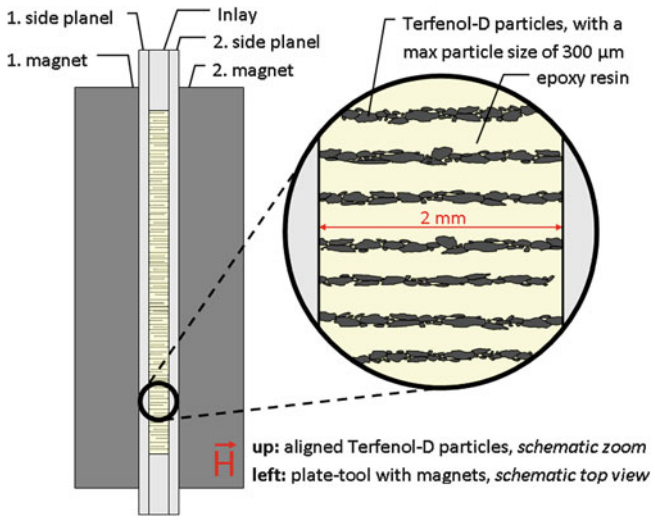

Fig. 2.10 Schematic illustration of the plate tool with magnets and enlarged view of particle alignment

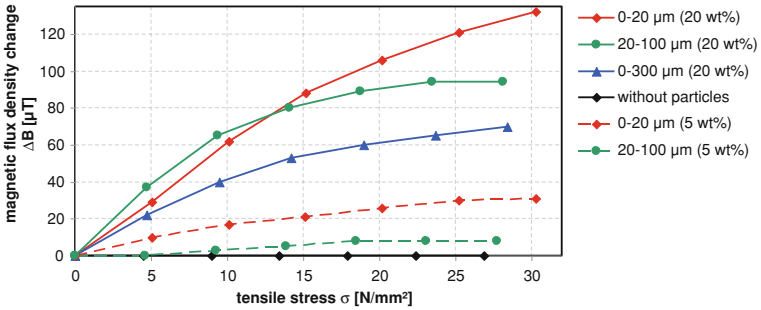

Fig. 2.11 Plots of the measured magnetic flux density change under tensile load

2.6 Summary and Conclusion

The following articles reflect a part of the research work that is dealing with the behavior of composite materials in combination with smart materials on different scales and how this knowledge can be used to enhance the performance by improving mechanical properties, by facilitating the production process of parts and by adding new functionalities.

The improvement of mechanical properties of fiber composites by adding nanoscaled particles materials will be addressed in two articles. Different particle types (epoxy-silica and boehmites) are investigated. Especially matrix dominated

properties of fiber composite laminates like shear strength, compression strength and damage tolerance are considered.

Another article discusses the use of carbon nanotubes (CNT's) as a new superior actuator material for smart composite structures. The goal is to transfer both the extraordinary mechanical properties of CNT's and the possibility to actuate them with very low voltages on a macroscopic composite material.

Piezocomposites and biological inspired piezoceramic honeycomb actuators, as reliable, robust and advanced possibilities to form smart material systems with macroscopic components are presented in two further articles.

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