

MATERIALS WITH VARIABLE STIFFNESS

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ABSTRACT

In this study different concepts to attain a material that can reduce its stiffness upon external stimulation were evaluated regarding their suitability for traffic safety applications.

All concepts rely on resistive heating of a carbon fibre reinforcement upon application of electric current through the fibres. The stiffness reduction is achieved by a phase transformation due to heating of the material. The phase transformation takes place either in a thermoplastic interphase, in a thermoplastic matrix or in a thermoset matrix, depending on the concept.

The different concepts were studied regarding their thermomechanical and processing properties and their ability to reduce their stiffness upon application of an electric current was tested. Moreover, the materials were evaluated regarding their potential for fast activation, which is crucial for applications in traffic safety. Stiffness-reduction was achieved upon application of an electric current, where the activation temperature was between 60 and 120°C and the extent of stiffness-reduction varied between 50 and 90%, depending on the material. The response time was found to depend to a large extent on the amount of material, which leads to the conclusion that smart design solutions are required for larger parts.

It is concluded that the concepts vary in their thermal, mechanical and processing properties as well as in their extent of stiffness-reduction upon activation. The results presented in this work prove the feasibility of the studied materials for traffic safety applications and the concepts allow further optimization of the materials for specific applications.

1 INTRODUCTION

Materials with adjustable stiffness have been studied in recent years mainly for applications in aerospace industry, such as morphing wings or deployable structures. The studied concepts rely on shape-memory alloys and polymers, as well as electroactive polymers, piezoelectric and magnetostrictive materials [1], [2]. Stiffness-modifiable composites have been investigated earlier, but the studied concepts were found to be too complicated [1].

The use of resistive heating of carbon fibres to achieve a phase transformation within a composite material was introduced by Bismarck and co-workers. Materials studied by the group reduce their stiffness upon application of an electric current through the carbon fibres. Two different concepts relying on this mechanism were studied: (i) a thermoplastic interphase between a thermoset matrix and its carbon fibre reinforcement [3] and (ii) thermoplastic layers in a carbon fibre reinforced epoxy laminate [4]. These concepts were designed to combine the good mechanical performance of thermoset resins with the large potential for stiffness-reduction of thermoplastics. However, to date the achieved mechanical performance of the studied materials does not match the performance of thermoset matrix composites, due to some unresolved issues, such as poor distribution of coated fibres

in the thermoset matrix and poor interaction between a thermoplastic layer and the epoxy matrix. Moreover, the studied materials are not suitable for medium or high volume production, as they involve hazardous chemicals and high effort.

Other applications for such materials are found especially in traffic safety. In the European project ENLIGHT, for example, possible solutions for such material and its implementation in a pedestrian-friendly hood design are studied. However, also other applications can be imagined that can reduce injury severity of vulnerable road users and occupants. For example, a softening front beam may be able to decrease the severity of upper leg injuries and a softening A-pillar may exhibit the potential to reduce injury risks for taller pedestrians and cyclists. Moreover, the use of the material in the car interior may reduce injury risks for occupants. Material with adaptable stiffness could even be used for controlling and redirecting damage in the crash structure of a car.

Here we report advances regarding the above mentioned concept relying on a thermoplastic interphase, as well as alternative stiffness-modifiable material concepts for automotive applications, where manufacturability at high volumes is crucial rather than mechanical performance.

A continuous fibre coating process was therefore developed and optimized to advance stiffness-modifiable materials with a thermoplastic interphase for industrial implementation, as reported earlier [5]. Moreover, the potential of conventional thermoplastic and thermoset matrix composites to reduce their stiffness by resistive heating of their carbon fibre reinforcement was studied. For automotive applications, the stiffness-reduction potential of thermoset materials or the mechanical performance of thermoplastics may be sufficient, while the manufacturing of these materials can be achieved at high volumes. Such added functionality is an additional driving force for the implementation of structural composites at large volumes in the automotive industry. In order to evaluate the feasibility of the studied materials for traffic safety applications, the response time was investigated by theoretical considerations and testing.

For the materials studied in the present work, the stiffness-reduction is achieved by the following principles:

- A thin thermoplastic interphase between a carbon fibre reinforcement and a thermoset matrix is heated above its T_g upon application of current through the fibres.
- The matrix of a thermoplastic matrix composite is heated above its T_g upon application of current through the fibres.
- The matrix of a thermoset matrix composite is heated above its T_g upon application of current through the fibres.

The concepts differ in their thermal, mechanical and processing properties as well as their chemical stability. Moreover, the achievable stiffness-reduction differs for the different concepts.

2 MATERIALS AND METHODS

2.1 Composite preparation

2.1.1. Thermoset composites

The composite materials were prepared from coated (see [5] for details) or commercial sized (Hexcel HexTow AS4C GP 12K) fibres by filament winding and vacuum infusion. Eight layers of fibres were automatically aligned unidirectionally on a winding plate with 3 mm bandwidth. The plates were then placed in a vacuum-bag and infused with the epoxy resin (Araldite LY5052/Aradur 5052) applying an infusion pressure of 100 mbar. The resin and tool temperature upon infusion were 40°C and the composite was cured for 15 hours at 600 mbar and 80°C.

2.1.2. Thermoplastic composite

The manufacture of the thermoplastic composite from commingled yarns (COMFIL 54C-LPET-1500) was achieved using filament winding and vacuum bagging. Four layers of commingled fibre bundles were wound unidirectionally on a plate with 2.25 mm bandwidth. The plates were then placed in vacuum-bags at 50 mbar and heated to 210°C for 1 hour.

2.2 Material characterization

2.2.1. Dynamic mechanical thermal analysis (DMTA)

DMTA measurements were performed on a Rheometric Scientific DMTA 4 analyzer in single cantilever bending mode. The free distance between the clamp and the drive shaft was 14 mm. Rectangular samples with fibres in 90 direction with dimensions 30x10x1.5 mm were heated from room temperature to 155°C with a heating rate of 5°C/min. The tests were performed in strain controlled mode with a frequency of 1 Hz (according to ASTM-D7028-07) and with a maximum strain of 0.1%. The specimens were clamped to the frame with an initial clamp torque of 30 cNm.

2.2.2. Testing with applied current

The stiffness-modifiable properties of the material were verified by 3-point bending tests with applied current. A special 3-point bending rig was developed in order to enable safe handling even if high current or potential is applied to the specimen (for a detailed description, see [5]).

In order to assure efficient current transfer to the carbon fibres in the specimen, they were treated according to a procedure described by Deierling [6] to reduce contact resistance upon application of current into carbon fibre reinforced composites. The specimen were polished at both ends with a sharp angle to expose all carbon fibre ends (fibres were oriented in 0 direction), followed by application of a conductive epoxy resin (CircuitWorks[®] conductive epoxy DW2400). The resin was cured and polished with fine grained polishing paper to maximize the contact area to the subsequently applied copper plates. The copper plates were contacted to the power supply using crocodile clamps. The thus achieved resistance of the specimen was around 400%-500% of the theoretical resistance.

A laser beam was used to measure the displacement of the sample and the temperature of the specimen was measured by taping a thermoelement to the specimen's surface. The thus prepared specimen was placed in the set-up and a weight of 6.5 kg was applied at room temperature to determine the initial flexural modulus. To heat the specimen an electrical current was applied using a DC Power Supply (TSX1820P from Thurlby Thandar Instruments) with a maximum output of 18V/20A.

3 RESULTS AND DISCUSSION

3.1 Material choice

3.1.1 Thermoplastic interphase

The stiffness-modifiable composite with a thermoplastic interphase is achieved by electrocoating of carbon fibres prior to infusion with a thermoset matrix. A continuous process for fibre-coating was developed within the European project ENLIGHT [5]. The process was optimized to use a minimum amount of toxic chemicals and energy. However, the mutagenic solvent dimethylformamide (DMF) could not be avoided and the process is rather time- and energy-intensive compared to the other concepts. The matrix was chosen to exhibit a glass transition temperature around 100°C. The RTM resin was a widely used commercial resin system with low viscosity for fast infusion. Hereafter, the material will be named EP/PMMA@CF.

3.1.2 Thermoplastic matrix composite

The thermoplastic composite material can be processed by compression moulding, which is a well-established, fast and cheap process for composite manufacturing. However, it is not straightforward to find commercial thermoplastic matrices that allow a significant reduction of stiffness and at the same time fulfil mechanical, thermal and chemical requirements posed by the automotive industry. In order to achieve fast stiffness-reduction, the thermoplastic matrix needs to exhibit a significant reduction of stiffness within a narrow temperature range. A highly amorphous polymer is desired, as only the amorphous part of a polymer is affected at the T_g . Moreover, in order to allow application in cars, the material should not change its properties below 80°C.

For the present work a commercially available commingled yarn of carbon fibres with amorphous PET fibres from COMFIL (54C-LPET-1500) was chosen as a system to prove the concept, even if the T_g of this PET was 65°C and thus too low for many automotive applications. A respective amorphous

thermoplastic PET exhibiting a higher T_g (81°C) is available, and the production of commingled yarns and/or LPET81 fabrics can be initiated by COMFIL upon industrial demand. Hereafter, the investigated material will be named LPET65@CF.

3.1.3 Thermoset matrix composite

Processing of carbon fibre reinforced thermoset composites can be achieved by RTM or compression moulding (for pre-preg materials), which are suitable for large-scale production. Moreover, many different thermoset matrices with a large variation of glass transition temperatures are commercially available and feasible for the purpose. However, upon choice of the matrix resin the production requirements (e.g. fast curing) need to be taken into account and limit the variety.

For the present work, a RTM resin material was studied. The RTM resin was the same as used for EP/PMMA@CF. Hereafter, the material will be named EP@CF.

3.2 DMTA

DMTA analysis of the materials was performed to verify and compare their ability to reduce the stiffness at temperatures above the glass transition temperature of the coating or the matrix. The obtained DMTA curves of LPET65@CF, EP/PMMA@CF and EP@CF are compared in Figure 1.

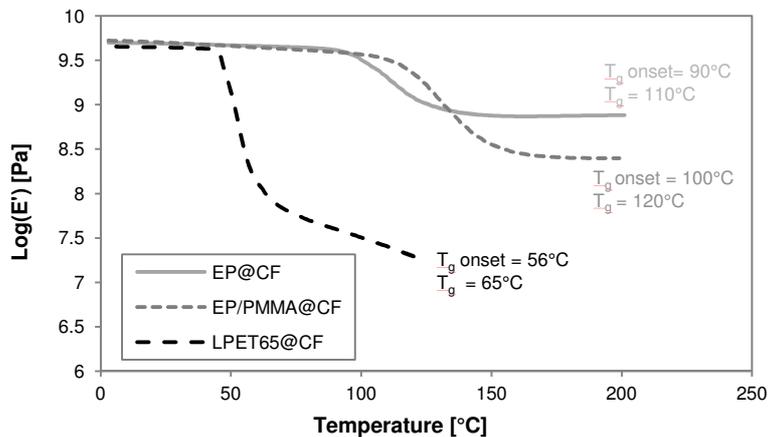


Figure 1: DMTA curves of EP@CF, EP/PMMA@CF and LPET65@CF (fibres in 90 direction).

It is seen from Figure 1 that the reduction of stiffness that can be achieved for the three materials increases in the order EP@CF < EP/PMMA@CF < LPET65@CF. It needs to be mentioned, however, that the values from DMTA at low stiffness are not exact, as the evaluation does not take into account changes in the clamping force above T_g . The achievable reduction in stiffness was therefore verified separately by 3-point-bending experiments.

Results from DMTA show moreover that the stiffness-reduction can be achieved within a small range of temperature for all studied materials, thus fulfilling a crucial requirement to allow for fast reduction of stiffness.

The glass transition temperature for LPET65@CF is significantly lower compared to the thermoset matrix composites. However, as mentioned above, a similar material with a higher T_g (81°C) is available. Due to the higher T_g of PMMA, the T_g for EP/PMMA@CF is increased slightly as compared to EP@CF.

3.3 3-point bending with applied current

The feasibility of this method was evaluated by comparing the stiffness at room temperature of EP@CF and EP/PMMA@CF obtained by this method with 3-point-bending measurements according to ASTM D790-03 (see Table 1). At least three specimens were tested for each material and the results are summarized in Table 1.

	Flexural modulus [GPa]					Maximum relative change
	ASTM	3-point bending set-up (with current)				
	RT	RT	80°C	100°C	Max. Temp*	
EP@CF	105	124 ± 7	93 ± 16	74 ± 3	62 ± 3	50%
EP/PMMA@CF	60	59 ± 8	37 ± 7	20 ± 7	16 ± 7	72%
LPET65@CF		74 ± 5			8 ± 2	89%

* maximal measured temperature for EP@CF samples: 114, 123, 152°C; for EP/PMMA@CF samples: 103, 118, 137, 147, 124°C; and for LPET65@CF samples: 63, 66, 66°C.

Table 1: Flexural modulus in correlation with the measured temperature during 3-point-bending experiments.

In Figure 2 the displacement measured at different temperatures during 3-point-bending tests with applied current is presented for the three studied materials. The corresponding reduction in bending stiffness was calculated to be 89% for LPET65@CF, 72% for the EP/PMMA@CF and 50% for EP@CF.

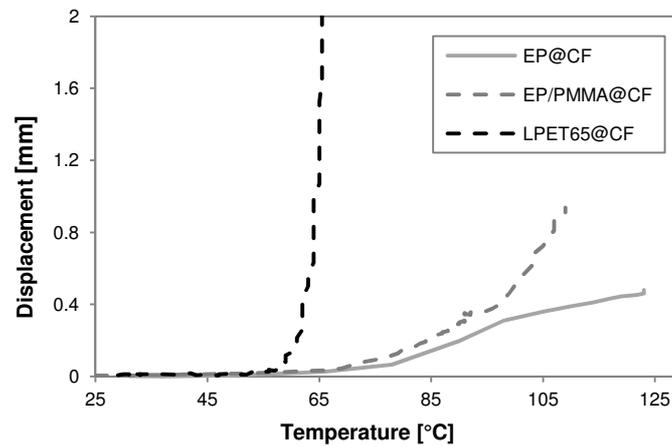


Figure 2: Displacement correlating with temperature measured during application of current in the above described 3-point-bending set-up.

The fibre volume fraction was lower for EP/PMMA@CF than for EP@CF, which results in a lower flexural modulus. The relative change in modulus, however, is assumed to be influenced only to a minor extent by the fibre volume fraction and the obtained results are therefore valid.

Hence, it was proven that stiffness-reduction in all investigated materials can be achieved by application of current through the composite. Moreover, the different efficiency of stiffness-reduction and mechanical performance for the different materials allows selecting the best suited material depending on the application.

3.4 Fast activation

3.4.1. Theoretical considerations

It is crucial for active safety systems in cars to be activated within a short time. Softening materials would make sense for situations, where the time between alert and collision is not sufficient to stop the vehicle. The required time to stop a car is calculated according to equation (1). The driver's reaction time is not considered, as braking will be induced by the same mechanism (either a collision warning system or manually) and thus at the same time as the softening material is activated.

$$t = \frac{v-u}{a} \quad (1)$$

where t is the time (s), v is the final velocity (in this case the vehicle is supposed to stop, hence $v = 0$ m/s), u is the initial velocity (m/s) and a is the deceleration. A usual value for the braking force (a) is ~ 8 m/s² [7], while minimum prescribed braking force is 5.4 m/s², according to European Regulations [8]. The stopping time calculated for normal braking force of 8 m/s² is presented in Table 2 for different speeds.

Initial velocity (km/h)	Stopping time (s)
40	1.4
50	1.7
70	2.4

Table 2: Required time to stop a vehicle depending on its speed (excluding reaction time).

Hence, the presented active safety system relying on a softening material would make sense for cases where time from alert to collision is shorter than 1.5 – 2 s. Therefore, the potential of the studied materials to reduce their stiffness within 0.5 to 1.5 s is investigated.

Only resistive heating of the fibres is considered, as the heat transfer within the matrix is instantaneous due to the short distances between the fibres. The required energy to heat a certain amount of material to different temperatures was calculated using the following equations (not taking into account convection/conduction):

$$mC_p\Delta T = \frac{V^2}{R} \quad (2)$$

$$E = mC_p\Delta T \quad (3)$$

where m is the mass of the carbon fibres (kg), C_p is the specific heat (J.kg⁻¹K⁻¹), ΔT is the aimed temperature difference (K), V is the volume of carbon fibres (m³) to be heated, R is the electric resistivity of the carbon fibres (Ω m) and E is the energy (J). The required power to heat the material within a certain time is calculated from the energy according to the following equation:

$$P = \frac{E}{t} \quad (4)$$

where P is the power (W) and t is the time (s).

It is obvious from these equations, that the required power depends largely on the aimed temperature and the amount of material that is to be heated. Therefore, the required power can be reduced by using a material with low activation temperature (low T_g) and by a smart design where the functional material is used in strategic locations rather than in the entire part.

The time-dependance of the resistive heating of the material is shown in Figure 3 (left) for 1 g of material and different power input. In the right hand graph in Figure 3, the time-dependance of the heating is shown for a power input of 86 kW, depending on the amount of material.

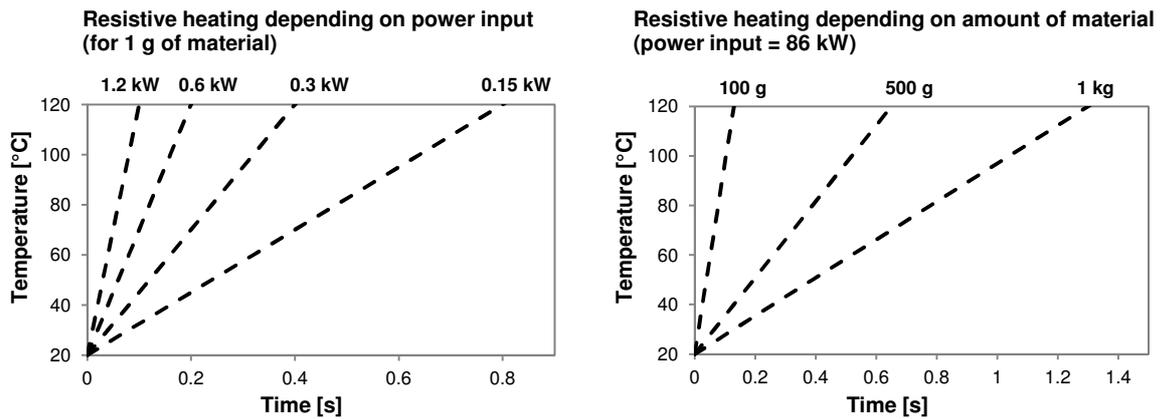


Figure 3: Time-dependance of the resistive heating of the material, depending on the power input (left) or the amount of material (right).

For a hood design as studied in the European project ENLIGHT between 400 and 600 g of material need to be activated. The mid-term goal for power available in an electric vehicle (for a 306 kg battery pack) is about 100 kW [9]. The activation of such hood would therefore be possible within 600 ms for all studied materials (as seen from Figure 2, all materials exhibit a T_g of $\leq 120^\circ\text{C}$). Hence, it could be shown that the activation of such material implemented in a smart hood design requires significantly shorter time than it takes to stop a vehicle from 40 km/h or faster (as seen from Table 2).

Activation of a softening hood is therefore achievable in a shorter time than the vehicle stopping time for relevant speeds. Further improvements of the design as well as the implementation of solutions to utilize the kinetic energy from breaking may allow even faster activation times.

For the evaluation of the concept for other safety-relevant applications it is crucial to consider and optimize the amount of required active material.

3.4.2. Testing of fast activation

The experimental verification of these values was achieved using the same 3-point-bending set-up as described in section 2.3.2. To achieve fast heating, a constant voltage was applied through a pulse generator using a pulse time of 1 s.

The response time of LPET65@CF was investigated to prove the concept. Five specimens with dimensions of 60x10x2 mm were tested. Based on equations (2)-(4), a theoretical voltage input of 2.5 V (power: 40 W) is required for such specimen to achieve stiffness-reduction within 1 s. Figure 4 shows a typical result obtained for such experiment. The actual used pulse voltage was 14 V.

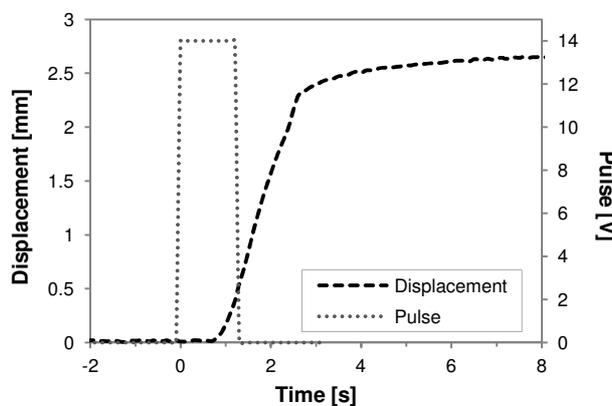


Figure 4: Typical curve for the development of the displacement with time in correlation to the applied potential pulse.

As seen from Figure 4, the material was successfully activated within 1 s. Even if the time to maximum displacement is 4 s, it is assumed that this delay is caused by inertia and does not need to be taken into account.

Higher voltage (about 500%) is required compared to the theoretical calculations. This is in concordance with the measured increased resistance of the specimen. It can thus be assumed that the required power to activate the hood would also be around 500% of the values presented in Figure 3 if the same method of contacting the material would be applied. Hence, it is crucial to optimize power input. Therefore, an alternative method based on establishing electrical contacting already during material processing is considered for future investigations. It is assumed that the contact resistance can be reduced significantly by this method, thus reducing the actual required power for stiffness-reduction within the aimed time.

4 CONCLUSIONS

Three different concepts to achieve a stiffness-variable material were studied. It was shown that an application of all proposed active materials for safety systems is feasible, as stiffness-reduction can be achieved within the required time frame using energy systems available in future electric vehicles. The three concepts resulted in four proposals for stiffness-modifiable materials with different processing requirements, activation temperatures, efficiencies regarding stiffness-reduction and mechanical properties. Hence, the best suited material can be chosen depending on the requirements for the considered application. The materials and their known properties are summarized in Table 3.

Material	T _g	Processing	Stiffness-reduction	Required power (kW) to activate hood within 0.5 s
EP@CF	110	RTM	50%	122
EP/PMMA@CF	120	Fibre coating + RTM	72%	140
LPET65@CF	65	Compression moulding	89%	61
LPET81@CF	81	Compression moulding	Not measured	83

Table 3: Thermo-mechanical and processing properties of the different considered materials and (theoretical) required power to activate a hood based on the respective material.

Although the achievable stiffness-reduction was not measured for LPET81@CF, it is assumed to be similar to LPET65@CF. If a lower reduction in stiffness is sufficient, also semi-crystalline thermoplastic matrices can be considered instead of L-PET, such as regular (semicrystalline) PET. Other common thermoplastics such as PA or PP exhibit glass transition temperatures below 60°C.

Other thermoset matrix materials ranging from different epoxies via polyester resins to polyurethanes can be considered, depending on the requirements for the aimed application. However, due to the crosslinking present in thermoset materials, the stiffness-reduction is generally lower compared to thermoplastic matrices or the studied concept with a thermoplastic interphase. Such limitations may be overcome by a lower degree of crosslinking or special designed matrix systems with longer polymer backbones.

Fast activation was successfully proven on the example of LPET65@CF. The activation time does, however, depend on the amount of material and on the quality of the electrical contact to the fibres. Hence, future investigations will focus on improved contacting to minimize losses due to contact resistance. For the implementation of the material in a traffic safety system it is recommended to consider smart design solutions to minimize the required amount of active material.

High-volume applications require a straightforward, fast and cheap processing method. Hence, the material concepts recommended for automotive industry are mainly thermoplastic matrix composites or – for applications requiring better mechanical and thermal performance – thermoset matrix composites. The matrix can be chosen depending on the requirements regarding mechanical and thermal properties and chemical stability, as well as process requirements and the aimed stiffness-reduction.

The concept based on a thermoplastic interphase is less suitable for large production volumes, due to the large effort and connected expenditure required for the fibre coating process. However, for advanced applications this solution may be interesting, as the material exhibits good mechanical performance and at the same time can achieve large reduction in stiffness.

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