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Dynamic-mechanical-thermal analysis of hybrid continuous–discontinuous sheet molding compounds

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ABSTRACT

Sheet molding compounds (SMC) are very promising for the production of lightweight structural components, due to the specific mechanical properties combined with the suitability for a large-scale manufacturing process. Automotive industry already implements SMC materials for structural components in their vehicle concepts. Polymeric materials, hence also fiber reinforced polymers, show a viscoelastic behavior and dynamic-mechanical-thermal analysis (DMTA) is an important method of determining the influence of temperature and loading speed of this material class. In this work, SMC which based on a novel hybrid resin system were examined under bending loads using a electric-dynamic test system to realize high-force dynamic-mechanical-thermal analysis. The examined SMC materials were either discontinuously (Dico) or continuously (Co) reinforced. In addition a hybrid continuous–discontinuous reinforcement was realized by stacking different SMC materials. The mechanical characterization aimed to investigate the influence of the reinforcement architecture and the effect of hybridization on the temperature- and frequency-dependent material properties. Glass transition temperature of the hybrid SMC was comparable to glass transition temperature of the discontinuous glass fiber reinforced component. Compared to the continuous carbon fiber SMC, the decrease of storage modulus of the hybrid SMC could be shifted to higher temperatures and damping was also significantly increased due to hybridization.

1. Introduction

Fiber reinforced polymers, providing the advantage of high specific stiffness and strength, are in great demand especially in the automotive industry to face the progressive climate change. This material class offers a high lightweight potential as it enables to reduce weight of components and, in a consequent next step, also CO_2 emissions. Besides high specific stiffness and strength, sheet molding compounds (SMC), which in general feature a discontinuous fibrous reinforcement, based on fiber bundles with a distinct length, stand out due to a cost-efficient manufacturing process offering high formability and design freedom. However, a disadvantage of discontinuously fiber reinforced composites is the limited mechanical performance. Unidirectional and continuous fiber reinforced composites impress with their mechanical performance, but, application is limited due to low formability and design freedom as well as high manufacturing time and costs. Hybrid composites, in general, aim to combine advantages of different types of composites to achieve superior properties for a distinct application. Recent contributions have already demonstrated a positive effect resulting from a combination of different fibrous architectures to reinforce SMC. For this purpose, chopped fiber reinforced SMC were combined with pre-impregnated, tailored continuous fiber reinforcements in a single-stage compression molding process [1–3]. This approach, however, has a decisive disadvantage due to high material and manufacturing costs. An alternative approach, presented by Gortner et al. [4,5] based on the integration of dry textile preforms in a standard SMC process. Although this approach led to enhanced mechanical performance, a critical point for resulting material properties was the impregnation of the textile preforms. To overcome high material costs as well as to ensure a sufficient impregnation Bücheler and Henning developed an adopted SMC process. A novel two-step curing resin system enabled to control the viscosity of the paste throughout the entire manufacturing process and SMC reinforced by unidirectional carbon fibers in form of a non-crimp fabric in a unidirectional layout could be manufactured on a slightly modified standard conveyor plant [6]. Based on this novel continuously reinforced SMC, the International Research Training Group (GRK 2078) of the ”Integrated engineering of continuous–discontinuous fiber reinforced polymers”, focuses on development and investigation of hybrid SMC materials [7].

Experimental characterization showed, that mechanical material properties significantly increased due to a combined discontinuous–continuous reinforcement [8]. In addition, hybridization also enhanced quasi-static and dynamic puncture properties, however, the effect of hybridization was less distinct for dynamic puncture, which was attributed to a negative rate dependence of the continuous carbon fiber reinforced SMC [9]. However, both studies were conducted at ambient temperature. In many applications, SMC materials must withstand oscillating forces at elevated temperatures while maintaining structural

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integrity. For this reason and to consider SMC for structural components in serial production, it is important to understand the influence of temperature and loading speed on mechanical performance. Since the material behavior of polymers is highly governed by viscoelasticity [10] an appropriate method to experimentally investigate dependence of frequency and temperature on material properties is a dynamic-mechanical-thermal analysis (DMTA), often also referred to dynamic-mechanical analysis (DMA). In general, DMA bases on applying an oscillating force to a specimen and analyzing the material's response to that force [11]. By recording the phase shift between the sine waves of the applied force and the material's response the temperature- and rate-dependent behavior of polymeric materials can be characterized. The technique is highly sensitive with respect to the movements of the polymer chains. Hence, it represents an ideal tool to measure transitions in polymers [12]. In particular, properties such as storage modulus (E'), loss modulus (E''), damping factor or damping, respectively ($\tan\delta$), and glass transition temperature (T_g) can be determined. Fibers embedded in a polymeric matrix influence the frequency- and temperature-dependent material behavior and a very first attempt to measure and analyze the dynamic-mechanical behavior of fiber reinforced polymers dates back to Gibson and Plunkett [13]. From that point, many publications on dynamic-mechanical properties of, mainly, unidirectionally fiber reinforced polymers aimed for example to investigate the influence of fiber type [14], fiber content [15], fiber orientation [14,16] or the effect of interfacial interactions [17,18] on mechanical performance of fiber reinforced polymers exposed to loads at different temperatures and varying frequencies. Experimental analysis also highlighted, that in fiber reinforced composites viscoelastic as well as viscoplastic material behavior, properties of the interface or interphase, respectively, and damage within the material influence the damping behavior [19,20]. Dynamic-mechanical analysis is also an appropriate tool to identify and describe the effect of dynamic strain [21] or frequency [15] on material behavior as well as to characterize damage evolution in fiber reinforced composites [22].

Recently, investigations also focused on dynamic-mechanical behavior of short and long fiber reinforced polymers and a dependence of elastic and viscous material parameters [23] as well as temperature-dependent stiffness and damping [24] on fiber content was highlighted. Dynamic-mechanical investigations of polypropylene reinforced with discontinuous short glass fibers (30 weight [wt.]%) and produced by injection molding, showed an anisotropic material behavior resulting from fiber orientation. In particular, the stiffness decreased with an increasing angle between the specimen orientation and the injection direction [25].

By means of DMA it is also possible to distinguish between non-linear viscoelastic effects and irreversible stress-induced damage, while both effects were responsible for stiffness reduction in long glass fiber reinforced polypropylene [26]. In general, effects of temperature and frequency on mechanical behavior is more pronounced for thermoplastic materials. However, composites based on thermosets, such as SMC, also show temperature-dependent and viscoelastic material behavior [25,27]. Hence, DMA is also a promising tool to investigate time-, temperature- and frequency-dependent material behavior of SMC materials. Hamdan et al., for example, investigated the variation of storage modulus and loss tangent as a function of storage time and curing of an unsaturated polyester-polystyrene based SMC reinforced with E-glass fibers (30 wt.%, fiber length = 25 mm) [28]. For a vinylester-based SMC composite featuring 50 wt.% of glass fibers, Shirinbayan et al. [29] identified the main transition temperatures due to molecular mobility as a function of temperature with dynamic-mechanical-thermal analyses. In greater detail, three distinct transition zones in a temperature range from 0 °C to 250 °C were found. According to Shirinbayan et al. [30] the first transition, at approximately 130 °C, was related to the glass transition of the SMC. Glass transition region extended from 70 °C to 200 °C. This effect was explained by a non-completed crosslinking, and a post cross-linking during DMTA, that prevented, together with the reinforcing glass fibers, a sudden decrease of the modulus in the glass transition region. The second transition, in the range of 59 °C, identified the brittle-ductile transition for the amorphous polymer, generally related to the mobility of small segments within the molecular chains. From what is understand by now, the origin of the third transition could not yet be clearly identified.

Composites, based on hybrid resins, have also already been investigated by means of dynamic-mechanical analyses. Yu et al. [31], for example, synthesized and characterized carbon fiber reinforced unsaturated polyester/urethane and showed that dynamic-mechanical analysis led to one unique glass transition temperature. This finding was explained by the fact, that the individual components, which were considered to form the hybrid resin, showed good compatibility during blending and probably formed a simultaneous interpenetrating network. In contrast, if components are incompatible, each component exhibits its own unperturbed relaxation process [32].

Static and dynamic mechanical properties of hybrid composites, which were realized by different stacking sequences of several layers made from plain weave glass or carbon fiber fabrics, both embedded in epoxy resin, have been investigated in [33]. Results highlighted that fiber type and stacking sequence had a decisive influence on thermo-mechanical behavior and carbon fibers generally increased storage modulus. In addition, as already resumed by Finegan and Gibson [34], glass fibers improved the damping performance of the composite laminate, whereas it was shown that the position of the glass fiber reinforced layers significantly affect the damping factor of the hybrid composite. Although some effort was already carried out to characterize the dynamic-mechanical-thermal behavior of composites based on hybrid resin systems or hybrid fibrous reinforcement, to the extent of the author's knowledge, this contribution is the first publication on a hybrid continuous–discontinuous (CoDico) SMC, which considers a hybrid resin system for the individual components and, in addition, a hybrid fibrous reinforcement architecture, so taking both aspects into account. As a first step, the study at hand aims to identify the influence of fiber orientation resulting from flow during manufacturing on dynamic-mechanical properties of discontinuous (Dico) glass fiber reinforced SMC, based on an unsaturated polyester-polyurethane hybrid (UPPH) resin system. Then, dynamic-mechanical behavior of a continuous carbon fiber reinforced SMC as well as of a hybrid continuous–discontinuous glass/carbon fiber reinforced (CoDico) SMC is evaluated and, in the end, compared to the non hybridmaterials. This procedure enables to properties of CoDico SMC are the effect of non-hybrid on storage modulus, loss modulus, damping and glass transition as well as temperature- and frequency dependence of the aforementioned properties.

Outline. The work at hand is structured as follows: in Section 2, the (hybrid) composite material is introduced and the specifications on composition and manufacturing are given. The methodology to perform a dynamic-mechanical-thermal analysis with a specifically adapted testing device is described in Section 3. In Section 4, the results of mechanical characterization are shown and discussed. Finally, in Section 5, a summary of the main results is given and conclusions are drawn.

Notation and abbreviations.

T_g	Glass transition temperature
E'	Storage modulus
E''	Loss modulus
E^*	Complex modulus with $E^* = E'(1 + i\tan\delta) = E' + iE''$
$\tan\delta$	Damping factor/damping ($\frac{E''}{E'}$)
θ	Temperature
Dico	Discontinuous
Co	Continuous
CoDico	Continuous–discontinuous
GF	Glass fiber
CF	Carbon fiber

Table 1
Components of resin system to manufacture continuous carbon fiber SMC.

Component	Product name	Supplier
UPPH resin	Daron®	Alyancis
Impregnation additive	BYK 9076	BYK
Release agent	BYK 9085	BYK
Styrene	Mono Styrol	BASF
Inhibitor	pBQ	Fraunhofer ICT
Peroxide	Trigonox 117	Akzonobel
Isocyanate	Lupranat M20R	BASF
Accelerator	BorchiKat 0243	Borchers
Carbon fiber non-crimp fabric	Panex35	Zoltek

Table 2
Components of resin system to manufacture discontinuous glass fiber SMC.

Component	Product name	Supplier
UPPH resin	Daron®	Alyancis
Release agent	BYK 9085	BYK
De-airing	BYK A530	BYK
Inhibitor	pBQ	Fraunhofer ICT
Peroxide	Trigonox 117	Akzonobel
Isocyanate	Lupranat M20R	BASF
Glass fiber	Multistar 272	Johns Manville

2. Materials and manufacturing

2.1. Resin system and semi-finished materials

Manufacturing of the discontinuously as well as of the continuously reinforced semi-finished materials were carried out on a flat conveyor plant (type: HM-LB-800 by Schmidt & Heinzmann, Bruchsal, Germany) at the Fraunhofer ICT in Pfinztal, Germany. The continuous as well as the discontinuous SMC considered within this study were based on an unsaturated polyester-polyurethane (UPPH) hybrid resin system by Aliancys, Switzerland. To meet manufacturing requirements of the discontinuous glass and continuous carbon fibers and to improve impregnation, the resin system was only slightly modified for the two SMC composites (cf. Tables 1 and 2).

Glass fiber rovings were cut to a length of 25.4 mm (1 in.) and dropped onto the conveyor belt on a foil covered by the UPPH resin to manufacture the discontinuous (Dico) glass fiber reinforced SMC. Speed of the cutting unit and conveyor belt were set to achieve a nominal fiber volume content of 23%. The resin and fibers were then covered with another polymeric film and foil to form a sandwich, rolled up and stored for several days to allow the polymeric material to mature.

Alternatively a unidirectional carbon fiber non-crimp fabric was fed to the conveyor belt to achieve a continuous carbon fiber reinforcement of the semi-finished sheets. A heating unit, installed at the conveyor belt, allows to control the paste's viscosity as well as to partially cure the resin system. The stack was then cooled down and the achieved B-stage allowed for handling and cutting of the continuously reinforced material. The continuous carbon fiber reinforced semi-finished sheets featured a nominal fiber volume content of 47%.

2.2. Compression molding and specimen preparation

After maturation of several days, the discontinuously and continuously reinforced semi-finished sheets were cut into plies. The plies were stacked and compression molded at approximately 2500 kN, 150 °C and 112 s mold closing time. To manufacture the discontinuously reinforced materials the stack of the Dico GF SMC was placed in the middle of a rectangular-shaped mold (800 mm × 250 mm), leading to a mold coverage of approximately 35%. During compression molding the material flew in one dimension to fill the mold.

The continuous carbon fiber SMC was compression molded with a mold coverage of 100% within the rectangular shaped-mold, hence the material did not flow.

Hybrid SMC was manufactured by stacking the two different semi-finished materials (Dico GF SMC and Co CF SMC) before compression molding within the rectangular-shaped mold in a one-shot process forcing the discontinuous component to flow between the two continuously reinforced face layers (cf. Fig. 1). All sheets featured a nominal thickness of 3 mm. Specimens for experimental investigation featured a length of 80 mm a width of 15 mm and were extracted by water-jet cutting.

3. Experimental characterization

To investigate dynamic-mechanical-thermal material properties, a specimen is subjected to a stress- or strain-controlled load. During the test, the load, the amplitude of the specimen's deformation and the phase shift between the sine waves of the load and the displacement are measured. Load capacity of standard testing equipment to carry out a dynamic-mechanical analysis (DMA) is limited. Standard testing apparatus only allow for the investigation of non-reinforced materials or of very small specimen sizes, which do not account for the heterogeneous microstructure. Hence, small specimens do not present a representative volume element of fiber reinforced polymers and certainly not for hybrid composites. Hence, within this study, dynamic-mechanical-thermal characterization was carried out with an Instron ElectroPuls E3000$^{\text{TM}}$ electric-dynamic test system with a dynamic nominal force of 3 kN and a load cell capacity of 5 kN. Setup of the machine and testing procedure was adapted to enable DMA measurements and to determine temperature and frequency dependence of mechanical performance. Deflection of the specimen was measured by recording the movement of the crosshead. Before testing was started, measured deflection (crosshead) was compared to deflection values which were determined by a laser position sensor (type: optoNCDT 2300, MicroEpsilon), which was mounted on an aluminum frame beneath the specimen. Deviation between measured distances by the laser and the crosshead was lower than 1%, hence negligible. A water-cooling system enabled to cool down the measurement devices during testing (cf. Fig. 2). The integrated temperature chamber allowed for variation of testing temperature in a range from −100 °C to 350 °C.

Specimens were loaded in three-point bending with a distance of the lower supports equal to 60 mm. As proposed in [35], a three-point bending load was chosen to measure temperature-dependent material behavior, as this loading type eliminates the need for clamping. In addition, it provides a realistic loading case of hybrid laminated SMC structures with continuous face layers. All experiments were strain-controlled. To avoid plastic deformation, mean strain was equal to 0.125%. Amplitude was set to 0.05%. As proposed in [36] quasi-static flexural pre-tests served as reference to define the region of elastic deformation of the considered SMC materials. In addition, pre-load was constant for every temperature step as it might influence material performance, such as T_g [21]. A preliminary test aimed to define a frequency range suitable to conduct DMA with the specially adapted testing device. For this purpose a specimen made of steel with same dimensions as the SMC material was dynamically loaded at room temperature with an increase in frequency. Up to 8 Hz, damping factor ($\tan\delta$) was approximately equal to 0.02 with no significant variation. From that point, up to 20 Hz, $\tan\delta$ slightly decreased to 0.01. Hence, for the DMA of (hybrid) SMC frequency domain was set in between 0.2 Hz and 6.3 Hz to avoid machine-related influences, which might falsify measured properties [14]. In addition to 0.2 Hz, 0.63 Hz, 2 Hz and 6.3 Hz, a frequency of 1 Hz was also considered relating to other studies (e.g. [29]). During testing of SMC materials, temperature within the chamber was increased stepwise within a temperature range of $\theta = -40$ °C to $\theta = 210$ °C. At every temperature

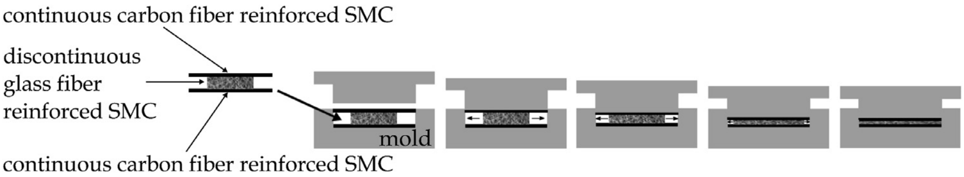

Fig. 1. Compression molding of continuous–discontinuous glass/carbon fiber SMC.

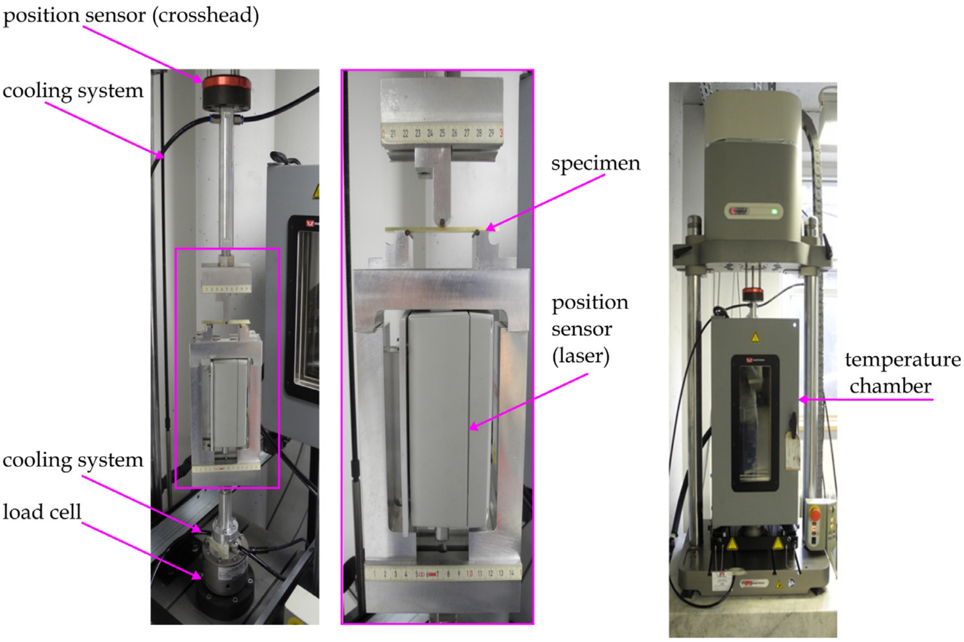

Fig. 2. Test setup to perform dynamic-mechanical-thermal analysis installed on Instron ElectroPuls^{TM} E3000 electric-dynamic test system.

step a frequency sweep was carried out considering 0.2 Hz, 0.63 Hz, 1 Hz, 2 Hz and 6.3 Hz. As T_g might depend from heating rate [19], temperature increase was precisely controlled for all tests. In the beginning of a test, the temperature chamber was cooled down to θ = -40 °C and temperature remained constant for 20 min to ensure homogeneous temperature within the chamber as well as of the specimen and bending device. From θ = -40 °C to θ = 120 °C temperature was increased by 5 K between the frequency sweeps. From θ = 120 °C to θ = 210 °C temperature was increased by 3 K between the frequency sweeps. At each temperature step, the specimen was loaded up to a static mean strain of 0.125% and cyclically loaded with increasing frequency. Unloading of the specimen during temperature adaption between two testing sequences ensured to avoid creep of the material. After each temperature increase a dwell time was realized to ensure acclimatization of the specimen within the temperature chamber ($\Delta\theta$ = 5 K: dwell time ≈ 120 s, $\Delta\theta$ = 3 K: dwell time ≈ 90 s). For the predefined frequencies, preliminary tests enabled to determine the number of cycles, which were necessary to obtain stable values of sought properties (E', E'', $\tan\delta$). Damping factor $\tan\delta$ is an indicator of how efficiently the material loses energy to molecular rearrangements and internal friction [11]. With recorded force and displacement values as well as the phase shift between the obtained sine waves, storage and loss modulus as well as damping factor were evaluated by considering several data points (20% of last recorded cycles at each step) and determining the arithmetic mean value. To define glass transition temperature (T_g) the evolution of $\tan\delta$ versus time was recorded and the maximum recorded value as well as four data points prior and four data points after this point were considered to define a polynomial fit of fourth order. Temperature value of the maximum of this fitted curve (hence analytical peak value of $\tan\delta$) was defined as T_g. Within the study at hand, calculation of arithmetic mean value and standard deviation bases on three individual specimens to quantitatively describe the material behavior.

4. Results and discussion

4.1. Frequency-dependent material behavior of discontinuous glass fiber reinforced SMC

Fig. 3 depicts the influence of frequency on storage modulus (E') and loss modulus (E'') as well as damping factor (tan δ) of two representative discontinuous glass fiber reinforced SMC specimens, which were extracted in flow direction (0°, cf. Fig. 3 top) or perpendicular to flow (90°, cf. Fig. 3 bottom), respectively. Temperature-dependent evolution of damping ($\tan\delta$) reveals that the unsaturated polyester-polyurethane resin system forms one network during curing with one distinct glass transition temperature. A comparable result was also presented by Kehrer et al. for the same hybrid resin system [25]. Regardless from fiber orientation, evolution of storage modulus can be divided in four different stages. From the beginning of the investigated temperature range up to θ ≈ 30 °C, a slight linear decrease of E' is observed independent from testing frequency. Then, up to θ ≈ 90 °C decrease of E' is still linear with a higher rate. A further increase in temperatures leads to a non-linear decrease of E' with increasing severity in glass transition region, indicating viscous material behavior. The higher the testing frequency the lower the temperature at which E' starts to significantly decrease. In the end of the observation ($\theta >\approx$ 170 °C) storage modulus remains constant at approximately 30% and 25% of the initial value for the 0°- and 90°-oriented specimen, respectively. A rate dependence of stiffness of the material is observed within a temperature range between θ ≈ 100 °C and θ ≈ 170 °C.

Due to the homogeneous microstructure of discontinuous glass fiber SMC, mechanical properties are generally determined by scatter. In addition, since bending testing applies a load only to a very small volume of material, local fiber volume content and local fiber orientation significantly affect mechanical performance and results do not directly repre-

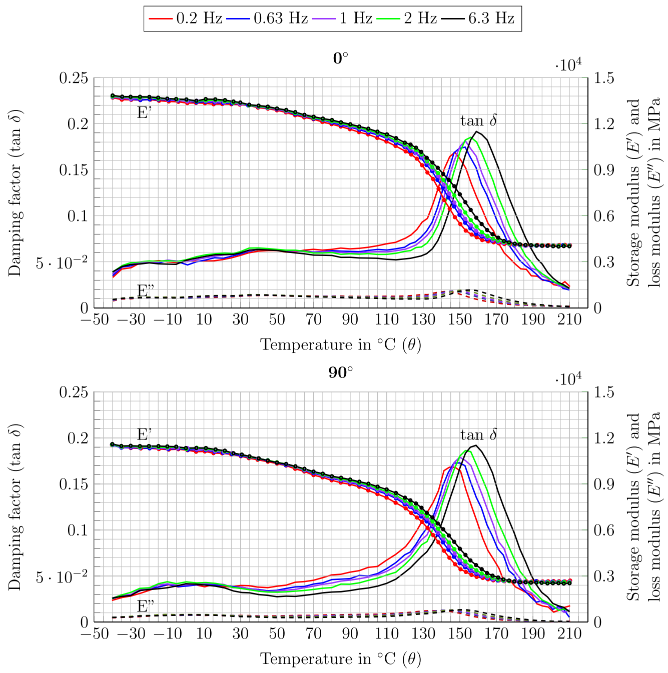

Fig. 3. Representative evolution of storage modulus (E'), loss modulus (E'') and damping factor ($\tan\delta$) of discontinuous glass fiber SMC extracted in flow direction (0°, top) and perpendicular to flow (90°, bottom) as a function of temperature and frequency.

sent mechanical performance of greater volumes of material. To identify the significance of scatter in greater detail, three specimens extracted in flow direction (0°, cf. Fig. 4 top) and perpendicular to flow direction (90°, cf. Fig. 4 bottom), respectively, were considered for mechanical characterization.

At room temperature ($\theta = 25$ °C) arithmetic mean value of storage modulus E' is equal to 13.2 GPa (0.2 Hz) and 13.5 GPa (6.3 Hz), with a standard deviation of ± 1.3 GPa (0.2 Hz) and ± 1.4 GPa (6.3 Hz) for 0°-oriented specimens. Coefficient of variation (CV) is equal to 10% (0.2 Hz) and 10.5% (6.3 Hz), respectively. For 90°-oriented specimens arithmetic mean value and standard deviation of E' are equal to 11.1 GPa ± 0.5 GPa (0.2 Hz, CV = 4.4%) and 11.3 GPa ± 0.5 GPa (6.3 Hz, CV = 4.5%) for 90°-oriented specimens, respectively. Despite of this scatter in mechanical performance, a global trend is clearly visible for all considered SMC specimens, independent from orientation, and E' decreases for increasing temperature. This effect is most severe in the glass transition region. Relative decrease of E' is comparable for different specimens of the two considered orientations. Furthermore, a frequency-dependent material behavior is only present in the vicinity of T_g.

Glass transition temperature (T_g) of Dico GF SMC shifts to higher values as testing frequency increases. For 0°-oriented specimens T_g is equal to 145.8 °C ± 1.2°C at 0.2 Hz. T_g increases to 159.6 °C ± 1.1 °C at 6.3 Hz (cf. Fig. 5). No significant variation was observed for 0°- and 90°-oriented specimens and it can be assumed that formation of glass transition region is not affected by slightly different fiber content or fiber orientation which is present in the inspected volume of material.

Glass transition temperature (T_g) is slightly lower than the values obtained by Kehrer et al. [25] for the same material. The aforementioned study considered a tension loading, hence a greater volume of the material was loaded and glass transition region might be shifted due to a variation in loading case as it results from a complex interaction of matrix and reinforcing fibers. In addition, as described in [37], T_g strongly depends on mode of evaluation and dynamic-mechanical investigation in general are very sensitive to testing equipment and setup [14].

The damping factor ($\tan\delta$), also considered solely as damping, is an indicator of how important molecular rearrangements and internal friction are at a given frequency- and temperature-load. At room temperature ($\theta = 25$ °C) $\tan\delta$ of 0°-/90°-oriented specimens was equal to $0.052 \pm 0.003/0.041 \pm 0.003$ (0.2 Hz) and $0.047 \pm 0.004/0.042 \pm 0.008$ (6.3 Hz). Thus, an increase in frequency only slightly affects damping of 0°-oriented specimens when loaded at room temperature. In addition, damping of specimens extracted parallel to flow (0°) was higher compared to 90°-oriented specimens and fiber orientation slightly influences damping properties of Dico GF SMC. As depicted in Fig. 5 damping of Dico GF SMC increases at higher temperatures especially in the vicinity of T_g. In the glass transition range $\tan\delta$ also slightly depends on frequency. At T_g $\tan\delta$ of 0°- and 90°-oriented specimens increases from 0.17 ± 0.005 at 0.2 Hz for both considered directions to 0.19 ± 0.005 (0°) and 0.19 ± 0.007 (90°) at 6.3 Hz. The damping factor in [25] was slightly lower compared to the obtained values in the study at hand.

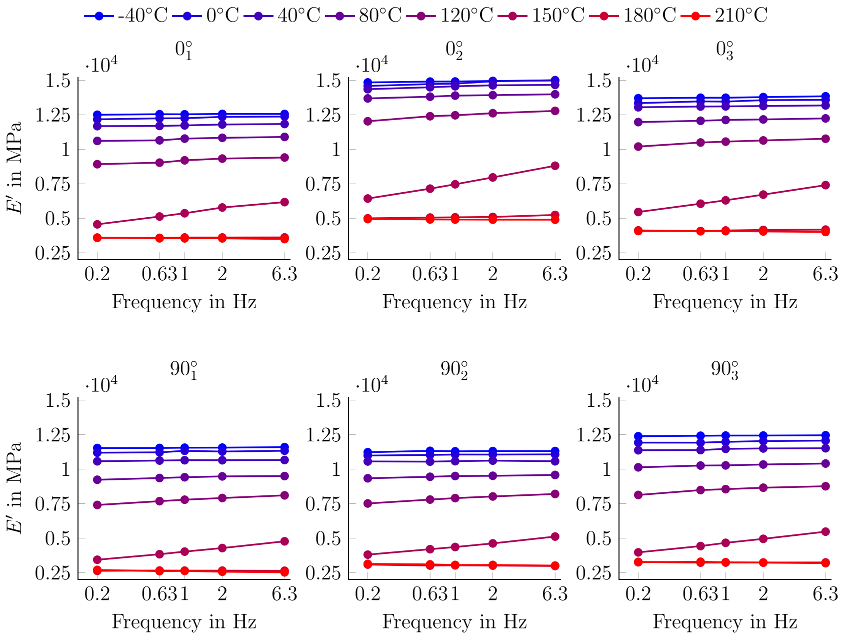

Fig. 4. Frequency dependence of storage modulus (E') at various temperatures. Depicted are results of six different specimens of discontinuous glass fiber SMC. Three specimens were extracted in flow direction (0°, top) another three specimens were extracted perpendicular to flow (90°, bottom).

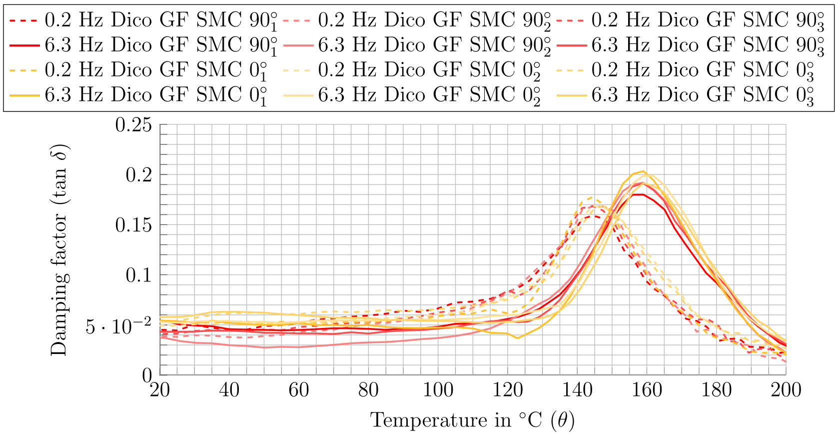

Fig. 5. Frequency dependence of damping factor (tan δ). Depicted are results of three different specimens of discontinuous glass fiber SMC, which were either extracted in flow direction (0°) or perpendicular to flow (90°), respectively. Indices indicate number of individual specimen.

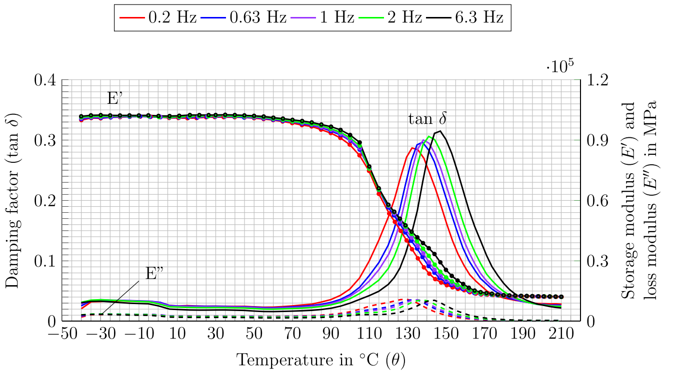

Fig. 6. Representative evolution of storage modulus (E'), loss modulus (E'') and damping factor (tan δ) of continuous glass fiber SMC as a function of temperature and frequency.

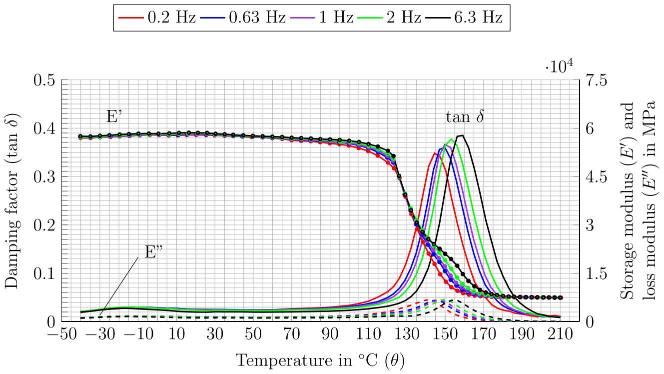

Fig. 7. Representative evolution of storage modulus (E'), loss modulus (E'') and damping factor (tan δ) of continuous–discontinuous glass/carbon fiber SMC as a function of temperature and frequency.

However, in the aforementioned study by Kehrer et al. specimens were loaded in tension and extensional damping factors have been shown to be lower than flexural damping factors [38].

4.2. Frequency-dependent material behavior of continuous carbon fiber reinforced SMC

Frequency-dependent material behavior of continuous carbon fiber reinforced SMC is depicted in Fig. 6. Continuous carbon fiber SMC shows a constant storage modulus E' for temperatures up to $\theta \approx 60$ °C. From that point, storage modulus starts to decrease slightly (in a linear way) up to $\theta \approx 90$ °C. Then, decrease gets non-linear before storage modulus significantly drops as a temperature of $\theta \approx 105$ °C is reached. At room temperature ($\theta = 25$ °C) arithmetic mean value of storage modulus E' is equal to 93.0 GPa (0.2 Hz) and 94.0 GPa (6.3 Hz), with a standard deviation of ± 7.4 GPa and a coefficient of variation (CV) of 8% for both frequencies. Thus, there is no frequency dependence of E' at room temperature for the investigated frequency range. Frequency dependence is only significant in the immediate vicinity of T_g but less pronounced compared to discontinuous glass fiber SMC. Glass transition temperature T_g depends on frequency and increases from 134.6 °C ± 1.1 °C (0.2 Hz) to 148.7 °C ± 1.4 °C (6.3 Hz). At T_g damping factor (tan δ) reaches higher values for higher testing frequencies and arithmetic mean values equals 0.286 ± 0.005 (0.2 Hz) and 0.303 ± 0.006 (6.3 Hz). Damping in the glass transition range is significantly more pronounced compared to discontinuous glass fiber reinforced SMC.

4.3. Frequency-dependent material behavior of continuous–discontinuous glass/carbon fiber reinforced SMC

Frequency-dependent material behavior of continuous–discontinuous glass/carbon fiber reinforced SMC is depicted in Fig. 7. At room temperature ($\theta = 25$ °C) arithmetic mean value of storage modulus E' of CoDico GF/CF SMC equal 58.5 GPa ± 2 GPa (0.2 Hz) and 59.0 GPa ± 1.8 GPa (6.3 Hz). Coefficient of variation is euqal to 3.4% (0.2 Hz) and 3.1% (6.3 Hz), hence, significantly lower compared to the pure Dico GF SMC (0°) or Co CF SMC. Comparable to Co CF SMC storage modulus of hybrid continuous–discontinuous glass/carbon fiber SMC remains constant for temperatures up to $\theta \approx 50$ °C before it slowly starts to linearly decrease up to a temperature of $\theta \approx 110$ °C. From that point E' shows a non-linear decrease and a significant drop at $\theta \approx 125$ °C. The temperature, at which E' significantly drops is shifted to a higher value compared to the E'-evolution of pure continuous carbon fiber SMC. The evolution of the storage modulus depends only significantly on frequency in the vicinity of T_g. Glass transition temperature increases from 144.9 °C ± 0.3 °C (0.2 Hz) to 158.4 °C ± 0.2 °C (6.3 Hz). At T_g damping factor (tan δ) reaches higher values for higher testing frequencies and arithmetic mean values equal 0.329 ± 0.019 (0.2 Hz) and 0.356 ± 0.022 (6.3 Hz). Absolute values, but also standard deviation of damping are higher for hybrid CoDico GF/CF SMC compared to pure Dico GF SMC (0°) or pure Co CF SMC.

4.4. Effect of hybridization on dynamic-mechanical behavior of SMC composites

4.4.1. Storage modulus (E')

At room temperature ($\theta = 25$ °C) continuous carbon fiber SMC face layers enhance E' of hybrid SMC compared to a pure Dico GF SMC by a factor of approximately 4.5. Evolution of relative storage modulus of discontinuous glass fiber SMC (Dico GF SMC), continuous carbon fiber SMC (Co CF SMC) and continuous–discontinuous glass/carbon fiber SMC (CoDico GF/CF SMC) is depicted in Fig. 8. It is visible that not only absolute values but also the evolution of E' is affected by the hybridization. In the investigated frequency range, storage modulus of

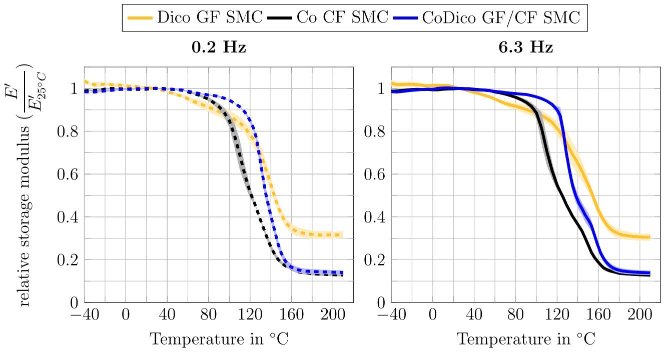

Fig. 8. Evolution of relative storage modulus of discontinuous glass fiber SMC (Dico GF SMC), continuous carbon fiber SMC (Co CF SMC) and continuous–discontinuous glass/carbon fiber SMC (CoDico GF/CF SMC) at 0.2 Hz and 6.3 Hz. Bold and dashed lines indicate mean value, shaded area indicates standard deviation.

Dico GF SMC already starts to decrease slightly for lowest considered temperatures. In contrast, E' of Co CF SMC and CoDico GF/CF SMC only starts to decrease for elevated temperatures and $\theta > \approx 60$ °C and $\theta > \approx 50$ °C, respectively. Hence, for the hybrid CoDico GF/CF SMC, the continuous fibrous reinforcement stabilizes E' up to $\theta \approx 60$ °C. Hybridization furthermore shifts the significant drop of E' to higher temperatures, compared to the pure continuous carbon fiber SMC and it can be concluded that the glass fiber reinforced core has also an decisive effect on the temperature-dependent evolution of E' of the hybrid CoDico GF/CF SMC.

Relative values allow to differentiate stiffness decrease of the specimens with different fibrous reinforcement. Values of E' are related to E' at room temperature ($\theta = 25$ °C). Above glass transition temperature, storage modulus of the Dico GF SMC is $\approx$ 30% of the initial value of E'. Stiffness decrease of Co and CoDico SMC is more severe, as E' drops to $\approx$ 15% if $\theta > T_g$. In the investigated frequency range, only a slight frequency dependence was observed for the relative stiffness evolution of the considered SMC materials in the vicinity of T_g (cf. Fig. 8). To sum up the findings, hybridization of SMC materials stabilizes E' for temperatures $\theta < T_g$ and leads to an increase of the temperature-related application range.

4.4.2. Glass transition and damping

Damping in composite materials depend on numerous factors like fiber type, fiber volume content, fiber length and orientation as well as interface properties. In Fig. 9 evolution of damping factor is shown for one representative specimen of discontinuous glass fiber SMC (Dico GF SMC), continuous carbon fiber SMC (Co CF SMC) and continuous–discontinuous glass/carbon fiber SMC (CoDico GF/CF SMC). At room temperature ($\theta = 25$ °C) $\tan\delta$ of Dico GF SMC equals 0.052 ± 0.003 (0.2 Hz) and 0.047 ± 0.004 (6.3 Hz), respectively. Damping of Co CF SMC is less pronounced (0.2 Hz: $\tan\delta = 0.025 \pm 0.001$, 6.3 Hz: 0.018 ± 0.002). These findings underline the fact, that lower fiber ratios (length/diameter) increase damping of composites due to shear stress concentrations at the fiber ends of the discontinuous fibers and the resulting shear-stress transfer to the viscoelastic matrix [39].

Although hybrid CoDico SMC mainly consists of Dico GF SMC (thickness of the face layers are only ≈ 0.3 mm, each), $\tan\delta$ at room temperature is only slightly increased compared to a pure continuous carbon fiber SMC (0.2 Hz: $\tan\delta = 0.028 \pm 0.003$, 6.3 Hz: 0.024 ± 0.004). Thus, for the hybrid SMC, damping at room temperature is mainly determined by the continuously carbon fiber reinforced face layers.

At 0.2 Hz glass transition regions of Co CF SMC and CoDico GF/CF SMC start approximately at $\theta \approx 90$ °C. Then, the increase in damping is more significant for Co CF SMC compared to hybrid CoDico SMC, which shows only a slight linear increase in damping up to $\theta \approx 120$ °C. From that point, damping of the hybrid CoDico GF/CF SMC also significantly increases. The mentioned temperature ($\theta \approx 120$ °C) coincides with the beginning of the glass transition region of Dico GF SMC and damping of CoDico SMC in the vicinity of T_g as well as peak values of $\tan\delta$ result from an overlay of temperature-dependent behavior of Dico GF SMC and Co CF SMC (cf. Fig. 9). The same trends holds true for mechanical performance at higher frequencies (6.3 Hz). Different sections are only shifted to higher temperatures. Another reason for the increased damping of the hybrid CoDico GF/CF SMC could be linked to a resin rich transition zone, which forms at the interface of continuously reinforced face sheets and discontinuously reinforced core [40].

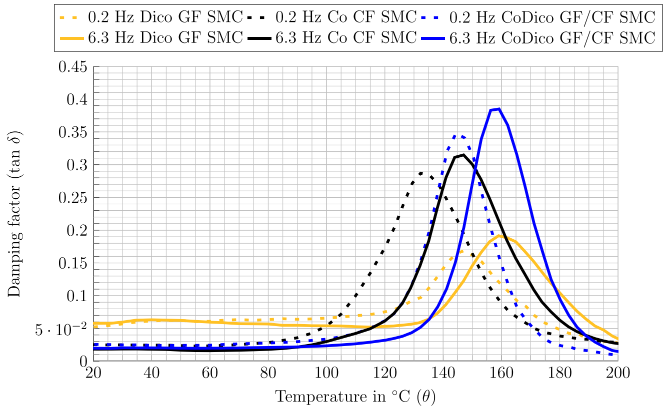

Fig. 9. Representative evolution of damping factor of discontinuous glass fiber SMC (Dico GF SMC), continuous carbon fiber SMC (Co CF SMC) and continuous–discontinuous glass/carbon fiber SMC (CoDico GF/CF SMC) at a frequency of 0.2 Hz and 6.3 Hz, respectively.

As depicted in Fig. 10 glass transition temperature increases with increasing frequency for all considered SMC materials. An increase of one decade leads to an increase of T_g of approximately 10 °C independent from fiber type and reinforcing architecture (cf. Fig. 10, right). No significant difference was observed for glass transition temperature of Dico GF SMC and hybrid CoDico GF/CF SMC. However, T_g of Co CF SMC was significantly lower, although a unidirectional fibrous reinforcement as well as a higher fiber content usually shift T_g to higher values [27]. Chukov et al. also pointed out, that a strong fiber-matrix interface increases immobilization of polymeric chains in thermoplastic materials and shifts T_g to higher temperatures [41]. This effect might also be present in thermosets and if one transfers this relationship to the results obtained in the study at hand, it could be concluded that the hybrid resin leads to a limited fiber-matrix adhesion with carbon fibers, hence, a weaker fiber-matrix interface and lower values of T_g. In addition, as described in [42] curing state directly influences glass transition temperature as uncompleted curing shifts this temperature to lower values. This relationship is well known for polymeric materials and can analytically be described by an approach as presented in [43]. The so-called DiBenedetto-equation, has also been shown to be a convenient constitutive equation for expressing the glass transition temperature as a function of curing for thermosetting polymers [44]. The particular characteristic of the resin system considered for the continuous carbon fiber SMC is a two-step curing process, which provides a chemically stable and highly viscous B-stage ideal for cutting, preforming and handling of the prepregs prior to molding [6]. The level of pre-curing, hence the B-stage, might also have an effect of curing of the composite in the mold and resulting glass transition temperature [45]. In addition, DMA analysis is not only a promising tool to identify the curing state of a polymeric material as it is very sensitive to chain mobility [11,46], uncompleted curing affects glass transition temperature in a more pronounced way compared to mechanical, e.g. bending, properties [46].

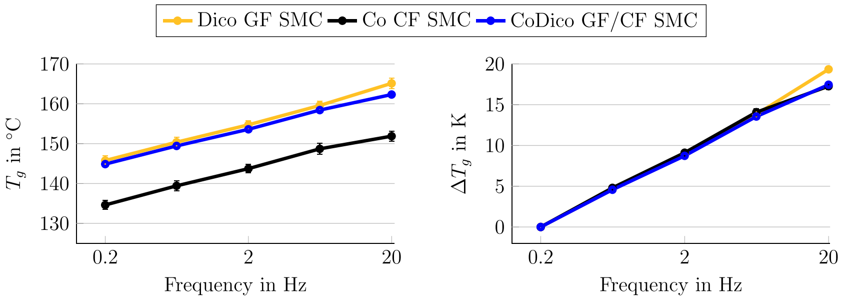

Fig. 10. Temperature- and frequency-dependent glass transition temperature of discontinuous glass fiber SMC (Dico GF SMC), continuous carbon fiber SMC (Co CF SMC) and continuous–discontinuous glass/carbon fiber SMC (CoDico GF/CF SMC). For this investigation a frequency of 20 Hz was additionally considered.

5. Summary and conclusion

The study at hand aimed to evaluate dynamic-mechanical-thermal properties of (hybrid) SMC composites. Mechanical characterization focused on evaluation of storage modulus (E') and damping ($\tan\delta$). These properties reflect temperature- and frequency-dependent stiffness and viscoelastic material behavior. In addition, glass transition temperature (T_g) was determined as the peak value of $\tan\delta$-temperature evolution. The following conclusions can be drawn:

- A novel testing device and adaption of an electric-dynamic loading-frame allowed to carry out dynamic-mechanical analysis with a higher load capacity compared to a standard DMA testing apparatus.
- All considered materials, which were based on a hybrid resin system, showed a temperature-dependent mechanical behavior and one distinct glass transition region.
- Frequency dependence of mechanical performance of considered SMC materials is only significant in the immediate vicinity of T_g. In general, frequency dependence is most distinct for discontinuously reinforced glass fiber SMC.
- Discontinuously glass fiber reinforced SMC composites show slightly anisotropic dynamic-mechancial properties, resulting from one-dimensional flow during molding and small volume loaded in bending configuration. Mechanical performance is also characterized by scatter.
- Hybridization leads to an increase of glass transition temperature T_g of continuous–discontinuous glass/carbon fiber SMC compared to pure continuously reinforced SMC composites.
- Storage modulus of the hybrid SMC remains constant in a greater temperature range below T_g compared to the pure discontinuous glass fiber SMC. As stiffness decrease limits applicability of composite materials, hybridization is promising to enlarge applicability of SMC composites as far as temperature-related application range of the material is considered.
- Hybrid continuous–discontinuous glass/carbon fiber SMC features highest damping of all considered materials resulting from superposition of damping of discontinuous and continuous components. This effect is possibly further reinforced due to a resin rich transition zone between face layers and core of the hybrid SMC.

Finally, to conclude the findings, hybrid continuous–discontinuous glass/carbon fiber SMC is a promising approach to ensure strucutral integrity in a larger temperature range compared to pure discontinuous SMC composites, but also to increase damping to offer increased design flexibility compared to continuous carbon fiber SMC. To evaluate the effect of hybridization on dynamical-mechanical properties in a greater detail and to get more quantitative data, a higher number of specimens, different loading cases and a variation of laminate layup should be considered for further investigations. In addition, testing device should be adapted to allow for higher testing frequencies. In the end, mechanical properties and experimental data could also be considered as input data for analytical [47] or mean-field based thermo-mechanical modeling of (hybrid) composites [25,26].

Data availability

The raw/processed data required to reproduce these findings cannot be shared at this time as the data also forms part of an ongoing study.

Declaration of Competing Interest

None.

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