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Effects of fibre orientation and content on the mechanical, dynamic mechanical and thermal expansion properties of multi-layered glass/carbon fibre reinforced polymer composites

Introduction

A competitive market situation supplemented by exponentially increasing technological advents are continuously pushing the demands for novel material combinations that would pose excellent to outstanding properties in terms of mechanical, dynamical, thermal or dielectric ones, unrestricted solely to high performance, high-strength or lightweight structures. Advanced multi-layered composite architectures appear to be one of the answers, especially for structural engineering applications driven by design factors like manufacturing costs, weight, environmental conditions or material compatibilities. Hybrid composites can be considered as another research path under the focus and extensive development and unfortunately, the source of confusion in a relatively large number of research articles, demanding further clarification and standardization.

Research conducted on hybrid composites, predominantly based on intermingled carbon and glass fibre fabrics, can be traced back to the early 1970s, from the work of Bunsell and Harris [1] or Summerscales and Short [2], that were among the first in the investigation of the mechanical properties of various material combinations in view of their prospective use as lightweight load bearing composite structures. Since then, a variety of combinations emerged as viable architectures to produce fibre reinforced polymer (FRP) composites. However, research conducted to retrieve the mechanical properties has largely been limited to configurations in which fibres have been intimately mixed within the matrix or stacked having various ply lay-up sequences [3].

The success key for developing advanced multi-layered polymer based composite architectures is to enhance the failure strain of their constitutive. The selection of the constitutive materials, their structural or geometry-related parameters, has a significant influence on the overall composite failure strain. Based on the aforementioned specificities, extensive studies were carried out with respect to the effects of different fibre fabrics multi-layering sequencing on the overall tensile and flexural properties of polymer based composites [4-9]. When considering the mechanical characteristics, flexural strength was the most common retrieved from three-point bending tests and reported. In the work of Mujika [10] it was reported discrepancy on the elastic modulus in bending while switching from three to four point bending configuration for different polymers based composite architectures. Although some investigations on the advanced polymer composites behaviour in failure have been carried out [11], published articles scarcely reported tensile and flexural properties in view of the unidirectional carbon and glass fibre multi-layered polymer based composites concerning the differences due to the presence, content and orientation of carbon fibres. The latter can be regarded to industrial data protection and may have been unreleased for public on large.

Dynamic mechanical analysis proved to be a useful tool in the study of polymer based composite materials' behaviour under various temperatures, frequency or external loading conditions. Temperature-dependent dynamic mechanical parameters, such as storage modulus, loss modulus and mechanical damping, allow a closer monitoring of the level of interactions between the polymer matrix and the reinforcements, and the derived Cole-Cole or Cole-Davisson plots proved to be the most expressive and useful data processing tools in sizing the constitutive influence.

Literature provides numerous references focusing on the fibre reinforced polymer composites and their dynamical material properties revealing their inherent structure related particularities and thermal history during their manufacturing [12]. Both organic and inorganic fibres were considered as high-potential reinforcement candidates for the different polymer based composite architectures and dynamic mechanical analysis (DMA) as the most-used testing method in order to size the overall material behaviour under shock and vibrations. Furthermore, it should be emphasised that carbon fibres were the most preferred reinforcements due to their outstanding material properties unattained so far by any other type of materials. This enabled a wide-range of engineering applications ranging from aerospace to marine, civil engineering to transport, sporting goods to automotive industry, etc. [13-16].

Next, the multi-layering concept allowed the advent of an entry in the polymer based composite material class and opened new research directions of studies. Extensive experimental studies were carried out and reported within literature on these types of materials, aiming the understanding of their structural behaviour while subjected to different design and loading constraints [17-19].

Further, rather contraction effects than expansion responses from constitutive were often considered in the composite design for those architectures intended to be developed to be used in harsh environments [20]. Supplementary, negative overall CTE response was retrieved for composite benchmarks (e.g. GFRP) whose polymer resins were exhibited low elastic modulus and high expansion material properties. A representative example of the aforementioned can be the glass fibre/polypropylene system developed by Ito et al. [21]. Carbon fibres naturally exhibit different CTE responses along their longitudinal and transversal directions and are usually selected as reinforcements for multi-layered polymer composite structures to tailor their overall CTE. Supplementary, several studies were reported in literature on the multi-layered polymer composites' material properties changes if pre-conditioned under extreme environments [22-24].

The herein contribution presents a comparative study centred on the design, development and material characterization of some tailored multi-layered polymer composite architectures reinforced with different content of uni-directional (UD) carbon and random glass fibres. The elastic and dynamic mechanical properties, along with their thermo-physical changes, were monitored and recorded within a temperature range below and beyond the glass temperature of the unsaturated polymer resin employed as the matrix material, and next analyzed and debated. Supplementary, it will emphasize the differences on the effective material properties under the focus due to the orientation of the UD carbon constitutive with respect to the general reference system. Moreover, the content of the UD carbon fibres ply-ups, that are balancing symmetrical or unsymmetrical the overall composite design, will be considered for further explanations.

Experimental research

Materials selection and specimens preparation

The composite specimens have been manufactured as a 5-ply laminate with different volume fraction ratio of the reinforcement materials – glass fibre chopped strand mats (n. GF; MultiStrat^{TM} Mat ES 33-0-25, Johns Manville, USA) and UD carbon fabrics (n. CF; Panex$^{®}$ 35 UD300, Zoltek Co., H). The reinforcements were bounded together by an unsaturated polyester resin (Synolite 8388-P-1, DSM Composite Resins, CH) and further cured at room temperature for 24 h before cut into specimens for the experimental tests. The curing agent methyl ethyl ketone peroxide (MEKP) was purchased from AKZO Nobel (Butanox M-50, NL). The multi-layer composites were labelled according to their relative volume fraction with the structure as GF:CF(100:0), GF:CF(60:40) and GF:CF(80:20), respectively, keeping constant the overall fibre loading (35 vol.%). All samples were cut in accordance to account for the 0° and 90° different carbon fibre orientation.

Material testing procedures

Mechanical testing. Tensile and 3-point bending loading were performed to assess the mechanical properties in accordance with SR EN ISO 527-1:2000 and SR EN ISO 14125:1998 standards. LS100 and LR5K Plus devices from Lloyd Instruments (UK) were used to retrieve the stress/strain dependencies, followed by elastic modulus, yield stress, ultimate strength recovery procedures to preliminary asses the multi-layered reinforced polymer composites' toughness. The final experimental data were statistically processed out of those recorded for each 10 representative specimens used.

Dynamic mechanical thermal analysis (DMA). The dynamic mechanical thermal analyses were performed using a DMA 242 C analyzer (Netzsch GmbH, D) running in a 3-point-bending mode at an oscillating frequency of 1 Hz. The storage modulus (E’) and loss modulus (E”) as well as the damping factor (tan δ) of each composite specimen were retrieved under the following conditions: a temperature range from - 40º C up to 150º C at scan rate of 3º C/min, a controlled atmosphere due to the liquid nitrogen used as a cooling agent. The samples were shaped having 60 mm in length and 10 mm in width, while the thickness were individually measured as dependent on the layering sequence.

Thermo-physical changes. Thermo-physical changes of specimens were performed on a differential dilatometer DIL 420 PC/1 from Netzsch GmbH (D), in accordance with ASTM E228-11 and DIN 53752-A standards. The specimens were shaped into rectangular bars of 25 mm in length and 5 mm wide while the transversal external surfaces were polished to guarantee plan-parallel surfaces for precise positioning within the measuring head. The temperature mode was set up as a dynamic heating ramp trend from 25° C up to 250° C. The heating rate imposed was 1° C/min.

Results and discussion

Mechanical properties

There is evidence that the individual constitutive fibre content has an influence on the composite's failure strain. The UD carbon fibres content increase and their different orientations with respect to the reference system led to a shift in the overall fracture event curve. A shift was encountered in the load-deflection curves retrieved from flexure tests on the GF:CF(100:0) and the GF:CF(80:20) specimens, on both directions of carbon fibre orientation as shown in Figure 1.

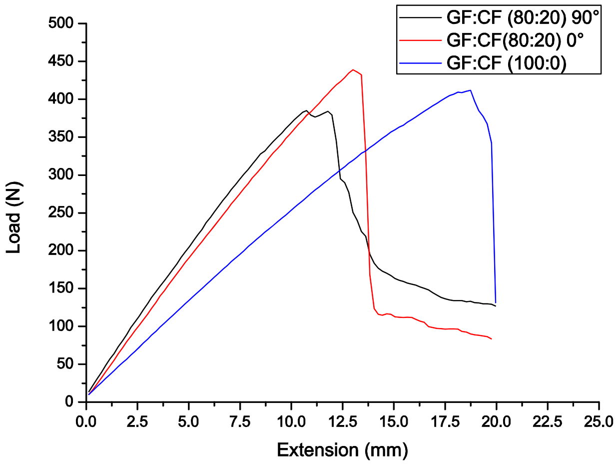

Figure 1. Load versus extension curves of the GF:CF(100:0) and GF:CF(80:20) composites in flexure.

The composite elastic modulus is another dependent material property on the fibre volume fraction; the experimental flexural modulus is much lower than the tensile modulus as the content of UD carbon fibres increases. This it can be seen from data provided in Table 1. The decrease in the flexural modulus can be regarded as a shear-deformation effect within these multi-layered polymer composite specimens, being one of the failure mechanisms that are particular about these types of materials. Furthermore, the effective elasticity modulus in flexure can be correlated with the fibre's length, especially for the UD carbon fibres. The aforementioned is acknowledged in the technical literature to dominate this elastic property [3].

Table 1. Tensile and flexural mechanical properties accounting for different content and orientation.

Sample architecture	UD CF orientation	Tensile		Flexural	
		Strength (MPa)	Modulus (GPa)	Strength (MPa)	Modulus (GPa)
GF:CF(100:0)		116.17 ± 0.04*	4.81 ± 0.03	38.10 ± 0.04	4.68 ± 0.03
GF:CF(80:20)	0°	295.6 ± 0.03	14.64 ± 0.09	25.15 ± 0.05	4.74 ± 0.07
	90°	95.8 ± 0.02	7.60 ± 0.05	32.40 ± 0.04	4.23 ± 0.07
GF:CF(60:40)	0°	331.37 ± 0.01	16.21 ± 0.07	108.12 ± 0.05	4.48 ± 0.05
	90°	93.15 ±0.04	7.22 ±0.05	118.14 ± 0.04	4.21 ± 0.04

* Standard error.

Inconsistencies between the experimental values, proven various UD carbon fibres orientation and number of layers, can be ranked as being relatively low for data retrieved based on the flexure tests in comparison with the tensile tests where a severe drop of approximately 50% in the elastic modulus was found to occur for the same composite architecture but different UD carbon fibre orientation. Moreover, the discrepancies between the multi-layer composites' overall strength are evenly more accentuated, especially in the composite architectures with longitudinal (0°) oriented UD carbon fibres. The effective mechanical properties of these advanced composites are being also influenced by the capacity of stress transferring between the fibre-matrix interfaces and fibres' different breakage mechanisms. Further inside on the above is beyond the purpose of the present study.

In Figure 2 was plotted the maximum bending stresses of the reference, and the multi-layered composite specimens considered in the herein study. Data scattering can be regarded to the increase of the carbon fibre reinforced content, different orientation directions and less to the uncontrolled random distribution of the glass fibres.

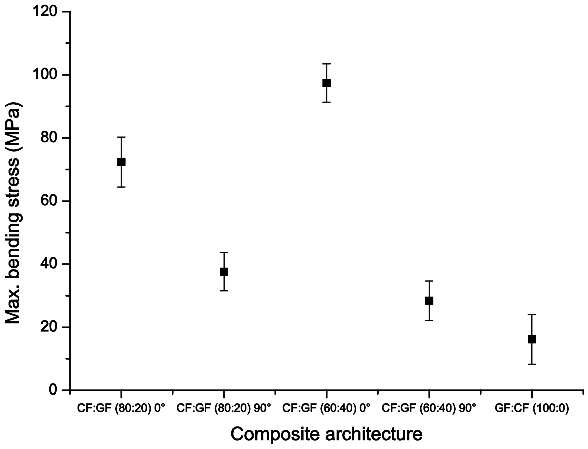

Figure 2. Mean maximum stress values in flexure retrieved for the multi-layered composites.

Dynamic mechanical thermal analysis (DMA)

Dynamic mechanical properties (DMA) of the multi-layered fibre reinforced polymeric composites can be regarded to reveal the same dependencies on several influencing factors as their benchmark, such as: *material-related dependencies* - nature of the matrix material, constitutive material type; *structure-related dependencies* - layer architecture (e.g. reinforcing fibre orientation), number of layers, nature of fibre-matrix interfaces; *loading conditions* – frequency, temperature range, type of load and temperature history [12].

In this section, a detailed analysis will be made with respect to the dynamic mechanical properties of the multi-layered polymer composite specimens investigated, and some of the above-mentioned influencing factors will be closely monitored and ranked on the retrieved experimental data.

Storage modulus (E'). The storage modulus is closely related to the load-bearing capacity of a material and is analogous to the flexural modulus measured as per ASTM-D 790 [12]. Figure 3 reveals the effect of temperature dependence of the storage modulus of GF:CF(100:0) and the multi-layered composite architectures under the focus. Accordingly, one might observe that E' values decrease as the number of the UD carbon fibre ply-ups increases and changes their orientation with respect to the general reference system. The latter can be regarded to the fact that the modulus of elasticity (i.e. Young's modulus) of the UD carbon fibre is much lower in the transversal direction than in the longitudinal direction. Addition of carbon fibre ply-ups was expected to lead to an increase of the E' modulus of these architectures of polymer based composites. Nonetheless, based on these simple observations, UD carbon fibres can be viewed as behaving better in standalone configurations than in combined multi-layered structures but are proving once again that their presence may contribute significantly to an efficient stress transfer within the structure as well as from the fibre constitutive to the matrix.

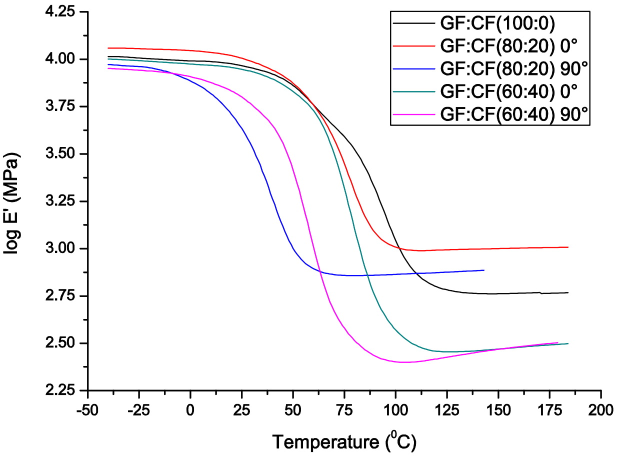

Figure 3. Temperature dependence of log E’ curves for all composites under the study.

As temperature increases, close to glass transition temperature (T_g), the E’ of the reference and multi-layered composites experience a sharp decrease indicating that the materials are passing through the glass/rubbery transition stage. Further, above T_g, the storage modulus of the GF:CF(100:0) is lower than that of GF:CF(80:20), having UD carbon fibres both 0° and 90° oriented but higher than that of GF:CF(60:40), of same type. The aforementioned can be regarded to the increases in molecular mobility of the polymer chains above T_g temperature, as well as to the UD carbon fibre content and temperature dependence.

Loss modulus (E’’). Generally, the loss modulus represents the viscous response of the polymer based materials or can be a measure of energy dissipated as heat/cycle under deformation [12]. As in the case of the storage modulus, the loss modulus preserves the same tendency of high values over the temperature range for reduced numbers of carbon fibre ply-ups embedded within the multi-layered composite architecture (see Figure 4). A discrepancy can be observed throughout the transition region, especially for the maximum E” in case of GF:CF(60:40) and GF:CF(100:0) specimens. This can be attributed to the inhibition of the relaxation process within the composite structures with the addition of the UD carbon fibres as a supplementary phase. However, GF:CF(80:20) multi-layered composite architecture has resulted in higher E” values within temperature range above it’s T_g.

Below the T_g values of each specimen, the E’’ curve for GF:CF(100:0) composite is closer to the curve retrieved for the GF:CF(80:20) architecture, whereas after these values, is getting nearer to the curve retrieved for the GF:CF(60:40) combination, proven the longitudinal orientation of the UD carbon fibres. In Table 2 was listed the glass transition temperature values retrieved from the E” curves, for all the specimens involved in the present study, revealing once again the influence of the number of UD carbon fibre ply-ups and orientation. Glass temperature values retrieved using aforementioned curve types were reported within literature to be more realistic compared to those obtained from damping factor plots and their associated experimental values [19].

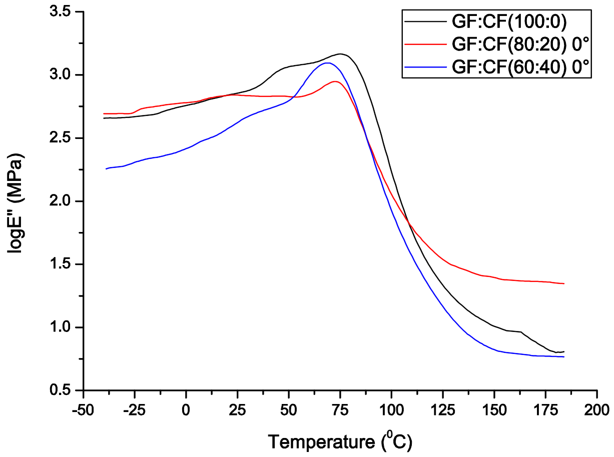

Figure 4. Temperature dependence of log E'' curves for composite specimens with 0° oriented UD carbon fibres.

Table 2. Peak height and glass transition temperatures (from tan δ, E'' and dL/L_0 curves) of multi-layered composite architectures.

Sample architecture	UD CF orientation	Peak height of tan δ curve	Temperature [° C]		
			T_g from tan δ	T_g from E"	T_g from onset dL/L_0
GF:CF(100:0)	-	0.3762	91.2	89.0	52.0
GF:CF(80:20)	0°	0.3030	87.4	85.0	55.0
	90°	0.3241	88.4	83.6	55.0
GF:CF(60:40)	0°	0.5182	86.2	81.3	58.0
	90°	0.4782	88.0	83.4	59.0

Damping factor (tan δ). The material loss factor or loss tangent is related to the impact resistance of a material and depends, as has already been shown in literature, on the applied frequency [12]. The damping properties of the investigated polymer based composite materials provide the balance between the elastic and viscous phases of the individual architectures. Generally, the higher tan δ peak values the better the material performances are in shock and vibrations, whereas the lower tan δ peak values the higher the load-bearing capacity properties are of the polymer based composite materials. It is widely acknowledged that a peak in tan δ curve represents an energy-absorbing transition, whereas the area beneath represents the amount of energy absorbed.

In Figure 5(a) was plotted the tan δ curves function of temperature, accounting for different UD carbon fibre content and orientation, whereas in Table 2 was provided the tan δ peaks and T_g values retrieved from tan δ for all the combinations considered. Figure 5(b) represents a detailed excerpt from the tan δ curves to reveal the influence of the UD carbon fibre content and orientation.

It is interesting to note that the loss factor experience, in almost all cases, a positive small shift as the UD carbon fibre content and orientation change. These positive shifts in T_g values show the effectiveness of the GF and CF fibres as reinforcing agents and will be not linked to any post-curing effects. For example, in case of GF:CF(60:40) multi-layered composites the shifting of T_g to higher temperatures may be associated with the decreased mobility of the polymer chains by the addition of supplementary carbon fibre ply-ups. Based on the diminishing in the tan δ, an improved fibre/matrix interface bonding behaviour can be observed in case of GF:CF(80:20) architecture while compared to the GF:CF(100:0) specimen. Moreover, in case of 90° UD carbon fibre configurations, the same tendencies hold with respect to the tan δ variations.

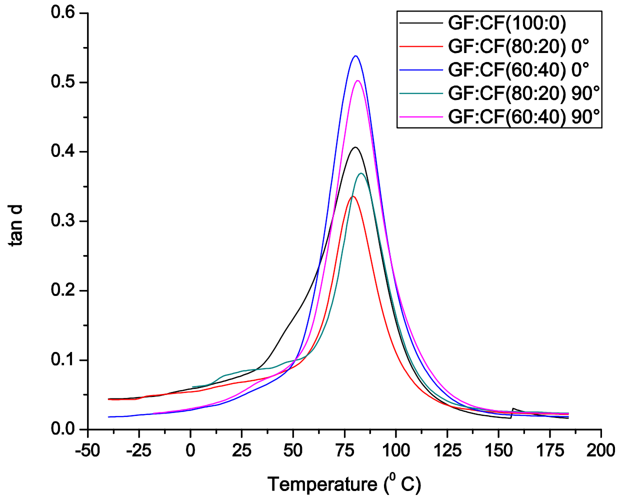

Figure 5(a). Temperature dependence of tan δ curves for all the composite specimens under discussion.

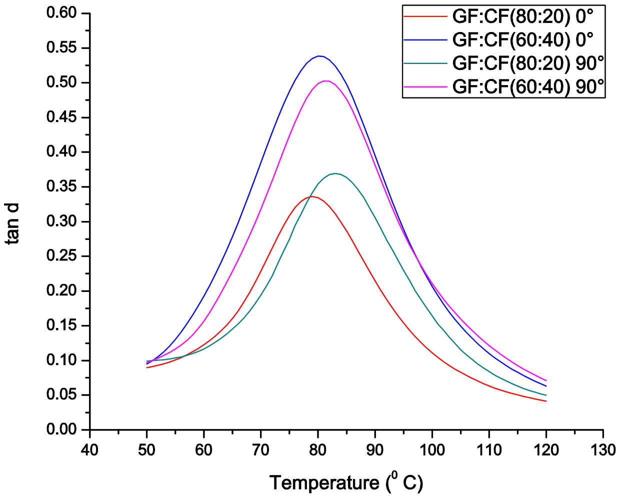

Figure 5(b). Zoom around tan δ peaks accounting for different UD carbon fibre content and orientation within the multi-layered composite architectures.

Cole-Cole plots. Generally, these types of graphs are reported to be good indicators of the homogeneity of the structures subjected to dynamical loading. Departure from the ideal circular shape reveals the heterogeneity of polymer based systems [12].

Figures 6, (a) and (b) shows the Cole-Cole plots of the loss modulus (E”) as a function of the storage modulus (E’) for the reference and multi-layered composite structures accounting for different UD carbon fibre content and orientation. As it can be seen, both content and orientation of the UD carbon fibre contribute to the Cole-Cole plot shape changes. While in case of the 0° disposed UD carbon fibres, the changes give rise to imperfect semicircular shapes to the Cole-Cole plots, for their reverse configuration (i.e. 90°), the form of plots changes to an irregular shape. These imperfections indicate that there is heterogeneity among the glass and carbon fibres, as well as the polymer matrix material. Consequently, the content and orientation of constitutive, supplemented by their individual material properties influence the shape of the Cole-Cole plots, thereby the tailoring process regarding the dynamical mechanical properties of these multi-layered composite architectures.

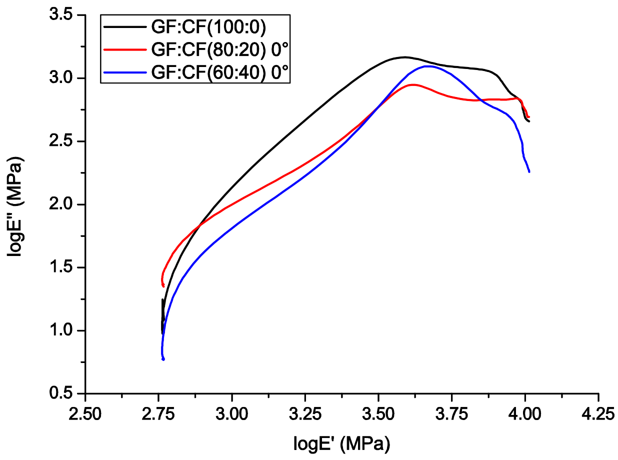

Figure 6(a). Cole-Cole plot of the composites with different content and 0° orientation of UD carbon fibres.

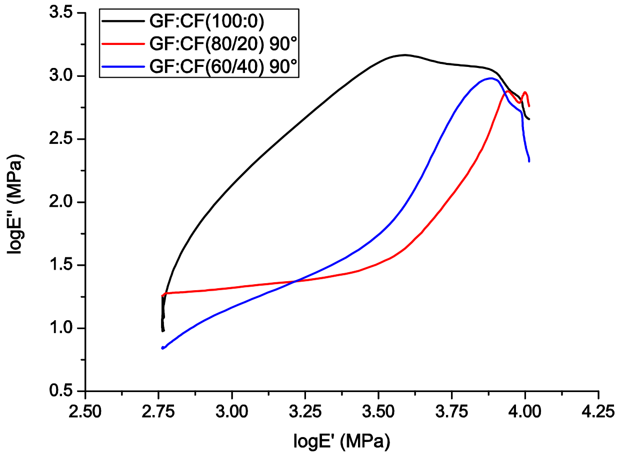

Figure 6(b). Cole-Cole plot of the composites with different content and 90° orientation of UD carbon fibres.

Thermo-mechanical analysis (CTE)

The instantaneous coefficient of thermal expansion (CTE) as well as the thermal strain field recorded for the polymer based composite specimens herein can be viewed in Figure 7 and Figure 8, respectively. These are accounting for different orientation and content of the UD carbon fibre constitutive. Supplementary, as it can be seen from Figure 7, the different layer architectures induce the occurrence of little peaks (i.e. noise) in the instantaneous overall CTE field over the temperature range, departing from the linear variation for temperatures higher than T_g values. These can be associated to the weak cross linked network of the unsaturated polymer matrix herein, to the transitions from the glassy state to the rubbery state as well to the different fibre/matrix interfaces.

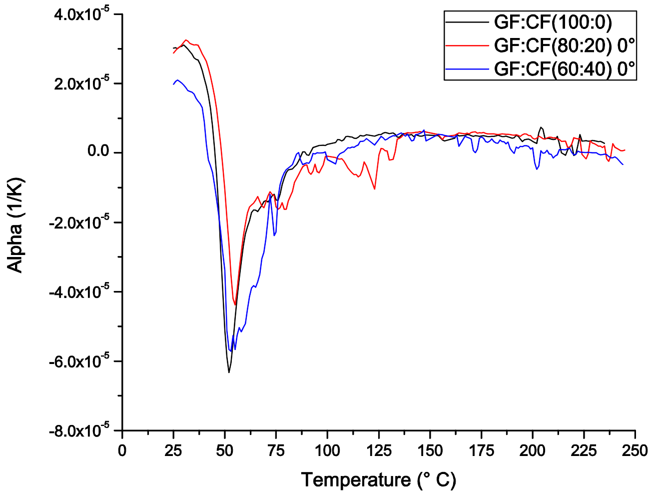

Figure 7. Instantaneous overall CTE temperature variations accounting different content and 0° orientation of UD carbon fibres within the composite architectures.

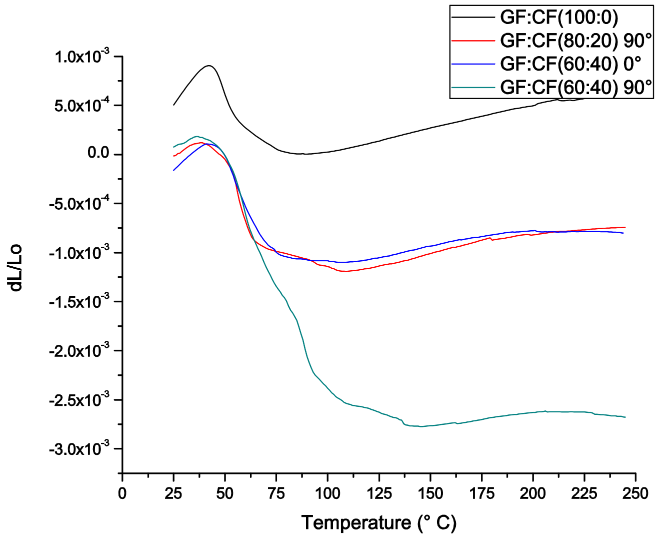

Figure 8. Thermal strain fields evolution from the composite specimens under the study.

With respect to dimension changes, the differences in the recorded variations reveal the influence of both UD carbon fibre content and orientation. One interesting observation that must be underlined herein, concerns the similarities revealed by the GF:CF(80:20) with 90° UD oriented carbon fibres and GF:CF(60:40) with 0° UD oriented carbon fibres during the temperature rise. It seems that the specimens exhibit almost identical behaviour in terms of dimension change over the temperature range, as shown in Fig. 8, and relatively close instantaneous CTE values within the same interval. This may be useful while manufacturing costs come as one of the major issues in the structures design and applications development.

Concerning the T_g values retrieved from the onset in the dL/L_0 curves, these are lower than the ones retrieved from the DMA runs, as has been reported also in Table 2. As it can be seen from the table, a shift of approximately 30° C from the DMA reported values was recorded for all values retrieved for the multi-layered composite specimens. This can be assigned mainly to the experimental device working principle that cannot be ranked as providing the best results concerning the T_g values comparatively with the DMA method. Any further debate on this issue is beyond the purpose of the herein article. It must be pointed out that the DIL measurements give good indicators on the compatibility of the reinforcements with the matrix materials as well as on the influence of the individual constitutive on the overall material properties of the composite architecture.

Conclusions

These architectures of advanced polymer reinforced composites exhibit unique material properties while compared with the benchmark polymer composites, either glass or carbon fibre reinforced. They were developed to replace the benchmark polymer composites in engineering applications where the manufacturing cost and weight are key issues in the lightweight structure's design.

High flexural properties and high-tensile strength were retrieved in case of GF:CF(60/40) multi-layered composite architecture. The increase in the carbon fibre reinforced content was found to contribute further to the improvement of the structural strength, both in tensile and flexure mode. Furthermore, carbon fibres reinforced ply-ups are revealing improved stress transfer behaviour between fibres and polymer matrix as compared to their concurrent (i.e. glass fibres based). Further studies should be considered to investigate the stress transfer mechanisms among the individual layers and fibre to matrix interfaces.

Furthermore, the GF:CF(60/40) architecture was found to exhibit improved resistance if subjected to shock and vibrations comparatively with the reference. It was also shown that the UD carbon fibre content and orientation with respect to the general reference system contribute to the shape changes in the Cole-Cole plots.

The instantaneous CTE responses were also found to be influenced by the UD carbon fibre content and orientations. Besides, the influence of the constitutive individual material properties on the effective CTE was also underlined and revealed in the retrieved experimental curves.

The automotive industry was identified as the primary industry that can benefit from the use of this advanced polymer based composite structures. One may also include the aerospace or marine structural components, sporting goods manufacturing, etc. proven the presence of the carbon fibre constitutive within the composite architectures and supplementary, their yearnings for development of lightweight structures.

Nevertheless, the advent of advanced polymer based composite architectures based on particle/particle, particle/fibre or fibre/fibre multiple tailoring largely opened the combination possibilities and allow outstanding achievements with respect to their effective material properties, manufacturing technologies and costs as well as their wide-spread application areas.

Acknowledgement

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