Experimental characterization of fibre-reinforced composites improved with nanofibres or nanotubes

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Abstract. A review is presented on the testing and mechanical properties of continuous fibre reinforced composites modified with nanotubes or nanofibres either dispersed in the resin or grown on the microfibres. The nano-level cross-links are shown to be able to (1) increase the fibre/matrix interfacial strength, (2) reduce the inter-fibre crack growth, and (3) improve the inter-ply delamination resistance. A positive influence on the thermal expansion is also detected. However, for unfavourable material constitutions, the strength properties can stay almost the same or even significantly deteriorate.

1 Introduction

Continuous fibre reinforced plastics (FRPs) are characterised by a high strength and stiffness at a low weight. However these advantages are obtained in the fibre direction, while the transversal mechanical properties are governed by a low-strength polymer matrix and fibre/matrix interface. Thus they are the weaker links in a composite structure, and improvements of the transversal properties, especially strength, would extend the applicability of FRPs.

To attain this goal, many efforts are put into improving the fibre-matrix interface by treatment of micro-fibres (MFs), their sizing, or "whiskerization". Meso-scale methods like z-pinning or structural stitching are also studied. During the last years, carbon nanotubes (CNTs) and nanofibres (CNFs) are recognized as another, probably the most perspective way. Two main methods exist to introduce CNTs/CNFs into a preform. Usually they are simply mixed with the resin or (rarely) integrated into films which are further dissolved. This technique is used in the most of known studies [1-16]. Sometimes electrical field [5,20] or natural resin flow [11] is used to align CNTs/CNFs in the matrix. The second and less used way is to grow CNTs/CNFs on MFs, e.g. by the catalytic decomposition of hydrocarbon gas over metal particles [27-32], or (rarely) to attach them onto MFs by electrophoresis [33]. Several studies deal with CNTs/CNFs employed as sizing for MFs [15,34].

The primary reinforcing phase is represented almost equally in the reviewed studies as glass [4,5, 7,8,11,15,17,18,20,21,23,24,26] or carbon [1–3,6,9,10,12–14,19,22,25,30,31,33] fibres. A few known studies [17,28,29,32] deal with other fibres: aramid, silicon carbide or alumina fabrics are used, respectively. The present review aims at a comparison of available test data and methods.

Notations: in the figures below, circle markers denote the test data for composites with grown CNTs, squares – for composites with nano-modified matrix, and triangles – for nanosizing. In the second case, the weight fraction of CNTs/CNFs is counted in respect to the resin.

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2 Tensile tests

The in-plane uniaxial tension is conducted according to ASTM D3039, D 638, ISO 527, or another appropriate method. It is anticipated that the nanoscale reinforcement most strongly influences the matrix-dominated mechanical properties, while the in-plane ones are typically dominated by the fibre reinforcement. Thus, the measured in-plane tensile moduli of hybrid and base composites are almost the same, Fig.1 (left), accounting for a natural statistical difference. There are three studies, [7,13,26], which do not follow this rule. But this discrepancy should mainly be attributed to a large thickness variation between different plates: 23-30% for tensile tests in [13] and up to 20% in [7]. Only a 37% improvement in the direction transversal to the glass fibres in [7] may partially be accounted for the nano-reinforcement. In other studies reviewed in this section, maybe except for [33], the difference in thicknesses was within 5% and thus should not play a prominent role.
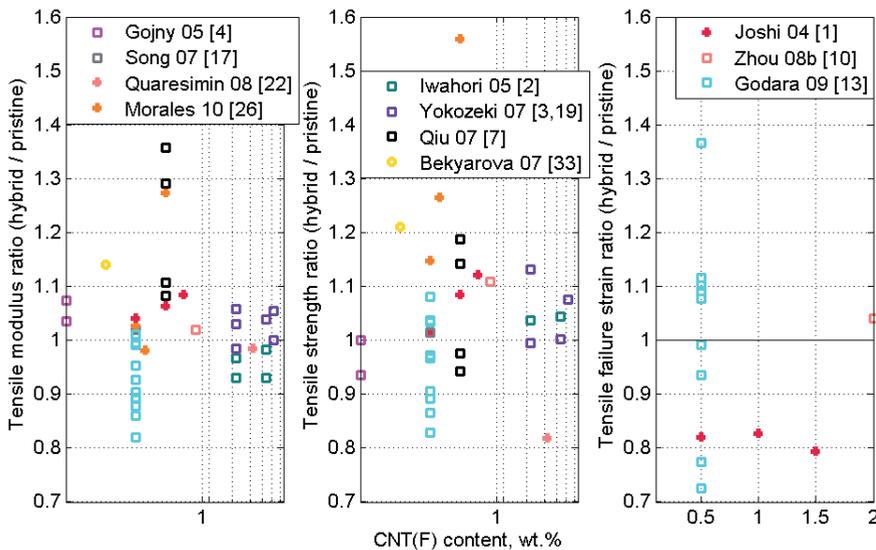

Fig. 1. Left to right: normalised Young's modulus, ultimate stress, and ultimate strain.

The thickness discrepancy should also be taken into account when considering the influence of a nano-reinforcement on the ultimate stress and strain under uniaxial tension, Fig.1. In [13], a systematic decrease is seen for the tensile strength and strain of a UD composite tested in the fibre direction, up to 20%. This is attributed to the known fact that agglomerates of CNTs can act as defects and therefore degrade the composite strength, even under the longitudinal loading. Under 45° or 90° loading, the trend towards increased ductility (higher transverse failure strain) can be explained on the hypothesis of improved crack bridging in the matrix. But since the corresponding ultimate stresses change within the range of the standard errors, it is more reasonable to treat the tests as not very reliable due to the thickness problem noted above.

In [3], besides the stiffness and strength values (which are slightly rising after CNFs addition), the damage accumulation is also investigated. A clear retardation of the matrix crack onset and accumulation is obtained in the specimens with CNFs compared to the pristine ones. It is suggested that CNF dispersion results in a fracture toughness improvement and residual thermal strain decrease. Using an SEM, the authors observe that the fracture surfaces after a 5 wt% and 12 wt% CNF dispersion are clearly rough compared to that of a 0 wt%. Using a chemical penetrant and X-ray radiography, the damage accumulation and cracking patterns are studied. It is observed that the cracks number in a hybrid CFRP is much less than that in the base one.

In [32], the tension-bearing (ASTM D953) behaviour is improved well, as well as the failure mode changes from the matrix dominated (no CNTs) to the fiber dominated one (with CNTs).

3 Compression

Amongst the published studies only [2,19] deal with the uniaxial in-plane compression. The tests lead to the conclusion that hybrid CFRPs (5 or 10 wt% of CNFs to the resin, 10 or 50 CNF length-to-diameter aspect ratio) exhibit higher compressive strength than the base CFRP, up to 15%. The stiffness is also slightly increased, up to 7%. No clear dependence on the CNF wt% or aspect ratio is detected. In [2], according to a photo shown in the paper, normal tensile setup was used in the compression regime. Of course, a specialized test set-up (ASTM D 3410 IITRI, or D 6641, or the one used in [19]) should be recommended, to provide a good specimen and load alignment.

4 Flexural strength and stiffness

Three-point bending is used in the known studies which generally follow ASTM D790, ISO 178 to obtain the flexural strength and modulus. The summary data are shown in Fig.2.
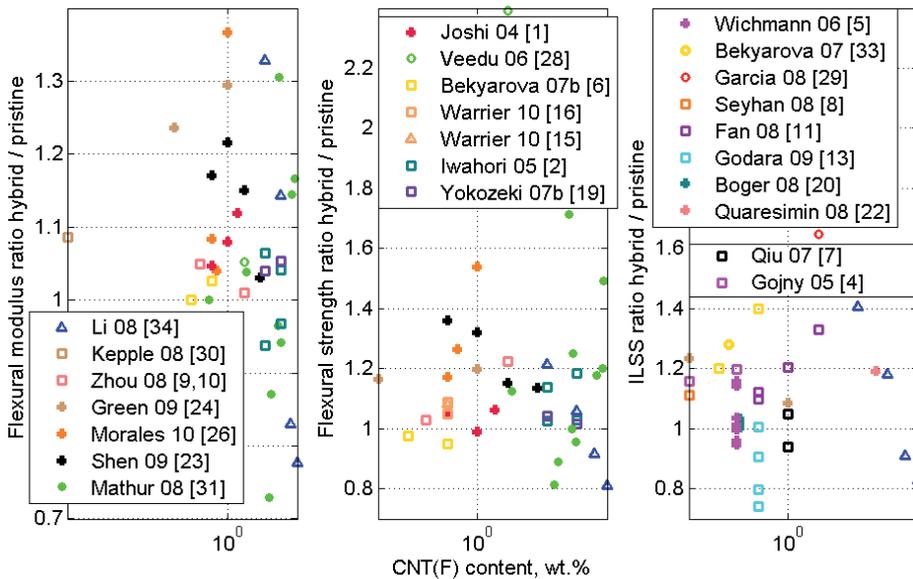

Fig. 2. Left to right: normalised Young's modulus, ultimate stress, and ILSS.

Similarly to the tensile stiffness discussed above, the flexural one is also mainly fibre dominated, and incorporation of CNTs/CNFs is not expected to change it much. Indeed, except for several data points [23,24,26,31] which could be due to a thickness discrepancy, the other data for the epoxy-based composites are situated within a ±10% statistic distortion range.

It should be pointed out that study [34] deals with a special case (unique amongst the others discussed in this paper) of carbon-carbon composites, when it is possible to use large (here, up to 20%) amount of CNFs, using immersion in a CNF-rich solution and further drying. In this case the matrix plays a much more important role for the stiffness, as revealed by a 30% improvement at a 5 wt.% CNF loading. The CNFs play a role of a bridge in the pores; then, it is more difficult for a crack to initiate and propagate. However in the additive range of 10–20 wt.%, both the flexural modulus and strength degrade; this may be due to larger porosity and aggregation in the final composite which are revealed by microscope observations.

For polymeric hybrid composites, better flexural strength is attributed also to a better bonding between MFs and matrix due to CNTs [15]. With further addition of CNTs the flexural strength is often further improved. In [28], besides the strength and stiffness, the flexural toughness is assessed also and shows a 424% enhancement compared with the base composite.

5 Interlaminar shear strength (ILSS)

Three-point bending is usually used, following ASTM D2344. It should be kept in mind that this popular short-beam shear test gives rather qualitative results due to a complex stress field [11] and, as it is written in the specification, "it is not generally possible to relate the short-beam strength to any one material property"; i.e., pure interlaminar shear failure rarely takes place. As shown in [11], the statistical confidence of this method can be very poor, and the standard deviation in the measurement data can be too large. Much more efficient practice is to use the double-notch compression testing (ASTM D3846) as done in [6] or compression shear test as done in [11].

The summary data are shown in Fig.2 (right). Even taking into account some unreliability of the short-beam shear test and thickness discrepancies in several data sets, it is clear that ILSS significantly increases in the hybrid composites. The most remarkable improvement (about 70%) is obtained in [29] and is attributed to the growth of relatively large (2 wt.%) amount of CNTs on MFs. This method is thus compares favourably with a matrix nano-modification or nano-sizing. This is because the well-aligned CNT forests provide better resin impregnation and arrange a more efficient structure than CNTs/CNFs randomly oriented in the resin or in the sizing (two latter methods are prone to form CNT agglomerates or MF bundlings which cause unimpregnated zones).

A carbon-carbon composite with moderate amount of CNFs, [34], also shows a well-improved ILSS, especially of the composite with a 5 wt.% CNF loading (nearly 40% rise in ILSS) where an extensive CNF breakage appears at the fracture surface. However when the additive content increases to 15 wt.% and higher, ILSS degrades because of detrimental porosity and aggregation.

In [6] it is observed with SEM that the fiber/matrix debonding is smooth in the base composites, while the hybrid ones show a reduced level of debonded MFs. This indicates again that CNFs contribute to resisting delamination by the crack bridging effect.

Two important points are touched in the literature when discussing ILSS tests. The first one is the role of CNTs type and their chemical functionalization, presumably resulting in a stronger CNT-matrix interface. In [4,5] the best results are achieved with DWCNTs. In [13] several CNT types are used to fill the resin. The test data show a systematic decrease of ILSS for the composite systems with MWCNTs, TWCNTs, or DWCNTs followed by an increase when functionalized MWCNTs are utilized. However functionalized DWCNTs show even larger ILSS degradation. Possible reasons are damages introduced to the CNT surfaces, decreased CNT aspect ratio, and the level of functionalization. In the earlier reported literature [4,7,11] the increase in ILSS is also seen as a results of a successful functionalization.

The second point is the role of CNT orientation. In [5] it is reported that the application of an electrical field in the thickness direction (while curing the composite) results in a slight increase of ILSS. In [11] is was observed that the preferential orientation of MWCNTs in the thickness direction (due to modified vacuum infusion method) is suspected to contribute to the increase in ILSS, although the measured difference consists only 2% .

6 Mode I interlaminar fracture toughness (G_{IC})

This characteristic (also named as Mode I critical strain energy release rate) is determined using the double cantilever beam (DCB) test, according to ASTM D-5528. In this test, a thin release film strip (e.g., Teflon) is embedded into the laminate to introduce a starter "crack". The crack onset value of G_{IC} is usually calculated at the point of a sudden load drop, as indicated by the load–displacement curve at a constant displacement rate. In the strict sense, this test method "is limited to use with composites consisting of UD... laminates with brittle and tough single-phase polymer matrices" but is often used for other composites too. Besides the analytical formula having a number of correction factors, the main drawback of this method is that G_{IC} onset may be affected by the starter "crack" features, and G_{IC} propagation values depend on the technique used to trace the crack tip (a travelling microscope should be used but, of course, it does not monitor the inside crack front). In [14] the

acoustic emission (AE) is employed to detect the crack tip position more accurately. At a 0.3 wt.% CNTs, this technique shows that G_{IC} propagation value increases by a 13 or 33% according to the conventional or AE based method, respectively. Thus, this is the very sensitive issue of DCB test.
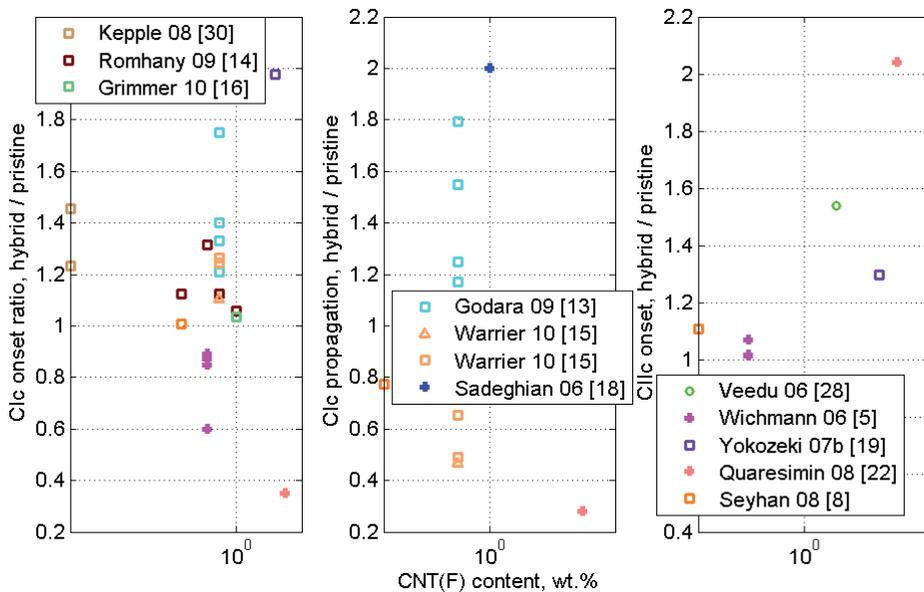

Fig. 3.
Left to right: normalised C_{IC} onset, C_{IC} propagation, and C_{IIC} onset. C_{IC} from [28] is not shown to keep a reasonable scale, since the hybrid composite shows a 348% rise compared with the base one.

Summary data are shown in Fig.3 (left and centre). It is generally seen that the hybrid composites show a significant improvement in G_{IC} onset level. The single and prominent exception is reported in [5] and could be explained by the testing difficulties mentioned above.

In [13], a consistent increase is observed for G_{IC} onset when MWCNTs, DWCNTs, TMWCNTs, or functionalized-MWCNTs are used for the hybrid composite. This highlights the role of optimization of the CNT-epoxy bonding, as well as a correct choice of CNT type. Normal MWCNTs are the least effective but still lead to a 21% increase, despite their poor interfacial interaction with the epoxy matrix and high chances for agglomeration. TMWCNTs and DWCNTs are believed to avoid extensive agglomeration.

When CNTs are used as a sizing [15], the hybrid composite shows even lower G_{IC} onset improvement, and G_{IC} propagation value falls even below the baseline. This may be attributed to the fibre bundling which impairs the crack bridging mechanism. The same is seen for G_{IC} propagation for several other hybrid composites studied in [15]; in this case, the degradation should perhaps be explained using [8] where the authors offer the following hypothesis. CNTs randomly oriented in the resin could limit the fiber nesting which is a major source of the crack bridging. Then the adhesion mechanism of two adjacent plies may be altered, and G_{IC} is thus impaired; it is supported by fractography revealing less extensive fibre-matrix debondings in the hybrid composites. This hypothesis could also explain some test cases in Fig.3 (left)

In [14] interesting AE results are presented. The acoustic activity reveals that the damage front advances in the form of multiple small cracks. When such a micro-crack shoots forward and meets a CNT, it often stops, and another micro-cracks starts to propagate if having a more favourable location. Above 0.5 wt.% CNT loading, another failure mechanism dominates; CNTs gather into agglomerates resulting in a lower AE activity, i.e. in a lower resistance against the crack propagation.

In [12], the composites are investigated both at a room and cryogenic (-150°C) conditions. Because of rapid crack propagation at the low temperature, the work of fracture is evaluated instead of G_{IC}. This dissipation energy is obtained by integrating the crack growth resistance curve (R-curve)

for a stable crack growth region. The results reveal that CNTs have a minor influence at the ambient conditions but a prominent influence at the cryogenic ones; e.g., the dissipation energy for 0.7 wt.% loaded specimens is by a 6.4% and 31% higher, respectively, than that of the baseline specimens.

In [12] the effect of CNTs on the crack density is measured too. After six thermo-mechanical cycles (0 to -150°C, 1/2 of the failure load), the hybrid specimens show a lower crack density (by a 18% for 0.2 wt.% or a 28% for 0.7 wt.%); this agrees well with the fracture toughness enhancement.

7 Mode II iterlaminar fracture toughness (G$_{IIC}$)

This characteristic (also named as Mode II critical strain energy release rate) is measured using the end notched flexure (ENF) test, when the specimen with an end starter "crack" is loaded in a three-point bending flexure, according to DIN EN 6034. The crack is then propagates in the shear sliding mode; however, the propagation is inherently unstable under the displacement control, and only G$_{IIC}$ onset values are usually used. This test method also has some drawbacks, often yet more pronounced than for Mode I DCB test discussed above [42].

Available test data is rather limited, Fig. 3(right). Only [22,28] demonstrate a significant improvement in G$_{IIC}$ after growing a 7.5% of CNFs or a 2 wt.% of CNTs. Three other studies [5,8,19] do not report a significant improvement for the tested hybrid composites.

In [8] it is observed that the specimens show a discontinuous crack growth by micro-crack coalescence which forms many hackles at the fracture surface. The CNT bundles are seemed to act as rigid fillers which arrest the cracks, preventing or delaying their expansion.

In a number of studies [32,35–41] G$_{IIC}$ (as well as G$_{IC}$) is measured for laminates where CNTs are introduced only in several iterlayers (or at the midplane only). The interfacial bonding is reported to be enhanced by 70-300%; the points are not shown in Fig.3, just to avoid too mixed picture.

8 Hardness index

This property is addressed only in [28] where the indentation modulus and hardness are measured using a nano-indenter. A consistent enhancement (about 15%) is observed for both properties for the hybrid composite compared with the base one.

9 Impact resistance

In [28] the notched impact strength was measured following ASTM D256 (resistance of plastics to a "standardized" pendulum hammer). An 8% improvement in the impact strength for the hybrid composite could be explained by the aforementioned crack bridging effect in the modified matrix. In [19], impact damage resistance and tolerance are studied; no clear improvement is observed. In [25] no difference is observed under a low-velocity impact but CAI strength is better by a 12-15%.

10 Fatigue

By now this issue is addressed only in [10] for tension–tension fatigue with a 0.1 R-ratio, in [16] for pre-cracked specimens under zero–tension fatigue, and in [25] for post-impact compression-compression fatigue, also with a 0.1 R-ratio. It is shown that a CNT-modified matrix can extend the specimen life by a factor up to three. The fracture surfaces evidence that CNTs restrain the crack propagation by its bridging, CNTs fracture, and CNTs pull-out; the roles of these phenomena are dependent on the applied cyclic strain energy.

11 Coefficient of thermal expansion (CTE)

The thermoelastic dimensional stability is tested usually under vacuum or in nitrogen atmosphere, using the thermal mechanical analysis (TMA) or ASTM E289 with a standard or laser interferometry. Summary data are shown in Fig.4 (left) for CTEs measured in the transversal direction (90° for UD composites or through-the-thickness for others). For UDs, the longitudinal CTE (0° direction) is fibre-dominated and thus does not change when CNTs are used [3].
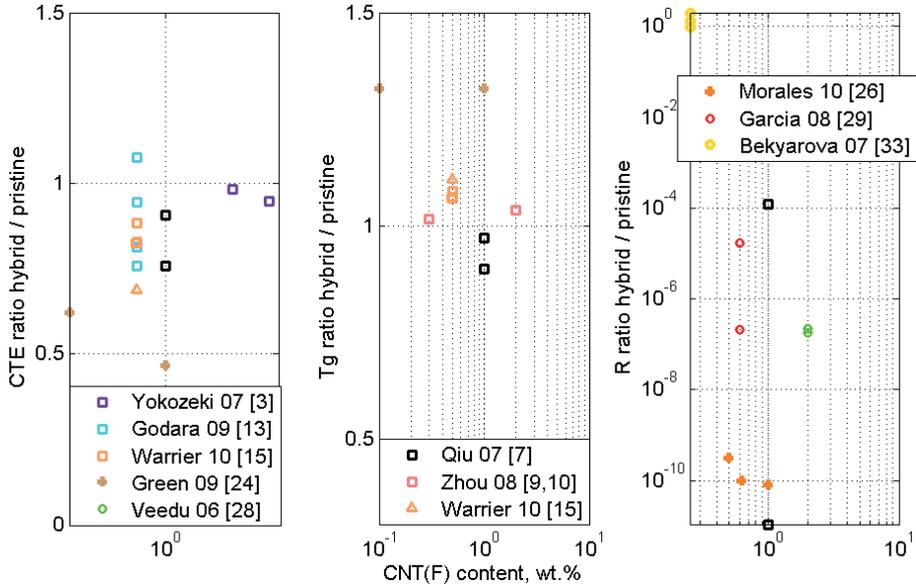

Fig. 4. Left to right: Thermal expansion coefficient CTE, glass transition temperature Tg, and electrical resistance R. Normalized to the baseline composite.

The transverse CTEs are usually much lower in a hybrid composite, although there is no clear dependency on the CNT content. Two ways are proposed to explain this phenomenon. The first one approaches this from the mechanical point of view and takes into account 1) a negative CTE of CNTs [7] and 2) fastened interfacial interactions of the composite plies [28].

In the second way, it is taken into account that CNTs are dimensionally similar to the polymer chain building units. Thus CNTs decrease the free volume of the polymer network, influence the chain alignments, and thereby reduce their thermally induced movements [15]. This influence depends on the CNTs type and their bonding with the surrounding matrix [13]. In [7] the lowest CTE (-25%) is obtained for the hybrid composite containing functionalized MWNTs, while the normal ones are much less effective. A similar effect is reported in [13]: MWCNTs are ineffective and even increase CTE by a 10%, probably because of a weak interaction with the matrix and high probability to agglomerate. TMWCNTs and DWCNTs reduce CTE by a 23 and 32%, respectively, maybe because they effectively block the thermally induced movements of the polymer chains, due to the reduced size and higher interaction. But the functionalized MWCNTs drop out of this tendency, since show only a 6% improvement. However one should remind of a significant thickness discrepancy of the specimens tested in [13].

In [15] CNTs added into the sizing decrease CTE by a 31% in comparison with the base composite; this is attributed to a better fibre/matrix interface. Addition of CNTs in the matrix only has a milder effect, by a 12%, and is believed to be the consequence of increased matrix rigidity. Further, the addition of CNTs both onto the MF surfaces and in the matrix results in an intermediate CTE improvement by a 17%. In [21], CNFs are shown to effectively reduce the residual thermal stresses in an L-shaped composite part.

12 Glass transition temperature (Tg)

There are a number of thermal analyzers and methods (TMA, DMA...) used to register the polymeric glass transition. The summary of available data is shown in Fig.4 (centre) and does not reveal a consistent difference between the base and hybrid composites. In [15] it is noted that Tg demonstrates the same trend as CTE: CNTs are the most efficient in sizing, then in the matrix; combination of the two lies in-between. But it is seen in Fig.4 (centre) that the difference between these combinations looks as insignificant. Nevertheless Tg increases that supports the theoretical considerations about a dimensional similarity between CNTs and the polymer chain building units that allows CNTs to influence the chain alignment and temperature-induced motion (their mobility reduces) and thereby to increase Tg [15].

In [9,10] the decomposition temperature is registered also, using the thermo gravimetric analysis (TGA) but the obtained difference is either very small (3.4°C) or almost zero. In [23] the heat distortion temperature does not change, while the weight loss temperature changes by 11°C.

13 Thermal and electrical conductivities

The through-thickness thermal conductivity is usually measured according to ASTM E1530. A data set is available only in [1,28]; these studies show that in a hybrid composite this property can increase up to a 50%, in comparison with the baseline.

As for the average electrical resistance, it is usually measured by ASTM D257 for relatively low-conducting materials (base composites here) or D4496 in the case of a higher conductivity [29]. The available test data are summarized in Fig.4 (right). While [28,33] report a similar decrease in the in-plane and through-the-thickness resistances, [29] shows the difference to be large. Also, while the hybrid composites containing SMCNTs and MWCNTs show a similar resistance contraction in [33], normal or chemically functionalized MWCNTs give quite different values in [7]. A possible reason for the latter effect is seen in a disrupted electrical structure of the functionalized MWCNTs.

In [7] it is also reported that the electrical conductivity of the hybrid composite obeys a percolation-like power law curve, with a low percolation threshold less than 1 wt.%. The current–voltage measurement exhibits a quantum tunnelling conduction mechanism.

Generally it is clear that the electrical conductivity can terrifically (by several orders of magnitude) be improved by CNTs/CNFs which create numerous conducting paths in all directions. A minor overall influence of CNTs in [33] is obviously explained by a low (0.25% wt.%) CNT fraction that is insufficient to build the paths. This study reports also a single decrease in the out-of-plane conductivity but this is attributed to different specimen thicknesses. The decrease may also originate from different morphologies of SWNTs (which easily agglomerate to form bundles and paths) and MWNTs (which are solitary and thus need to be numerous to form a network).

Studies [4,5] also report the electrical resistance measurements but only for the hybrid composites, so no comparison is possible with the base ones. The interesting finding is that the in-plane resistance behaviour is anisotropic [4]. The percolation threshold is determined to be below 0.1 wt.%. The in-plane resistance is more than an order of magnitude lower than the out-of-plane one, obviously due to longer conductive paths which are forced to pass around the glass fibres in the latter case. It is also reported that a post-curing reduces the overall conductivity, probably by breaking the conductive paths due to shrinkage [4].

In [5] a comparison is done between the conductivity measured in the thickness direction for a composite with the matrix containing 0.3 wt.% of DWCNTs which are spatial or aligned by an electrical field during the manufacturing. In the former case a $1 \cdot 10^{-6}$ S/m conductivity is detected, while aligned CNTs increase this by more than one order of magnitude ($3 \cdot 10^{-5}$ S/m). The increase in conductivity (or decrease in the percolation threshold) in the direction parallel to the electrical field has already been reported for neat CNT/epoxy composites.

In [20] the electrical conductivity is successfully used to monitor the stress-strain state and damage development during mechanical tests (ILSS, incremental tension, and fatigue).

14 Conclusions

It may be concluded that the use of CNTs/CNFs can be an effective way to improve strength, thermal/electrical conductivity and some other properties of the fibre-reinforced composites. However in many cases the published datasets do not give a broad view. The following points can be mentioned to highlight the ways for a further research:

1. An optimal configuration regarding to the processing technique, resin type, CNT or CNF aspect ratio and mass fraction, etc. is hardly observed in the most of the existing data sets. As can be seen in Figs.1-4, a good deal of the points fall within a ±10% range near the baseline; this means that these material configurations are ineffective. Thus, parametric studies are desirable. The wt.% parameter is systematically varied in [1,11,14,21,23,26,31,34] but every of these studies covers only one or at best two load cases. In this respect, as well as with regard to some manufacturing features, a more consistent research is presented in [13,15] but for a single wt.% case.

2. In particular, hybrid composites with normal or chemically functionalized CNTs are compared only in [7,13]. Although some properties are shown to be increased in the latter case, the improvements are still questionable due to scattered specimen thicknesses used in these studies. Nevertheless a CNT surface modification is seen as a challenging way to optimize the CNT-matrix contact and, therefore, the interfacial strength.

3. There is still no clear experimental comparison of the impregnation ability and mechanical performance for a composite material either with grown CNTs/CNFs or with these dispersed in the matrix. If the difference is mild, the cost efficiency and processibility (especially due to much harder drapability of preforms with grown CNTs/CNFs) would dictate the use of dispersed CNTs/CNFs. Or maybe it is efficient enough to interlay a preform with CNF/CNF-rich soluble or melting films?

4. CNFs are utilized in [1-3,18,19,21,22,24,26,27,34] and only [27] utilizes them in the form of grown nanoforests (as a component for a carbon-carbon composite). Thus the on-fibre-grown CNFs are still unexplored in combination with a polymeric resin. CNFs have lower mechanical properties if compare with CNTs but can be produced at a milder temperature and thus allow for lower production costs. This means there is space for certain optimization in this way too.

5. Such important characteristic as the fire resistance (time-to-ignition, heat release rate...) can also be an interesting issue. It is known that for the neat nanoreinforced polymers these parameters are improved. One may expect the same for the considered hybrid composites case too. By now it is addressed only in [23], and a good improvement is detected.

6. As already noted, in several studies significantly different thicknesses and fibre volume fractions are obtained in the base and hybrid composites. This does not allow for a consistent interpretation of the test data. It should be pointed out that this is an inherent feature in the case of grown CNT/CNF forests (with a relatively large wt.%) and vacuum-assisted manufacturing. The reason is in a high resistance of the forests to compression; as a result, the pressure needed to achieve the target fibre volume fraction can increase drastically [43].

7. In all the studies, except for [23], thermoset matrices are used, and the ability of thermoplastic ones is still almost unexplored. The main reason for this is the impregnation obstacles, since CNTs mixed even with an epoxy resin drastically increase its viscosity [17]. Then a flow blockage can be faced already with a 0.5 wt.% CNTs in a polymer [11]. However the thermoplastic resins still have a good chance to be used, e.g. with the help of solvents as it is proved for the neat CNT-thermoplastic composites [44,45].

15 Acknowledgements

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