

# Novel uses of carbon composites for the fabrication of external fixators

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## Abstract

A novel bone fractures external fixator has been developed by using carbon fibre reinforced epoxy resin components. Aim of the project was the design and fabrication of an external fixator lighter than metallic ones, X-rays transparent, with suitable mechanical properties, and sufficiently stable under vapour sterilization conditions. The hydrothermal stability of carbon fibre epoxy resin composites has been assessed by measuring the elastic and ultimate properties of a properly selected carbon fibre-epoxy resin system after prolonged simulated sterilization treatments in water at 120 °C. The suitability of filament winding as a technique appropriate to produce the tubular elements of the designed fixator has been investigated by determining the relationship between winding angle and required mechanical properties of the tubes. Filament wound composite tubes have been obtained using a high tenacity PAN based carbon fibre and a DGEBA epoxy resin cross-linked by an anhydride based curing agent. Winding angles have been changed in the range from  $\pm 5^\circ$  to  $90^\circ$ . The axial tensile modulus of composite tubes can be reasonably well predicted on the basis of the lamination theory, while the most promising among the analyzed failure criteria in terms of fitting with the experimental axial tensile strength data, seemed to be the Tsai-Wu criterion.

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## 1. Introduction

Fixation systems are usually necessary for the treatment of unstable long bone fractures in order to support the broken bone. One method available is external fixation whose clinical use received a substantial impulse in the 1960s and 1970s mainly for the contributions of Vidal and Burnyand [1]. External fixators are frames designed to hold the fractured fragments rigidly and in anatomically alignment until the repair and healing of the soft tissue is complete [2]. External fixators can be of the following types: (a) unilateral (they neutralize only rotatory and axial loading), (b) bilateral (they neutralize rotatory, axial, and varus valgus forces but not all anteroposterior forces), (c) frames (they neutralize movements in all planes).

The early fixators were very rigid and were claimed to be responsible, in some cases, of non- or delayed union [1]. A number of reports stated that micromotions of

axial loading at a fracture site could enhance bone healing [3–5], and consequently, starting from the early eighties, the so called dynamic external fixators (DAFs) were introduced. Such devices allowed axial dynamization with improved clinical results [6–9]. A number of companies are currently producing DAFs based on various mechanical design solutions, like Orthofix, Mono-Tube Howmedica, EBI×Fix DFS, Hoffmann, Heidelberg Zimmer, etc. . . Among them one of the most widely used is the DAF system by Orthofix [10] whose micromovements can be obtained by releasing a locking screw that allows an inner rod to slide in the axial direction [6,9].

Most of the DAFs are currently made in various metal alloys (like stainless steel, aluminium or titanium alloys) since good mechanical properties are generally required (such as high stiffness and strength [11]) and cause metals allow the devices to be reused after a sterilization cycle that usually consists of a vapour treatment in autoclave at 121 °C for at least 15 min [12], as recommended by the international pharmacopoeia. An unresolved problem commonly encountered in the use of all commercially available metallic external fixa-

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tors is the visibility of the fracture site from a radiographic point of view [13]. In fact, since metallic materials are radiopaques they can obstruct the fracture visualization. Main consequences of this fact are the increasing radiation exposure time for both patient and surgical team. Another drawback in the use of metallic materials is that fixation systems generally result quite heavy, especially when stainless steel is used. The use of radiolucent materials (like some polymer matrix composites) should permit an easier and more accurate radiographic evaluation of the bone healing process [13], and results in much lighter systems [14]. To our knowledge, only a very limited number of commercial external fixators are currently made (partially or totally) of polymeric composites, like Monotube Triax Carbon Tube and Ultra-X (Howmedica Osteonics, Corp., Rutherford, NJ, USA), OrthoFrame/Mayo Wrist Fixator (Orthologic Corp, Phoenix, AZ, USA), or Jet-X (Smith&Nephew, Memphis, TN, USA).

In the framework of an extended research project in collaboration with Orthofix srl (Bussolengo, Verona, Italy), the design and development phases of a new external fixator partially made of polymeric composite have been recently performed by this research group [15]. A picture of a prototype of the new external fixator is reported in Fig. 1. The central part of the new fixator, consisting of four tubes and three plaques, is intended to be realized in a radiolucent composite material. In the prototype reported in Fig. 1 these elements are realized with epoxy/carbon composites tubes and plaques, obtained by filament winding and manual lamination, respectively.

The aim of the present paper is to investigate the suitability of a carbon fibre reinforced epoxy composite for the fabrication of the above described external fixator in terms of: (i) tensile mechanical properties of its tubular components as a function of the winding angle and (ii) hydrothermal stability of composite after repeated sterilization cycles.

## 2. Experimental

### 2.1. Materials

The reinforcing fibre used in this work was a high tenacity PAN based Torayca-Soficar T-300 (Toray Ltd) carbon fibre with an epoxy compatible sizing. In particular, filament wound tubes have been produced by using FT 300B-3000-40B continuous roving carbon fibres (lot No. F110064), while plane laminates were produced by manual impregnation by using a 0/90 balanced carbon fabric CC 202 by SEAL (Legnano, MI, Italy) with the following characteristics: weight: 204 g/m<sup>2</sup>, tow size: 3 K, weave: plain).

The polymeric matrix was an epoxy system produced by Camattini S.p.A (Collecchio- Parma) consisting of a DGEBA based epoxy resin (MC 102), 93 phr of an anhydride based curing agent (WH 102) and 0.2 phr of 1-methyl-imidazol (IG 847) as a catalytic agent. Viscosity data at various temperatures and times have been obtained by a Brookfield viscometer DVIII at 100 rpm. Epoxy matrix dog-bone specimens (ASTM D638 type) were prepared by pouring the liquid resin into the cavities of an open silicone mould. The optimal curing cycle suggested by the resin producer, is schematized in Fig. 2.

### 2.2. Processing

Composite tubes were obtained on a three controlled axes filament winding machine model ALAB 0102 (cod. 012/00FW) by T.EL.MEC. s.r.l. (Olginate LC, Italy). Fibre tows were impregnated by passing through a resin bath kept at 50 °C just before they were wound onto a cylindrical Teflon coated stainless steel mandrel. Helical winding was used for obtaining winding angles  $\theta$  of  $\pm 5^\circ, \pm 10^\circ, \pm 20^\circ, \pm 30^\circ, \pm 45^\circ, \pm 60^\circ$ . The troublesome point encountered with the lowest winding angles (i.e.  $\theta < 20^\circ$ ) was to prevent the fibre slippage of the tow during winding. The solution was to use two Teflon pin



Fig. 1. Picture of a prototype of the new external fixator.

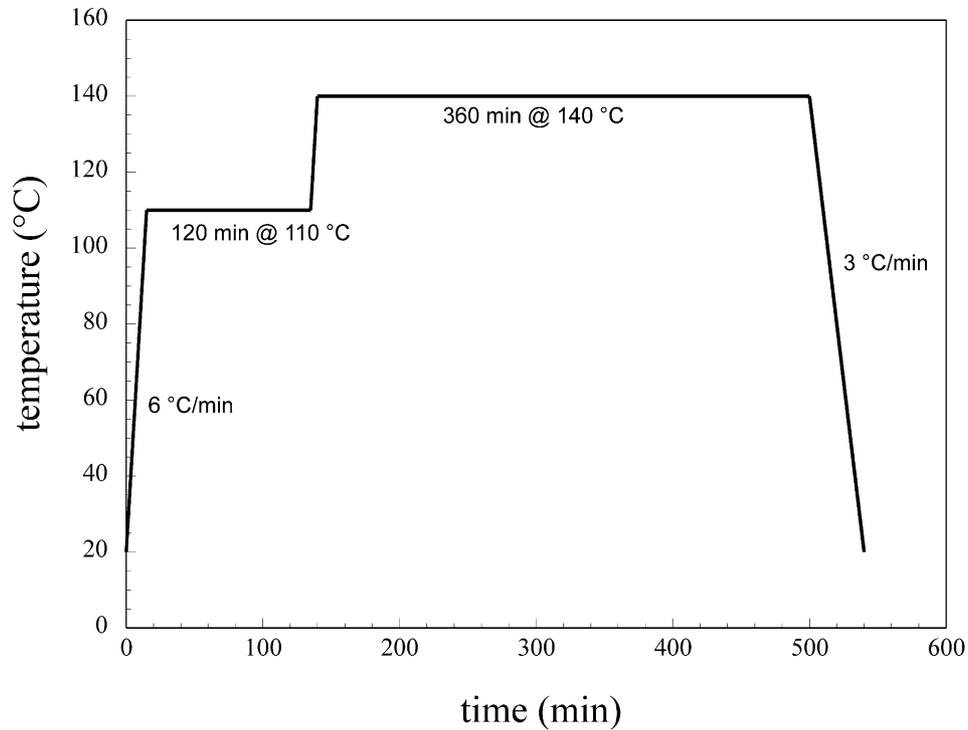


Fig. 2. Temperature profile adopted for a standard curing cycle.

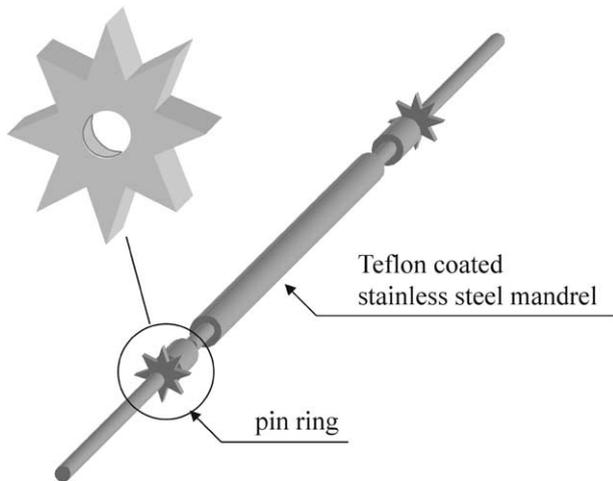


Fig. 3. Mandrel with pin rings for low angle helical winding of composites tubes.

rings, as those represented in Fig. 3, to catch the fibre tow at the end of each passage. Hoop winding was also performed in order to obtain a winding angle of  $90^\circ$ . Composite tubes were cured according to the cycle schematized in Fig. 2, removed from the mandrels, and cut down to specimens by eliminating the final end parts. Average dimensions of the obtained tubular specimens are: length 270 mm, internal diameter 7.5 mm, thickness 0.6 mm (three layers of).

Plane laminates have also been obtained by manual impregnation of five carbon fibre fabric plies. The

assembly (dimensions  $180 \times 160 \text{ mm}^2$ ) was consolidated in a 10 tons Carver Laboratory Press under a pressure of 0.2 MPa following the curing cycle schematized in Fig. 2 thus obtaining a laminate of about 1 mm in thickness. Rectangular specimens ( $35 \times 8.5 \times 1 \text{ mm}^3$ ) were machined from the cured laminates.

### 2.3. Testing procedures

The effects of a typical vapour sterilization procedure have been investigated on samples treated in a home made autoclave capable of maintaining the samples exposed to steam at  $120\text{--}125^\circ\text{C}$  for the desired time.

Samples removed from the sterilizing autoclave were let to cool down at room temperature and the surface moisture was wiped out. Weight variations of samples have been assessed by a Gibertini E-42 analytical balance with an accuracy of  $\pm 0.1 \text{ mg}$  on at least three specimens. The same balance has been used to measure the density of pure epoxy resin and its carbon fibre composites according to ASTM D792, i.e. by a water displacement method. By assuming a fibre density  $\rho_f = 1.760 \text{ g/cm}^3$  [16] the fibre volume fraction of composites ( $V_f$ ) was estimated by the following expression [17]:

$$V_f = \frac{\rho_c - \rho_m}{\rho_f - \rho_m} \quad (1)$$

Tensile mechanical properties of pure epoxy resin and single carbon fibres have been measured with an Instron 4502 tensile tester. All measurements were performed at

room temperature, at a cross-head speed of 0.2 and 2 mm/min for the single carbon fibres and for the epoxy resin dog-bones, respectively. Tensile tests on single fibre specimens have been conducted in accordance to ASTM standard D3379. Tensile strength and modulus were measured at various gage lengths (10, 15, 20, 25 mm) on 40 monofilaments randomly extracted from a bundle. The average fibre diameter as determined by scanning electron microscopy observations is in accordance with the value of 7  $\mu\text{m}$  reported on the product data sheet. Tensile tests on resin dog-bone specimens have been performed at room temperature with a strain gauge extensometer (Instron, model 2620; gage length 25 mm), on at least five specimens.

Tensile mechanical properties on carbon fibre composites tubes were determined at room temperature using a 25 kN closed loop servo-hydraulic MTS 858 Mini Bionix testing machine at a displacement rate of 1 mm/min on at least three specimens. Steel rods were inserted in both ends of composite tubular specimens to protect them against chuck failure in hydraulic grips. Specimens axial displacement was measured by a strain gauge extensometer (MTS, model 634.31F-24; gage-length 20 mm).

Differential scanning calorimetry (DSC) measurements were performed by a Mettler DSC-30 calorimeter at a heating rate of 10  $^{\circ}\text{C}/\text{min}$  in a nitrogen flux of about 200 ml/min.

Rectangular samples of carbon fibre laminate were characterized by dynamic mechanical thermal analysis (DMTA) measurements on a Polymer Laboratories Ltd (UK) model MkII dynamic thermal analyser in the single cantilever configuration. DMTA test conditions

were: temperature range from 30 to 200  $^{\circ}\text{C}$ ; heating rate of 3  $^{\circ}\text{C min}^{-1}$ ; frequency of 1 Hz; peak to peak displacement of 32  $\mu\text{m}$ .

### 3. Results and discussion

#### 3.1. Fibre and matrix properties

The viscosity of the epoxy resin mixed with the anhydride based curing agent and the catalytic agent is initially decreasing with temperature as reported in Fig. 4, that also shows how the curing reaction is relatively slow even at 50  $^{\circ}\text{C}$ . This is an important feature for the selected epoxy system since it allows the filament winding process to be successfully conducted at 50  $^{\circ}\text{C}$  with a good fibre impregnation while maintaining a relatively low viscosity. The curing reaction can be analysed by differential DSC analysis on freshly mixed epoxy resin, as reported in Fig. 5. The first scan shows that the curing reaction is starting from about 65  $^{\circ}\text{C}$  with an exothermic peak having a maximum located at about 156  $^{\circ}\text{C}$ , and a total energy release of about 312 J/g. The second scan, performed on the same sample rapidly cooled to room temperature, shows a clear glass transition temperature located at about 154  $^{\circ}\text{C}$  (inflection point) with no evidence of further chemical reactions. DSC analysis performed on a sample treated according to the curing cycle schematized in Fig. 2 (suggested by the resin producer), shows a thermogram practically identical to the second scan of Fig. 5. The glass transition temperature is located at about 159  $^{\circ}\text{C}$ , that is indicating the completion of the curing reaction. The

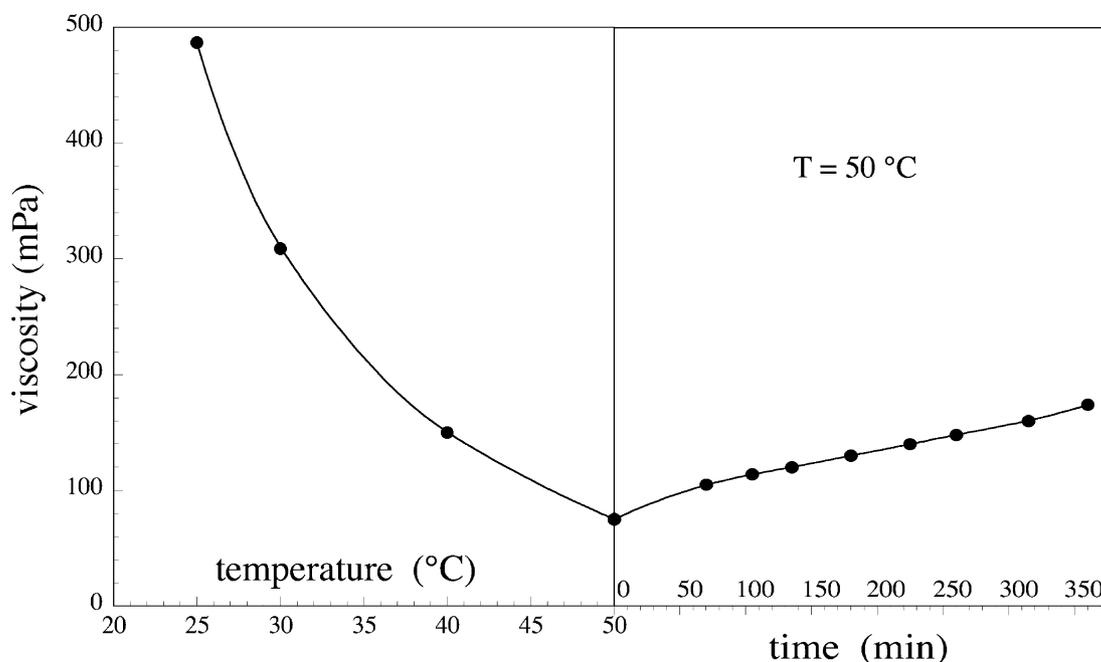


Fig. 4. Brookfield viscosity of the epoxy resin at various temperatures and at various times at 50  $^{\circ}\text{C}$ .

density of the fully cured resin resulted equal to 1.191 g/cm<sup>3</sup>.

Tensile tests conducted on the fully cured epoxy resin and on the single carbon fibres indicate that both composite constituents behave in a brittle manner with

completely linear stress-strain curves up to fracture. The epoxy resin shows a modulus of  $3.0 \pm 0.1$  GPa, a strength of  $60 \pm 5$  MPa and an strain to failure of  $2.130 \pm 0.085\%$ . As expected, carbon fibres display a marked dependence of their tensile mechanical properties on the sample

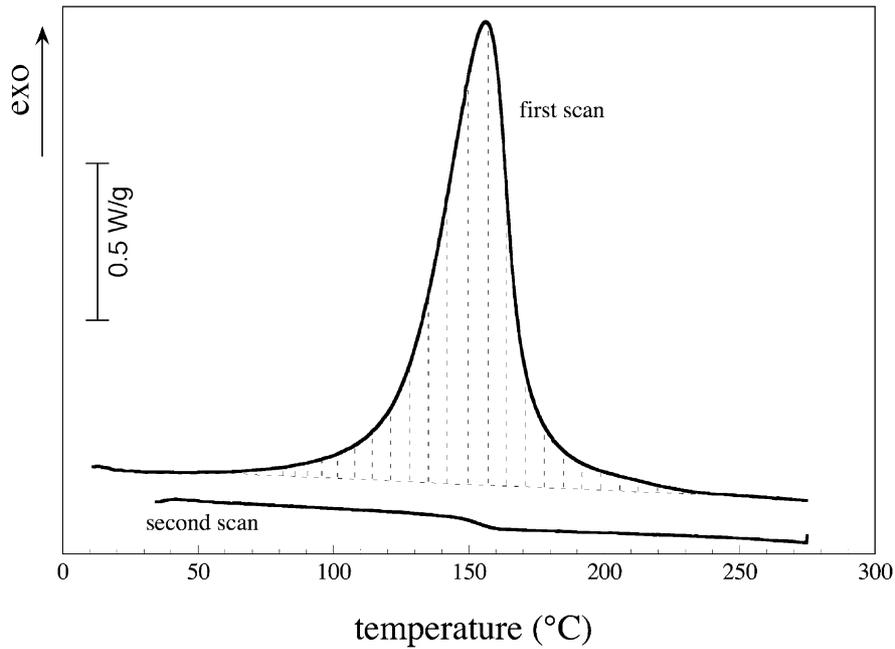


Fig. 5. First and second DSC scan on a freshly mixed epoxy resin.

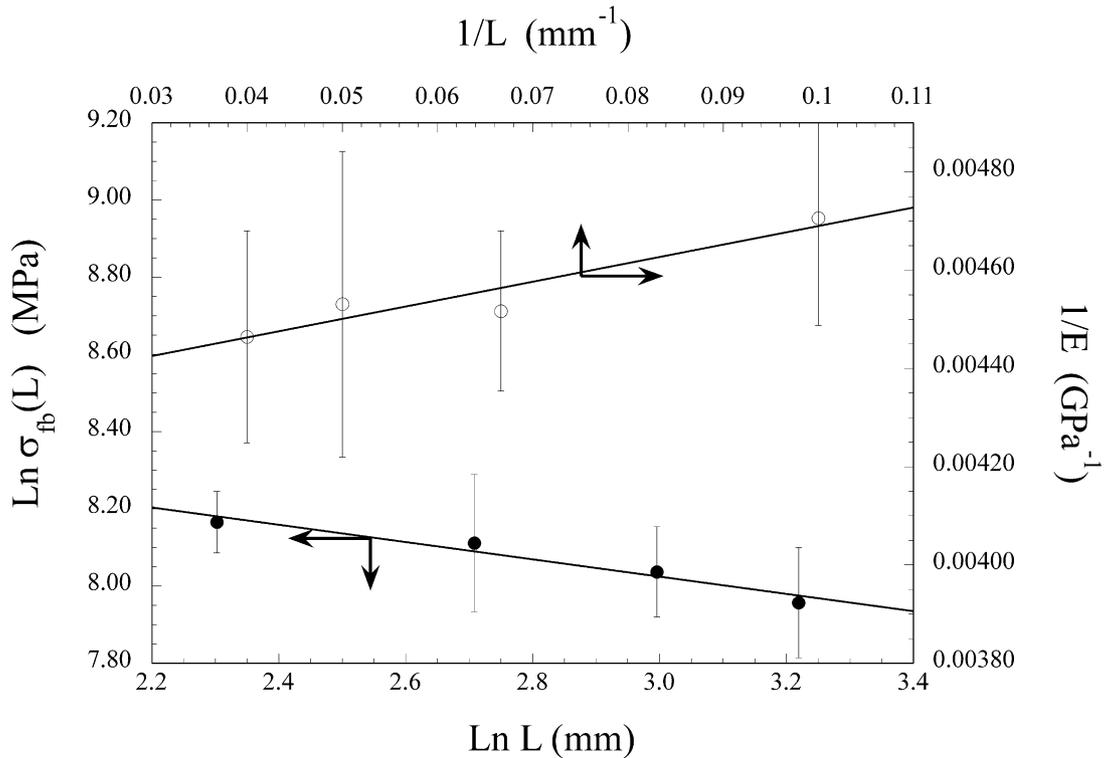


Fig. 6. Gage length dependencies of the carbon fibre strength,  $\sigma_{fb}(L)$ , and compliance,  $1/E$ .

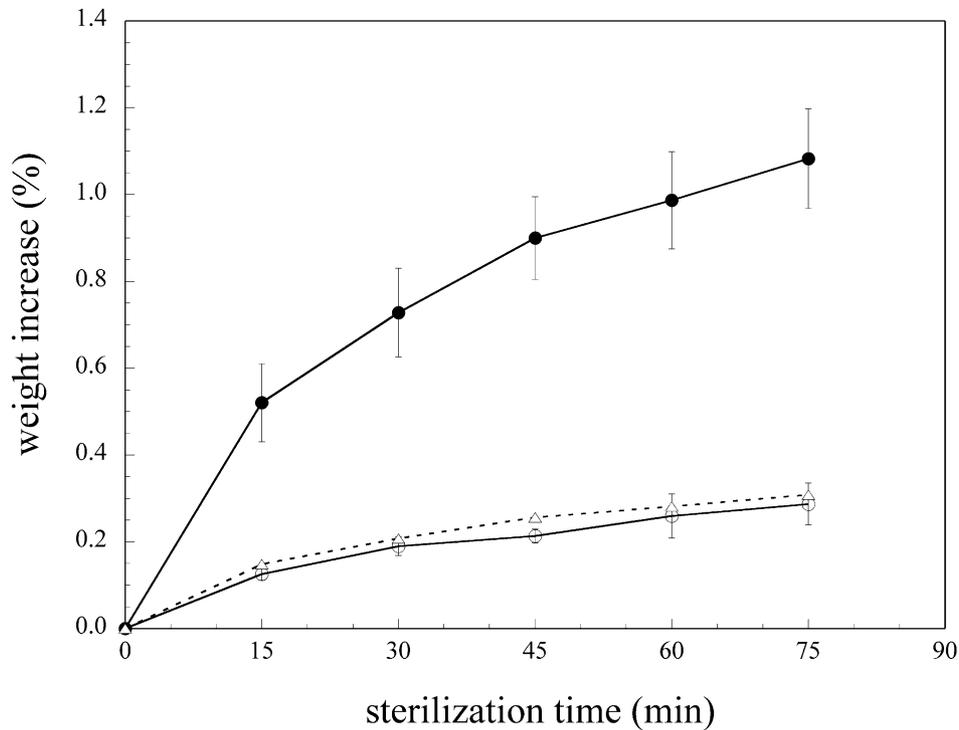


Fig. 7. Weight increase of the pure epoxy resin (full circles), and the carbon fibre laminates (open circles). Open triangles refer to the estimated weight increase of a quantity of pure epoxy resin equal to the matrix volume fraction in the composite laminates.

Table 1

Glass transition temperature,  $T_g$ , corresponding to the maximum of  $\tan\delta$  peak, storage modulus  $E'$  at two different temperatures after several sterilization cycles

Number of sterilization cycles (15 min in water at 120 °C)	0	1	2	3	4	5
$T_g$ (°C)	150.5	149.7	145.7	144.7	140.6	139.6
$E'$ at 30 °C (GPa)	32.4	32.3	30.3	27.7	24.4	28.5
$E'$ at 120 °C (GPa)	32.3	31.9	30.0	27.3	23.5	27.4

length, that is evident from Fig. 6 for as concern the tensile strength. The dependence of the fibre strength on its length can be computed according to the Weibull approach [18]:

$$\sigma_{fb}(L) = \sigma_0 L^{-\frac{1}{m}} \Gamma\left(1 + \frac{1}{m}\right) \quad (2)$$

where  $\sigma_{fb}(L)$  is the mean fibre strength,  $L$  the fibre length,  $\Gamma$  the gamma function and  $\sigma_0$  and  $m$  are the Weibull scale and shape parameters, respectively. Eq. (2) can be modified as follows:

$$\ln\sigma_{fb}(L) = \ln\sigma_0 - \frac{1}{m} \ln L + \ln\Gamma\left(1 + \frac{1}{m}\right) \quad (3)$$

From Eq. (3) it is evident that scale and shape parameters can be obtained by a linear regression of the  $\ln\sigma_{fb}(L)$  vs.  $\ln L$  data. Parameters  $\sigma_0$  and  $m$ , referred to a fibre length of 1 mm, result to be 6552 MPa and 4.469, respectively, in good accordance with other data

obtained in a similar way but regarding different carbon fibres [19]. According to the ASTM standard D3379, the true fibre modulus can be evaluated through the zero extrapolation of the fibre compliance as a function of the inverse of the gage length (see Fig. 6). This procedure gives a fibre tensile modulus of 232 GPa.

### 3.2. Effects of sterilization process on epoxy matrix and plane laminates

The weight variation of the pure epoxy matrix and of the carbon fibre laminates after several sterilization cycles are reported in Fig. 7. After each sterilization process (consisting of a treatment in water at 120 °C for 15 min) the epoxy resin absorbs a certain amount of water progressively approaching a saturation level. It is interesting to observe that the carbon fibre laminates absorb an amount of water that is proportional to the matrix weight fraction of the composites, that is equal

to 0.285 (as assessed by the density measurements). This result is suggesting that the fibre-matrix adhesion in the carbon fibre composite is sufficiently strong and stable to impede a preferential absorption at the fibre-matrix interface. The effects of sterilization treatment on the

thermo mechanical stability of laminate composites have been investigated by the dynamic mechanical thermal analysis. The DMTA thermograms obtained on a laminate non-sterilized or exposed to five sterilization cycles, are reported in Fig. 8. The material glass

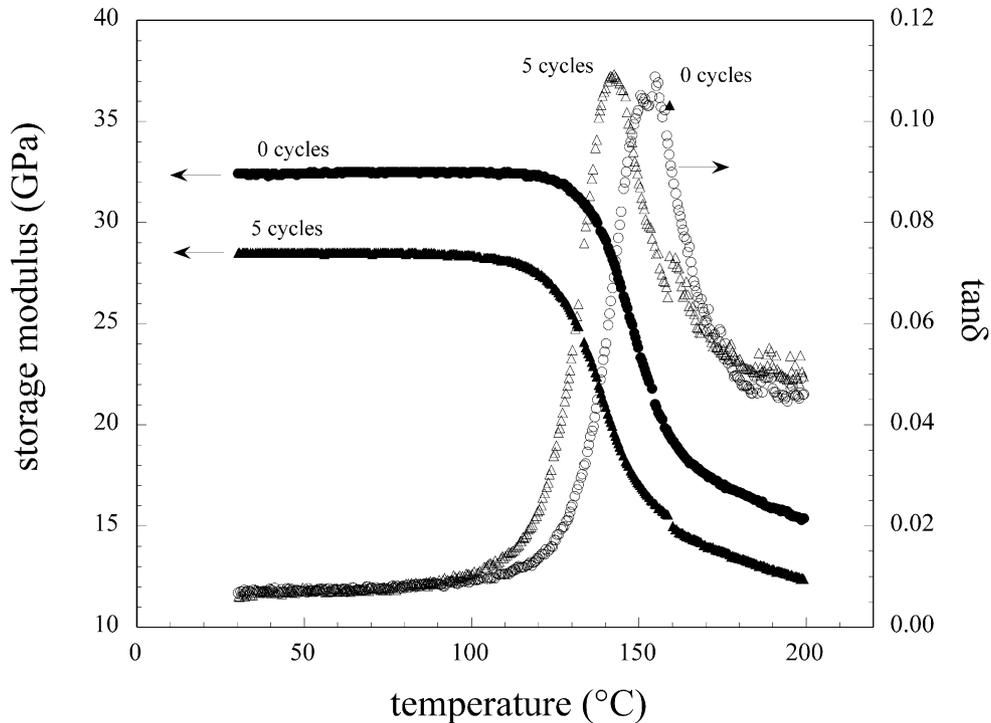


Fig. 8. Temperature dependence of storage modulus (full points) and loss factor (open points) for laminates non-sterilized (circles) and after five sterilization cycles (triangles) as measured by dynamic mechanical thermal analysis measurements.

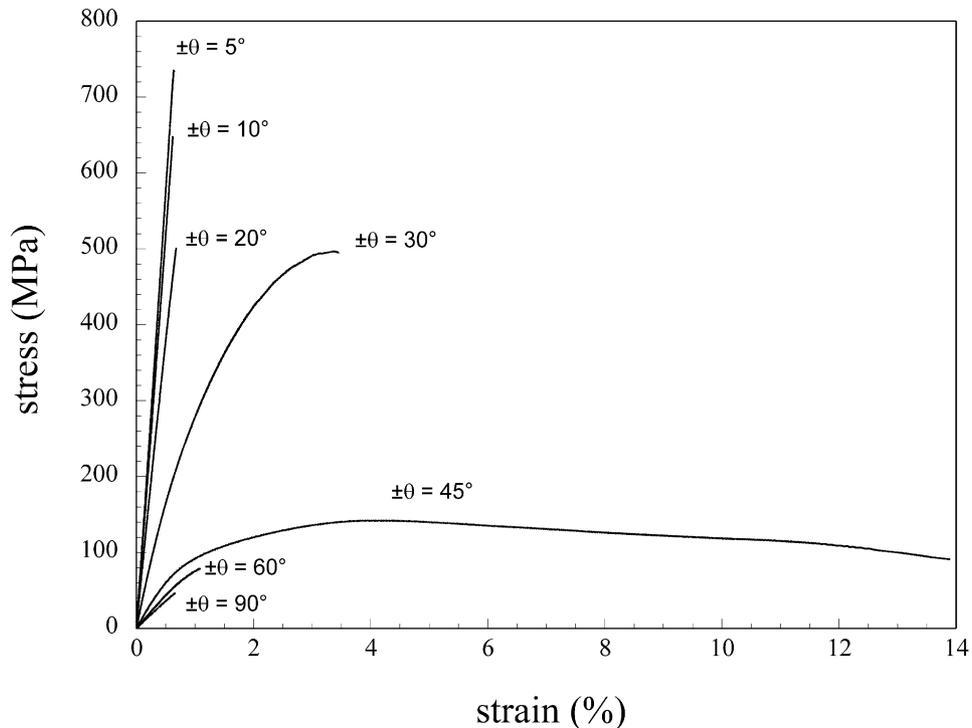


Fig. 9. Stress–strain curves of filament wound tubes with various winding angles tested under tensile configuration.

transition can be conventionally evaluated as the temperature corresponding to the  $\tan\delta$  peak. The main effects of repeated sterilization treatments are summarized in Table 1. The material glass transition temperature decreases after each sterilization cycle, most probably due to the combined plasticizing and degradative effects of the absorbed water. For as concern the storage modulus values, a decrease can be observed in the measured values after each sterilization cycle even if the laminate stiffness is not seriously compromised by the sterilization procedure. Moreover the storage modulus values evaluated at 30 and 120 °C are in any case very similar thus indicating the substantial stability of the material at the typical sterilization temperatures.

### 3.3. Mechanical properties of composite tubes and effects of sterilization

The density measurements performed on sections (10 mm long) of the tubular composite specimens indicate an average composite density of  $1.450 \pm 0.008 \text{ g/cm}^3$ , independently from the winding angle. This density corresponds to a fibre volume fraction of  $0.455 \pm 0.015$ , that is the value used in the analytical models of the mechanical properties throughout the paper.

The stress–strain curves, obtained from testing the composite tubes under tensile configuration, are reported in Fig. 9 as a function of the winding angle. It can be observed that a linear stress–strain curve followed by a brittle fracture is preserved up to a winding angle of  $\pm 20^\circ$ . It is quite interesting to note that for winding angles of  $\pm 30^\circ$  and  $\pm 45^\circ$  a peculiar stress–strain curve is obtained, characterized by a marked deviation from linearity and by an increase of the strain to break. The peculiar behaviour of these samples (especially those with a winding angle of  $\pm 45^\circ$ ) is related to the transition from a fibre-dominated to a matrix-dominated mechanical behaviour. The occurrence of an extensive matrix damage and the development of a steady state neck propagation is associated to this failure mode switch, as evidenced in Fig. 10. A similar behaviour has been recently observed for uniaxial tensile tests performed on braided composite tubes [20].

The tensile modulus values as a function of the winding angle are reported in Fig. 11 for non-sterilized tubes together with the values obtained after a sterilization time of 75 min (corresponding to the total time of five sterilization cycles). First of all it is quite interesting to observe that the prolonged sterilization process does not affect the stiffness of the composite tubes. Since the mechanical behaviour of filament wound composites is analogous to that of other angle-ply laminates, the analytical methods developed for laminates can be applied to filament wound structures [21]. The analytical prevision of the composites moduli according to

the lamination theory have been computed by the software Computer Aided Design Environment for Composites (CADEC) developed by E.J. Barbero [22,23] using the matrix and fibre properties experimentally measured and previously reported. It is worth noting that the stiffness analytical predictions, computed on the basis of the lamination theory for the non-sterilized composite tubes, are in quite good accordance with the experimental results.

The winding angle dependence of the stress at break is reported in Fig. 12 for non-sterilized and sterilized (for 75 min) composite tubes. Again, the effect of a sterilization time corresponding to five typical sterilization cycles is practically negligible and in some cases (winding angles of  $\pm 20^\circ$  and  $\pm 30^\circ$ ) a beneficial effect can also be observed with a slight strength increase. This effect could be probably ascribed to a plasticizing effect due to the absorbed water. For the non-sterilized composites the output of various analytical failure criteria

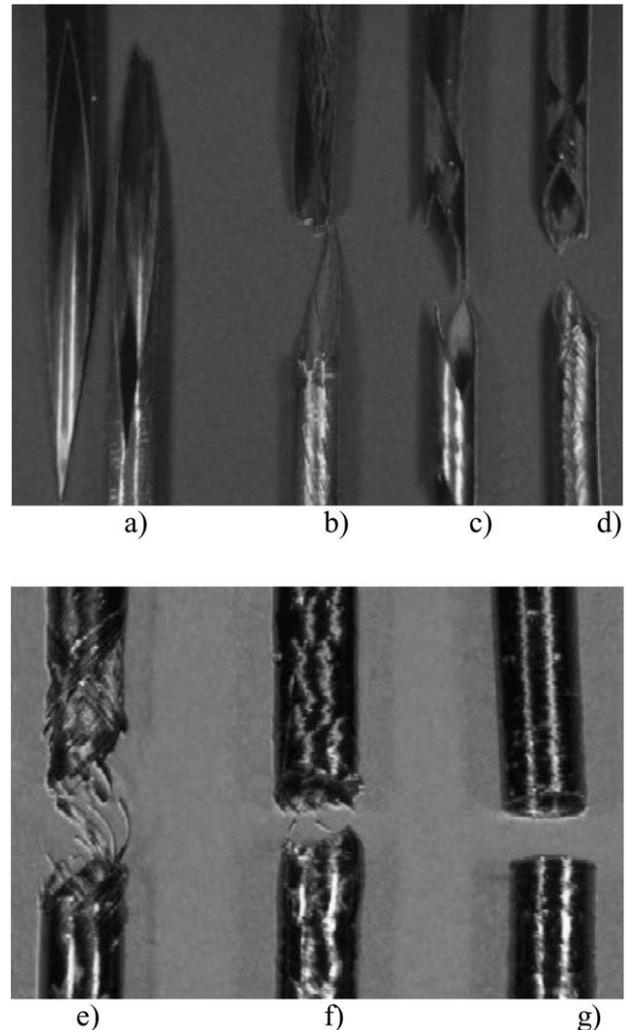


Fig. 10. Pictures of the specimens at various winding angles after the tensile tests: (a)  $\pm 5^\circ$ , (b)  $\pm 10^\circ$ , (c)  $\pm 15^\circ$ , (d)  $\pm 30^\circ$ , (e)  $\pm 45^\circ$ , (f)  $\pm 60^\circ$ , and (g)  $\pm 90^\circ$ .

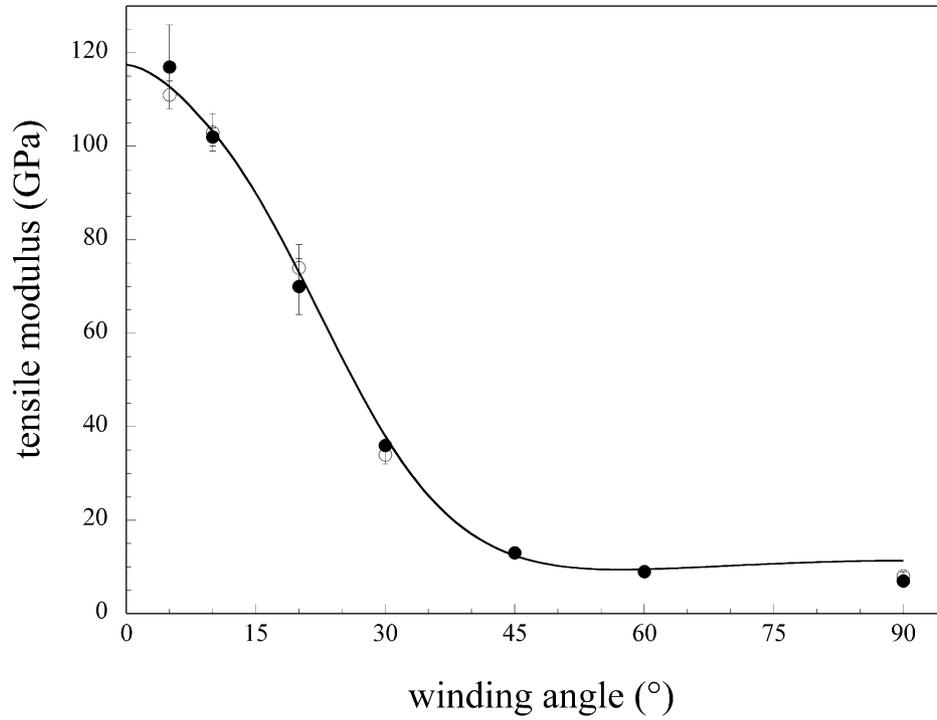


Fig. 11. Tensile modulus of filament wound composite tubes non-sterilized (full circles) and after a sterilization time of 75 min (open circles). Full line represents the analytical prevision according to the lamination theory.

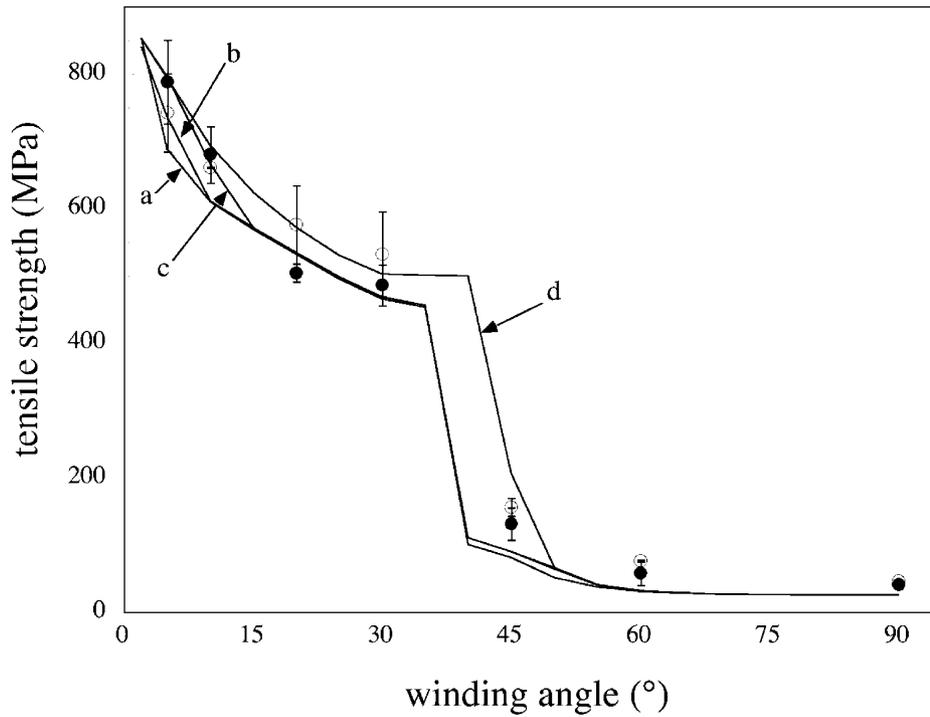


Fig. 12. Stress at break of filament wound composite tubes non-sterilized (full circles) and after a sterilization time of 75 min (open circles). Full lines represent the analytical prevision according to the following failure criteria: (a) maximum strain, (b) Tsai-Wu, (c) maximum stress, (d) truncated maximum strain.

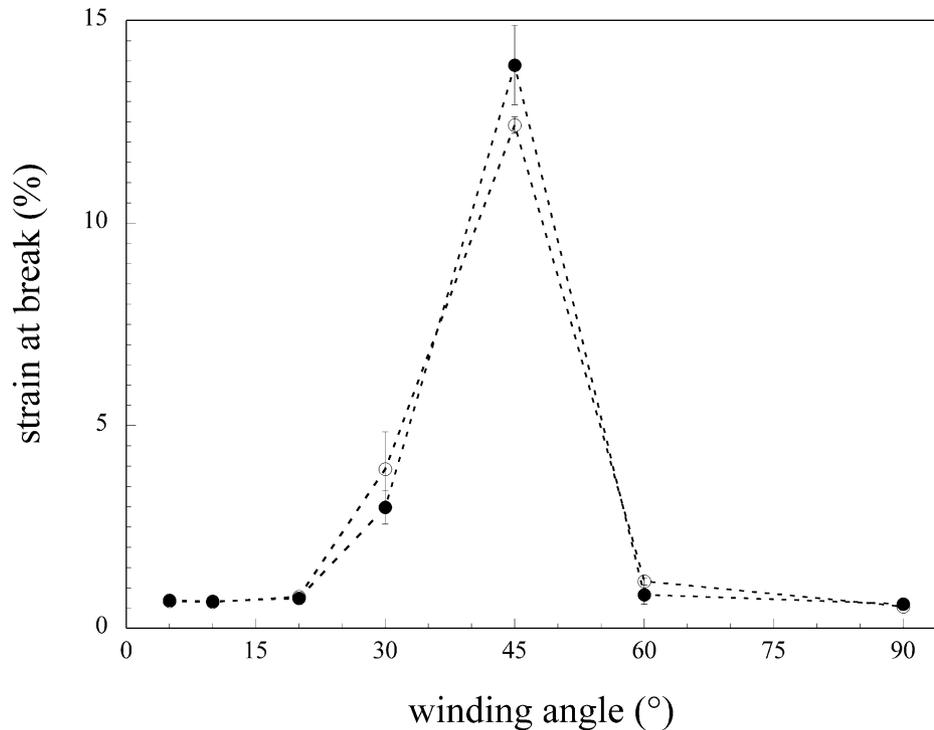


Fig. 13. Strain at break of filament wound composite tubes non-sterilized (full circles) and after a sterilization time of 75 min (open circles).

(i.e. maximum strain [24], maximum stress [25,26], Tsai–Wu [27], and truncated maximum strain [22]) has been verified and compared with the experimental results. All these models are implemented in the software CADEC [23] that also accounts for a micromechanics section where the response of a single layer can be predicted from the properties of single constituents and their volume fractions. An attempt has been made to use a fibre strength value, estimated through the Weibull distribution, for the fibre length of the composite tubes. Nevertheless, the resulting analytical prediction largely underestimated the experimental values probably because the strength values obtained from Eq. (2) cannot be extrapolated at fibre length too much different from those of the tested fibres [28]. As a consequence, in all the failure criteria, the fibre strength was chosen as the value measured on the longest tested fibre sample (i.e. the strength value of  $2856 \pm 405$  MPa evaluated on the 25 mm long fibre lot). Among the analyzed failure criteria, the most promising in terms of fitting with the experimental data seems to be the Tsai–Wu criterion. All the considered criteria show a clear discontinuity in correspondence to a strength value that is probably associated to the transition from a fibre- to matrix-dominated behaviour. Depending on the failure criterion, this transition occurs at winding angles in the range from  $\pm 35^\circ$  to  $\pm 40^\circ$ . From a practical point of view this is suggesting that a proper selection of winding angle for the construction of the external fixator tubular arms should be a value lower than  $\pm 35^\circ$ .

Strain at break values as a function of the winding angle are reported in Fig. 13 for non-sterilized and sterilized (for 75 min) composite tubes. It is quite interesting to observe that the strain at break passes through a maximum for a winding angle of  $\pm 45^\circ$  and that the strain at break is not markedly affected by sterilization for 75 min.

The above results clearly indicated the suitability of carbon fibre-epoxy system for the fabrication of external fixators, provided that both matrix and fibres are properly selected in order to achieve the necessary hydrothermal stability and the required mechanical properties, i.e. stiffness and strength. In our case, the overall bending stiffness of a fixator prototype, fabricated by using the components described above, complied with the requirements evaluated by comparison with metallic commercial models.

#### 4. Conclusions

The selected epoxy resin has been proven to be suitable for the production of carbon fibre filament wounded tubes from the point of view of the viscosity and gel time. Moreover the relatively high glass transition of the fully cured epoxy resin (located at  $159^\circ\text{C}$ ) is such that the composite tubes can be repeatedly sterilized in water at  $120^\circ\text{C}$  with slight variations of their thermomechanical stability as assessed by dynamic mechanical thermal analysis.

Winding angles of the obtained composite tubes have been changed in the range from  $\pm 5$  to  $90^\circ$ . The axial tensile modulus of composite tubes can be reasonably well predicted on the basis of the lamination theory. Various failure criteria have been considered in order to predict the experimental axial tensile strength data, the most promising being the Tsai–Wu criterion. It is important to underline that both axial tensile modulus and strength data did not were significantly affected by a sterilization treatment in water at  $120^\circ\text{C}$  prolonged for as long as 75 min.

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