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The fiber volume fraction influence on mechanical properties of multi-layered carbon tubes

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1. Introduction

Carbon fiber composites, particularly those with polymeric matrices, have become the dominant composite materials for automotive industry, aerospace industry, sporting goods and other applications due to their low density, high strength, high fracture toughness, high thermal conductivity, low thermal expansion, high creep resistance, high energy absorption on impact, corrosion resistance and reasonable cost [1, 2]. Nowadays the carbon fiber composite materials are of great interest as new materials essential for the development of modern sports equipment [3]. The importance of the composites is growing due to the high strength to weight ratio. It is expected that further development of carbon fibres will take place involving obtaining fibres with improved mechanical properties with the use of nanotechnology [4].

A common view is that carbon fiber is very stiff and brittle materials that break when a certain load is reached. However, carbon fiber should be known as carbon component structures that can be designed to work in a elastic and flexible way. Currently, carbon fibers are derived from several precursors, with polyacrylonitrile (PAN) being the predominant precursor used today [5]. PAN is an organic polymer composed of a mixture of acrylonitrile ($CH_2 = CHCN$) as the main component, mixed with other monomers. Composite materials consist of two or more different materials of different physical properties, which in microscopic level are combined into a third material. The different materials give unique properties to the composite, but they do not dissolve or blend into each other [6]. It can be easily identified in macroscopic level by naked eye, conversely as traditional plastics and metals, which are homogenous. The structural properties of composite materials are primarily derived from the carbon fibers reinforcement, whereas bonding matrix holds the reinforcement and distributes the load among the fibers. The carbon fibers are very useful materials in application as low weight combined with high specific tensile strength and modulus is required. PAN-based carbon fiber tows consists of many continuous filaments, therefore, mechanical properties along and perpendicularly to the filament direction are different. These fibers may be short (typically <3 mm in length), or long (3-25 mm in length), and they may be aligned in the directions where loading will be greatest. This anisotrophy principle was observed and widely applied in tubes constructions aiming to ensure high modulus and strength, as well as relatively low weight. Epoxies are one of the most common materials used to form the matrix in carbon-fiber fabrication. Epoxies offer high strength, low shrinkage, chemical and solvent resistance [7]. They wet the material easily and the composite can be processed using a variety of methods.

The bonding between fibers and matrix is created during the manufacturing phase of the composite material. For carbon fiber reinforced composites, the interface between carbon fiber and the matrix has fundamental influence on controlling the mechanical properties of the composite material [8, 9]. Even when one component of the composite dominates, both the components generally must work together to obtain optimal performance. Accordingly, carbon fiber producers make products that are similar but not identical. This should be taken into account while selecting the carbon fiber tubes for any type of design, as material properties of the tubes vary from manufacturer to manufacturer. In this regard carbon fiber tubes are different, if compared to the metal tubes, so any type of tube needs to be carefully thought out and manufacturing process and testing on the specific tubes are highly recommended. The strength and stiffness of composite tubing is very difficult to compare to metal tubing because of the possible variation in fiber orientation during the layup.

This work is carried out in cooperation with Lithuanian-Hungarian Ltd. Brača-Sport. The company produces its own carbon fabric layers and is the leading oars manufacturer in the world of sprint canoe, kayak racing and academic rowing. An experience shows that oar weight, stiffness and strength has a huge impact on athletes‘ performance. An oar is a stick loaded in bending. The key points are that an oar should be strong enough under bending, exerted by the sportsman, ensure the particular stiffness, matching the personal characteristics of the user, providing the right „feel“, necessary lightness and durability. To reduce a weight while maintaining both the structural integrity and proper mechanical properties is one of the key challenges for manufactures.

Manufactures, which play in professional rowers market, understand the importance to combine low weight and high stiffness while maintaining the strength. Most tubes used in sports equipment commonly are made with approx. 25...30% content of binder of total product mass due to several reasons. First, the decrease in the binder matrix amount may cause the appearance of the spots with filler drawback, as well as unpredictable properties of the structure. The second reason mainly is related to the technological constraints, i.e. having the low content of resin it is difficult to ensure necessary impregnation of the fiber. The resin is necessary component, but too much or too less amount of it can really weaken the structure of the tube.

Optimum ratio of carbon fiber and epoxy resin is the main interest of this investigation.

2. The preparation of carbon fiber tubes

The manufacturing processes of all tubes made of composite materials involve mixing of matrix materials with the reinforcing materials. It is necessary to adjust the filament winding, vacuum resin infusion or hand lay-up processes using carbon and glass fiber. The manufacturing process consists of four basic steps: wetting (impregnation), lay-up, consolidation and solidification. PAN fibers are formed by a process called wet spinning. The dope is immersed in a liquid coagulation bath and extruded through holes in a spinneret made from precious metals. Filament winding consists of drawing resin-impregnated fibers that are wound over a rotating mandrel (Fig. 1).

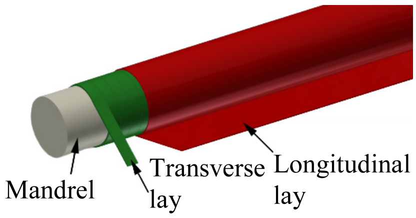


Fig. 1 Tube layers orientation and structure: longitudinal lay with 160 g/m^2 areal weight of carbon fiber, transverse lay with 120 g/m^2 areal weight of carbon fiber

Taking into account the required properties of the product winding patterns are selected during the design stage. Generally the vacuum infusion process (VIP) is used to draw resin throughout a dry fibre laminate in a one sided mould. By a typical hand lay-up process, reinforcements are laid into a mould and manually wet out using rollers, brushes or through other means. To create moulds for composite tubes a thin layer of fiberglass is laminated over a smooth metal pipe or tube that is used as a mandrel (Fig. 1).

The composite mechanical properties are mainly defined by the fiber architecture. The optimum design for maximum strength and stiffness is a unidirectional layup of carbon fibers which are located parallel to the loading axis [6]. In this investigation the hand lay-up producing process is chosen. Tube structure is based on longitudinal and transverse lays of unidirectional carbon fiber films. The longitudinal and transverse layers were winded on 26.7 mm diameter mandrel on modified lathe. The longitudinal layers were winded manually (Fig. 1), while the transverse filaments were winded on the rotating mandrel using approximately 6 mm wide tape of preimpregnated fibers, i.e. so called prepregs (Fig. 2).

The ratio of longitudinal and transverse layers was used 4:3, therefore each tube contained seven layers of the braid (Table 1). The prepreg moulding process, which gives better resin-to-fiber control, was used for the tube specimens. Prepregs are made with precise resin and fiber contents and orientations, and careful wetting of the fibers [10].

As previously mentioned, PAN-based Toray T700 carbon fiber tow with filament diameter of 7 μm combined with different impregnation of modified low-viscous epoxy resin CeTePox 152 R based on bisphenol-A was used.

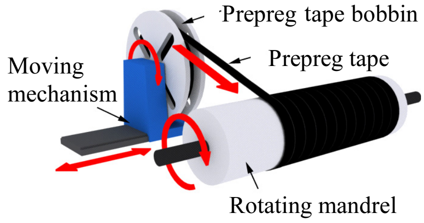


Fig. 2 The preparation scheme of tube transverse prepreg layer

Table 1

Tube specimens' layers structure

Layer No	Areal weight, g/m^2	Epoxy resin content, %			
		Number of group of specimen			
		I	II	III	IV
1	160	16%	18%	20%	22%
2	120				
3	160				
4	120				
5	160				
6	120				
7	160				

The longitudinal orientation layers, which are marked by the odd numbers (Fig. 3), provides the tube stiffness and tension strength while the transverse even layers give strength and impact resistance, therefore tubes specific characteristics can be achieved by modelling the layers orientation scheme.

Fig. 3 Layers structure of experimental tube: *1, 3, 5* and *7* are longitudinal layers; *2, 4* and *6* - transverse layers

After all the lamina layers preparation, the polypropilene film tape was winded by the same manner as the transverse carbon fiber braid. This polypropilene layer provides the compression and helps to remove the air voids as well as stops the leakage of the epoxy resin, which at the higher temperatures (approximately to 50-60^{o}C) becomes relatively fluid.

3. Materials

The experiments were carried out using four type tube specimens with the same structure, but with the different volume of fiber fraction, i.e. 16, 18, 20, and 22%. All test tubes have seven layers of braided composite.

As it is seen from Table 2, carbon fiber composites are very attractive materials where strength-to-weight of stiffness-to-weight ratios are fundamental. Since the carbon fiber density is also low, the overall performance to weight ratio of a carbon composite is higher than that of the other materials.

Table 2

Mechanical properties of materials

Material	Tensile strength, ultimate, MPa	Young's modulus, GPa	Elongation at break, %	Density, g/cm^3
Low carbon steel ASTM A36	440 ... 550	200	20 (200 mm) 30 (50 mm)	7.80
Aluminum alloy (6000 series)	310	69	17 (12.7 mm diameter)	2.70
Titanium alloy (Ti-6-4)	950	114	14	4.43
PAN-based carbon fiber (type Toray T700S)	4900	230	2.13	1.80
Pitch-based carbon fiber (mesophase pitch P25)	1400	160	0.9	1.90
Kevlar 49 (aramid fiber)	3620	131	2.8	1.45

The ultimate tensile strength is defined as the maximum stress that a tube can withstand before failure. Carbon fiber is used most efficiently when loaded in tension. Young's modulus can be used as an indicator of the stiffness of the tube. The design of the oars is based on stiffness, i.e., it is aimed to ensure specified elastic deflection under a given load. As can be seen from Table 2, carbon fiber shows highest strength, Young's modulus, but very small elongation at break in comparison with the other materials. Among the high performance carbon fibers, PAN-based carbon fibers can obtain greater elongation than those based on pitch, because pitch is more graphitizable than PAN, and the oriented graphitic structure causes the fibers to be more sensitive to surface defects and structural and flaws [1]. However, the tensile strength of a carbon fiber depends on its elongation which, in turn, is determined by the degree of defects. Therefore the elimination of the impurities is one of the most important operations. Exist of these impurities cause to decrease the tensile strength of the carbon fiber. One of the ways to increase the carbon fiber elongation at break is the use an epoxy resin of high elongation.

4. Evaluation of mechanical properties

To identify the mechanical properties of the carbon braided composite tubes, materials tests are conducted by a variety of test methods i.e., flexural, compression (longitudinal and transverse) tests, tensile tests. Testing conditions specify the loading direction with respect to the components (axial or transverse) and loading rate (static or dynamic).

Specimens for mechanical tests on composites are usually taken in three different forms: pultrusion, tubes, and flat sheet [11]. Composite crush testing can be divided into three categories: coupon, element, and structure testing [6]. The tests can be performed on the entire sample or on coupons of specified dimensions. The tubes specimens are convenient in testing taking into account the manufacturing process, which has a noticeable impact on the composite material performance in real application. Hence it is possible to conclude, that testing using the close to realistic configuration may be considered as leading to more reliable results.

The mechanical tests were performed using 50 kN "Amsler" testing machine and HBM testing equipment. All tests were conducted in the laboratory environment (at 20±2°C and 50±5% relative humidity).

In materials testing, flexural strength is most commonly determined through a three-point bend test, in which a loading nose deflects a specimen at a set span and loading rate until fracture [12]. When a tube is loaded in bending, some fibres are subjected to tension while others to compression.

Specimens for flexural strength testing were prepared by following the procedure outlined for PAN-based Toray T700 carbon fiber tow combined with impregnation of epoxy resin matrix at room temperature with a gauge length of 600 mm, outer diameter of 29 mm, inner diameter 26.7 mm and wall thickness of 1.15 mm. The tests were performed applying the three-point flexure testing method with 480 mm distance between the supports, therefore the span to diameter ratio was 16:1 (Fig. 4).

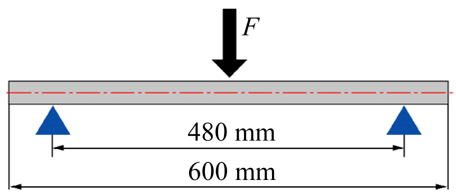


Fig. 4 Three-point flexure testing scheme of the tube specimen

The load was applied on the top between supports (Fig. 5). This test followed the procedures of ASTM D790M-93, which allow a wide freedom of choice in term of specimen dimensions [13].

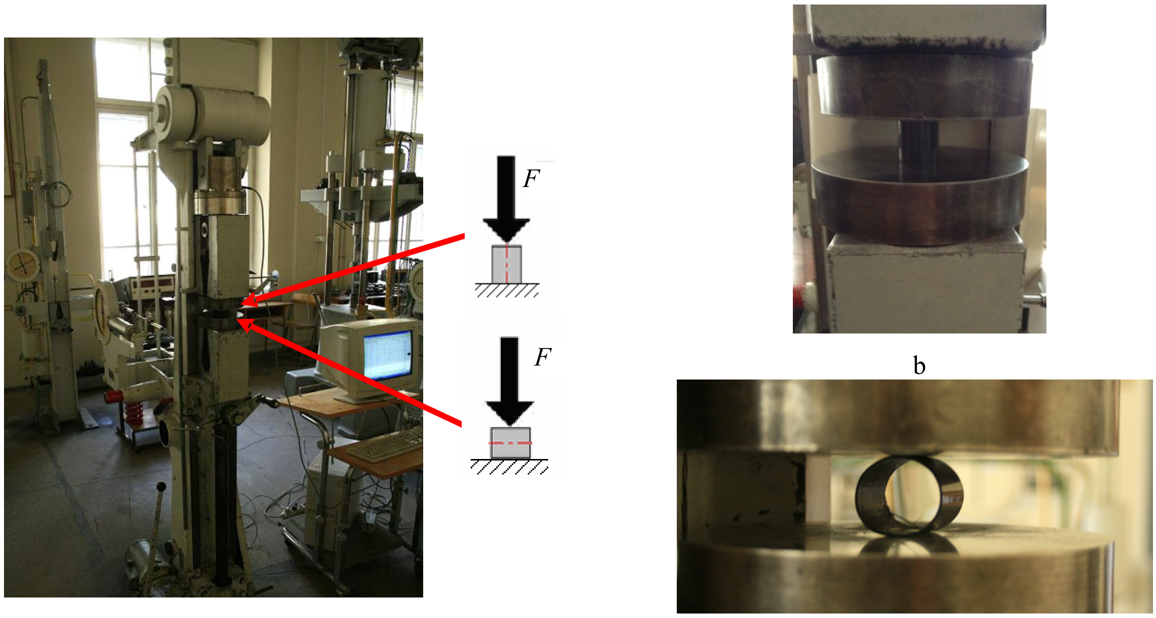
Fig. 5 The experimental stand for flexural testing of multi-layered structure carbon fiber specimens

The average loading rate for this analysis was in the order of 5 mm/min. As it is noted in Table 1, the longitudinal layers were made of 160 g/m^2 areal weight of carbon fiber and transverse layers were made of 120 g/m^2. A minimum of 4 tests were performed for each group of specimens with the different volume fiber fraction (16, 18, 20 and 22%).

Most carbon fiber tubes are really strong in tension, but they are not as strong in compression. The Young's Modulus of PAN-based Toray T700 carbon fiber in transverse direction equal 28 GPa versus 230 GPa in axial direction.

Compression failure in fibre reinforced composites is of much interest and is often a limiting factor in load application because of the lower compressive strength relative to tensile strength [14].

The structure and the volume fiber fraction of the specimens for compression tests was the same as for flexural testing specimens. The specimens were cut using a standard buzz-saw cutter to a specimen length of 30 mm. The length to diameter ratio is 1.0. Such a length of the specimen ensures that it does not fail prematurely in a buckling mode. Unfortunately, machining the specimen ends tends to leave the micro cracks and a roughened surface in which many of the fibres protrude from the resin surface. In order to reduce these cracks and potential delamination areas, the additional specimens' ends polishing was done. Each specimen was carefully positioned between the rigid, parallel compression test platens and the load applied at a constant strain rate, causing failure in about 20 s in longitudinal direction (Fig. 6, b), and approximately 60 s in transverse direction (Fig. 6, c).



a c

Fig. 6 Quasi-static compression: a) general view; b) longitudinal (axial); c) transverse test experimental setup on an "Amsler" testing machine with HBM testing equipment

A minimum of 16 tests were conducted for each configuration of specimens and the average values and variations were determined and presented. The specimens were tested to failure by compressing them between two plates at a rate of 3 mm/min (in longitudinal direction), and at a rate of 10 mm/min (in transverse direction). During damaging, the load-displacement curves were received directly from the testing machine.

5. Experimental results and discussion

The motivation for the research reported here was to compare the mechanical properties of multi-layered structure carbon fiber tubes with different impregnation of epoxy resin matrix. The mechanical and physical properties of carbon fibers vary according to the precursor material combined with different impregnation of epoxy resin matrix [1, 5, 15, 16].

The loading nose in the flexural testing was pressed towards the specimen tube (Fig. 6, a). The load and displacement data were recorded from HBM testing equipment and the load-displacement (F-Δl) curves for four type tube specimens were made (Fig. 7). As it is seen from Fig. **7**, the load applied to the tube specimen was almost linearly proportional to the displacement until the failure. The linear elastic range shows the stiffness (elastic modulus) of the specimen. The specimen with 22% volume fiber fraction has the highest value of bending strength, and failure of this specimen occurred at 3.0 kN load.

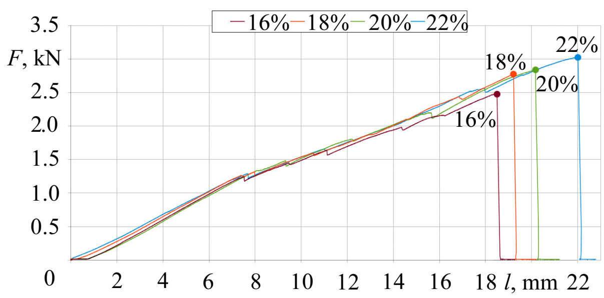


Fig. 7 Engineering load F versus displacement l curves of flexural tests for carbon fiber tubes having different volume of fiber fraction

As indicated in Fig. **7**, the difference in bending strength between specimens with 16 and 22% volumr fiber fraction is approximately 20%. The curve with 16% of epoxy resin content impregnation up to 1.7 kN load is analogous with the other curves, but above this magnitude the specimen with 16% of epoxy resin content impregnation lost stiffness, it means that above 1.7 kN load the tube bends more than the others with the higher content of impregnation and at the same time it has lower flexural strength.

Small fluctuations of the curves show some cracks in the structure of the fiber before the total failure. These cracks arise due to the failure of the microfibers constituting the fiber. The curves showed that more fluctuations have specimens with the less content of epoxy resin. A photograph of typical failure behaviour after flexural tests in these specimens is shown in Fig. 8.

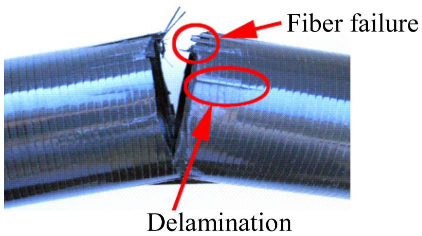


Fig. 8 Specimen after flexural test

Because of mixed layers orientation, there appeared transverse failure fracture with small areas of interlaminar (usually referred as delamination) shear modes of top longitudinal layer of the fracture area. The results presented in Fig. 7 show that decrease in epoxy resin content till 18% has effect just on the flexural strength, while the stiffness is influenced marginally. The impregnation below this magnitude has negative effect both on the stiffness and bending strength. It could be explained by the interlaminar bonding structure of the defects. Matrix fills the spaces between carbon fiber filaments, therefore, the decrease in the filler amount may result the appearance of voids and air gaps. Consequently, this causes the weaker interlaminar bonding and results delamination defects and fracture.

Obtaining results of flexural tests showed that in structures the epoxy resin content may be decreased till 18% without losing the stiffness and strength, whereas the structure with 16% content of epoxy resin can lose approximately 20 percent in strength. The epoxy resin impregnation process requires high accuracy and present-day prepreg manufacturers can guaranty just ±2% resin content tolerance, so it is necessary to use at least 20% prepreg to avoid the fluctuations.

There are three basic methods introducing a compressive load in to specimen: direct load to the specimen end (longitudinal compression test), loading specimen by shear (transverse compression test) and mixed direct and shear loading [17]. It is established that the compressive strength is generally lower than the tensile strength [18]. Aiming to design the composite structure of efficient and safe behaviour under compressive loading, it is of great importance to predict the compressive strength of the composite and consider the possible failure modes of the structure.

Fig. 9 shows load-displacement curves of transverse compression experiment. As can be seen from Fig. 9, all the specimens exhibited a linear relationship up to failure. The load F is increased until the maximum compressive stress reaches the compressive strength. A curve can be divided into two parts. The first corresponds to the linear region, which is dependent on the shear stiffness of the specimen. In the second part, the sudden failure of the specimen can be seen, proving the fracture behaviour of the specimen. The compressive loading acts on the fibers of transverse layers and epoxy resin rather than on axial fibers. The rate sensitivity of the epoxy resin should be reflected in the transverse compressive response. As it was expected, carbon fiber composite tube with 22% epoxy resin impregnation showed higher strength and stiffness. The load corresponding ultimate strength value is equal 750 N. The results showed that decrease in volume fiber fraction from 22% to 16% has a negative effect both on the strength and stiffness.

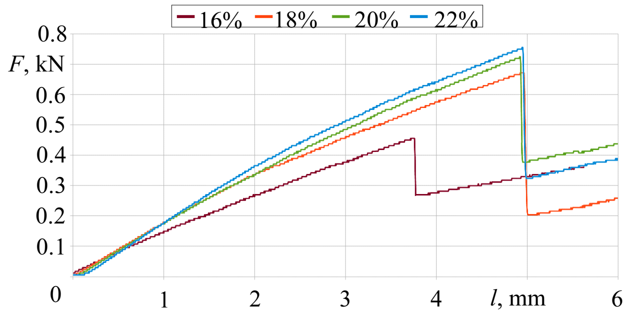


Fig. 9 Engineering load F versus displacement l curves of transverse compression tests for carbon fiber tubes having different volume of fiber fraction

Longitudinal compression testing was carried out in the same manner as for the previous specimens for transverse testing machine "Amsler" compression testing. In this experiment direct end loading method was used. It is important to keep in mind that both end surfaces perpendicularity to the symmetry axis of the tube has a big influence on the final result. Obviously, if this condition is not satisfied, loading nose at prime position acts just on the part of end surface. The specimens showed gradual decrease in load after the point of maximum load due to individual fiber breakage. It is seen from Fig. 10 that after reaching maximum load corresponding ultimate strength, the load decreased till lower value and fluctuated in relatively small area. In this area the fiber layers splaying and fiber fracture occurs.

Looking from strength and stiffness perspective from longitudinal compressive test results it is visible that with increased epoxy resin content the stiffness increases also, though strength values are very similar (comparing the obtained experimental results of the specimens with 20% of epoxy resin content with 22% and 18% with 16% respectively).

The curves in Fig. 10 show marginal non-linearity near specimen fracture at high strain values. The appearing non-linearity of the curves indicates the beginning of the specimen‘s fracture, i.e. indicates the diminishing stiffness. The loading process initiates the microcracking of matrix, the fibre composites exceed the limit of proportionality and the peak load results the total failure of fibres.

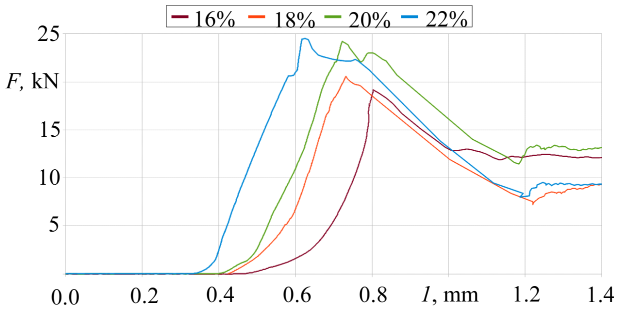


Fig. 10 Longitudinal compressive load F versus displacement l curves for the carbon fiber tubes having different volume of fiber fraction

Fig. 11 Fractured specimens after longitudinal compression test

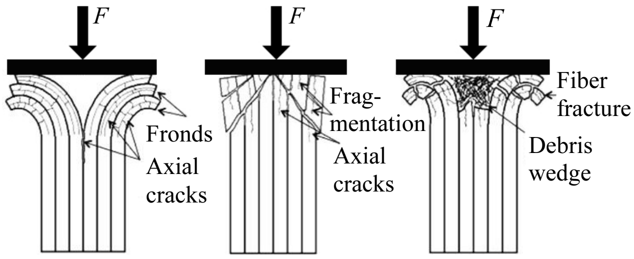


Fig. 12 Typical crush failure modes of carbon fiber composite tubes: a - fiber splaying; b - fragmentation; c - brittle fracture [19]

A photograph of some failed specimens is shown in Fig. 11. Failures consistently occurred at the end of the specimens with a complete breakdown of the matrix, the fibers splaying out to give a brush – like appearance. Fig. 12 shows a typical cross-section of a tube wall that has failed as brittle fracture mode of a composite cross-section. The results of longitudinal compression tests show that for the specimens with up to 22% of the volume fiber fraction the brittle fracture is the predominate crush failure mode of composite. This failure mode is essentially a combination of the fiber splaying and fragmentation modes and has the following common characteristics: formation of intermediate length axial cracks, fronds development and fracture, and rising of large debris wedge.

Long axial cracks are typical for cross-section of a tube wall that has failed by fiber splaying, while short axial cracks shows the fragmentation mode of a composite cross-section. The cracks separate the fibers into bundles, referred to as fronds [6]. These fronds are divided and bent either to the inside or outside of the tube wall. The initial cracks propagation starts first in the matrix, because fibres present more strength and strain to failure.

6. Conclusions

1. Experiments showed how critical the resin-to-fiber ratio is. In order to reduce the oar weight, the content of epoxy resin should be minimal, but the structure and properties of the tube should remain unimpaired. Obtained experimental results lead to conclusion that at least 20% of volume fiber fraction with constant carbon fibre quantity should be used.

2. Carbon fiber is used most efficiently under tension loading. When the load applied transverse to the tube with the different orientation of the layers, some of the fibers experience tension while the others experience compression. Experimental results showed that applying the load transverse to the fibre direction, the strength and stiffness of the composite are lower, if compared to those under axial loading.

3. The results of the compression response and failure are influenced by the fiber volume fraction and quality of the interface between the fiber and matrix. The higher volume of fiber fraction results increase both in flexural properties and compressive strength.

4. The tube materials failure occurs in several forms, i.e. microcracking of the epoxy resin matrix, carbon fibers breakage, separation from the matrix and separation of longitudinal and transverse orientation layers, i.e. the delamination.

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ANGLIES PLUOŠTO STRUKTŪROS ĮTAKA DAUGIASLUOKSNIŲ VAMZDŽIŲ MECHANINĖMS SAVYBĖMS

R e z i u m ė

Šiame darbe nagrinėjamos daugiasluoksnių anglies pluošto vamzdžių mechaninės savybės esant apkrovai. Kompozicinių medžiagų mechaninėms savybėms svarbiausią reikšmę turi anglies pluošto ir jį impregnuojančiojo rišiklio santykio nustatymas, nulemiantis gaunamo junginio charakteristikas. Pagrindiniu tyrimo objektu buvo vamzdžių, pagamintų iš vienos krypties anglies pluošto juostos, impregnuotos epoksidine derva, mechaninės savybės, suirimo procesas esant įvairioms apkrovoms. Tiriamųjų bandinių gamybai buvo naudojamas vidutinio stiprumo poliakrilnitrilo pagrindu pagamintas anglies pluoštas Toray T700, įmirkytas skirtingos koncentracijos epoksidinėje dervoje. Įvertinus bandinių gniuždymo ir lenkimo stiprumines charakteristikas, nustatytas optimalus impregnuojančiojo rišiklio kiekis.

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THE FIBER VOLUME FRACTION INFLUENCE ON MECHANICAL PROPERTIES OF MULTILAYERED CARBON TUBES

S u m m a r y

This paper considers development and analysis of the mechanical behaviour of tubes which are made of composite materials under loading. Composite materials interface between layers have an essential influence on mechanical characteristics and are hardly predictable before combining this mix. Presented analysis focuses on tubes, made of unidirectional carbon fiber, epoxy resin, considering various structure combinations and influence on mechanical behaviour under critical circumstances. The specimen tubes were reinforced using the polyacrylonitrile-based Toray T700 carbon fiber tow, of intermediate strength and modulus, combined with epoxy resin matrix of different impregnation, in order to analyse these variations effect on stiffness and bending strength characteristics.

Keywords: carbon fiber composites, volume fiber fraction, mechanical properties.

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