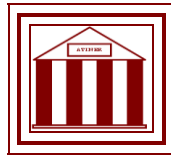


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**Specimen Preparation and Material
Characterization of Filament Wound Composite
Tubes**

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Specimen Preparation and Material Characterization of Filament Wound Composite Tubes

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Abstract

Filament wound composite structures are widely used in the field of pressure vessels, tubes, pipelines or rocket cases. The mechanical behavior of these structures is typically different from those of flat laminated structures due to an alternating lay-up sequence, winding tension and manufacturing induced imperfections. However, design and analysis issues require the same engineering data as used for laminated structures in general. It has therefore become necessary to establish an accompanying quality assurance procedure following the production process to identify the material properties of the manufactured tubes especially for the single layer. Consequently, there are three different approaches of determining the elastic moduli and tensile strengths of a filament wound laminate. Either specimens are resected from a curved tube, from a tube with plane areas or standardized flat specimens are manufactured under deviating production conditions. All approaches entail disadvantages, whether in terms of manufacturing or testing parameters including geometry, lay-up sequence, porosity, fiber tension and load direction. This study presents the discrepancies in the determination of mechanical properties of a filament wound glass-fiber-reinforced polymer tube on curved or cylindrical specimens and flat specimens produced to meet the specifications of international standards. In order to obtain material properties not only in longitudinal but also in transverse direction of the tubes, the so-called split-disk tensile test modeled after ASTM Standard D 2290, is used with tube segments. The procedures of specimen production and preparation are described in detail. Material properties such as the fiber volume and void content of the composite specimens are conducted in order to consider quality and production differences. Finally tensile tests are performed and the results are compared and discussed.

Keywords: Composite, Fiber-reinforced polymer, Filament winding, Split-disk, Tensile tests, Tubes.

Introduction

Due to the low density, high strength and stiffness, fiber-reinforced polymers (FRP) are usually associated with high-performance lightweight structures. Beyond these evident characteristics, FRPs are successfully used in engineering applications wherein lightweight properties occupy an outlying significance. Dependent on the type of fiber and matrix, these materials show excellent chemical and electrical performance. They feature dimensional stability, resistance to corrosion, outstanding damping properties and high fatigue durability. Today, for instance in chemical and process industries, traditional materials are successfully replaced and an increasing number of pipes, tanks, reservoirs and pressure vessels are made of composite materials (Putic et al., 2009). These products are usually manufactured using a filament winding process, at which oriented rovings (narrow bundles of fibers) are consolidated with a thermoset matrix system and wound around a rotating mandrel. Subsequently, the fiber-reinforced part is cured at certain temperatures and the mandrel is removed. The fabrication process can be highly automated and integrated into a continuous chain of production with a rate up to 500 kg of composite per day (Gay, 2015). With respect to the output in Europe of 2014, about 7.6 % of the mass of glass-fiber-reinforced polymers (GFRP) were produced by filament winding (Witten, 2014). In general, this production technology is progressing in various industries including aerospace, transport, electrical, consumer, construction and marine.

Despite of the high degree of automation, the individual process conditions and a variety of fabrication variables are affecting the mechanical properties as well as the quality of the composite material. Therefore, the engineering constants and strength values must be individually determined concerning design, dimensioning and calculation issues. However, the majority of international standards regarding the determination of FRP properties require flat and even specimens. Hence, many different approaches of obtaining the needed material properties have been developed, balancing a proximity to the manufacturing process with testing simplicity.

In the present paper three of the most promising approaches are investigated after a short overview of the filament winding process and the most-influential production parameters in relation to the mechanical properties of tubes. These procedures, among others, are described in detail whether in terms of manufacturing or testing. All investigations focus on the experimental determination of the elastic modulus and tensile strength of uniform-layered tubes in longitudinal and transverse direction by tensile testing. Ring specimens are moreover tested by means of a split-disk test. A comparison of the procedures is made aiming to find the most reliable methods for the determination of said parameters.

Filament-Winding

The filament winding process is based on a geometrical calculation of set-down patterns for one or more fiber-rovings onto a rotating mandrel, which serves as positive or male mold for the tube. In case of wet-winding, these rovings are usually impregnated with a thermoset matrix by passing through a resin bath or over a resin-soaked roll. After completion of the set-down pattern for the desired geometry and lay-up, the product is cured under rotation and defined temperature cycles.

The quality and material properties of filament-wound tubes depend on a variety of parameters. Numerous ways of classifying these parameters have been established. Cohen (1997) for example categorized influential parameters into five groups, which are resin, fiber, fabrication process, design and equipment. Five parameters were investigated with the result of fiber tension and winding time influencing the elastic modulus as well as the lay-up sequence, winding tension and winding tension gradient influencing the tensile strength in the hoop direction (Cohen, 1997).

Faria (2013) classified eleven parameters which can be controlled or influenced by the manufacturer, consisting of the basic materials (fiber and matrix), the mandrel geometry, the initial fiber tension, the bandwidth and/or thickness of the roving strands, the path of the fiber onto the mandrel, the winding angle, the initial degree of impregnation, the processing temperature, the winding speed, the lay-up sequence and the curing time after winding.

As it is apparent, it is not possible to expand on all parameters in this paper. However, the most significant ones are described in the following sections.

Influence of Lay-up and Basic Materials

As is generally known for FRP, the fiber angle, lay-up and layer thickness show the most significant influence on the mechanical properties, as countless works pointed out (Cohen, 1997; Vargas, 2004; Kaynak, 2005). Erdiller (2004) affirmed this statement by pointing out that a rise of the winding angle from 45° to 90° in split-disk samples increases the tensile strength from approx. 200 up to 1000 MPa and the tensile modulus from 20 up to 150 GPa. Aforementioned experimental investigations also showed that the variation of fiber material constitutes a larger influence than that of the resin. The hoop tensile strength of ten composite tubes with five different fibers, two different resin systems and a winding angle of $\pm 65^\circ$, was detected by split-disk tests. The resin system altered the strength in a range of 0.87 % up to 3.31 %, whereas an increase of nearly 10 % occurred by changing the type of glass fiber (Erdiller, 2004). This effect enlarges for tubes with greater winding angles owing to an overlap of fiber and load direction.

Some of these affective basic material parameters besides their mechanical properties are the type of roving, curing temperature and sizing of the fibers. The wettability, in dependence of the permeability of the rovings and the

viscosity of the resin, should also be taken into account. Low wettability leads to a bad impregnation and therefore to high void and low resin contents. This can be prevented by heating the resin, lowering the winding speed or lengthening the impregnation path (Balya, 2004).

Influence of the Manufacturing Process

The filament winding process is the subject of many investigations regarding its influence on mechanical properties. Lee and Springer (1990) established an analytical model of a filament wound tube which considers the effects of process variables such as winding speed, fiber tension and applied temperature on the mechanical properties. Erdiller (2004) experimentally examined the influence of fiber tension on tensile tests using complete tubes with the result, that an increase from 0 N to 15 N improves the obtained longitudinal tensile strength in a range from 1 % to 20 % depending on the type of fiber used.

Exact positioning of the rovings on the mandrel, fiber volume content and fiber tension are the most defining manufacturing parameters for quality and mechanical properties of filament wound tubes. The prestress or initial fiber tension is usually applied on the roving mounting via band brakes and should provide a uniform fiber tension, independent of the thread eye position or the volume of the roving coil. However, the fiber tension is also influenced by the number of deflections and their turning radii along the distance from roving mounting to mandrel. The strongest deflections usually occur within the impregnation unit and at the thread eye, the last guidance point, shortly before the set-down on the mandrel. Therefore, the fiber tension as well as the width and height of the roving strands can change locally, especially for increasing deflections on the tread eye due to low fiber angles. A constant width and height of the roving strands is essential for a precise positioning and filling without gaps or overlaps along the surface of the mandrel. Gaps may result in quality restrictions, such as voids and resin accumulations, while overlaps lead to local changes in the tube thickness and fiber orientation.

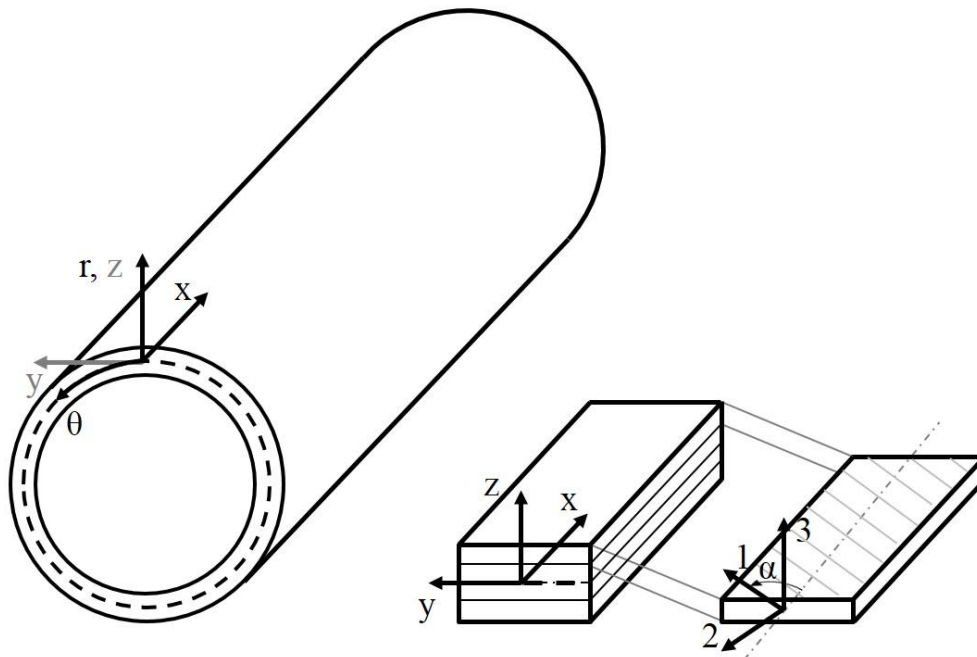
Achieving the desired fiber volume content without fluctuations throughout the volume of the tube or specimens is rather challenging. In general, rovings are soaking up too much resin during the impregnation. Thus, the superfluous resin has to be stripped off the roving strands. This can be realized e. g. by adding two rolls with adjustable distance after the impregnation unit. Nevertheless, an accumulating layer of resin will occur on the outer surface of the tube, which grows with increasing wall thickness.

Li et al. (2009) focused on numerical investigations of the subsequent production step of resin curing and in detail on the viscosity, degree of cure and curing rate under variation of the hold temperature from 438 K to 478 K in steps of 10 K. The thermo-mechanical model showed a decrease of all three mentioned parameters for lower hold temperatures influencing the residual stresses and warpage deformations of the tube.

Determination of Material Characteristics

All aforementioned factors affect the tube or specimen quality as well as their characteristics. The quality is usually audited by the void and fiber volume content on smaller accompanying samples of the tubes, whereas the determination of mechanical properties, in particular the elastic moduli and tensile strengths of the anisotropic material, is more challenging. The focus of this study lies on the characteristics in longitudinal and transverse direction of unidirectional-layered tubes, commonly designated as local 1- and 2-direction. This local coordinate system is transformed using the fiber angle α into a translational global laminate system x-y-z or cylindrical tube system x- θ -r with the designation as axial, hoop and thickness direction. A distinction between the local and global tube system is crucial due to the fact that for a winding angle of 0° longitudinal and axial direction and vice versa for 90° longitudinal and hoop direction are corresponding. Characteristics in the thickness direction r, z or 3 are generally difficult to determine and neglected for thin specimens. A comparison between the cylindrical coordinate system of the tube and the translational coordinate system of the laminate and single layers is given in Figure 1.

Figure 1. *Coordinate Systems of Tube and Flat Specimen in Comparison. From Left to Right: Tube, Laminate and Single Layer*



Fundamentally, there are three ways for the manufacturing process of test specimens. Either the specimens are directly resected from the cylindrical tube, manufactured using a polygonal mandrel with plane surfaces or manufactured in a different production technique, e. g. by hand lay-up. All three possibilities

have their advantages and disadvantages, whether in terms of manufacturing or testing.

Specimens from Cylindrical Tubes

The advantages of resecting the specimens from the actual tubes or products refer to the manufacturing process. There is no need to adjust the manufacturing process in any way, which implies that tubes and specimens share the same conditions. Quality, lay-up sequence and fiber volume content are not differing. The drawbacks of this method occur in a field of testing due to the curved specimens.

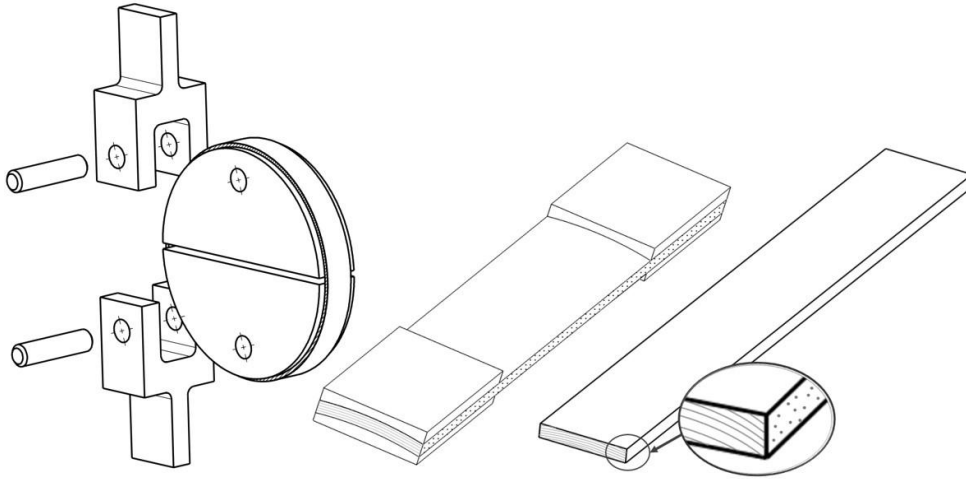
A curved geometry leads nonetheless to a multi-axial stress state, especially for the axial cut specimens and characteristics in a transverse direction. These specimens can be obtained from a tube in classical rectangular shape, according to standard DIN EN ISO 527-4 (1997) Type 2 or 3, with curved cross section. Because of this curvature load input elements have to be applied on the specimens, see Figure 2 middle, to generate plane surfaces in order to clamp the specimens in a testing machine. Another manner of generating those surfaces is by machining, see Figure 2 right. Here the curvature only occurs in the form of fiber directions of the single layers. Axial testing methods on entire tubes are also known, e. g. ASTM D 2105-1 (2014).

The determination of characteristics in hoop direction, typically fiber direction, could be examined by internal pressure tests, e. g. Karpuz (2005) or Frieß et al. (2010), or the more often used split-disk tests according to standard ASTM D 2290-04 (2012). The internal pressure or burst test is often unfeasible, because of the need for another testing machine, the difficulty of applying only radial pressure and the elusiveness of the hoop tensile strength. According to Knight (1977) and the stated standard, the split-disk test can only be utilized for the hoop tensile strength not the hoop modulus. Several research works such as Kaynak (2005), Srebrenkoska et al. (2015) and Yoon et al. (1997) disproved this statement in experimental and numerical investigations. However, the determination of strain as well as the calculation of the elastic modulus differs. All three mentioned investigations stated, that the friction between fixture and ring should be reduced, e. g. by applying teflon band, carbon powder or liquid lubrication (ASTM D 2290-04, 2012, Srebrenkoska et al., 2015; Yoon et al., 1997). Yoon et al. (1997) used strain gauges in various positions along the disk in combination with a load hysteresis to calculate the modulus, with the result that only the area of local bending at the gap in the fixture should be avoided. Srebrenkoska et al. (2015) determined the elastic modulus by measuring the displacement of the disks via an extensometer according to the equation (1) provided by Kinna (1964):

$$E_{exp} = \frac{0.1257 r_{mean}^3 F}{wt^3 \Delta} \quad (1)$$

The hoop tensile modulus E_{exp} is estimated using the maximum load prior to failure F , the mean radius r_{mean} , the displacement Δ and the thickness t and width w of the ring sample. Figure 2 shows the principle test setup for the split-disk test. The test fixture is designed to cause a failure of the ring specimens in the areas where the loading is nearly uniaxially, however an additional bending moment in the gap area cannot be prevented completely.

Figure 2. *Specimens Taken Directly from a Tube. From Left to Right: A-0 with Split-disk Setup, A-90K and A-90.*



Specimens from Mandrels with Plane Surfaces

In order to approach a similar manufacturing process for flat specimens to determine mechanical properties according to classical methods, mandrels with plane surfaces are used. Here, rovings are impregnated and handled according to the actual cylindrical product with only the mandrel affecting manufacturing changes in fiber tension and set-down quality. Relevant investigations according to the literature use a variety of mandrels, such as dihedrons (Sankar Reddy et al., 2015; Radulovic, 2008) or those with rectangular (Peel, 1998; Schultz, 1998), hexagonal or octagonal cross sections. This procedure is described in standard ISO 1268-5 (2001). The more the shape deviates from the circle, the more fluctuations occur in the fiber tension. The fiber or roving tension on cylindrical mandrels is constant throughout the winding process. Mainly two forces act on the roving yarn, the pretension in hoop direction and an additional force in radial direction caused by the rovings being pressed against the mandrel surface. For polygonal mandrels the radial force is maximized on the turning points and minimized in between. Therefore, the resin accumulates in the mid sections of the planes and no contact of rovings and mandrel may occur. This leads to smaller thicknesses and higher fiber volume contents close to the edges (Riteska et al., 2014; Schultz, 1998). Flat mandrels are moreover producing a higher void content, especially around the

edges (Perillo et al., 2014). In some cases, the specimens can also be slightly curved due to residual stresses caused by the mandrel shape or fiber tension.

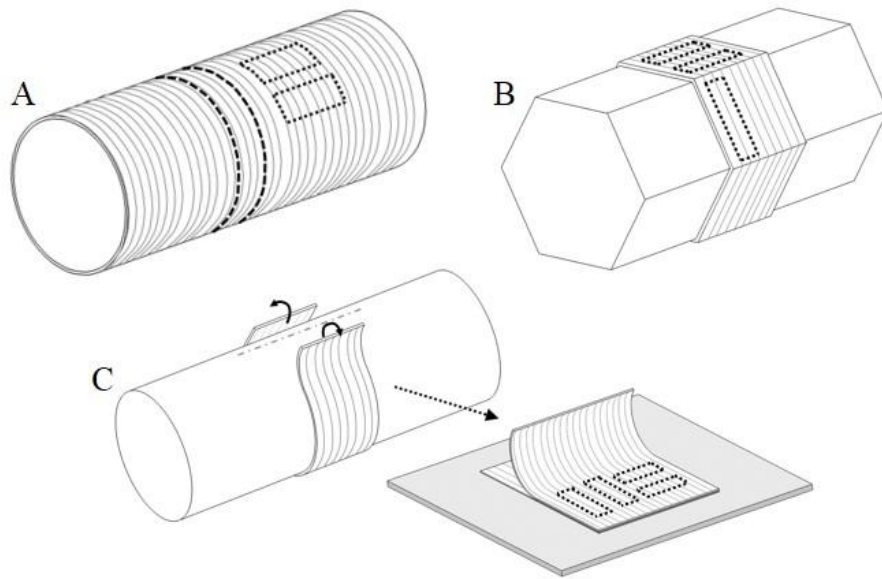
In order to generate plane specimens by using a cylindrical mandrel, in some works such as Frieß et al. (2010) or Phillippidis et al. (1999), the laminate is cut off the mandrel and placed on a flat mold before curing. Hence, the manufacturing induced fiber tension and radial stress is lost during this procedure.

Specimens from Other Production Techniques

This method is the least feasible from a manufacturing point of view, but provides the most standardized, flat specimens for the determination of the characteristics. The specimens are produced as accompanying samples besides the filament-winding process with the same basic materials. Quality and manufacturing parameters differ from those of the filament-wound tubes, irrespective of the used production technique (e. g. hand laminating, vacuum infusion or autoclave procedure). Especially a comparable adjustment of layer thickness, fiber volume content, void volume content and fiber positioning is very laborious. Even if this is achieved, there are still differences in the fiber tension and the ondulation of the layers. During the filament-winding process a kind of cross weaving occurs due to the simultaneous placement of two layers with opposite angles $[\pm\alpha]$. A recreation of this ondulation by hand laminating is time-consuming and requires a very precise placement of the rovings. However, the big advantage of this method lies in the standardized testing. An approach according to standard DIN EN ISO 527-4 (1997) for the determination of the elastic moduli and tensile strengths in plane direction is sufficient and the uniaxial stress state is secured.

Experimental Investigations

Three main kinds of specimens were investigated and referred to as type A, B and C. The first-mentioned are obtained from a cylindrical tube, the second from a hexagonal tube and the last from a plate, which was sliced from the cylindrical mandrel before curing, see Figure 3. The rotation of the mandrel was stopped for the latter method after the automatized set-down pattern. Then the tube was wrapped in foil to support stability during the manual repositioning of the laminate. A cut was made by a scalpel along a previously placed metal sheet to prevent the damage of the mandrel. In all cases, specimens in longitudinal (fiber) direction (0) and transverse (matrix) direction (90) were used to determine the elastic moduli and tensile strengths.

Figure 3. *Schematic Presentation of the Investigated Types of Specimen*

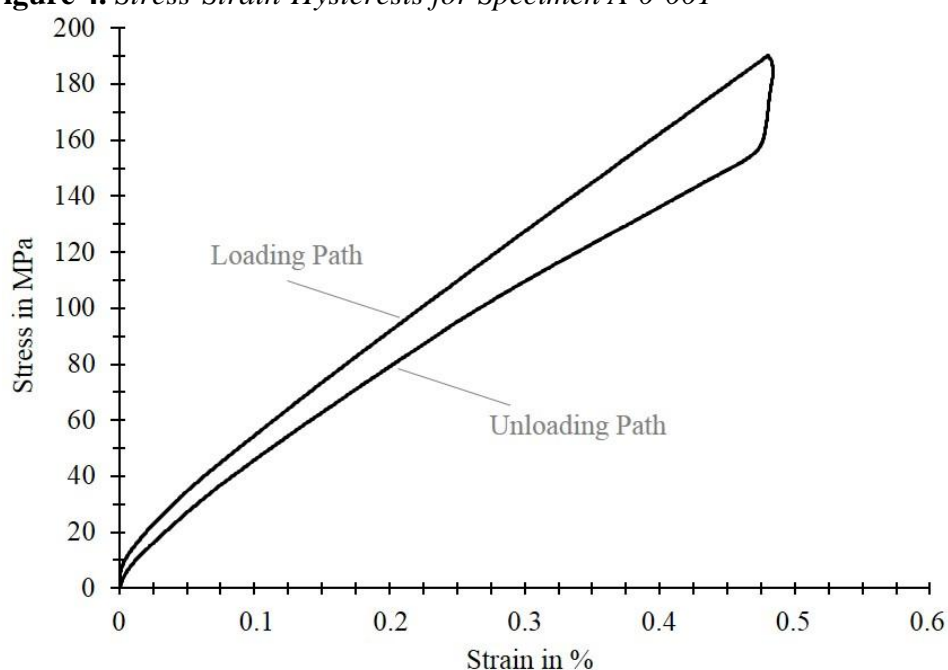
All types of specimens were manufactured using a filament winding machine with three controllable axes. The basic materials are glass fibers of the type PPG FiberGlass HYBON 2002 and an epoxy matrix system consisting of three components, the resin MY 740, hardener HY 1102 and accelerator DY 062 of the company Huntsman. Additionally, two defoamers with the product designations BYK-A 501 and BYK-A 525 were used. All specimens were wound with an angle of nearly 90° , the so-called hoop winding, and milled from cured tubes or plates in case of type C, respectively. The curing cycle included three temperature levels 110°C , 150°C and 170°C , which were successively passed with a heating rate of 2 K/min. and held for 40 minutes each. The cylindrical mandrel had a diameter of 150 mm and the areas of the hexagonal mandrel an edge length of 300 mm each. Specimens for the determination of the transverse properties were manufactured with larger thicknesses, considering the lower expected strength values.

Ten rings of width 25 mm, referred to as A-O, were prepared for the split-disk test and five of them (A-0-001 to A-0-005) equipped with strain gauges in longitudinal direction. For an investigation of the curvature influence, two specimen forms of type A for the transverse direction, termed as A-90 and A-90K, were obtained from the cylindrical tube. Whereas specimens of the type A-90K maintain their curved geometry as shown in the middle picture in Figure 3, specimens of the type A-90 are milled off pursuant to the right picture to achieve flat surfaces. Five to six specimens were prepared for types B and C, with widths of 12 mm for the longitudinal and 25 mm for the transverse direction. Load input elements were bonded on each specimen type except the split-disk rings.

All tensile tests were carried out on a 250 kN universal testing machine by applying a constant speed of 1 mm/min. Three specimens of types A-90, A-

90K, B-0, B-90, C-0 and C-90 were equipped with strain gauges in load direction. The tensile strength and the elastic modulus were determined from the stress-strain-graphs according to DIN EN ISO 527-4 (1997), using the least squares method for the latter. The split-disk specimens of type A-0 were examined following a hysteresis; see Figure 4, from 30 N preload to a load of 30,000 N. In accordance to the other specimens, the gradients of the loading and unloading path in the strain-stress-curve were determined using the least squares method in a range from 0.05 to 0.25 % strain. The obtained gradients were averaged for receiving the elastic modulus in longitudinal direction (Yoon et al., 1997).

Figure 4. *Stress-Strain-Hysteresis for Specimen A-0-001*



Additionally, the quality, expressed by fiber and void volume content, is determined on small quadratic samples of the tubes and plates according to the international standards DIN EN ISO 1172 (1998), DIN EN 2559 (1997) and DIN EN ISO 7822 (2000). The density was examined according to DIN EN ISO 1183-1 (2013).

Results

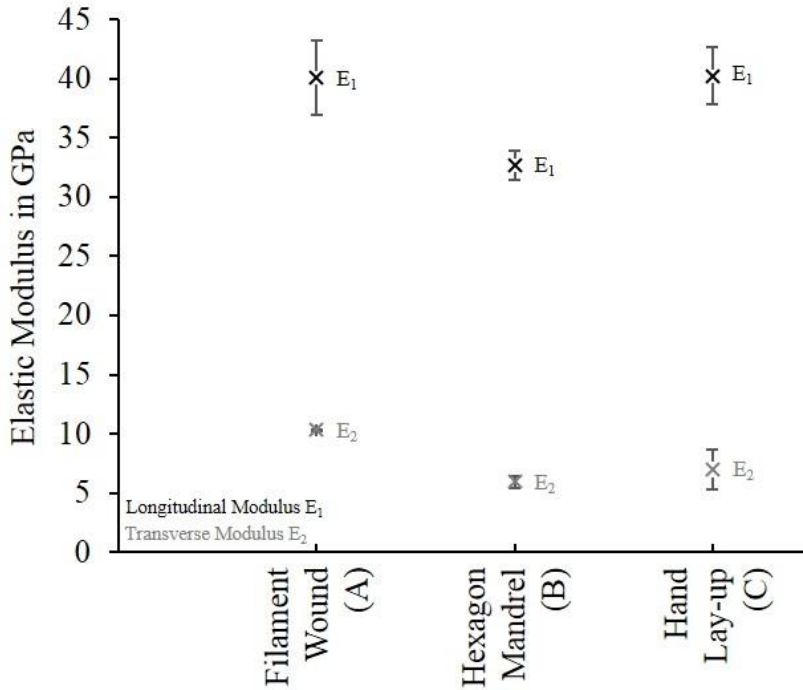
It should be noted, that the manufacturing process of the specimens of type C strongly depends on an accurate handling and placing of the sliced off laminate. However, the overall preparation and testing of specimens A is most laborious.

Specimens of the type A-90K with curvature cannot be evaluated due to delusively high values for the tensile strength and elastic modulus caused by an

additional axial curvature. This deformation is presumably caused by residual stresses. Therefore, the applied tensile load initially compensates this effect before stretching the specimen and the load cell registers a misleading high force level.

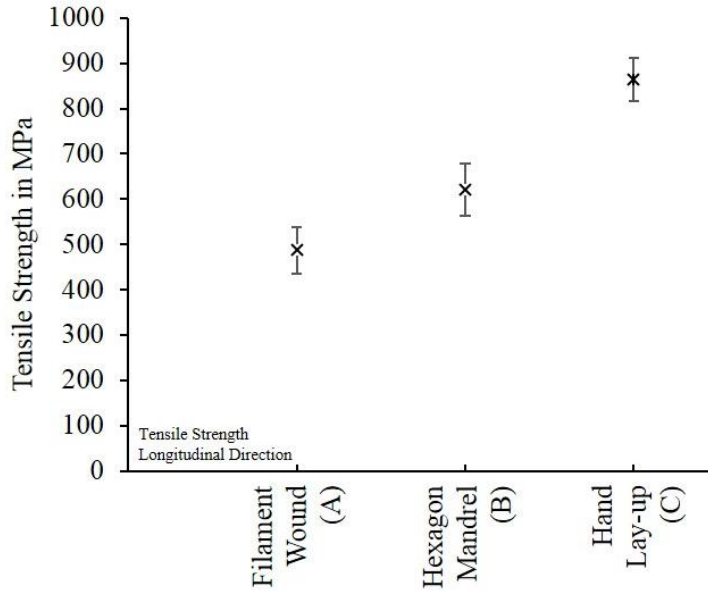
Figure 5 displays a summary of all determined elastic moduli for the three preparation procedures. Apparently, the longitudinal moduli E_1 for procedures A and C are sharing similar mean values and standard deviations, while the specimens from the hexagonal mandrel evince a significant drop of 18.7 % from 40.2 to 32.7 GPa. A similar trend is evident for the transverse modulus E_2 , although a discrepancy from procedure A to C can be perceived. The higher standard deviations for longitudinal direction are related to a higher dependency on an exact accordance of fiber, load and strain gauge orientation.

Figure 5. Longitudinal and Transverse Modulus Expressed by Mean Values and Standard Deviation



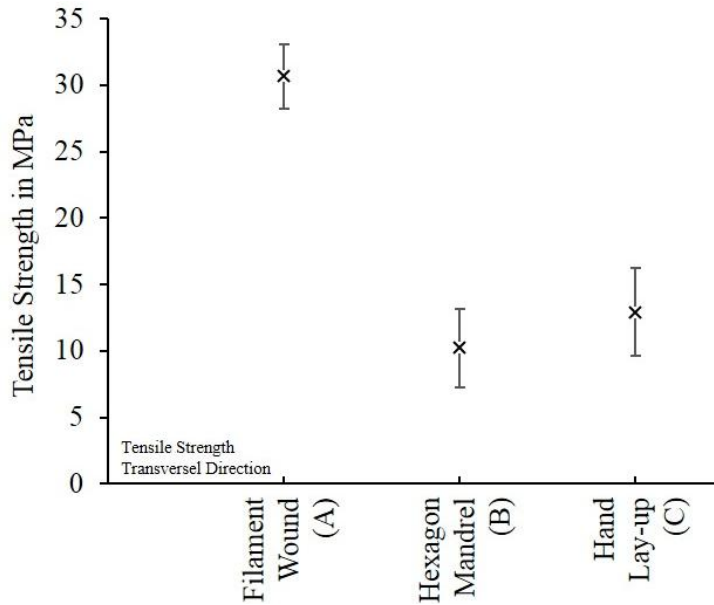
Regarding the longitudinal tensile strength, a clear ascending order from A to C is visible in Figure 6. In consideration of the corresponding elastic moduli E_1 obtained from the procedures A and C, it is obvious to raise a similar expectation for the tensile strength in a longitudinal direction. Nevertheless, a gap of approximately 376 MPa is recognizable, mainly resulting from the general set-up of the split-disk test, which is utilized for the specimens of type A-0. This result accords to the investigations of Knight (1997) concerning lower strength values owing to the additional bending moment and notch stress in the free area of the specimen where the failure occurs.

Figure 6. *Longitudinal Tensile Strength Expressed by Mean Values and Standard Deviation*



Conversely, the transverse tensile strength, depicted for all procedures in Figure 7, shows the highest values of 30.6 MPa for the specimens of type A-90 of the cylindrical tube. The low values for procedures B and C correspond to the substantially higher void contents up to 10 % of these specimens.

Figure 7. *Transverse Tensile Strength Expressed by Mean Values and Standard Deviation*



Conclusions

Specimens from the hexagonal tube in procedure B showed significantly lower elastic moduli and tensile strengths both in longitudinal and transverse direction due to about 10 % lower fiber volume contents. Consequently, this procedure appears inadequate for a material characterization.

The fact that the process induced fiber tension and radial force of filament-wound tubes was totally lost in procedure C-0 and the elastic modulus E_1 nevertheless corresponded with those of the split-disk tests of A, proved an independency of these two parameters. Therefore, both methods can be applied for the modulus in a longitudinal direction. However, procedure C is to be preferred for the examination of the longitudinal tensile strength due to the occurring bending moment and notch effect at the split-disk test.

Method A-90 with machined areas provides the best results for modulus E_2 and the tensile strength in transverse direction because of the lowest void content (< 2 %).

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