INVESTIGATION OF INFLUENCE OF CARBON FIBER DELIVERY SYSTEM FOR FILAMENT WINDING PROCESS WITH NOL-RING SPECIMEN TESTS

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Abstract

The aim of this study is exploring the impact of delivery system for filament winding process, including the impregnation bath on the characteristics of NOL-Ring carbon fiber/epoxy resin specimens. Tensile strength and break force of NOL-ring specimens was measured according to ASTM D2290. Meanwhile, scanning electron microscopy (SEM) was used in analysis of the interfacial behavior and breaking of fibers. Also, it will be presented the relationship between parameters of specimens winding and mechanical properties. The results show that, besides the winding parameters the carbon fiber delivery system used for manufacturing of specimens has influence on the tensile strength of the samples. The tensile strength of NOL-ring made from unidirectional carbon fiber/epoxy composites in this work reached maximal value of 2868MPa.

1. Introduction

Composite systems which consist of fibers and resins have high usage in industrial areas as new structural materials. Composites are strong and light, thus they are mostly used where the mobility is important. One of the important composite forming processes is the filament winding (FW) process. Although this process is very simple in concept, high path control of the continuous fibers is needed for high quality product.

For evaluation of filament wound composite structures NOL-rings test is usually used in Institute of Materials Science and Applied Mechanics, Laboratoire National d'Essais [1,2,3,6]. Proposed ring shape of samples may be applied in axial tension test, internal pressure test, etc., as well as their combinations. Hoop tensile strength of NOL-ring specimens can be determined with help of split-disk test [7].

With help of design of experiment (DOE) can be investigated the complex interaction between filament winding manufacturing and design variables, which affect tensile strength and composite quality of NOL-ring specimens [4,5,6]. Tension control is important factor for better winding patterns, which is the main reason for composite high strength [8].

Therefore, tension control system was designed and manufactured to understand the effect of it parameters on the end product. There are a number of parameters that affect the breaking of fiber during it transport in FW process, which influence the composites mechanical properties [9,10,11,12,13,14,15].

In this study will be considered some variables. Split-disk tests were used to determine the hoop tensile strength. These tests have lower cost than hydrostatical tests and are very efficient in determine the performance of tubular structures which are usually used under internal pressure developing high hoop. This research will present and discuss some of delivery control system variables and final properties of NOL-ring specimens, manufactured with conventional filament winding equipment.

2. Experimental

2.1. Materials and equipment

This study used highest strength, standard modulus single carbon fiber Torayca T700S 24K from Toray, adequate for high tensile applications like pressure vessels, recreational and industrial. Carbon fiber was impregnated into commercially available epoxy resin system with anhydride hardener and amine accelerator for filament winding processing from Huntsman, Araldite® LY 1135-1 / Aradur® 917 / Accelerator 960-1. Wet winding process was carried out on laboratory filament winding machine MAW FB 6/1 with six axes, roller type resin bath and electrical creel manufactured from Mikrosam A.D. Carbon fiber from the electrical creel with constant 20N winding tension was passing through different delivery system from N^{o}1 to N^{o}8 (figure 3) and through constant resin bath, causing fiber impregnation. Resin quantity on the impregnation roller was held constant with help of knife during the winding of all ring samples, whereas the temperature of the resin bath was constant, 37^{o}C. Winding speed was 3.3m/sec or 15% of the machine's maximal speed. Steel mandrel with five molds made especially for this research (figure 1) was used for the winding.

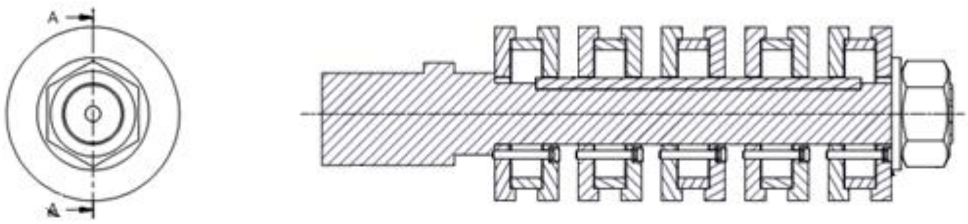
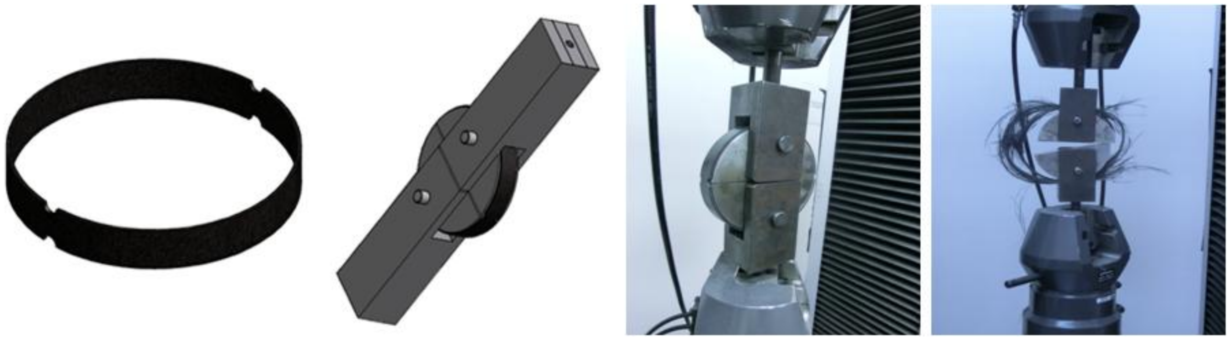


Figure 1. Technical drawing of the mould mandrel used for NOL-ring specimens winding.

Before winding, mandrel was coated with mold release agent from Huntsman, QZ-13 in order to ensure trouble-free de-molding of the samples. After the winding, fabricated samples were cured with help of industrial heater at 100^{o}C for 6 hours. According to ASTM D2290 [12] were made NOL-ring specimens with 146 mm inside diameter, 22,86mm width and winding angle ±90^{o}. Specimens were winded with 21 layers of carbon fiber and after the surface treatment their thickness was around 3.0mm. Prepared specimens had two reduced arias located 180^{o} apart with minimum width in the reduced sections of 13.97mm.

2.2. Mechanical analysis

Tensile tests of NOL-ring samples were carried out at room temperature using universal testing machine (AG-Xplus Series, Shimadzu) with max load of 250kN and loading speed of 5mm/min. Width and thickness of each NOL-ring specimen was measured with help of micrometer with reading to at least 0.0254mm. In this way prepared specimens were elongated till rupture with help of test fixture, made according to ASTM D2290. NOL-ring specimens and used test fixture are given in figure 2.



a) b) c) d)

Figure 2. NOL-ring specimens and test fixture a) NOL-ring sample b) Test fixture c) NOL-ring sample placed in text fixture d) NOL-ring testing

2.3. Design of experiment (DOE)

To optimize the filament winding process and production of NOL-ring specimens, design of experiment (DOE) has been followed.

Symbol	Factor	Level	
		-1	**+1**
A (x_1)	Number of rollers	4	7
B (x_2)	Roller diameter	60 mm	80 mm
C (x_3)	Sensor's angle	90^o	120^o

Table 1. Level of process parameters.

N^o of experiment	Factor		
	A	**B**	**C**
1	4	60	90
2	7	60	90
3	4	80	90
4	7	80	90
5	4	60	120
6	7	60	120
7	4	80	120
8	7	80	120

Table 2. Factorial design 2^3.

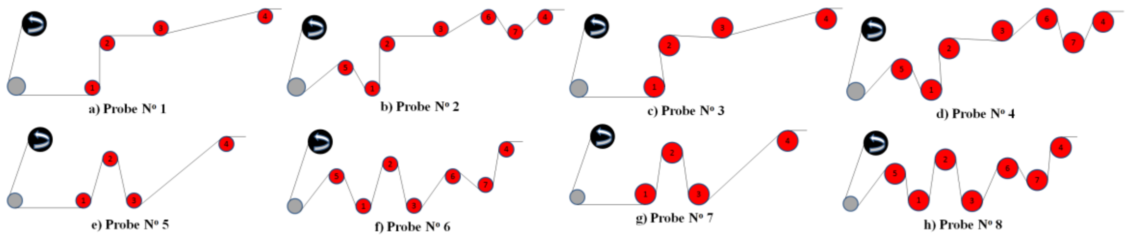


Figure 3. Schematic drawing of delivery system design (a) Sample N^{o}1, b) Sample N^{o}2, c) Sample N^{o}3, d) Sample N^{o}4,e) Sample N^{o}5, f) Sample N^{o}6, g) Sample N^{o}7, h) Sample N^{o}8).

DOE has been well known for its efficiency and allow gaining a maximum of information from a minimum amount of experiments. As process parameters - variables in fabrication of NOL-ring specimens in this research were used: number of rollers, roller's diameter and sensor's angle in two different levels (table 1) with number of permutations 2^3 and number of experiments 8. Factorial design type 2^3 was used and is represented in table 2. Design of carbon fiber delivery system with different winding angle, diameter of rollers and number of rollers is shown in figure 3.

2.4. Fractographic analysis

Fractured surfaces obtained from mechanical tests were examined at different magnification by using scanning electron microscope (SEM) from Tescan type Vega3, in order to observe fracture behavior of the specimens, fibers orientation and crack interaction of these domains.

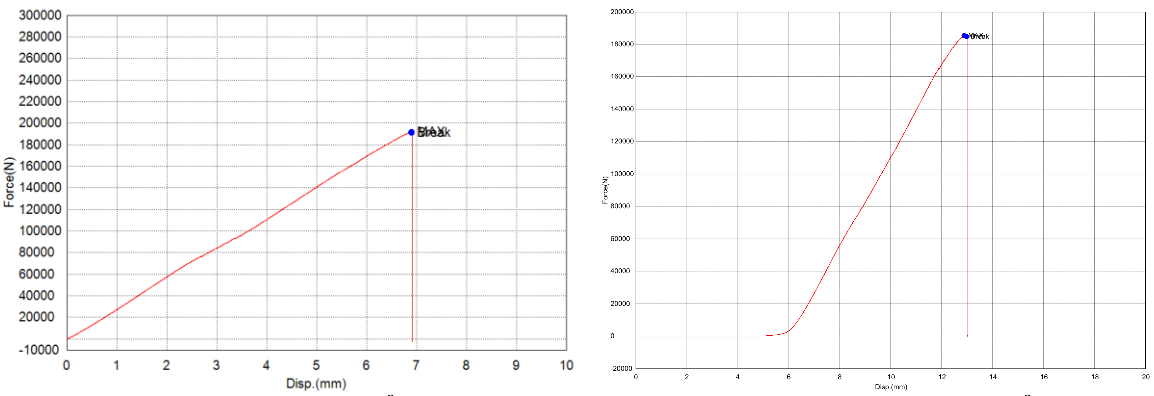
3. Results and discussion

3.1. Hoop tensile strength

It should be noted that the tensile strength, σ can be determined with help of equation 1.

$$\sigma = \frac{F}{2tw} \tag{1}$$

In equation (1) F is the ultimate burst force recorded in Newton (N), whereas t and w are the thickness and width of the NOL-ring sample in millimeter (mm), respectively according to ASTM D2290 standard.



a)Force and displacement of sample N^{o}7-4 b)Force and displacement of sample N^{o}3-1

Figure 4. Force and displacement graphs of NOL-ring samples from universal tensile testing machine.

Figure 4 shows a typical force-displacement diagram at ambient temperature for samples N^{o}7-4 and N^{o}3-1. The tensile strength of composite rings were determined for four specimens for each experiment according to equation (1). The average values for each experiment are shown in table 3. Displacements of all samples were around 6-7 mm of the tensile tests.
From the results shown in table 3 can be observed, that NOL-ring specimen N^{o}7 with tensile strength of 2646.4MPa had shown the best results. This sample N^{o}7 was winded with 4 rollers with 80mm outside diameter and angle 120^o. In contrary, specimen N^{o}4 winded with 7 rollers with 80mm outside diameter and angle 90^o had shown the worst results, 33% lower value than tensile strength from sample N^{o}7. According to [8], control system in filament winding technology is important factor in better winding patterns which is the main reason of

high strength. The influence of main winding parameters during manufacturing of full section composite parts is reported in [5].
If comparison is made between specimens N^{o}6 and N^{o}2 and specimens N^{o}7 and N^{o}3, it can be seen that sensors angle has an influence from 1,9% to 21% in mechanical properties of NOL-ring specimens. Further, rollers diameter have shown an effect from 6,88% to 19% in specimens N^{o}1 and N^{o}3 and specimens N^{o}7 and N^{o}5, respectively. Number of rollers has demonstrated an influence between 2,2% and 17% in mechanical properties for specimens N^{o}1 and N^{o}2 and specimens N^{o}7 and N^{o}8.

Specimen N^o	**Break force mean [N]**	**Tensile strength [MPa]** ($y_{exp.}$)	**Tensile strength mean [MPa]**	**Specimen N^o**	**Break force mean [N]**	**Tensile strength [MPa]** ($y_{exp.}$)	**Tensile strength mean [MPa]**
1-1	162475.0	1891.46	1885.97	5-1	180300.0	2205.67	2127.64
1-2		1864.04		5-2		2247.66	
1-3		1871.17		5-3		2010.40	
1-4		1917.20		5-4		2046.81	
2-1	165381.2	2011.69	1929.27	6-1	168950.0	2137.38	1967.94
2-2		1910.24		6-2		1975.63	
2-3		1887.94		6-3		1896.02	
2-4		1907.21		6-4		1862.74	
3-1	179286.6	2067.15	2071.84	7-1	210955.4	2707.51	2646.46
3-2		1957.57		7-2		2582.36	
3-3		2133.07		7-3		2867.49	
3-4		2129.56		7-4		2428.47	
4-1	151311.8	1685.27	1770.26	8-1	190968.7	2204.12	2191.00
4-2		1740.62		8-2		2189.14	
4-3		1777.98		8-3		2238.27	
4-4		1877.15		8-4		2132.49	

Table 3. NOL-ring results from tensile testing for each design of delivery system.

Ring shape of samples, were cut from manufactured composite cylinders and were tested until failure using pressurized ring test method to measure composite vessel hoop strength and stiffness [4]. Burst pressure between 21,1MPa and 20,1MPa was detected by the tests. Further, NOL-ring samples tested at ambient temperature manufactured with carbon fiber/epoxy resin are investigated in [6], where average burst pressure of samples was 1578.9MPa.
In [11] tensile strength of carbon fiber/epoxy resin specimens was measured with help of split disk test. Specimens winded with 90^o winding angles were prepared according to ASTM D2290. It was concluded that, carbon fiber (800tex)/epoxy resin specimens have average tensile strength between 1157.60MPa and 1201.20MPa, whereas carbon fiber (2190tex)/epoxy resin specimens have average tensile strength between 964.46MPa and 1033.38MPa.
Similar mechanical testing of epoxy resin/glass fiber split-disk-test specimens cut from winded composite tube were conducted in [13], where tensile modulus of 2.75GPa and fractural stress from 731MPa were reported. Furthermore, for all samples fracture occurred in the reduced section too. However, in [13] is described separation between the laminas, regions of delamination, where the fracture of fibers occurs in blocks.

3.2. Design of experiment (DOE)

The results for tensile strength, dispersion and minimal value of parameter's final coefficients for factorial design 2^3 in this research are shown in table 4. According to table 4, minimal calculated value of parameter's final coefficients is 42.54. Parameter's function and their interaction with 5% mistake are represented with equation 2.

Specimen	y_{exp}	y_{cal}	S_y^2	S_y^2sum	S_y^2mid	S^2b_i	Δb_i
1	1885.9	1891.13	232.86	81874.02	10234.25	426.4	42.54
2	1929.2	1922.13	3116.68				
3	2071.8	2066.71	6720.37				
4	1770.2	1777.39	6529.35				
5	2127.6	2122.49	13590.74				
6	1967.9	1975.09	15003.17				
7	2646.4	2651.59	34736.71				
8	2191.0	2183.87	1944.15				

Table 4. Results from design of experiment (DOE)

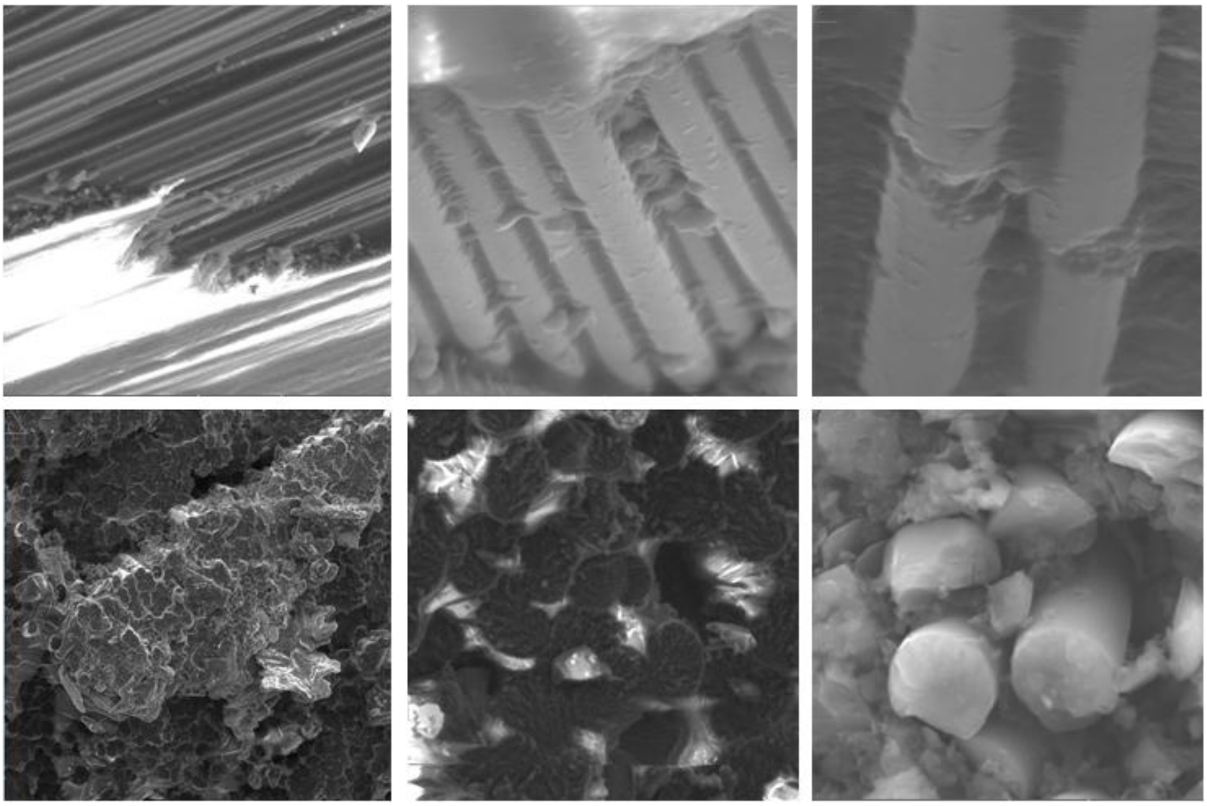
$$y = 2073.8 - 109.18x_1 + 96.09x_2 + 159.46x_3 - 80.08x_1x_2 - 44.6x_1x_3 + 89.38x_2x_3 \qquad (2)$$

From design 2^3 were calculated Cochran criteria (G_{cal}) with value 0.42 and Fisher criteria (F_{cal}) with value 0.12, which fulfill the rule $G_{cal} < G_{tab}$ and $F_{cal} < F_{tab}$ [16,17]. According to this, the hypothesis for model 2^3 is acceptable with 5% mistake.
In [4] is represented the effect of processing and design variable on strength, stiffness and quality of composite vessels using design of experiment. From five variables, it was concluded that tension gradient and stacking sequence show the highest effect on vessels burst pressure, whereas winding tension and the interaction between tension gradient and winding time exhibit smaller but significant influence. Cut or uncut helicals, as variable, execute no effect on cylinders burst pressure.

3.3. Scanning electron microscopy (SEM) analysis

Scanning electron microscopay (SEM) analysis were performed to see impregnation quality of carbon fibers into the epoxy resin during filament winidng process. Also, demages on carbon fiber surfaces were observed, due to the influence of the delivery control system. In figure 5 are presented SEM analysis from longitudinal and cross section of NOL-ring specimens with differnet magnification after tensile testing.
From SEM results can be seen cracks in the epoxy resin. It is assumed that, during the testing after the load is transferred to the fibers, fiber breaking occurs and at the end it is observed breaking of the NOL-ring sample. It can be stated from figure 5 (a) and (b) that, carbon fibers are well adhered by the matrix, but they deform during the tensile testing. Further, in figure 5 (c) can be noticed broken carbon fibers in longitudinal section. These damages have occurred during filament winding process, due to the larger number of rollers in the control system where the breach of fibers is often until they pass the machine delivery system.
SEM analyses of unloaded and loaded NOL-ring specimens were done in [7]. NOL-ring specimens, made from PPS and carbon fibers, were prepared according to ASTM D2290. Irregular distribution of matrix and fibers was seen, due to distribution of fibers within the prepreg and the filament winding process using in-situ consolidation of the thermoplastic

polymer by laser heating. In addition, single carbon fiber filaments breakage and inter-fiber matrix cracks was observed [7].



a) b) c)

Figure 5. SEM photographs of failure section for NOL-rings (deforming and breaking of fibers after testing)

4. Conclusions

The experimental procedure described in the present work is suitable to study the influence of fibers delivery system on mechanical characteristics of NOL-ring specimens.

From conducted mechanical testing can be concluded, that the best results in tensile strength and break force were obtained from delivery system with 4 rollers with 80mm outside diameter and sensor angle 120^o. Change of sensor angle will cause variation from 1.9% to 21% in final mechanical results, whereas roller diameter in delivery system show an influence from 6.88 % to 19.6%. Following, different number of rollers have an effect from 2.24% to 17.21% in the mechanical results.

The obtained function from the factorial design lead to conclusion that researched parameters and the interaction between them has to be taken into account in FW process.

SEM analysis exhibit good merger between carbon fibers and epoxy resin system in the NOL-ring specimens. Also, the breaking of the NOL-ring specimens took place at the reduced areas, as expected. In addition, damages on the fibers surface were detected, due to the larger number of rollers in the delivery control system.

It is assumed that, lower mechanical properties of NOL-ring specimens are caused from fiber breach or void content in the final composite, which were not analyzed in this research. Statistical analysis of the data showed some very significant results, which should be very helpful in improving composite pressure vessel performance and quality. Some of the more significant findings relate to how composite rings strength was affected by the manufacturing and design variables.

5. References

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