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Study of Voids Effect on Tensile Strength of Carbon Fiber Reinforced Composites for Structural Applications

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Abstract:** Advanced carbon fiber reinforced polymer composites (CFRP) are being particularly used for aerospace structural applications because of their high strength, stiffness and light weight. To design a structural component thorough characterization of composite material is required. In the present study the high strength carbon T700 12K fiber with bi-functional epoxy resin LY556 and hardener HY5200 was selected for fabrication of NOL ring specimens on a cylindrical mandrel by filament winding technique. Voids are inevitable during the fabrication in the fiber reinforced composites. The matrix-dominated properties such as: inter laminar shear strength, bending properties, tensile strength and modulus, fatigue and fracture toughness are influenced by voids, while the fiber-dominated mechanical properties are not significantly influenced by voids. Different thicknesses of NOL rings of 1.3, 1.4, 1.6 and 1.7mm are made and defects like voids were identified through non-destructive testing methods. Ultrasonic test is used to identify the voids in the NOL ring specimens. Ultrasonic wave transmission was observed at a particular zone which gave the resemblance of induced voids in the specimens. Composite properties are directly proportional to the fiber volume fraction. Physical properties of different thickness of laminates were evaluated. It is observed from the test results composite with less void content gave better compaction and strength compared with more void content. The results also showed that resin mix viscosity influences the void formation and strength of the filament wound composite.

Keywords:** CFRP, high strength, NOL ring, Ultrasonic test, Voids, filament wound composite

I. INTRODUCTION

The mechanical characterization of fiber reinforced composites are determined by the interfacial adhesion properties between fiber and matrix. The transfer of the stress is through the combination of physical adhesion, chemical bonding and mechanical inter locking. However effective fiber matrix interface is required to transfer the stress from the matrix to the load bearing fibers. If any void/porosity are present in fiber matrix interface the bonding strength will decrease which results in strength decrease[1-6]. To avoid the voids during fabrication of specimens thorough mixing of resin and hardener is required and no bubble should be formed during impregnating the fiber in the matrix. Composite materials are heterogeneous materials they are prone to have certain defects inside the composite laminates. Many factors are associated with strength characteristics of composites such as resin content, fiber volume fraction, void content, loading direction and loading rate etc. Other factors contribute to the formation of void including the curing pressure, resin system, environmental conditions and so on, some of which are almost unavoidable. The mechanical properties of composite directly related to the reinforcing fiber content. It is therefore important that the fiber ratio should be monitor during the manufacturing of structural component. The role of fiber in a composite plays a vital role. The main factors that govern the fiber contribution in composite are the mechanical properties of fiber, the surface interaction of fiber and resin (Interface), the amount of fiber in the composite (Fiber volume fraction) and orientation of fibers in the composites. The strength and stiffness of a composite laminates will increase in proportion to the amount of fiber content. Although literature indicates that mechanical properties decrease as the void content increases[7-8], the magnitude of the effect of void content on the cylindrical shape filament wound CFRP is not evident. Hence in this study particularly carbon fiber reinforced filament wound composite specimens fabricated with varying process parameters. Another important factor which effects the strength of the composite is viscosity of resin mix[9]. To derive the full potential of the fiber in composite matrix viscosity play an important role. In this work, effect of physical properties on filament wound composite specimens were studied.

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II. EXPERIMENTATION

A. Materials

The reinforcing material is carbon fiber T-700 12K and the epoxy resin is diglycidyl ether of bisphenol –A (DGEBA) type of bi functional epoxy resin LY556 which cannot be cured on its own. A curing agent like organic amines HY5200 are mixed thoroughly with epoxy resin. The resin mixture undergoes cross linking reaction at elevated temperature and curing will be completed within 5 to 6 hours.

1) Technical Specification:

Table.1 Carbon fiber T700

Parameters	**Specifications**
GradeTow size	T-700 12K, No Twist
Tensile Strength	4.9 GPa
Tensile Modulus	230GPa
% Elangation	2%
Density	1.8g/cc
Carbon content	94%
Fiber diameter	6-8 microns
Sizing	Epoxy compatible

Table.2 Resin LY556 and Hardener HY5200

Parameters	**Specifications**	
Grade	LY556	HY5200
Colour	Pale yellow, clear liquid	Brown, clear liquid
Specific gravity	1.1-1.2	1.0-1.1
Viscosity	8000 -12000 CPS	150 -180 CPS
Epoxy content	5.0 – 5.9 Eq/kg	-
Volatile content	0.75 % by weight	-
Shelf life	Two years from the date of manufacture	Three years from the date of manufacture

B. Sample Preparation

The high strength carbon roving T700 12K and epoxy resin LY556 and suitable hardener HY5200 was selected for fabrication of NOL ring specimens by wet filament winding technique. The ratio of resin to the hardener is in proportion of 100:24 by weight was maintained. The carbon fiber strands are un wind and passes continuously through resin tank, these resin impregnated strands are passed on to a rotating cylindrical mandrel. The strands are wound around the mandrel in a control manner and specific orientation. The temperature of resin is maintain around 50^0c in order to bring the resin viscosity optimum range this will enables volume fraction of fibers.

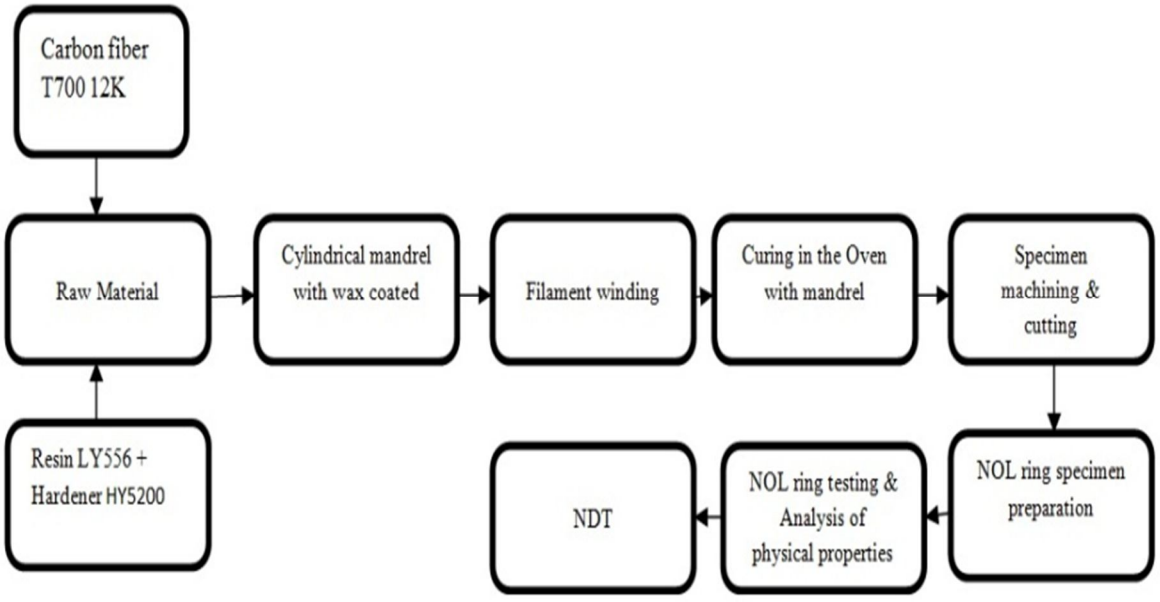


Fig.1 Flow chart of manufacturing of NOL ring specimens

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Fig: 2 Filament winding machine with resin bath



Fig: 3 Filament winding machine with winding

C. Specimen Curing

The oven is used for curing the filament wound NOL ring mandrel. The temperature of oven is 0 to 250 ^{0}c and chamber internal dimensions are 15000 X 3000 X3000 mm was selected for curing the component. The oven air temperature must be controlled with digital temperature indicator cum controller. The oven should have features to keep the job rotating during curing. The following cure cycle was followed.

Set the temperature of the oven increased from room temperature to 90^{0}c @ 1^{0}c /minute (90min)
Minimum component temperature 80^{0}c hold starts and maintains component temperature 80^{0}c ± 2^{0}c for 2hrs.
After completion hold period set the temperature to 170^{0}c @ 1^{0}c /minute (90min)
Minimum component temperature reaches 160^{0}c hold starts maintain component temperature 160^{0}c ± 2^{0}c for 4hrs.
After completion hold period set the temperature to 190^{0}c @ 1^{0}c /minute (30min)
Minimum component temperature reaches 180^{0}c hold starts and maintains component temperature 180^{0}c ± 2^{0}c for 4hrs.
After completion hold start cooling the oven from 180^{0}c to 60^{0}c @ 1^{0}c /minute (120min)
When temperature reaches 60^{0}c switch off the oven allow the component to cool to room temperature within 2 to 4 hrs.
Open the oven door and the remove cured mandrel from the oven.

D. Specimen Cutting

NOL ring specimens are made from cured filament wounded mandrel. The mandrel is placed between the chuck and the tail stock of the lathe machine by using single point cutting tool turning operation is carried out on the wounded mandrel. A fine cut has been given for removal excess resin formed during winding process. NOL ring specimens can be made by using parting tool according to ASTM 2290 standards.

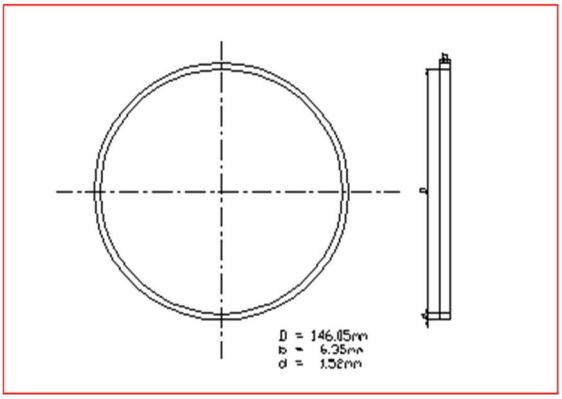


Fig.4 Dimensions of NOL ring specimen



Fig.5 NOL Ring after cutting

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E. Specimen Testing

Different tests were performed to know the physical and mechanical properties of the samples. NOL ring specimen was tested to find hoop tensile strength, specimens of each one with different thickness was made to test tensile strength. The hoop tensile strength was carried out by using universal testing machine (UTM). The tensile behavior prepared samples were determined at room temperature according to ASTM D3039 standards. The test specimens has been placed in the fixture (Four way split) of adjustable grips of 100KN computerized UTM with electronic extensometer. The test was repeated 5 times of each thickness of specimen and average value was considered to calculate the tensile strength.



Fig.6 Universal testing machine (UTM)



Fig.7 NOL Ring Test Fixture

III. TEST METHODS

A. Determination of fiber content

The mechanical characterization of a laminate is directly related to the fiber reinforcing content. It is therefore important that the fiber ratio should be monitored during the process.

The reinforcement weight percentage content (Mf)

$$Mf = \frac{Mass\ of\ the\ reinforcement\ in\ the\ laminate}{Total\ mass\ of\ the\ laminate} x100$$

The resin weight percentage content (Mm)

$$Mm = \frac{Mass\ of\ the\ resin\ in\ the\ laminate}{Total\ mass\ of\ the\ laminate} x100$$

If no voids are present $Mf\ \% + Mm\% = 100\%$ or $Mf + Mm = 1$

In the same way the reinforcement volume percentage content (Vf)

$$Vf = \frac{Volume\ of\ the\ reinforcement\ in\ the\ laminate}{Total\ Volume\ of\ the\ laminate} x100$$

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$$Vm + Vf = 1$$

Vm = 1- Vf , weight and volume fractions are related

$$Vf = \frac{\frac{Mf}{\rho f}}{\frac{Mf}{\rho f} + \frac{Mm}{\rho m}} \qquad Mf = \frac{Vf x \rho f}{(Vf x \rho f) + (Vm x \rho m)}$$

ρf and ρm being the specific gravity of the fiber and matrix.

The specific gravity of the laminate ρs can be related to the fiber and matrix content.

$$\rho s = (Vf x \rho f) + (Vm x \rho m)$$

B. Determination of Fiber Volume Fraction of Carbon Fiber

The fiber volume fraction of carbon fiber composite can be evaluated by thermo gravimetric analysis (TGA). The laminated specimen approximately 5 -10 mg was cut in to small pieces and placed in a platinum crucible and heated. To protect from oxidation the carbon fibers a constant nitrogen flow of around 40ml/min was maintained. The heating of crucible from room temperature to 800^0c @ 10^0c per minute can be carried out and kept at 800 ^{0}c for 30 minutes. The mass percentage of matrix Xm was obtained from Tg curves. The fiber mass percentage was calculated by Xf = 1-Xm. The fiber volume fraction of the laminate was calculated by using

$$Vf = \frac{Mf\ X \rho m}{(Mf X \rho m) + \rho f(Mc - Mf)} \text{X } 100$$

Where

Vf is Volume fraction of fibers

$\rho m\ is$ Density of epoxy matrix

$\rho f is$ the volume density of carbon fiber

Mc is the total mass of the sample

Mf is the fiber mass of the sample

Mf = Mc x Xf = Mc x (1-Xm)

Another chemical method to determine the Volume fraction of the carbon-epoxy composite is by Acid digestion method as per ASTM D3171. In this method, take 50mg composite sample in 400ml beaker and pour in 200 ml of the Nitric acid. Then heat the beaker on a hot plate around 80ºC temperature until the acid slowly fumes. Stir occasionally with a glass rod carefully. Continue heating until the matrix is dissolved and the sample disintegrates, leaving bare fibers. Insert a funnel into large flask attached to the vacuum system and transfer the acid and the fibers into the funnel,. Turn on the vacuum pump and wash the fibers three times with 20 ml of nitric acid and then follow with a distilled water wash. Remove the funnel and the fibers and dry them in an oven at 100ºC for at least 90 min. Remove the funnel and fibers and let them cool in a desiccator. Weigh the funnel containing the fibers. Calculate the Fiber volume fraction:

$$Vf = \frac{Wf\ X\ \rho m}{(Wm \rho m) + \rho f Wm}$$

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Where it is assumed that the void content of the composite is negligible.

C. Void Content

During the incorporation of fibers into the matrix or during the manufacturing of laminates air or other volatiles may be trapped into the material. The trapped air / volatiles exist in the laminate as micro voids which may significantly affect sum of its mechanical properties. A high void content (greater than 5% by volume) lead to low fatigue resistance and increased variation in mechanical properties. A void content in a composite laminate can be estimated by comparing the theoretical density with its actual density.

$$V_{void} = (\rho_{ct} - \rho_{ce})/\rho_{ct} \times 100$$

Where

ρ_{ct} is theoretical density of the composite material.

ρ_{ce} is experimental density of the composite material.

Especially handmade laminates causes air bubbles to congregate between layers, If successive layer have not been properly consolidated. The higher the mass per unit area of the woven fabric having air bubbles are voids between the layers. These voids may cause stress concentration and delamination. It is therefore necessary to check the voids in the laminates.

The voids can be calculated by

$$Vv = [(100 - Mf)\frac{\rho s}{\rho f} + (100 - Mf)\frac{\rho s}{\rho m}]$$

Where

Vv = void content

Mf = fiber content expressed as mass percentage

ρf = Density of fiber

ρm =Density of matrix

ρs= Density of laminate

D. Non- Destructive Testing (NDT)

NDT is used to detect defects in laminated composites. In this testing component is not damaged in any way by this test procedure. The aim of the test is to detect poor compaction leading to resin rich areas or low fiber loading or internal defects like lack of reinforcement, delamination, cracks, failure of adhesive bond and inclusions.

E. Ultrasonic Testing (UST)

Low frequency ultrasonic wave (0.2 – 10 MHz) can propagate through the composites. The anisotropic, heterogeneous material causes alteration and dispersion of the beam. Theoretical point of view propagation is a complex phenomenon because two phase nature of system (fiber and resin). Structural defect interface with propagation of UST and partially reflect the sound waves. The presents of voids in the laminate reduces the transmission of UST and the method used to quantify the void content. Transmission speed of UST increases with increasing the fiber loading. In through transmission method an ultrasonic transmission is used on one side of the material while the detector is placed on the opposite side of the material. Scanning of the material using by this method will result in the location of defects, flaws and inclusions in the X-Y plane. This method is used for non-destructive testing of multi layered materials. It has been shown that ultrasonic characteristics are influenced by the voids in composite. Ultrasonic transducers with low frequencies were employed to determine how the ultrasonic characteristics varied with changes in the void volume fraction.

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IV. TEST RESULTS AND DISCUSSION

Table: 3 NOL Tensile strength at average density of 1.40

Sl. No	Dimension in (mm)	Density (g/cc)	Resin content (% by weight)	Fiber content (% by weight)	Fiber volume fraction (Vf)	Void (%)	NOL Ring Tensile Strength (MPa)
1	6.41x1.7	1.42	30.14	69.86	57.70	1.90	1300
2	6.45 x1.7	1.42	27.78	72.22	57.62	1.84	1324
3	6.36 x1.7	1.38	54.54	75.46	58.51	1.75	1317
4	6.48 x1.7	1.38	31.43	68.57	56.25	1.97	1289
5	6.51 x1.7	1.40	32.09	67.91	56.85	1.98	1311
Average		1.40	35.19	70.80	57.38	1.88	1308

Table: 4 NOL Tensile strength at average density of 1.46

Sl. No	Dimension in (mm)	Density (g/cc)	Resin content (% by weight)	Fiber content (% by weight)	Fiber volume fraction (Vf)	Void (%)	NOL Ring Tensile Strength (MPa)
1	6.43x1.6	1.46	31.88	68.12	55.88	1.92	1445
2	6.35 x1.6	1.49	32.48	67.52	56.52	1.84	1493
3	6.40 x1.6	1.44	30.23	69.77	56.45	1.88	1458
4	6.38 x1.6	1.46	31.05	68.95	56.56	1.96	1502
5	6.47 x1.6	1.45	31.38	68.62	55.90	2.38	1491
Average		1.46	31.40	68.59	56.26	1.99	1477

Table: 5 NOL Tensile strength at average density of 1.49

Sl .No	Dimension in (mm)	Density (g/cc)	Resin content (% by weight)	Fiber content (% by weight)	Fiber volume fraction (Vf)	Void (%)	NOL Ring Tensile Strength (MPa)
1	6.39x1.4	1.49	30.63	69.37	56.51	1.78	1620
2	6.32 x1.4	1.48	31.66	68.34	56.83	1.83	1763
3	6.40 x1.4	1.48	31.71	68.29	56.02	1.67	1720
4	6.38 x1.4	1.49	28.93	71.07	55.90	1.72	1741
5	6.42 x1.4	1.49	30.32	69.68	55.98	1.79	1657
Average		1.49	30.65	69.35	56.24	1.75	1700

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Table: 6 NOL Tensile strength at average density of 1.52

Sl .No	Dimension in (mm)	Density (g/cc)	Resin content (% by weight)	Fiber Content (% by weight)	Fiber volume fraction (Vf)	Void (%)	NOL Ring Tensile Strength (MPa)
1	6.40x1.3	1.54	25.34	74.66	64.60	0.83	1950
2	6.39 x1.3	1.49	23.74	76.26	63.84	0.79	1973
3	6.45 x1.3	1.53	25.49	74.51	64.05	0.85	1957
4	6.37 x1.3	1.52	22.75	77.25	65.97	0.72	1946
5	6.50 x1.3	1.56	25.69	74.31	65.13	0.75	1891
Average		1.52	24.60	75.39	64.71	0.78	1943

Table: 7 Effect of resin mix viscosity on Tensile Strength

Sl. No.	Resin mix	Viscosity (cP)	Fiber Volume fraction (Vf)	Voids (%)	NOL Ring Tensile Strength (MPa)
1	LY556+HY5200	6000 at 30°C	58.20	1.92	1520
2	LY556+HY5200	1000 at 50°C	65.13	0.75	1950

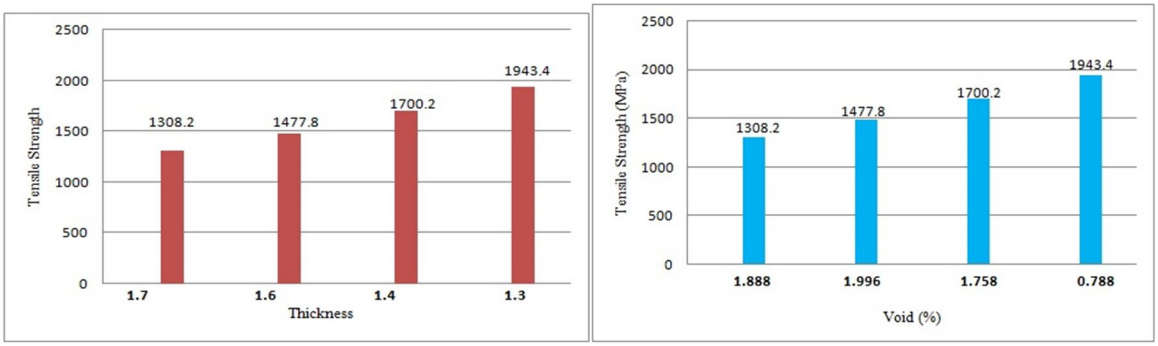


Fig.8(a): Tensile strength Vs Voids (b) Tensile strength Vs Thickness

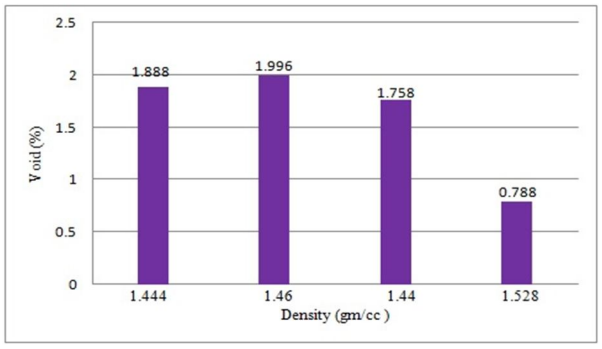


Fig.9: Voids Vs Density

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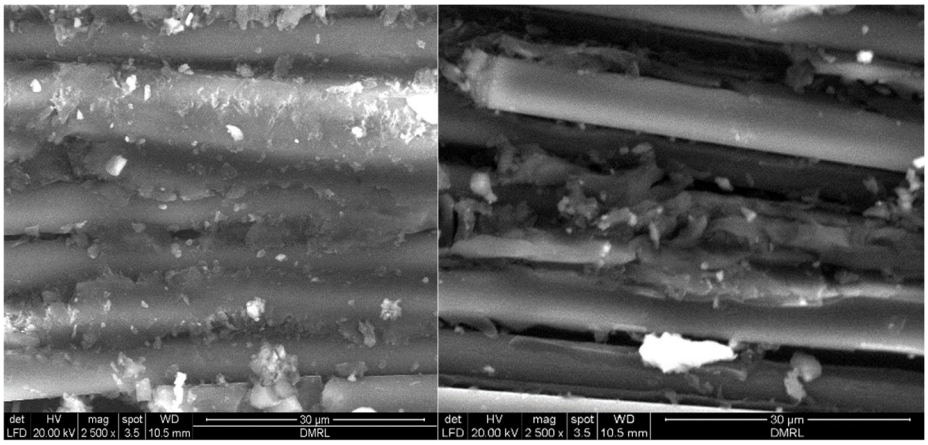


Fig.10: SEM images of fiber wettability with low viscous and high viscous resin mix

The composite rings with different densities and porosities were made with varying process parameters during filament winding. Voids are closely related to composite structural property. The results indicate that voids content and density of the composite is process dependent. Lower density resulted in lower tensile strength due to poor consolidation leading to premature failure in the composite. Good consolidation in composite with higher density and fiber volume fraction resulted higher tensile strength and full potential of fiber strength is realized.

Matrix mix viscosity plays a vital role in wetting of fibers and also void/porosity formation in the composite. Lower optimum viscosity of resin mix have better penetration and wetting of fibers. A resin mix with high viscosity will produce voids in a composite. It is difficult for a matrix with high viscosity to penetrate the original spaces between adjacent fibers. Matrix mix viscosity on wettability of fibers is evident from SEM pictures. Lower mix viscosity not only improved the wetting of fibers but also fiber volume fraction which is directly proportional to tensile strength of the composite. On the other hand, due to high mix viscosity of resin system, resulted in non-homogenous wettability of carbon fibers observed in SEM image. Therefore, unlike the static process of unidirectional composites, a matrix system with low viscosity and good wettability to carbon fibers is more necessarily required for the dynamic filament winding process.

V. CONCLUSION

A good quality product is one which is free from voids / defects. Process parameters influences the quality of the composite. Voids are inevitable in the fabrication of fiber reinforced composites and have detrimental effect on mechanical properties of composites. This study, revealed several CFRP specimens with wide ranges of voids and densities prepared by varying process parameters. Voids in composite specimens were identified by NDT technique and void content in composite was quantified by chemical analysis. It is observed from the test data better compaction lowered void content and resulted in high tensile strength. High percentage of porosity in NOL ring specimen led to premature failure in composite and resulted low tensile strength. Composite density calculated theoretically may not always same with experimentally determine density. This is due to voids present in the laminate. The differences in densities indicate the void content. Composite for aerospace structural application should have less than 1% voids. Matrix viscosity had profound effect on void formation and strength of the composite. A matrix system with low viscosity and good wettability to carbon fibers is more necessarily required for the dynamic filament winding process.

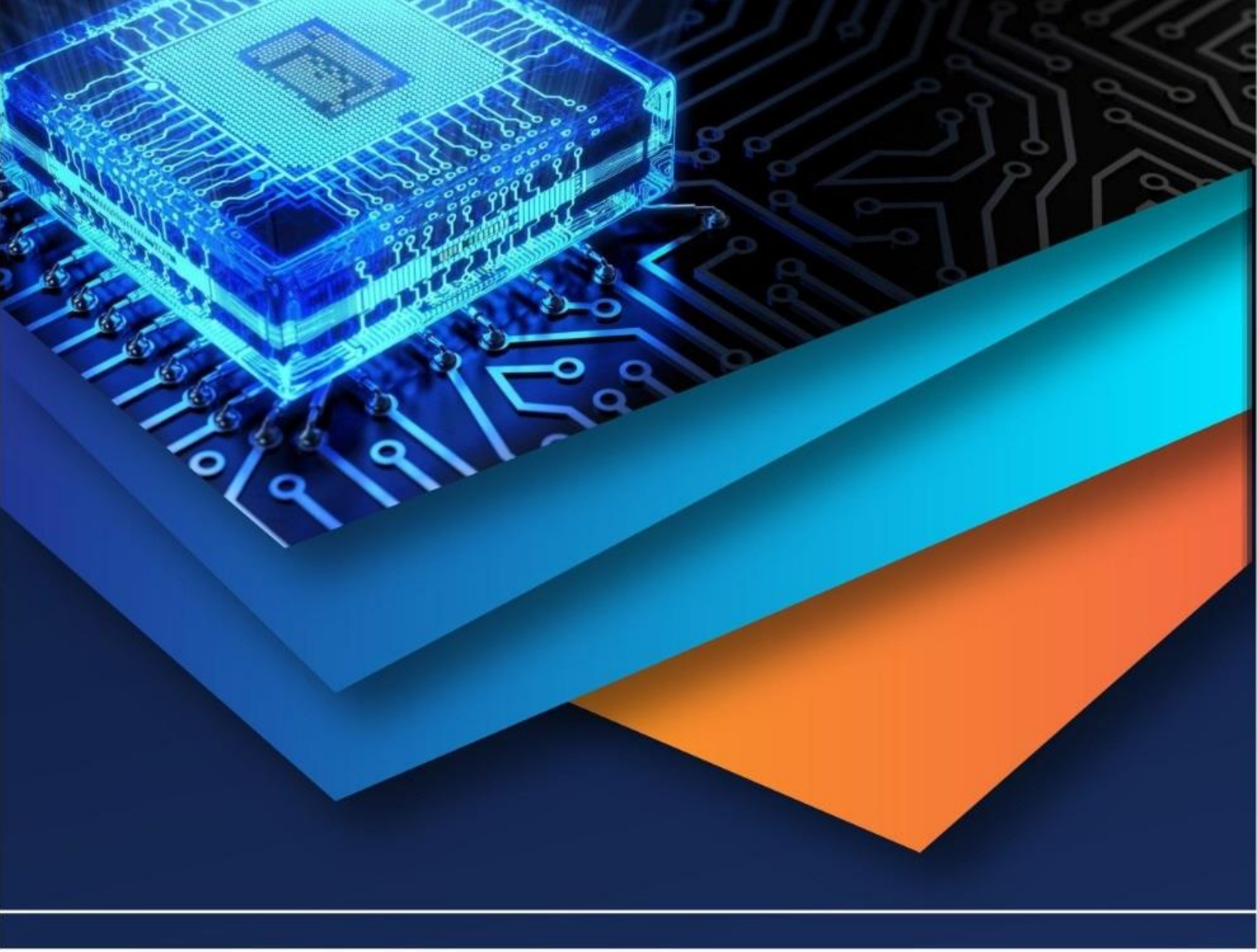
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