International Journal of Engineering and Advanced Technology (IJEAT)
ISSN: 2249-8958 (Online), Volume-8 Issue-6, August, 2019

Characterization of PAN based Carbon Tow for Structural Composites

G. Rama Rao, Ajmeera Ramesh, S. Kannan, P. Chandrasekhar

Abstract: ***Fibers are load carrying component in a fiber reinforced polymer composite materials. Tensile properties of composite are affected by the properties of the fiber. Polyacrylonitrile (PAN) based carbon fibers are widely used as a reinforcement in composite materials because of its high tensile strength and tensile modulus. Stringent characterisation of PAN based carbon tow is paramount for the fabrication of high performance advanced composite products. This paper presents characterization of PAN based carbon tow for structural composites. The physical and chemical parameters of carbon tow namely tex, density, diameter of single fiber, sizing content and carbon content were characterized for carbon fiber. Tensile properties of PAN based carbon tow can be determined by mono filament test and liquid resin impregnated tow tensile test. In the present study, tensile strength and modulus of PAN based fiber was evaluated by mono filament test. It was observed that tensile failure load was varying from filament to filament for same lot of fiber in mono filament test. High scattering in tensile strength values of filaments was observed due to statically distributed surface flaws on the filaments. Coefficient of variance for tensile strength of filament by mono filament tensile test was varied from 12-25 % from one lot to other lot of carbon tow. Liquid resin impregnated tow tensile test was conducted with specified resin system with low temperature cure cycle, to get more consistent and more accurate values of fiber tensile strength, modulus and % strain. Carbon tow was characterized by laminate level mechanical properties namely NOL ring hoop tensile strength and ILSS with high temperature curing epoxy resin system, which is used for fabrication of actual structural composite product.***

Index Terms: ***Carbon Tow, Mono filament test, Liquid resin impregnated tow test, NOL ring hoop tensile strength, ILSS***

I. INTRODUCTION

Polyacrylonitrile (PAN) based carbon tow is widely used as reinforcement in composite materials because of its high tensile strength and tensile modulus. Stringent characterisation of PAN based carbon tow is paramount for the fabrication of high performance advanced composite products. The mechanical properties of carbon fibres vary according to the precursor material and heat treatment conditions. PAN based carbon tow shows high strength, high modulus and low density (1.70-1.85 g/cm^3). Extensive work has been carried out for evaluation of mechanical properties of PAN based carbon tow. Carbon fibres were surface treated and applied during their production to improve handling and to promote fiber-resin bonding. The amount of sizing and its compositional consistency are significant in quality control of the fiber and measurement of these parameters is part of the fiber evaluation [1-7].

Tensile properties of PAN based carbon tow can be determined by mono filament test [8] and liquid resin impregnated tow tensile test [9]. Liquid resin impregnated tow tensile test was conducted with specified resin system with low temperature cure cycle, to get more consistent and more accurate values of fiber tensile strength and modulus. Resin system used in impregnation tow test was different from the resin system used in the fabrication of actual composite product. Resin system used in liquid resin impregnated tow test has low resin viscosity, higher percentage of elongation, low temperature curing, low glass transition temperature and has low residual stress. Whereas resin system used in the fabrication of composite product has high resin viscosity, higher percentage of elongation, high temperature curing, high glass transition temperature and has high residual stress. The liquid resin which has low residual stress is able to transfer maximum percentage of fiber tensile strength in liquid resin impregnated tow tensile strength than that of resin system has higher residual stress. However, higher temperature curing resin system is used for fabrication of composite product, which requires high glass transition temperature for its operational usage in aerospace applications. Composite properties namely Tensile strength and tensile modulus of unidirectional laminate [10], NOL ring hoop tensile strength [11] and ILSS [12] of PAN based carbon tows were evaluated with high temperature curing epoxy resin system, which is used for fabrication of actual structural composite product. NOL ring test gives apparent hoop tensile strength which is of direct relevance to pressure vessel application. NOL ring simulates fiber bending effect in cylindrical component made by filament winding technique. NOL ring with split disk test fixture gives conservative value of tensile strength (σ_{11}) among different methods. ILSS test gives interface bonding between carbon fiber and resin system. These tests are used as for quality control parameters for acceptance of PAN based carbon tow [13,14].

This paper describes test methods used for characterisation of PAN based carbon tow, which is used in the development of high performance of structural composites in aerospace application.

Revised Manuscript Received on October 30, 2019.

* Correspondence Author

G. Rama Rao*, Directorate of Composite Product Development Centre, Advanced Systems Laboratory, DRDO, Kanchanbagh, Hyderabad-*58.*

Ajmeera Ramesh, CMR Technical Campus, Hyderabad

S. Kannan, Directorate of Composite Product Development Centre, Advanced Systems Laboratory, DRDO, Kanchanbagh, Hyderabad-*58.*

P. Chandrasekhar, Directorate of Composite Product Development Centre, Advanced Systems Laboratory, DRDO, Kanchanbagh, Hyderabad-*58.*

© The Authors. Published by Blue Eyes Intelligence Engineering and Sciences Publication (BEIESP). This is an open access article under the CC BY-NC-ND license (http://creativecommons.org/licenses/by-nc-nd/4.0/)

II. MATERIALS

Carbon fibres used in this study were high strength and standard modulus PAN-based Carbon tow with epoxy compatible sizing. PAN based carbon fibre tow is shown in the figure no.1

Fig.No.1: PAN based Carbon Tow

III. EXPERIMENTAL WORK

The following tests were carried out for characterisation of the chemical, physical, and mechanical properties of PAN based carbon tow for structural composites.

A. Chemical properties of carbon tow

a) Carbon content

Typically, standard and intermediate modulus PAN based carbon tow has 93-94% by weight carbon content, with remaining being nitrogen. Carbon content of carbon fiber was measured by instrumental method of analysis using CHNSO Elemental analyzer instrument. The instrument was calibrated with analytical grade of Biphenyl to match with the carbon content in carbon tow.

b) Sizing content

The sizing content on the carbon tow is expressed as a percentage of the dry sized fiber weight. Sizing content on carbon tow was measured as per procedure given in the ASTM D4018. Evaluation of Sizing content on carbon tow is based on pyrolysis technique at 450°C for 15 minutes in inert atmosphere. The Sizing content was also determined by Thermogravimetric analysis (TGA) in inert atmosphere.

B. Physical properties of carbon tow

a) Tex

Tex of the roving is expressed as weight in gram per km length of roving. It is also expressed as linear density of roving which is weight per unit length. The Tex of the roving was determined by weighing 1000 m length of the roving in a calibrated microelectronic weighing balance with an accuracy of 0.1 mg.

b) Density

The density of carbon fiber was measured as per ASTM D3800 using Trichloroethylene solvent.

c) Fibre diameter

The average diameter of fibres in carbon tow was estimated by using tex, density and number of filaments in the tow. Scanning electron microscopy (SEM was usee for measuring fiber diameter and cross-sectional characteristics. SEM is also useful technique for determining morphological characteristics (deposits, pits) of fiber surfaces.

C. Mechanical properties of carbon tow

a) Tensile strength, Tensile modulus and % strain

Tensile strength, Tensile Modulus and % strain of carbon fiber were determined by Monofilamnet Tensile test, Liquid resin impregnated tow tensile test

b) Mono filament tensile test

Mono filament tensile properties were determined as per ISO 11566. The monofilament specimen is show in the figure No.2.

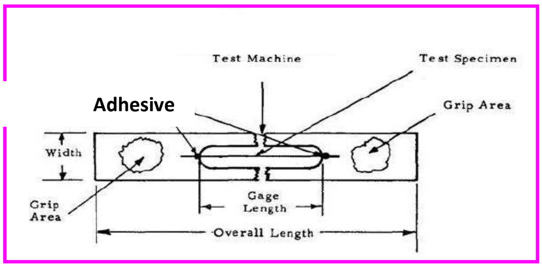

Fig.No.2: Mono Filament Tensile Test Specimen

The strain is measured by using cross head displacement, gauge length and assuming system compliance was negligible.

c) Liquid resin impregnated tow tensile test

Liquid resin impregnated tow tensile properties were determined as per ASTM D 4018. The impregnated tow properties depend on the choice of resin, therefore to minimize the effects of the impregnating resin on the tensile properties, the resin should be properly selected such that the impregnated tow failure is initiated by fibers and the impregnated tow is able to realize the full potential of carbon tow. Therefore, during qualification of incoming PAN based carbon tow, the impregnated tow test should always be conducted using a specified resin system with specified cure cycle. Low temperature curing Epoxy resin system used for impregnation of carbon tow. The resin impregnated tow specimens are show in the figure No.3.

Fig: 3(a)

Retrieval Number F8471088619/2019©BEIESP
DOI: 10.35940/ijeat.F8471.088619
Journal Website: www.ijeat.org

Published By:
Blue Eyes Intelligence Engineering
& Sciences Publication

Fig: 3(b)

Figure No.3 (a) & (b): Liquid Resin Impregnated Tow specimens winding on the mould and Glass fabric / epoxy tabs bonded on specimens (from left to right order).

Glass fabric /epoxy tabs were bonded on the specimens to get acceptable failure as per ASTM D 4018. Extensometer was attached to impregnated tow specimen at the middle of gauge length to measure strain of specimen for calculation of tensile modulus. Ultimate strain (%) was calculated as ratio of ultimate tensile strength to tensile modulus.

d) Laminate level mechanical tests

\Mechanical properties of cured laminates depend on epoxy resin and fiber volume fraction of the laminate. Composite properties of incoming PAN based carbon tow were evaluated with high temperature curing epoxy resin system, which is used for fabrication of actual structural composite product.

Hoop Tensile Strength of cured laminate was carried out as per ASTM D 2290 using four split disks with self–aligning test fixture to minimize the effect bending.

Tensile strength and tensile modulus of unidirectional laminate was carried out as per ASTM D 3039. Inter laminar shear strength was evaluated using a three-point test fixture with short beam specimens. The test was carried out as per ASTM D2344. The material direction under investigation must be oriented along the length dimension of the specimen. The test pieces require a span/depth (l/d) ratio low enough to minimize the influence of bending deformation and to achieve failure in shear rather than in bending. Laminate level mechanical test specimens were shown in the figure no.4.

Fig: 4(a)

Fig: 4(b)

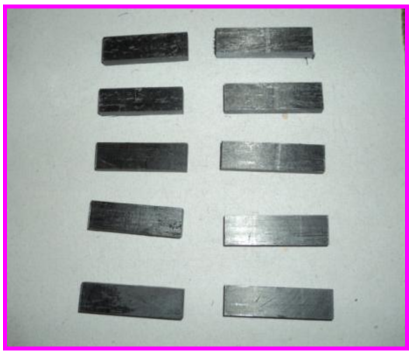

Fig: 4(c)

Figure No.4 (a), (b) & (c): Laminate Level Mechanical Test Specimens of NOL Ring, UD Tensile, and ILSS (from left to right in that order)

IV. RESULTS AND DISCUSSIONS

A. Chemical properties of carbon tow

The carbon content in standard modulus of PAN based carbon tow was 93 % by weight, Nitrogen content was 6 % by weight as measured by CHNS Elemental analyser. The amount of sizing in carbon tow was found 0.8% by weight.

B. Physical properties of carbon tow

The density of standard modulus of PAN based carbon tow was about 1.78 g/cc. Diameter of filament was measured by Scanning Electron Microscope (SEM). The diameter of filaments was varying from 6.197 to 6.580microns. Surface of carbon fiber was studied by Scanning Electron Microscope and found that no major surface damage & discontinuities of the filaments. The SEM scan of carbon fibre was shown in the figure No.5.

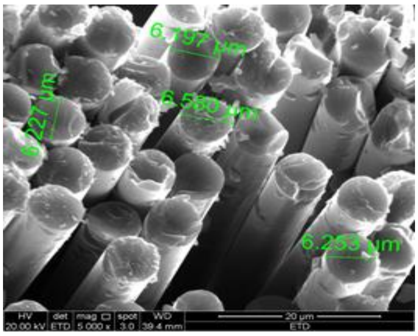

Figure No. 5: The SEM scan of carbon fibre

C. Mechanical Properties of carbon tow

Monofilament tensile test is the only test that gives accurate assessment of fiber strength and modulus. Stastical data for tensile strength and modulus of carbon tows by monofilament test was evaluated for different lots of carbon tow. It was observed that the tensile failure load was varying from filament to filament for same lot of carbon tow and hence it resulted in high scattering in monofilament tensile strength values. Coefficient of variance varied from 12-25 % from one lot to another lot of carbon tow for tensile strength of filament by mono filament tensile test.

Liquid resin impregnated tow tensile test provides reasonably representative test for average value of filament tensile strength in a roving or tow (Tow quality like number of breakages/ load Sharing can be captured). Liquid resin impregnate tow tensile specimens were prepared with low viscosity resin system and low temperature curing cycle. Impregnated resin has higher % elongation than fiber in order to transfer maximum fiber strength in to the tow. Resin content was measured in tow tensile specimens and it was found that about 40-50 % by weight of resin is present in impregnated tows. Tow specimens failed in lateral failure in gauge length. It was observed that about 100 % fibre strength was transferred in liquid resin impregnated tow specimens. Tow tensile test, specimen's failure mode and stress vs strain curve were shown in the figure no.6

Fig: 6(a)

Fig: 6(b)

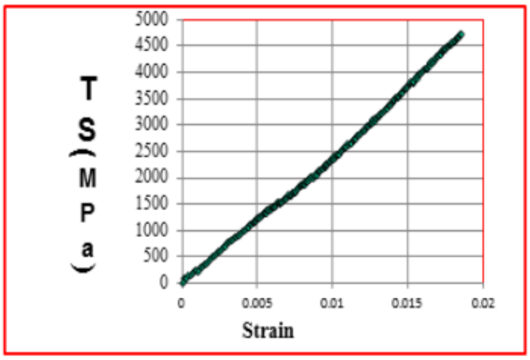

Fig: 6(c)

Figure No.6 (a), (b) & (c): Liquid Resin impregnated Tow Tensile Test in UTM with extensometer, Failure modes of tow specimens & Stress vs strain plot for tow tensile test (from left to right in that order).

Composite properties namely NOL ring hoop tensile strength, UD tensile strength and tensile modulus and ILSS of incoming PAN based carbon tow were evaluated with high temperature curing epoxy resin system, which is used for fabrication of actual structural composite product. NOL ring specimens failed in hoop failure mode and delamination failure mode. It was observed that about 40 % fibre strength was transferred in NOL ring composite at fiber volume fraction of 60 % with high temperature curing epoxy resin system. Failure modes of NOL ring specimens and stress vs strain curve for NOL ring hoop tensile strength were shown in the figure no.7

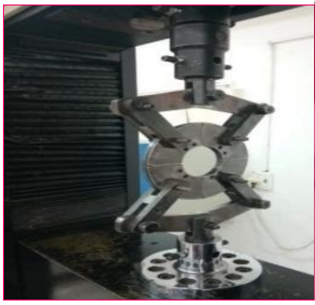

Fig: 7(a)

Fig: 7(b)

Fig: 7(c)

Figure No.7 (a), (b) & (c): NOL ring hoop tensile strength. Failure modes of specimens & Stress vs Strain plot for NOL ring test (from left to right in that order)

UD laminate tensile testing was carried out as per ASTM D3039. Tensile specimens were bonded with glass fabric/ epoxy end tabs. Rosette Strain gauge was bonded on longitudinal tensile specimens as per standard procedure of strain gauge bonding for measurement of strain of composite. It was observed that about 44 % fiber strength was transferred in UD composite at fiber volume fraction of 60 % with high temperature curing epoxy resin system. Specimens failed in explosive failure mode. ILSS value for carbon fibre/Epoxy resin UD laminate was about 50 -60 MPa. Test results of different lots of carbon tow were shown in the table No.1.

Table No.1 Test Results of different lots of PAN based carbon tow

Lot No	Mono Filament Tensile Test				Resin Impregnated Tow Tensile Test		NOL ring Hoop Tensile Strength (MPa)	ILSS (MPa)
	TS *(MPa)		TM *(GPa)		TS (MPa)	TM (GPa)		
	Avg. Value	% CoV*	Avg. Value	% CoV				
I	**4385**	**24.85**	**200**	**6.50**	5.10	226	2186	62
II	**4290**	**16.18**	**213**	**5.20**	4.98	220	2003	58
III	**4312**	**12.52**	**207**	**7.80**	5.05	228	2100	60
1V	**4059**	**18.37**	**209**	**6.75**	4.90	230	1923	56

TS* = Tensile Strength, TM*= Tensile Modulus and COV* = Coefficient of Variance

V.CONCLUSIONS

Stringent characterisation was carried out for use of PAN based carbon tow in fabrication of structural composite products for aerospace applications.

REFERENCES

1. E. Fitzer, "PAN-based carbon fibers-Present state and trend of the technology from the viewpoint of possibilities and limits to influence and to control the fiber properties by the process parameters," Carbon, 27[5] 621-45 (1989).
2. Torayca carbon fibre technical information, Toray Industries, Inc. 2005.
3. Hexcel carbon fibre technical information 2010.
4. S.Chand, "Review-Carbon fibers for composites," J. Mater. Sci.,35[6] 1303-13 (2000).
5. L. G. Rosa, A. Colella and C. A. Anjinho, "Effect of paraffin oil used as lubricant in tensile tests of carbon fibre bundles," Mater. Sci. Forum 514-51672-6 (2006).
6. S. Kumar and Y. Wang, "Fibers, fabrics, and fillers"; pp. 51-100 in Composites engineering handbook, Dekker, New York, 1997.
7. M. Guigon and A. Oberlin, "Preliminary studies of mesophase-pitch-based carbon fibres: Structure and microtexture," Compos. Sci. Technol.,25[3] 231-41 (1986).
8. ISO 11566: Carbon fibre — Determination of the tensile properties of single-filament specimens.
9. ASTM D 4018: Standard test methods for properties of Continuous filament carbon and Graphite fiber tow.
10. ASTM D 3039/ 3039 M: Standard Test Method for Tensile Properties of Polymer Matrix Composite Materials.
11. ASTM D 2290: Standard Test Method for Apparent Hoop Tensile Strength of Plastic or Reinforced Plastic Pipe by Split Disk Method.
12. ASTM D 2344 /D2344 M: Standard Test Method for Short-Beam Strength of Polymer Matrix Composite Materials and Their Laminates.
13. Composite materials Hand book, CMH-17-1G, Volume 1, Sage International publications.
14. S. Biswas, Micromechanics based damage mesomodel for unidirectional fibrous Laminated composite, Masters Thesis, Department of Aerospace Engineering, Indian Institute of Technology Kanpur, India, 2011.

AUTHORS PROFILE

Shri G. Rama Rao is working as Scientist in Advanced Systems Laboratory, DRDO, Ministry of Defence, Government of India, Hyderabad. He is working in the field of fiber reinforced polymer composites. His areas of interest are characterization of carbon fiber for structural composites, characterization of composite materials for generation of design input data and development of resin systems for composite products for aerospace applications.

Shri Ajmeera Ramesh, Assistant professor in Mechanical Engineering Department, His Qualification is M. Tech, (Ph. D). He has seven years of teaching experience. His Area of Specialization is light weight advanced composite materials.

Shri S. Kannan is working as Technical Officer in Advanced Systems Laboratory, DRDO, Ministry of Defence, Government of India, Hyderabad. He is working in the field of fiber reinforced polymer composites. His areas of interest are characterization of resins, fibers and composite materials for physical, mechanical and thermal properties

Shri Chandrasekhar is working as Senior Technical Officer in Advanced Systems Laboratory, DRDO, Ministry of Defence, Government of India, Hyderabad. He is working in the field of fiber reinforced polymer composites. His areas of interest are characterization of resins, fibers and composite materials for physical, mechanical and thermal properties.