

**STUDIES ON CARBON/POLYPROPYLENE COMPOSITES MADE
THROUGH ELECTROSTATIC POWDER COATING AND OTHER
METHODS**

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METHODS**

by

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Submitted

In fulfillment of requirements for the awards of the degree of Doctor of Philosophy

to the



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Dedicated Gratefully To

To My Gurus,

My Adored Grandmother, Parents, Sister, Extended Family

&

Friends

CERTIFICATE

This is to certify that the thesis entitled “**Studies on Carbon/Polypropylene Composites made through electrostatic powder coating and other methods**” submitted by **Mr. Vijay Anil Goud** to the **Indian Institute of Technology Delhi** for the award of the degree of **Doctor of Philosophy** in the **Department of Textile Technology**, is a record of bonafide research work carried out by him. Mr. Vijay Anil Goud has worked under our guidance and supervision.

The results contained in this thesis are original and have not been submitted in partial or full, to any other university or institute for the award of any degree or diploma.

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ABSTRACT

Though the processing steps for the manufacture of thermoplastic composites are simpler and faster when compared to those for thermoset composites, there are difficulties due to high melt viscosity of thermoplastic resins in accomplishing thorough infusion of resin into fibers and fibrous structures. Tremendously high viscosity for thermoplastic resin leads to numerous problems in the manufacturing process of thermoplastic composites. Impregnation processes such as melt impregnation or solution impregnation, tend to form a stiff towpreg that cannot be used for any textile preforming operation. Powder coating can be a cost effective as well as efficient processing technology to produce a flexible towpreg. A method for production of flexible towpregs for thorough infusion of resin into fibers using electrostatic spray gun, which involves powder coating a bundle of filaments is designed and developed.

The effects of process variables of the electrostatic spray process such as corona voltage, fluidizing air pressure, conveying air pressure, distance of gun from tow and tow velocity on maximum powder deposition are investigated and reported for carbon/polypropylene towpreg. The univariate studies are followed by multivariate studies in order to see the interaction effect of process parameters on the fiber volume fraction. Experimental designs including central composite design and three level designs such as Box Behnken and full factorial designs are quite popularly used. Box and Behnken based design of experiments is a proven technique to optimize the output response with minimal number of experiments. Box Behnken design allows interpretation of quadratic models, building of sequential designs, identification of lack of fit and use of blocks. Box Behnken design is reported to be more efficient design than central composite design and three level full factorial design, where efficiency is defined as number of coefficients in estimated model divided by number of experiments. Moreover, the prediction of mathematical model along with the interaction effect between the input parameters can help in obtaining accurate results when

compared to that of univariate studies. The univariate studies will fail, if the interaction effect is larger as it does not consider the dependency of one parameter to that of the other. Thus, the electrostatic spray coating process was optimized for fiber volume fraction using Box-Behnken experimental design. An empirical multi-parametric equation was obtained to define the relationship between the process parameters (electrostatic voltage at the gun, the distance between the tip of gun and towpreg and velocity of the carbon towpreg) through response surface methodology. Experimental verification of the model showed a validation of ~93%. The velocity of the carbon towpreg was found to have a more profound effect on the output response of fiber volume fraction when compared to other process parameters.

On optimization of the powder coating process in order to obtain towpregs with different fiber volume fraction, a fiber volume fraction of 50% was chosen and the towpregs were accordingly produced for future studies. The powder coating process was then compared with other methods of DREF spinning which produces similar core-sheath structure to that of powder coating and film stacking in which layers of films were stacked between different layers of unidirectionally wound carbon tow. An investigation into the flexural properties of unidirectional composites (UDC) produced from blending of carbon fiber and polypropylene (PP) matrix in varying forms of fiber, powder and film are reported. Dr. Ernst Fehrer (DREF) friction spun hybrid yarns are manufactured from PP fibers and carbon tows and are consolidated to yield UDC-D. PP powder is layered on carbon tows by electrostatic spray coating and are consolidated to form UDC-P. A third laminate, UDC-F is manufactured by the consolidation of PP films stacked between carbon tows. The fiber volume fraction is ~ 50 % in all the three UDCs. Better mechanical properties of UDC-P are observed in 3-point bending and short beam tests. Analysis by micro-computed tomography and scanning electron microscopy indicate better impregnation of PP powder in UDC-P despite higher viscosity of

PP powder than PP fiber, and thereby higher interlaminar strength. This is the first work reported on comparison of laminates from powder coating versus DREF and film stacking.

Further, the literature available on comparison of 2D composites to that of 3D composites using thermoplastic matrices are limited. Moreover, literature on comparison of 2D and 3D fabric composites produced by the process of electrostatic spray coating and DREF spinning among and between themselves is inadequate. Thus, for the aforesaid reason to surpass the poor impregnation of highly viscous thermoplastics, pre-forming techniques such as Dr. Ernst Fehrer (DREF) spinning and electrostatic spray coating are used. The pre-formed yarns are woven into 2D and 3D fabrics (angle inter-lock and orthogonal weave), which are then consolidated to yield 2D and 3D composites. In the tensile and flexural tests, 2D composites are observed to be better than the 3D composites owing to its weave pattern that deflects the line of material separation. The superior notch impact properties of 3D composites are attributed to the presence of the binder warp ends that hold the filler and stuffer warp ends. Further, the orthogonal structure's closer wrapping improves the shock absorption capability of the composite than the angle interlock composite. From micro-CT scans, porosity is a common feature of DREF spun composites. Also, composites made from powder coated towpregs performed better irrespective of the weave structure.

सार

यद्यपि थर्मोप्लास्टिक कंपोजिट के निर्माण के लिए प्रसंस्करण चरण सरल और तेज होते हैं, जबकि थर्मोसेट कंपोजिट के लिए तुलना में, फाइबर और रेशेदार संरचनाओं में राल के पूरी तरह से जलसेक को पूरा करने में थर्मोप्लास्टिक रेजिन की उच्च पिघल चिपचिपाहट के कारण कठिनाइयां होती हैं। थर्मोप्लास्टिक राल के लिए अत्यधिक उच्च चिपचिपापन थर्मोप्लास्टिक कंपोजिट की निर्माण प्रक्रिया में कई समस्याओं का कारण बनता है। पिघला हुआ संसेचन या समाधान संसेचन जैसे संसेचन प्रक्रियाएं, एक कठोर टोपरग का निर्माण करती हैं, जिसका उपयोग किसी भी कपड़ा सुधार कार्य के लिए नहीं किया जा सकता है। पाउडर कोटिंग एक लागत प्रभावी और साथ ही एक लचीले टॉपरग का उत्पादन करने के लिए कुशल प्रसंस्करण तकनीक हो सकती है। इलेक्ट्रोस्टैटिक स्प्रे बंदूक का उपयोग करके फाइबर में राल के पूरी तरह से जलने के लिए लचीले टॉपरगेट्स के उत्पादन के लिए एक विधि, जिसमें पाउडर कोटिंग शामिल है फिलामेंट्स का एक बंडल डिज़ाइन और विकसित किया गया है।

इलेक्ट्रोस्टैटिक स्प्रे प्रक्रिया के प्रक्रिया चर जैसे कि कोरोना वोल्टेज, वायु के दबाव को कम करने, हवा के दबाव को व्यक्त करने, अधिकतम पाउडर जमाव पर टो और टो वेग से बंदूक की दूरी के प्रभावों की जांच और कार्बन / पॉलीप्रोपाइलीन टॉवेलग के लिए रिपोर्ट की जाती है। फाइबर वॉल्यूम अंश पर प्रक्रिया मापदंडों के अंतःक्रियात्मक प्रभाव को देखने के लिए बहुभिन्नरूपी अध्ययनों के बाद अविभाजित अध्ययनों का पालन किया जाता है। केंद्रीय मिश्रित डिजाइन और तीन स्तर के डिजाइन जैसे कि बॉक्स बेहेनकेन और पूर्ण तथ्यात्मक डिजाइन सहित प्रायोगिक डिजाइन काफी लोकप्रिय हैं। प्रयोगों की न्यूनतम संख्या के साथ आउटपुट प्रतिक्रिया को अनुकूलित करने के लिए बॉक्स और प्रयोगों के बेकन आधारित डिजाइन एक सिद्ध तकनीक है। बॉक्स बेहेनकेन डिजाइन द्विघात मॉडल की व्याख्या, अनुक्रमिक डिजाइन का निर्माण, फिट की कमी की पहचान और ब्लॉकों के उपयोग की अनुमति देता है। बॉक्स बिहेनकेन डिजाइन को केंद्रीय समग्र डिजाइन और तीन स्तर पूर्ण फैक्टोरियल डिजाइन की तुलना में अधिक कुशल डिजाइन बताया गया है, जहाँ दक्षता को अनुमानित मॉडल में गुणांक की संख्या के रूप में परिभाषित किया गया है जो प्रयोगों की संख्या से विभाजित है। इसके अलावा, इनपुट मापदंडों के बीच बातचीत प्रभाव के साथ गणितीय मॉडल की भविष्यवाणी एकतरफा अध्ययनों की तुलना में सटीक परिणाम प्राप्त करने में मदद कर सकती है। अविभाज्य अध्ययन विफल हो जाएगा, अगर बातचीत प्रभाव बड़ा है, क्योंकि यह एक पैरामीटर की निर्भरता को दूसरे के बारे में नहीं मानता है। इस प्रकार, इलेक्ट्रोस्टैटिक स्प्रे कोटिंग प्रक्रिया को बॉक्स-बिहेनकेन प्रयोगात्मक डिजाइन का उपयोग करके फाइबर वॉल्यूम अंश के लिए अनुकूलित किया गया था। प्रतिक्रिया सतह कार्यप्रणाली के माध्यम से प्रक्रिया मापदंडों (बंदूक पर इलेक्ट्रोस्टैटिक वोल्टेज, बंदूक की नोक के बीच की दूरी और टॉपिंग और कार्बन टॉपरेज के वेग) के बीच संबंधों को परिभाषित करने के लिए एक अनुभवजन्य बहु-पैरामीट्रिक समीकरण प्राप्त किया गया था। मॉडल के प्रायोगिक सत्यापन ने ~ 93% का सत्यापन दिखाया। कार्बन टोपरग के वेग को अन्य प्रक्रिया मापदंडों की तुलना में फाइबर वॉल्यूम अंश की आउटपुट प्रतिक्रिया पर अधिक गहरा प्रभाव पाया गया।

विभिन्न फाइबर मात्रा अंश के साथ टॉपरगेट्स प्राप्त करने के लिए पाउडर कोटिंग प्रक्रिया के अनुकूलन पर, 50% का फाइबर वॉल्यूम अंश चुना गया था और टॉपरगल्स तदनुसार भविष्य के अध्ययन के लिए उत्पादित किए गए थे। पाउडर कोटिंग प्रक्रिया की तुलना DREF कटाई के अन्य तरीकों से की गई थी जो पाउडर कोटिंग और फिल्म स्टैकिंग के समान कोर-म्यान संरचना का उत्पादन करती है जिसमें फिल्मों की परतों को यूनिडायरेक्ली रूप से घाव कार्बन टो की विभिन्न परतों के बीच स्टैक किया गया था। फाइबर, पाउडर और फिल्म के विभिन्न रूपों में कार्बन फाइबर और पॉलीप्रोपाइलीन (पीपी) मैट्रिक्स के सम्मिश्रण से उत्पादित यूनिडायरेक्शनल कंपोजिट (यूडीसी) के फ्लेक्सुरल गुणों की जांच रिपोर्ट की जाती है। डॉ। अर्नस्ट फेहरर (डीआरईएफ) घर्षण स्पून हाइब्रिड यार्न पीपी

फाइबर और कार्बन टो से निर्मित होते हैं और यूडीसी-डी उपज के लिए समेकित होते हैं। पीपी पाउडर को इलेक्ट्रोस्टैटिक स्प्रे कोटिंग द्वारा कार्बन टो पर स्तरित किया जाता है और यूडीसी-पी बनाने के लिए समेकित किया जाता है। एक तीसरा टुकड़े टुकड़े, UDC-F कार्बन टो के बीच खड़ी पीपी फिल्मों के समेकन द्वारा निर्मित है। तीनों यूडीसी में फाइबर वॉल्यूम अंश ~ 50% है। यूडीसी-पी के बेहतर यांत्रिक गुणों को 3-बिंदु झुकने और लघु बीम परीक्षणों में मनाया जाता है। माइक्रो-कंप्यूटेड टोमोग्राफी और स्कैनिंग इलेक्ट्रॉन माइक्रोस्कोपी द्वारा विश्लेषण, पीपी फाइबर की तुलना में पीपी पाउडर की उच्च चिपचिपाहट के बावजूद पीपीडी पाउडर के बेहतर संसेचन का संकेत देता है, और इस तरह उच्च इंटरलामिनर ताकत। पाउडर कोटिंग बनाम डीआरईएफ और फिल्म स्टैकिंग से टुकड़े टुकड़े की तुलना में यह पहला काम है।

इसके अलावा, थर्मोप्लास्टिक मेट्रिसेस का उपयोग करते हुए 3 डी कंपोजिट की तुलना में 2 डी कंपोजिट की तुलना में उपलब्ध साहित्य सीमित हैं। इसके अलावा, इलेक्ट्रोस्टैटिक स्प्रे कोटिंग और DREF के बीच और खुद के बीच कटाई की प्रक्रिया द्वारा उत्पादित 2D और 3D कपड़े कंपोजिट की तुलना में साहित्य अपर्याप्त है। इस प्रकार, अत्यधिक चिपचिपा थर्मोप्लास्टिक के खराब संसेचन को पार करने के लिए पूर्वोक्त कारण के लिए, डॉ। अन्स्ट फेहरर (डीआरईएफ) कटाई और इलेक्ट्रोस्टैटिक स्प्रे जैसी पूर्व-निर्मित तकनीकें लेप का उपयोग किया जाता है। पहले से बने यार्न को 2 डी और 3 डी कपड़ों (कोण इंटर-लॉक और ऑर्थोगोनल बुनाई) में बुना जाता है, जो तब 2 डी और 3 डी कंपोजिट के लिए समेकित होते हैं। तन्यता और लचीले परीक्षणों में, 2 डी कंपोजिट को 3 डी कंपोजिट से बेहतर माना जाता है जो इसकी बुनाई पैटर्न के कारण होता है जो सामग्री पृथक्करण की रेखा को विक्षेपित करता है। 3 डी कंपोजिट के बेहतर पायदान प्रभाव गुणों को बाइंडर ताना छोरों की उपस्थिति के लिए जिम्मेदार ठहराया जाता है जो फिलर और स्टफर ताना समाप्त होता है। इसके अलावा, ऑर्थोगोनल संरचना की निकटता रैपिंग कोण इंटरलॉक समग्र की तुलना में मिश्रित की सदमे अवशोषण क्षमता में सुधार करती है। माइक्रो-सीटी स्कैन से, पोरसिटी डीआरईएफ स्पून कंपोजिट्स की एक सामान्य विशेषता है। इसके अलावा, पाउडर लेपित टॉपरगैस से बने कंपोजिट ने बुनाई की संरचना के बावजूद बेहतर प्रदर्शन किया।

Contents

Certificate	i
Acknowledgements	iii
Abstract	vii
Contents	xi
List of Figures	xvii
List of Tables	xxiii
Abbreviations	xxv
Chapter 1 Introduction and Objectives	
1.1 Introduction	3
1.2 Motivation	5
1.3 Objectives	6
1.4 Organization of thesis	7
Chapter 2 Literature Review	
2.1 Introduction	15
2.2 Composites	15
2.3 Constituents of thermoplastic composites	16
2.3.1 Reinforcement	16
2.3.2 Matrix	16
2.3.3 Embodiment of thermoplastic matrix and fiber	17
2.4 Single step impregnation techniques	19
2.4.1 Melt impregnation process	19
2.4.2 Solution coating	21
2.4.3 Surface polymerization	22
2.5 Multi step impregnation process	22
2.5.1 Ring spinning	22
2.5.2 Rotor spinning	24
2.5.3 DREF spinning	26

2.5.4	Film stacking	28
2.5.5	Wrap spinning	28
2.5.6	Parallel winding	30
2.5.7	Braiding	30
2.5.8	Air-jet texturing	31
2.5.9	Commingling	32
2.6	Powder coating technologies	34
2.6.1	Conventional powder bed	35
2.6.2	Conventional fluidized bed	36
2.6.3	Recirculating powder bed	37
2.6.4	Slurry coating	38
2.6.5	Acoustically fluidized bed	39
2.6.6	Electrostatic fluidized bed	41
2.6.7	Electrostatic spray coating process	44
2.7	Studies on comparison among different single and multi-step impregnation process	46
2.8	Electrostatic spray coating v/s powder coating technologies	50
2.9	Research Gaps	51
Chapter 3 Materials and Methods		
3.1	Introduction	55
3.2	Materials	55
3.3	Methods	56
3.3.1	Manufacturing of towpreg using electrostatic spray gun system	56
3.3.2	Manufacturing of hybrid yarn by DREF spinning	56
3.3.3	Manufacturing of Unidirectional and 2D, 3D woven composites	57
3.4	Material Characterization	59
3.4.1	Thermal analysis	59
3.4.2	Rheology – melt flow index and melt viscosity	60

3.4.3	Particle size distribution	60
3.5	Determination of composite properties	
3.5.1	Void Content	61
3.5.2	Tensile Properties	61
3.5.3	Flexural strength by three-point bend test	62
3.5.4	Interlaminar shear strength by short beam test	63
3.5.5	Notch impact properties	64
3.5.6	Analysis of samples using micro-computed tomography	65
3.5.7	Determination of flexural rigidity	
Chapter 4	Dry Electrostatic Spray Coated Towpregs for Thermoplastic Composites	
4.1	Introduction	69
4.2	Process flow chart for electrostatic spray coating	69
4.3	Fabrication aspects	70
4.3.1	Let-off zone and surface modification zone	71
4.3.2	Powder coating zone	72
4.3.2.1	Powder hopper	72
4.3.2.2	Venturi pump	73
4.3.2.3	Electrostatic spray gun	75
4.3.2.4	Powder coating booth	76
4.3.2.5	Powder recovery device	77
4.3.3	Sintering zone	78
4.3.4	Take up	79
4.4	Conclusions	82
Chapter 5	Process Parameters of Electrostatic Spray Coating Set-Up	
5.1	Introduction	87
5.2	Materials and Methods	89
5.2.1	Materials	89
5.2.2	Methods	89

5.3	Results and discussion (Univariate studies)	90
5.3.1	Effect of voltage on powder deposition	91
5.3.2	Effect of conveying air pressure on powder deposition	92
5.3.3	Effect of tow speed on powder deposition	93
5.3.4	Effect of gun distance on powder deposition	94
5.3.5	Effect of fluidization pressure on powder deposition	96
5.4	Multivariate studies	97
5.5	Results and discussion	100
5.6	Effect of heater temperature on flexural rigidity of towpreg	105
5.7	Conclusions	108
Chapter 6	Influence PP Matrix (Fiber, Powder And Film States) on Carbon-Polypropylene Composites	
6.1	Introduction	111
6.2	Materials and Methods	111
6.2.1	Materials	111
6.2.2	Methods	112
6.2.2.1	Manufacturing of hybrid yarn by DREF spinning	112
6.2.2.2	Manufacturing of towpreg using electrostatic spray gun system	113
6.2.2.3	Manufacturing of UDC-D, UDC-P and UDC-F	113
6.3	Material characterization	113
6.4	Determination of composite properties	113
6.5	Results and Discussion	114
6.5.1	Thermal analysis	114
6.5.2	Rheological properties	115
6.5.3	Particle size distribution of PP powder	116
6.5.4	Flexural strength by three-point bend tests	117
6.5.5	Interlaminar shear strength by short beam tests	118
6.5.6	Microstructural analysis	119
6.6	Conclusions	125

Chapter 7	Mechanical Properties of Carbon/Polypropylene 2D and 3D Woven Composites	
7.1	Introduction	129
7.2	Materials and Methods	131
7.2.1	Materials and characterization	131
7.2.2	DREF hybrid yarn used in 2DWC-D, 3DWAC-D and 3DWOC-D	132
7.2.3	Powder coated towpreg	133
7.2.4	Production of fabrics and composites	134
7.2.4.1	Production of fabrics	134
7.2.4.2	Production of composites	137
7.3	Determination of composite properties	137
7.3.1	Tensile properties	137
7.3.2	Flexural strength by three-point bend test	138
7.3.3	Notch impact properties	138
7.3.4	Determination of fiber volume fraction	138
7.3.5	Analysis of samples using Micro-Computed tomography	138
7.4	Results and Discussion	138
7.4.1	Tensile properties	138
7.4.2	Flexural properties	142
7.4.3	Notch impact properties	145
7.4.4	DREF based composites versus powder coated towpregs based composites	147
7.5	Conclusions	149
Chapter 8.0	Summary and conclusions	
8.1	Background	155
8.2	Summary and conclusion	156
8.2.1	Dry electrostatic spray coated towpregs for thermoplastic composites	156
8.2.2	Process parameters of electrostatic spray coating set-up	157

8.2.3	Influence of PP matrix (fiber, powder and film states) on carbon-polypropylene composites	158
8.2.4	Mechanical performance of carbon/polypropylene 2D and 3D woven composites	159
8.3	Future Scope of Work	160

References

Biodata

List of Figures

Caption	Name	Page No
Figure 2.1.	Hot melt impregnation Process	20
Figure 2.2.	Solution impregnation	21
Figure 2.3.	Ring spinning technology for production of hybrid yarns	23
Figure 2.4.	Rotor spinning process for production of hybrid yarn	25
Figure 2.5.	DREF III spinning process for production of hybrid yarn	27
Figure 2.6.	Film stacked laminate	28
Figure 2.7.	Wrap Spinning for production of hybrid yarn	29
Figure 2.8.	Parallel winding for production of hybrid yarn	30
Figure 2.9.	Process of braiding for production of hybrid yarn	31
Figure 2.10.	Air texturing process for production of hybrid yarn	32
Figure 2.11	Commingling process for Production of hybrid yarn	33
Figure 2.12	Conventional powder bed for production of towpreg	35
Figure 2.13	Recirculating fluidized bed for production of towpreg	38
Figure 2.14	Slurry coating for production of towpreg	39
Figure 2.15	Acoustically fluidized bed for production of towpreg	41
Figure 2.16	Electrostatic fluidized bed	42
Figure 2.17	Electrostatic spray coating for production of towpreg	45
Figure 3.1	Schematic diagram of hybrid yarn manufacturing through DREF III spinning.	57
Figure 3.2	(a) Wrapping of DREF hybrid yarns or towpregs around a metallic plate	59
	(b) placement of wrapped yarns or towpregs in the female mold cavity. Male mold shown in the right extreme	59
Figure 3.3	Tensile test using Universal Testing Machine	62
Figure 3.4	(a) Dimensions of notch specimen	65
	(b) schematic of the notch impact tester	65

Figure 4.1	Flowchart for electrostatic powder coating process	70
Figure 4.2	Illustration of electrostatic dry powder coating process	71
Figure 4.3	Let-off device, surface modification bath and pneumatic spreader	72
Figure 4.4	Diagram of powder feed hopper (Courtesy Dipole powder Coating)	73
Figure 4.5	Powder venturi pump (Courtesy Dipole powder Coating)	74
Figure 4.6	Electrostatic spray gun with cascade generator (Courtesy Dipole powder coating)	75
Figure 4.7	Powder coating booth	77
Figure 4.8	Hot air oven	79
Figure 4.9	Take up device	79
Figure 4.10	Developed powder coating set up	81
Figure 4.11	SEM image for coated and uncoated carbon tow	82
Figure 5.1	Electrostatic Voltage v/s Powder deposition	91
Figure 5.2	Powder deposition v/s conveying air pressure	93
Figure 5.3	Powder deposition v/s fiber velocity	94
Figure 5.4	Powder deposition v/s gun distance	95
Figure 5.5	Powder v/s Fluidization pressure	96
Figure 5.6	Predicted and actual fiber volume fraction	101
Figure 5.7	Output response of F_{vf} to input process parameters of V_g and T_v	102
Figure 5.8	F_{vf} response to D_g and T_v	103
Figure 5.9	Towpreg with fiber volume fraction of 50% sintered at oven temperature of (a) 180 °C (b) 200 °C (c) 220 °C	105
Figure 5.10	Towpreg with fiber volume fraction of 60% sintered at oven temperature of (a) 180 °C (b) 200 °C (c) 220 °C	106
Figure 5.11	Effect of oven temperature on flexural rigidity	107
Figure 6.1.	DSC plot of PP fiber, PP powder, and PP granule	114
Figure 6.2.	TGA plot for PP fiber, PP powder, and PP granule	115
Figure 6.3.	Viscosity plot for PP fiber, PP powder, and PP granule	116
Figure 6.4.	Particle size distribution	117

Figure 6.5.	Load-deflection plot in (a) three-point bend test	118
	(b) short beam test for UDC-D, UDC-P and UDC-F	118
Figure 6.6.	(a1) SEM image of cross-section of UDC-D	120
	(a2) SEM image at higher magnification of the encircled/ highlighted area of (a1).	120
	(b1) SEM image of cross-section of UDC-P	120
	(b2) SEM image at higher magnification of the encircled/ highlighted area of (b1)	120
	(c1) SEM image of cross-section of UDC-F	120
	(c2) SEM image at higher magnification of the encircled/ highlighted area of (c1)	120
Figure 6.7.	Micro-CT image of the composites in 3 point bending	122
	(a) UDC-D	
	(b) UDC-P	122
	(c) UDC-F	122
Figure 6.8.	(a1) SEM image of the DREF hybrid yarn before consolidation to form UDC-D	123
	(a2) schematic of PP fibers around carbon tow in the form of sheath	123
	(a3) schematic of the impregnation of sheath during consolidation	123
	(b1) SEM image of the PP powder coated towpreg before consolidation to form UDC-P	123
	(b2) schematic of powder coated carbon tows	123
	(b3) schematic of the impregnation of powder during consolidation	123
Figure 7.1	(a) Thermal treatment of DREF III spun hybrid yarn	133
	(b) SEM image of DREF III spun hybrid yarn	133
	(c) SEM image of DREF III spun heat set hybrid yarn	133
Figure 7.2.	Three-dimensional view of (a) 2D weave pattern for 2DWF	135
	(b) 3D angle interlock weave pattern for 3DWAF	135
	(c) 3D orthogonal weave pattern for 3DWOF	135

Figure 7.3	Peg Plan for production of	135
	(a) Plain woven fabric	
	(b) 3D woven orthogonal structure	135
	(c) 3D woven angle interlock structure	136
Figure 7.4.	(a) Loom for the production of 2D and 3D woven fabrics	137
	(b) beam arrangement for stuffer and binder warp ends	137
Figure 7.5.	(a) Stress-strain plot for 2DWC, 3DWAC and 3DWOC	140
	(b) Tensile strength with error bar	140
	(c) Tensile modulus with error bar	140
Figure 7.6	(a1) Warp breakage in 2DWC	141
	(a2) stuffer warp pull out and binder warp breakage in 3DWAC	141
	(a3) stuffer and binder warp breakage in 3DWOC	141
	Float length and material separation line in (b1) 2DWC	141
	(b2) 3DWAC	141
	(b3) 3DWOC	141
Figure 7.7	Load-deflection plot in 3-point bend test for 2DWC, 3DWOC, 3DWAC	143
	Flexural strength with error bar	144
	Flexural modulus with error bar	144
Figure 7.8.	(a) Notch-impact energy for all the composites	146
	Micro-CT images of post notch-test samples of 2D and 3D woven composites from DREF hybrid spun hybrid yarn;	146
	sectioned slice showing fiber breakage and porosity (b) 2DWC-D	
	(c) 3DWAC-D	146
	(d) 3DWOC-D	146
	(e) 2DWC-P	146
	(f) 3DWAC-P	146
	(g) 3DWOC-P	146
Figure 7.9.	(a) SEM image of the carbon tow	155

(b) SEM image of the carbon tow wrapped with PP fibers by DREF III spinning	155
(c) SEM image of the carbon tow coated with PP powder by electrostatic spray coating process.	155

List of Tables

Caption	Name	Page No.
Table 2.1	Comparison of different powder coating technologies	50
Table 3.1	Properties of carbon fiber	55
Table 3.2	Properties of PP fiber	56
Table 3.3	Properties for PP powder	56
Table 5.1	Experimental conditions for investigation of process characteristics for maximum powder deposition	90
Table 5.2	Process parameters and levels used for Box-Behnken design of experiment	98
Table 5.3	Actual and coded values (in parenthesis) for experimental response and corresponding F_{vf}	99
Table 5.4	Analysis of response model	100
Table 5.5	Effect of oven temperature on flexural rigidity	106
Table 6.1.	Unidirectional composites	112
Table 6.2.	Process parameters for manufacturing of DREF spun carbon/PP hybrid yarn	112
Table 6.3.	Powder coating machine parameters	113
Table 6.4.	Three-point bend test results of UDC-D, UDC-P and UDC-F	118
Table 7.1.	2D and 3D woven composites	131
Table 7.2.	Specifications of 2D and 3D woven fabrics	136
Table 7.3.	Tensile properties for 2DWC, 3DWOC and 3DWAC	139
Table 7.4.	Flexural properties for 2DWC, 3DWOC and 3DWAC	143
Table 7.5.	Notch Impact properties for 2DWC, 3DWAC and 3DWOC	146

Abbreviation and Symbols

N/Tex	Newton per Tex
%	percentage
kg/m ³	Kilogram per cubic meter
g/cm ³	Gram per cubic centimeter
μm	micrometer
g/den	gram per denier
g/10min	gram per 10 min
°C	degree Celsius
rpm	revolutions per minute
m/min	meter per minute
mm/min	millimeter per minute
cm	centimeter
mm	millimeter
m	meter
MPa	Megapascal
GPa	Gigapascal
μCT	Micro computed Tomography
kV	kilovolt
rad/s	radian per second
Pa.s	Pascal second
ρ_f	Density of fiber
ρ_m	Density of matrix
v_f	Volume fraction of fiber
$Q_3(x)$	volume proportion of particles smaller in size than x μm in relation to the total volume
V_g	The electrostatic voltage applied at the gun
D_g	Distance between the tip of the gun and the reinforcement or towpreg
T_v	The Velocity of the carbon towpreg

F_{vf}	Fiber volume fraction
g cm^2	Gram square centimeter
UDC-D	Unidirectional Composites from DREF spun hybrid yarn
UDC-P	Unidirectional Composites from powder coated towpreg
UDC-F	Unidirectional Composites from film stacking
2DWC-D	Two dimensional woven composites from DREF spun hybrid yarn
3DWAC-D	Three-dimensional woven angle interlock composites from DREF spun hybrid yarn
3DWOC-D	Three-dimensional woven orthogonal composites from DREF spun hybrid yarn
2DWC-P	Two-dimensional woven composites from powder coated towpreg
3DWAC-P	Three-dimensional woven angle interlock composites from powder coated towpreg
3DWOC-P	Three-dimensional woven orthogonal composites from powder coated towpreg