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THESIS ABSTRACT (ENGLISH)

NAME: MOHAMMED RIYAZUDDIN FAROOQUI
TITLE: INFLUENCE OF CARBON NANOTUBE (CNT) ON THE MECHANICAL PROPERTIES AND PROCESSIBILITY OF POLYETHYLENE TERAPHTHALATE (PET)/CNT NANOCOMPOSITE FIBERS
MAJOR: MECHANICAL ENGINEERING
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Polyethylene terephthalate (PET) is one of the mostly used polyester for fiber production. It is spun into filament, string or rope and used as a component of composite materials. In order to enhance their strength and broaden their application, PET fibers are blended with carbon nanotubes (CNT) which are known for high strength and stiffness. However, to produce good quality of PET/CNT nanocomposite fibers two major challenges were faced, one to obtain good distribution of CNT and the other to achieve good alignment of CNT along the fiber direction. In the present work, good distribution of CNT in PET matrix was achieved using twin screw melt extrusion process and the CNT alignment was obtained using melt spinning technique. The present study investigated the effect of process parameters on the mechanical properties of two sets of fibers. The first set of fibers is extruded in the form of large diameter fibers in the range of 220 to 700 μ m. The second set of fibers was melt spun into fine fibers of diameter in the range of 20 to 50 μ m. The extruded fibers showed good improvement in their mechanical properties with respect to CNT content in the range of 0.1 to 7.5wt%. The highest improvement of

mechanical properties was achieved with 0.1 wt% CNT content. Using this concentration in the nanocomposite fibers, the tensile strength was enhanced by about 90%, compared to pure PET fibers (51MPa). In addition, this composition significantly enhanced the strain at break by about 173 % and toughness by about 285 %. Other CNT concentration showed moderate improvements.

The second set of fibers which were melt spun fibers also showed good enhancement in the strength, stiffness and toughness with respect to CNT content in the range of 0.1 to 2.7wt%. The best improvement in the mechanical properties was observed with 0.1 and 0.5wt% CNT. Incorporating 0.1 and 0.5 wt% CNT in spun PET/CNT nanocomposite fibers of 20 μm diameters showed remarkable increment in the modulus by about 16% and 56%, respectively, compared to pure PET fibers with a modulus of 8.6 GPa. The tensile strength of PET/CNT nanocomposite fibers improved by about 40% (532MPa) due to the addition of 0.1wt% CNT in PET/CNT nanocomposite, compared to pure PET fibers of 380MPa. In addition, incorporating 0.1wt% CNT content in PET/CNT nanocomposite fibers of 50 μm diameter resulted in good improvement in toughness i.e. about 26% (689MPa). The improvements in the mechanical properties were based on the good mixing, distribution and alignment of CNT in PET/CNT nanocomposite fibers, as observed with scanning electron microscope

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THESIS ABSTRACT (ARABIC)

الاسم: محمد رياض الدين فاروقي

العنوان: تأثير أنابيب النانو الكربونية (سي إن تي) على الخواص الميكانيكية و القدرة العملية للبولي ايثيلين تيرافثالات (بي إي تي) ذات الألياف (سي إن تي) نانوية التركيب.

التخصص: الهندسة الميكانيكية .

التاريخ: مارس 2012

البولي ايثيلين تيرافثالات (بي إي تي) هو من اهم المركبات البوليمرية لصناعة الألياف. حيث تنسج الى خيوط أو حبال لتستخدم بعد ذلك كعنصر للمواد المركبة. و من أجل تعزيز قوتها و توسيع مجالات الاستعمال بها يمزج ال (بي إي تي) مع أنابيب النانو الكربونية (سي إن تي) حيث تعرف هذه الأنابيب بقوتها و قدرتها على التحمل العاليتين. ولكن حتى ننتج جودة عالية من هذه الألياف نانوية التركيب واجهنا اثنين من المصاعب الرئيسية. الأول هو الحصول على التوزيع الجيد لل (سي إن تي) و الثاني هو السعي وراء تحقيق المواءمة الجيدة لل (سي إن تي) على نفس اتجاه الألياف. في الوقت الحالي تم تحقيق التوزيع الجيد لل (سي إن تي) في ال (بي إي تي) من خلال اجراء عملية *twin screw melt extrusion process* و تم تحقيق مواءمة ال (سي إن تي) من خلال اجراء تقنية غزل المذاب. الدراسة الحالية ناقشت تأثير عملية المعالم على الخصائص الميكانيكية من مجموعتين من الألياف. المجموعة الاولى تقذف على شكل ألياف كبيرة القطر في مدى ٢٢٠ الى $700 \mu m$. المجموعة الثانية غزل مصهورها الى ألياف دقيقة بأقطار في مدى ٢٠ الى $50 \mu m$.

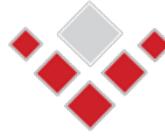
الألياف المقذوفة اظهرت تحسن ملحوظ في خواصها الميكانيكية بالنسبة الى نسبة ال (سي إن تي) في المركب من ٠.١ الى ٧.٥ % . أعلى معدل تحسن في الخواص الميكانيكية تم الحصول عليها في مركب ٠.١ وزن % من ال (سي إن تي) . باستخدام هذه النسبة من التركيز في الألياف نانوية التركيب، قوة الشد تحسنت بمقدار ٩٠ % ، مقارنة مع ال (بي إي تي) النقي ($51 MPa$). أيضا هذا التركيز حسن بشكل كبير كل من التوتر عند الكسر بنسبة ١٧٣ % و الصلابة بنسبة ٢٨٥ % . نسب التركيز الأخرى لل (سي إن تي) أظهرت معدلات تحسن معتدلة.

المجموعة الثانية من الألياف التي غزل مصهورها هي الأخرى أظهرت تحسن جيد في القوة، القساوة و الصلابة بالنسبة لتركيز ال(سي إن تي) في المدى ٠.١ الى ٢.٧ وزن %. أفضل التحسنات في الخواص الميكانيكية لوحظت في ٠.١ و ٠.٥ وزن % من (سي إن تي). مزج ٠.١ و ٠.٥ وزن % من ال(سي إن تي) في (بي إي تي) \ (سي إن تي) ألياف نانوية التركيب ذات قطر $20 \mu m$ أظهرت زيادة ممتازة في معامل. بنسبة % ١٦ و % ٢٥ على الترتيب. مقارنة مع الألياف (بي إي تي) النقية ذات معامل يونج بمقدار 8.6 GPa . قوة الشد للألياف (بي إي تي) \ (سي إن تي) نانوية التركيب تحسنت بمقدار % ٤٠ (532 MPa) نظراً لزيادة ٠.١ وزن % من (سي إن تي) في (بي إي تي) \ (سي إن تي) نانوية التركيب، مقارنة بالألياف (بي إي تي) النقية بمقدار 380 MPa . أيضاً، مزج ٠.١ وزن % من (سي إن تي) الى الألياف (بي إي تي) \ (سي إن تي) نانوية التركيب ذات قطر $50 \mu m$ نتج عن ذلك تحسن جيد في الصلابة % ٢٦ (689 MPa). التحسنات في الخواص الميكانيكية بنيت على اساس المزج الجيد، التوزيع و الموامعة لل(سي إن تي) في ألياف (بي إي تي) \ (سي إن تي) نانوية التركيب، كما لوحظ باستخدام المجهر الإلكتروني.

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TABLE OF CONTENTS

ACKNOWLEDGEMENT	iv
LIST OF TABLES	ix
LIST OF FIGURES	x
THESIS ABSTRACT (ENGLISH)	xiv
THESIS ABSTRACT (ARABIC)	xvi
CHAPTER 1	1
INTRODUCTION	1
1.1 Research Background	1
1.2 PET	1
1.3 CNT.....	3
1.4 PET/CNT nanocomposite	6
1.5 Melt Extrusion Spinning.....	7
1.6 Objectives	9
1.7 Challenges.....	9
1.8 Motivation.....	9
CHAPTER 2	10
LITERATURE REVIEW	10
2.1 Effect of CNT on PET/CNT nanocomposite	10
2.2 Effect of CNT on other Polymers	20

CHAPTER 3	27
3.1 Experimental Procedure.....	27
3.1.1 Poly (ethylene terephthalate) Material	27
3.1.2 CNT Material	27
3.1.3 CNT heat treatment	28
3.1.4 Moisture removal	30
3.1.5 Extrusion and Melt Spinning Process	31
3.2 Characterization Techniques.....	34
3.2.1 Fiber Diameter Measurements	34
3.2.2 Thermogravimetry Differential Scanning Calorimetry (TG-DSC).....	35
3.2.3 Scanning Electron Microscopy (SEM).....	36
3.2.4 Mechanical Testing	36
3.2.5 Work Plan.....	39
CHAPTER 4	41
RESULTS AND DISCUSSION.....	41
4.1 Effect of Die Temperature on Mechanical Properties of PET/CNT Nanocomposite Fibers.....	41
4.1.1 SEM Analysis.....	42
4.1.2 Mechanical Properties	42
4.2 Effect of spinning speed on mechanical properties of PET/CNT nanocomposite fibers.....	51
4.2.1 Mechanical Properties	51

4.3 Influence of CNT concentration on the Mechanical properties of extruded PET/CNT nanocomposite fibers	58
4.3.1 TG-DSC Analysis.....	58
4.3.2 SEM Analysis.....	59
4.3.3 Mechanical Properties	67
4.4 Effect of CNT concentration on the mechanical properties of spun PET/CNT nanocomposite fibers.	74
CHAPTER 5	84
CONCLUSIONS.....	84
5.1 Die Temperature Effect.....	84
5.2 Melt Spinning Effect.....	85
5.3 CNT Concentration Effect	86
CHAPTER 6	87
FUTURE WORK.....	87
NOMENCLATURE	89
REFERENCES	91
VITAE.....	96

LIST OF TABLES

Table 1.1 Characteristics of PET [4].....	4
Table 1.2 Characteristics of CNT[14].....	6
Table 2.1 Literature review.....	26
Table 4.1 Averaged Mechanical Properties of PET/CNT nanocomposite fibers	83

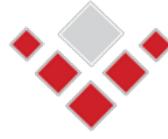
LIST OF FIGURES

Figure 1.1 Repeating unit of PET	2
Figure 1.2 Polymer condensation	2
Figure 1.3 SWCNT.....	5
Figure 1.4: MWCNT.....	5
Figure 1.5: Melt extrusion set up.....	8
Figure 1.6: Melt-spinning process	8
Figure 2.1 Mechanical properties [10].....	12
Figure 2.2 Elongation at break [10].	12
Figure 2.3 Mechanical properties [15].....	14
Figure 2.4 PBT/CNT stress strain Curves [25].....	23
Figure 3.1 SEM micrographs of aligned MWCNT's.	29
Figure 3.2 Furnace for heat treatment of CNT.	30
Figure 3.3 Vacuum oven for drying PET pellets and CNT materials.....	31
Figure 3.4 Second feeder for blending.....	33
Figure 3.5 Optical Microscope.	34
Figure 3.6 TG- DSC instrument.....	35
Figure 3.7 SEM.....	37
Figure 3.8. Lloyd tensile testing machine.....	38
Figure 3.9 Work plan	40
Figure 4.1 Distribution of 1wt% CNT in PETmatrix of PET/CNT nanocomposite fibers at die temperature 265°C obtained at (a) lower and (b) higher magnifications.	43

Figure 4.2 Distribution of 1wt% CNT in PETmatrix of PET/CNT nanocomposite fibers at die temperature 280°C at (a) lower and (b) higher magnifications.....	44
Figure 4.3 Distribution of 1wt% CNT in PETmatrix of PET/CNT nanocomposite fibers at die temperature 290°C at (a) lower and (b) higher magnifications.....	45
Figure 4.4 Influence of Die temperature on tensile stress strain curve of PET/CNT nanocomposite fibers	47
Figure 4.5 Influence of Die temperature on modulus of PET/CNT nanocomposite fibers.	48
Figure 4.6 Influence of Die temperature on yield strength of PET/CNT nanocomposite fibers.	48
Figure 4.7 Influence of Die temperature on tensile strength of PET/CNT nanocomposite fibers.	49
Figure 4.8 Influence of Die temperature on strain at break of PET/CNT nanocomposite fibers.	49
Figure 4.9 Influence of Die temperature on toughness of PET/CNT nanocomposite fibers.	50
Figure 4.10 Influence of Die temperature on spinnability of PET/CNT nanocomposite fibers.	50
Figure 4.11 Influence of spinning speed on the diameters of PET/CNT nanocomposite fibers.	52
Figure 4.12 Influence of spinning speed on the modulus of PET/CNT nanocomposite fibers.	55
Figure 4.13 Influence of spinning speed on the yield strength of PET/CNT nanocomposite fibers.	55
Figure 4.14 Influence of spinning speed on the tensile strength of PET/CNT nanocomposite fibers.	56

Figure 4.15 Influence of spinning speed on the strain at break of PET/CNT nanocomposite fibers.	56
Figure 4.16 Influence of spinning speed on the toughness of PET/CNT nanocomposite fibers.	57
Figure 4.17 TGA- Thermograms showing the weight change of pure PET, pure CNT and PETCNT 1wt%.	60
Figure 4.18 TGA calibration and the CNT concentrations of the produced nanocomposite fibers.	60
Figure 4.19 Fracture surface of Pure PET fibers at (a) lower and (b) higher magnifications.....	61
Figure 4.20 Distribution and alignment of 0.5wt% CNT in PET/CNT nanocomposite fibers at (a) lower and (b) higher magnifications.....	62
Figure 4.21 Distribution and alignment of 1wt% CNT in PET/CNT nanocomposite fibers at (a) lower and (b) higher magnifications.....	63
Figure 4.22 Distribution and alignment of 2.7wt% CNT in PET/CNT nanocomposite fibers at (a) lower and (b) higher magnifications.....	64
Figure 4.23 Distribution and alignment of 3.0wt% CNT in PET/CNT nanocomposite fibers at (a) lower and (b) higher magnifications.....	65
Figure 4.24 Distribution and alignment of 7.5wt% CNT in PET/CNT nanocomposite fibers at (a) lower and (b) higher magnifications.....	66
Figure 4.25 Influence of CNT concentration on the Mechanical properties of extruded PET/CNT nanocomposite fibers.....	70
Figure 4.26 Influence of CNT concentration on the modulus of extruded PET/CNT nanocomposite fibers.	71
Figure 4.27 Influence of CNT concentration on the yield strength of extruded PET/CNT nanocomposite fibers.....	71

Figure 4.28 Influence of CNT concentration on the tensile strength of extruded PET/CNT nanocomposite fibers.....	72
Figure 4.29 Influence of CNT concentration on the strain at break of extruded PET/CNT nanocomposite fibers.....	72
Figure 4.30 Influence of CNT concentration on the toughness of extruded PET/CNT nanocomposite fibers.....	73
Figure 4.31 Influence of CNT concentration on the modulus of spun PET/CNT nanocomposite fibers.....	80
Figure 4.32 Influence of CNT concentration on the yield strength of spun PET/CNT nanocomposite fibers.....	81
Figure 4.33 Influence of CNT concentration on the tensile strength of spun PET/CNT nanocomposite fibers.....	81
Figure 4.34 Influence of CNT concentration on the strain at break of spun PET/CNT nanocomposite fibers.....	82
Figure 4.35 Influence of CNT concentration on the toughness of spun PET/CNT nanocomposite fibers.....	82



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BY
MOHAMMED RIYAZUDDIN FAROOQUI

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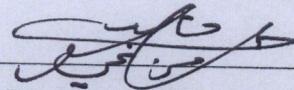
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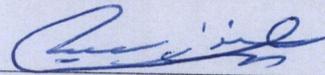
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This thesis, written by Mohammed Riyazuddin Farooqui under the direction of his thesis advisor and approved by his thesis committee, has been presented to and accepted by the Dean of Graduate Studies, in partial fulfillment of the requirements for the degree of **MASTER OF SCIENCE in MECHANICAL ENGINEERING.**

Thesis Committee



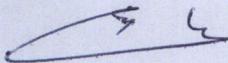
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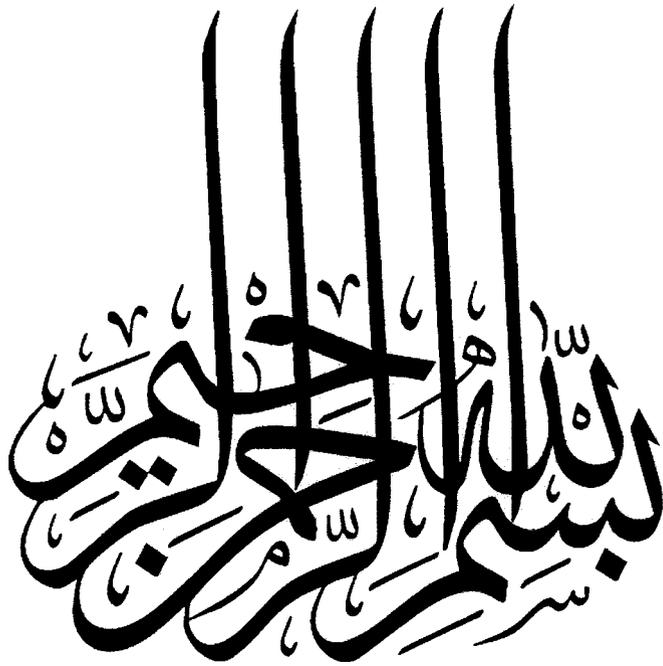
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Dean of Graduate Studies

8/5/12

Date





Dedicated to

*My beloved parents, brothers and sisters for their
duas and constant support and encouragement
throughout my life*

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TABLE OF CONTENTS

ACKNOWLEDGEMENT	iv
LIST OF TABLES	ix
LIST OF FIGURES	x
THESIS ABSTRACT (ENGLISH)	xiv
THESIS ABSTRACT (ARABIC)	xvi
CHAPTER 1	1
INTRODUCTION	1
1.1 Research Background	1
1.2 PET	1
1.3 CNT.....	3
1.4 PET/CNT nanocomposite	6
1.5 Melt Extrusion Spinning.....	7
1.6 Objectives	9
1.7 Challenges.....	9
1.8 Motivation.....	9
CHAPTER 2	10
LITERATURE REVIEW	10
2.1 Effect of CNT on PET/CNT nanocomposite	10
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CHAPTER 3	27
3.1 Experimental Procedure.....	27
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3.1.2 CNT Material	27
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3.1.4 Moisture removal	30
3.1.5 Extrusion and Melt Spinning Process	31
3.2 Characterization Techniques.....	34
3.2.1 Fiber Diameter Measurements	34
3.2.2 Thermogravimetry Differential Scanning Calorimetry (TG-DSC).....	35
3.2.3 Scanning Electron Microscopy (SEM).....	36
3.2.4 Mechanical Testing	36
3.2.5 Work Plan.....	39
CHAPTER 4	41
RESULTS AND DISCUSSION.....	41
4.1 Effect of Die Temperature on Mechanical Properties of PET/CNT Nanocomposite Fibers.....	41
4.1.1 SEM Analysis.....	42
4.1.2 Mechanical Properties	42
4.2 Effect of spinning speed on mechanical properties of PET/CNT nanocomposite fibers.....	51
4.2.1 Mechanical Properties	51

4.3 Influence of CNT concentration on the Mechanical properties of extruded PET/CNT nanocomposite fibers	58
4.3.1 TG-DSC Analysis.....	58
4.3.2 SEM Analysis.....	59
4.3.3 Mechanical Properties	67
4.4 Effect of CNT concentration on the mechanical properties of spun PET/CNT nanocomposite fibers.	74
CHAPTER 5	84
CONCLUSIONS.....	84
5.1 Die Temperature Effect.....	84
5.2 Melt Spinning Effect.....	85
5.3 CNT Concentration Effect	86
CHAPTER 6	87
FUTURE WORK.....	87
NOMENCLATURE	89
REFERENCES	91
VITAE.....	96

LIST OF TABLES

Table 1.1 Characteristics of PET [4].....	4
Table 1.2 Characteristics of CNT[14].....	6
Table 2.1 Literature review.....	26
Table 4.1 Averaged Mechanical Properties of PET/CNT nanocomposite fibers	83

LIST OF FIGURES

Figure 1.1 Repeating unit of PET	2
Figure 1.2 Polymer condensation	2
Figure 1.3 SWCNT.....	5
Figure 1.4: MWCNT.....	5
Figure 1.5: Melt extrusion set up.....	8
Figure 1.6: Melt-spinning process	8
Figure 2.1 Mechanical properties [10].....	12
Figure 2.2 Elongation at break [10].	12
Figure 2.3 Mechanical properties [15].....	14
Figure 2.4 PBT/CNT stress strain Curves [25].....	23
Figure 3.1 SEM micrographs of aligned MWCNT's.	29
Figure 3.2 Furnace for heat treatment of CNT.	30
Figure 3.3 Vacuum oven for drying PET pellets and CNT materials.....	31
Figure 3.4 Second feeder for blending.....	33
Figure 3.5 Optical Microscope.	34
Figure 3.6 TG- DSC instrument.....	35
Figure 3.7 SEM.....	37
Figure 3.8. Lloyd tensile testing machine.....	38
Figure 3.9 Work plan	40
Figure 4.1 Distribution of 1wt% CNT in PETmatrix of PET/CNT nanocomposite fibers at die temperature 265°C obtained at (a) lower and (b) higher magnifications.	43

Figure 4.2 Distribution of 1wt% CNT in PETmatrix of PET/CNT nanocomposite fibers at die temperature 280°C at (a) lower and (b) higher magnifications.....	44
Figure 4.3 Distribution of 1wt% CNT in PETmatrix of PET/CNT nanocomposite fibers at die temperature 290°C at (a) lower and (b) higher magnifications.....	45
Figure 4.4 Influence of Die temperature on tensile stress strain curve of PET/CNT nanocomposite fibers	47
Figure 4.5 Influence of Die temperature on modulus of PET/CNT nanocomposite fibers.	48
Figure 4.6 Influence of Die temperature on yield strength of PET/CNT nanocomposite fibers.	48
Figure 4.7 Influence of Die temperature on tensile strength of PET/CNT nanocomposite fibers.	49
Figure 4.8 Influence of Die temperature on strain at break of PET/CNT nanocomposite fibers.	49
Figure 4.9 Influence of Die temperature on toughness of PET/CNT nanocomposite fibers.	50
Figure 4.10 Influence of Die temperature on spinnability of PET/CNT nanocomposite fibers.	50
Figure 4.11 Influence of spinning speed on the diameters of PET/CNT nanocomposite fibers.	52
Figure 4.12 Influence of spinning speed on the modulus of PET/CNT nanocomposite fibers.	55
Figure 4.13 Influence of spinning speed on the yield strength of PET/CNT nanocomposite fibers.	55
Figure 4.14 Influence of spinning speed on the tensile strength of PET/CNT nanocomposite fibers.	56

Figure 4.15 Influence of spinning speed on the strain at break of PET/CNT nanocomposite fibers.	56
Figure 4.16 Influence of spinning speed on the toughness of PET/CNT nanocomposite fibers.	57
Figure 4.17 TGA- Thermograms showing the weight change of pure PET, pure CNT and PETCNT 1wt%.	60
Figure 4.18 TGA calibration and the CNT concentrations of the produced nanocomposite fibers.	60
Figure 4.19 Fracture surface of Pure PET fibers at (a) lower and (b) higher magnifications.....	61
Figure 4.20 Distribution and alignment of 0.5wt% CNT in PET/CNT nanocomposite fibers at (a) lower and (b) higher magnifications.....	62
Figure 4.21 Distribution and alignment of 1wt% CNT in PET/CNT nanocomposite fibers at (a) lower and (b) higher magnifications.....	63
Figure 4.22 Distribution and alignment of 2.7wt% CNT in PET/CNT nanocomposite fibers at (a) lower and (b) higher magnifications.....	64
Figure 4.23 Distribution and alignment of 3.0wt% CNT in PET/CNT nanocomposite fibers at (a) lower and (b) higher magnifications.....	65
Figure 4.24 Distribution and alignment of 7.5wt% CNT in PET/CNT nanocomposite fibers at (a) lower and (b) higher magnifications.....	66
Figure 4.25 Influence of CNT concentration on the Mechanical properties of extruded PET/CNT nanocomposite fibers.....	70
Figure 4.26 Influence of CNT concentration on the modulus of extruded PET/CNT nanocomposite fibers.	71
Figure 4.27 Influence of CNT concentration on the yield strength of extruded PET/CNT nanocomposite fibers.....	71

Figure 4.28 Influence of CNT concentration on the tensile strength of extruded PET/CNT nanocomposite fibers.....	72
Figure 4.29 Influence of CNT concentration on the strain at break of extruded PET/CNT nanocomposite fibers.....	72
Figure 4.30 Influence of CNT concentration on the toughness of extruded PET/CNT nanocomposite fibers.....	73
Figure 4.31 Influence of CNT concentration on the modulus of spun PET/CNT nanocomposite fibers.....	80
Figure 4.32 Influence of CNT concentration on the yield strength of spun PET/CNT nanocomposite fibers.....	81
Figure 4.33 Influence of CNT concentration on the tensile strength of spun PET/CNT nanocomposite fibers.....	81
Figure 4.34 Influence of CNT concentration on the strain at break of spun PET/CNT nanocomposite fibers.....	82
Figure 4.35 Influence of CNT concentration on the toughness of spun PET/CNT nanocomposite fibers.....	82

THESIS ABSTRACT (ENGLISH)

NAME: MOHAMMED RIYAZUDDIN FAROOQUI
TITLE: INFLUENCE OF CARBON NANOTUBE (CNT) ON THE MECHANICAL PROPERTIES AND PROCESSIBILITY OF POLYETHYLENE TERAPHTHALATE (PET)/CNT NANOCOMPOSITE FIBERS
MAJOR: MECHANICAL ENGINEERING
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Polyethylene terephthalate (PET) is one of the mostly used polyester for fiber production. It is spun into filament, string or rope and used as a component of composite materials. In order to enhance their strength and broaden their application, PET fibers are blended with carbon nanotubes (CNT) which are known for high strength and stiffness. However, to produce good quality of PET/CNT nanocomposite fibers two major challenges were faced, one to obtain good distribution of CNT and the other to achieve good alignment of CNT along the fiber direction. In the present work, good distribution of CNT in PET matrix was achieved using twin screw melt extrusion process and the CNT alignment was obtained using melt spinning technique. The present study investigated the effect of process parameters on the mechanical properties of two sets of fibers. The first set of fibers is extruded in the form of large diameter fibers in the range of 220 to 700 μ m. The second set of fibers was melt spun into fine fibers of diameter in the range of 20 to 50 μ m. The extruded fibers showed good improvement in their mechanical properties with respect to CNT content in the range of 0.1 to 7.5wt%. The highest improvement of

mechanical properties was achieved with 0.1 wt% CNT content. Using this concentration in the nanocomposite fibers, the tensile strength was enhanced by about 90%, compared to pure PET fibers (51MPa). In addition, this composition significantly enhanced the strain at break by about 173 % and toughness by about 285 %. Other CNT concentration showed moderate improvements.

The second set of fibers which were melt spun fibers also showed good enhancement in the strength, stiffness and toughness with respect to CNT content in the range of 0.1 to 2.7wt%. The best improvement in the mechanical properties was observed with 0.1 and 0.5wt% CNT. Incorporating 0.1 and 0.5 wt% CNT in spun PET/CNT nanocomposite fibers of 20 μm diameters showed remarkable increment in the modulus by about 16% and 56%, respectively, compared to pure PET fibers with a modulus of 8.6 GPa. The tensile strength of PET/CNT nanocomposite fibers improved by about 40% (532MPa) due to the addition of 0.1wt% CNT in PET/CNT nanocomposite, compared to pure PET fibers of 380MPa. In addition, incorporating 0.1wt% CNT content in PET/CNT nanocomposite fibers of 50 μm diameter resulted in good improvement in toughness i.e. about 26% (689MPa). The improvements in the mechanical properties were based on the good mixing, distribution and alignment of CNT in PET/CNT nanocomposite fibers, as observed with scanning electron microscope

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THESIS ABSTRACT (ARABIC)

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العنوان: تأثير أنابيب النانو الكربونية (سي إن تي) على الخواص الميكانيكية و القدرة العملية للبولي ايثيلين تيرافثالات (بي إي تي) ذات الألياف (سي إن تي) نانوية التركيب.

التخصص: الهندسة الميكانيكية .

التاريخ: مارس 2012

البولي ايثيلين تيرافثالات (بي إي تي) هو من اهم المركبات البوليمرية لصناعة الألياف. حيث تنسج الى خيوط أو حبال لتستخدم بعد ذلك كعنصر للمواد المركبة. و من أجل تعزيز قوتها و توسيع مجالات الاستعمال بها يمزج ال (بي إي تي) مع أنابيب النانو الكربونية (سي إن تي) حيث تعرف هذه الأنابيب بقوتها و قدرتها على التحمل العاليتين. ولكن حتى ننتج جودة عالية من هذه الألياف نانوية التركيب واجهنا اثنين من المصاعب الرئيسية. الأول هو الحصول على التوزيع الجيد لل (سي إن تي) و الثاني هو السعي وراء تحقيق المواءمة الجيدة لل (سي إن تي) على نفس اتجاه الألياف. في الوقت الحالي تم تحقيق التوزيع الجيد لل (سي إن تي) في ال (بي إي تي) من خلال اجراء عملية *twin screw melt extrusion process* و تم تحقيق مواءمة ال (سي إن تي) من خلال اجراء تقنية غزل المذاب. الدراسة الحالية ناقشت تأثير عملية المعالم على الخصائص الميكانيكية من مجموعتين من الألياف. المجموعة الاولى تقذف على شكل ألياف كبيرة القطر في مدى ٢٢٠ الى ٧٠٠ μm . المجموعة الثانية غزل مصهورها الى ألياف دقيقة بأقطار في مدى ٢٠ الى ٥٠ μm .

الألياف المقذوفة اظهرت تحسن ملحوظ في خواصها الميكانيكية بالنسبة الى نسبة ال (سي إن تي) في المركب من ٠.١ الى ٧.٥ % . أعلى معدل تحسن في الخواص الميكانيكية تم الحصول عليها في مركب ٠.١ وزن % من ال (سي إن تي) . باستخدام هذه النسبة من التركيز في الألياف نانوية التركيب، قوة الشد تحسنت بمقدار ٩٠ % ، مقارنة مع ال (بي إي تي) النقي (٥١ MPa). أيضا هذا التركيز حسن بشكل كبير كل من التوتر عند الكسر بنسبة ١٧٣ % و الصلابة بنسبة ٢٨٥ % . نسب التركيز الأخرى لل (سي إن تي) أظهرت معدلات تحسن معتدلة.

المجموعة الثانية من الألياف التي غزل مصهورها هي الأخرى أظهرت تحسن جيد في القوة، القساوة و الصلابة بالنسبة لتركيز ال(سي إن تي) في المدى ٠.١ الى ٢.٧ وزن %. أفضل التحسنات في الخواص الميكانيكية لوحظت في ٠.١ و ٠.٥ وزن % من (سي إن تي). مزج ٠.١ و ٠.٥ وزن % من ال(سي إن تي) في (بي إي تي) \ (سي إن تي) ألياف نانوية التركيب ذات قطر $20 \mu m$ أظهرت زيادة ممتازة في معامل. بنسبة % ١٦ و % ٢٥ على الترتيب. مقارنة مع الألياف (بي إي تي) النقية ذات معامل يونج بمقدار 8.6 GPa . قوة الشد للألياف (بي إي تي) \ (سي إن تي) نانوية التركيب تحسنت بمقدار % ٤٠ (532 MPa) نظراً لزيادة ٠.١ وزن % من (سي إن تي) في (بي إي تي) \ (سي إن تي) نانوية التركيب، مقارنة بالألياف (بي إي تي) النقية بمقدار 380 MPa . أيضاً، مزج ٠.١ وزن % من (سي إن تي) الى الألياف (بي إي تي) \ (سي إن تي) نانوية التركيب ذات قطر $50 \mu m$ نتج عن ذلك تحسن جيد في الصلابة % ٢٦ (689 MPa). التحسنات في الخواص الميكانيكية بنيت على اساس المزج الجيد، التوزيع و المواءمة لل(سي إن تي) في ألياف (بي إي تي) \ (سي إن تي) نانوية التركيب، كما لوحظ باستخدام المجهر الإلكتروني.

درجة الماجستير في العلوم

جامعة الملك فهد للبترول و المعادن

الظهران المملكة العربية السعودية

CHAPTER 1

INTRODUCTION

1.1 Research Background

1.2 PET

At the end of nineteenth century, only natural fibers such as cotton, wool, and silk were available[1]. Later new classes of fibers made from polymer and ceramics were developed. In the class of polymeric fibers, these include polyester, nylon, polyethylene, polypropylene, acrylic, spandex, Nomex®, Kevlar®, and Zylon®. The class of other fibers includes carbon fibers, silicon carbide, alumina, glass, alumina-borosilicate, and boron. The first man-made fiber was Nylon which replaced the silk in parachutes and military uses.

Today, the polyester industry makes up about 18% of world polymer production and is third after polyethylene (PE) and polypropylene (PP). Of all the other type of polyester the most significant in fibers production is Polyethylene terephthalate (PET), its repeating unit is shown in Figure 1.1. PET was first developed by Whinfield and Dickson in 1939[2] by chemical reaction between terephthalic acid (TPA) and ethyl glycol (EG)

shown in Figure 1.2 (known as poly condensation process). PET is a stiff, hard, strong, has good gas barrier properties and good chemical resistance except to alkalis. Its crystallinity varies from amorphous to fairly high crystalline. It can be vastly transparent and colorless [3].

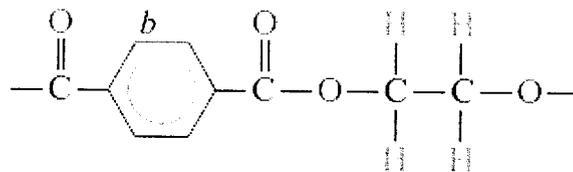


Figure 1.1 Repeating unit of PET

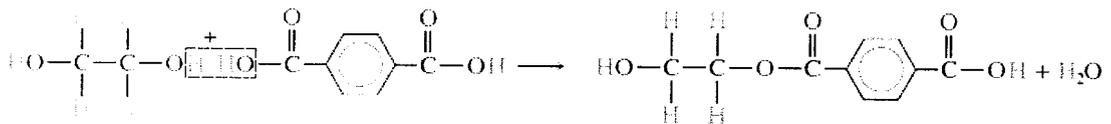


Figure 1.2 Polymer condensation

Thermal and mechanical properties of the PET are shown in Table 1.1. Molecular weight of repeat unit is 192.2 g/mol. [3]. Pattabiraman et.al [4] has shown that mechanical properties of virgin PET had the strength of 57MPa and modulus of elasticity of 2.3GPa. Another research group[5] has spun the PET Fibers, of size 21 μ m to 27 μ m, through twin Screw extruder and they reported the tensile strength of 350MPa to 510 MPa and the young modulus was in the range of 7 GPa to 13 GPa. Hence, Owing to its versatile properties, it has found applications in textile, automobile and in shipping industry.

Globally the production of PET synthetic fibers is 60 % of the world's PET production. It has been seen that several fine synthetic PET fibers are used for manufacturing mooring ropes which are used in ships [6-7]. PET also found significant used in construction industry as concrete-reinforcing fiber [8]. Moreover, PET is produced locally in Saudi Arabia in large quantity. Hence, enhancing the mechanical strength of PET can also improve the economy of the local market of the Saudi Arabia. In addition, PET with improved properties can be used in variety of industrial and household applications. They can be spun into filaments, string or rope, used as a component of composite materials. Efforts are being made to increase the range of applications of PET nanocomposites fibers, such as in medicine, electronics, biomaterials and energy production. The enhancement in the properties of PET composite fibers is achieved by modifying with organic and inorganic nanomaterials (1nm to 100nm) [19-36]. Nanomaterials include carbon nanotubes (CNT), carbon nanofiber, nanoclay, nanosilica and others.

1.3 CNT

CNT were first reported by Iijima in the year 1991[9]. After that, many investigations and developments are taking place to understand the full ability of CNT. In addition, it has been observed that CNT are ideal material for PET fiber reinforcement[10]. They are hexagonal network of carbon graphene sheets rolled into seamless cylinder with caps at the ends which are composed of half of the fullerene molecule. CNT are produced as single wall-CNT(SWCNT) Figure 1.3 or Multi wall-CNT shown in Figure 1.4. They are highly isotropic[11]. It was predicted that even the weak carbon nano tubes have the

strength in the order of several giga Pascals and young's modulus in the tera Pascal's [12] with low weight. In addition, CNTs have unique electrical properties and are good conductors of heat which make them novel material in various fields such as electrical field, nanotechnology, aerospace & material science [11].

Table 1.1 Characteristics of PET [4]

Melting temperature	250°C
Glass transition temperature	76°C
Crystallinity	33%
Tensile strength	Bulk 57 MPa
young modulus	2321.59MPa
Molecular weight of repeat unit	192.2 g/mol
PET Fibers [5]	
Fiber diameter	21 µm to 27 µm
Young's modulus	7GPa to 13GPa
Tensile strength	350MPa to 510MPa

The diameter of the nanotubes ranges from 1nm-100nm and length in millimeters and their density 1.3 gm/cm³ [13-14]. Young modulus measured to be 1.25TPa [12, 14] i.e. five times stiffer than that of the steel (210 GPa) and among all the properties of CNTs, the tensile strength that make them apart from other material were observed to be 63GPa i.e. fifty times stronger than the steels. Table 1.2 summarizes the CNTs' characteristics.

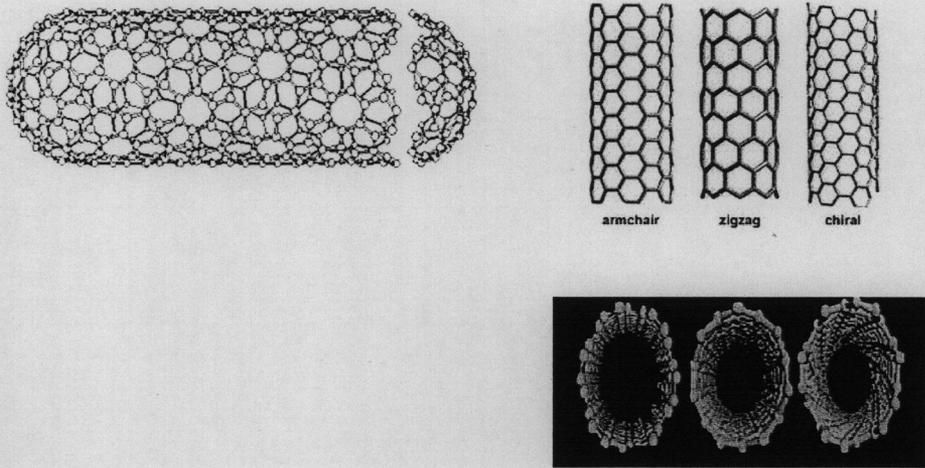


Figure 1.3 SWCNT.

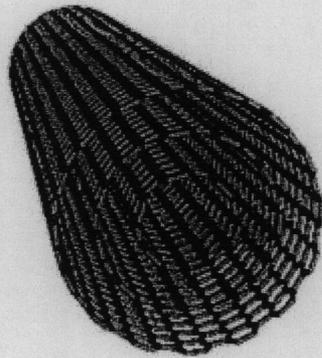


Figure 1.4: MWCNT

Table 1.2 Characteristics of CNT[14]

Tensile strength	63 GPa
young modulus	1.25TPa
Density	1.3 gm/cm ³
Diameters range	1nm-100nm

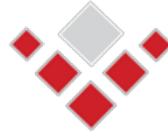
1.4 PET/CNT nanocomposite

Recent studies have shown that PET have excellently worked as a matrix in which small amount of CNT added in the PET enhances the thermo mechanical and electrical properties [10, 15-17]. Some groups of researchers have studied the improvement in the Properties of PET by different techniques [13-34]. Good dispersion and alignment of CNT in the PET nanocomposite fibers has good impact on improving their properties. Since, CNT are produced in agglomeration form, different techniques needed to separate them, disperse and distribute them in the matrix. Nanocomposite fiber produced with such configuration will have good performance. Today, the two major challenges to any researcher in order to develop high performance PET/CNT nanocomposite fibers are the distribution and the alignment of CNT in the PET matrix. To achieve well aligned, good adhered and uniformly distributed CNT in the PET matrix, the nanocomposite fibers are processed either via in situ polymerization [18] or melt extrusion spinning processes[10].

1.5 Melt Extrusion Spinning

Melt extrusion spinning process is the most simple and economical process. The uniform distribution of CNT in the PET matrix can be obtained by using twin screw extruder with mixing elements in melt extrusion process. In addition, the alignment of CNT in nanocomposite fibers can be accomplished by further melt spinning or drawing process.

Melt extrusion process setup of Twin screw extruder is schematically shown in the Figure 1.5. In order to obtain the different proportion of PET and CNT, two separate computerized control feeders will be used for feeding PET (matrix) and CNT (reinforcement) into the Extruder (barrel). The temperature throughout the barrel will be maintained above the melting point of the matrix. PET/CNT nanocomposite melt will be extruded through the die consisting of 10 holes spinneret as shown in Figure 1.6. The extrudate will be cooled by air flowing perpendicular to the flow of filaments. The solidifying polymers are drawn further by the winders.



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الرواد في قواعد المعلومات العربية

Influence of carbon nanotubes " CNT " on the mechanical properties of the polyethylene theraphthalate " PET" cnt nanocomposite fibers	العنوان:
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**INFLUENCE OF CARBON NANOTUBES (CNT) ON THE
MECHANICAL PROPERTIES OF THE POLYETHYLENE
THERAPHTHALATE (PET)/CNT NANOCOMPOSITE FIBERS**

BY
MOHAMMED RIYAZUDDIN FAROOQUI

A Thesis Presented to the
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DHAHRAN, SAUDI ARABIA

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Requirements for the Degree of

MASTER OF SCIENCE

In

MECHANICAL ENGINEERING

APRIL 2012