

## SHORT TERM SCIENTIFIC MISSION (STSM) SCIENTIFIC REPORT

This report is submitted for approval by the STSM applicant to the STSM coordinator

**Action number:** CA17107

**Host Institution:** RWTH Aachen University, Faculty of Mechanical Engineering, Institut für Textiltechnik (ITA), Germany

**STSM Request reference number:** ECOST-STSM-Request-CA17107-45683

**STSM title:** Melt Spinning and Characterization of Carbon Nanotube/Polymer Nanocomposite Filament Yarns

**STSM start and end date:** 13/01/2020 to 14/02/2020

**Grantee Name:** Müslüm Kaplan

### PURPOSE OF THE STSM:

Textile fibers containing carbon nanotubes (CNTs) have gained great interest due to their high mechanical, electrical, magnetic and thermal properties recently [1]. Thus, their property profile depends on nanocomposite properties, dispersion and orientation aspects, interfacial adhesion and polymer properties which can widely vary [2]. By using electrically conductive carbon nanomaterial (such as carbon black (CB), carbon nanotubes (CNT) and graphite nanoplatelets (GNP)) modified polymer composites as a great way of making textile fibers electrically conductive without compromising their comfort and flexibility.

Even though there are a variety of methods to produce electrically conductive nanocomposite polymer fibers, bicomponent melt spinning process should be emphasized [3], which is the subject of this work. Applying bicomponent technology with a home-made filter innovation of ITA may provide the possibility to produce fibers from conductive polymer composites (CPCs) with a high filler concentration [4].

In the scope of this STSM, at first, we examined the influence of the nanocomposite core material feeding parameters on the electrical conductivity of melt-spun fibers, incorporating nanofiller into a PA6 matrix by a pilot-scale bicomponent melt spinning. In the second, we investigated electrical conductivity of the single-wall carbon nanotube (SWCNT)/polyethylene (PE) nanocomposite melt-spun fibers.

### DESCRIPTION OF WORK CARRIED OUT DURING THE STSMS:

#### **Materials**

The polymers as matrix used was polyamide 6 (PA6), which is a typical semi-crystalline thermoplastic polymer with a wide range of engineering applications, and carbon-based nanofillers used were MWCNT, CB and SWCNT. The characteristics properties of the used PA6 and PE polymers and nanofillers are shown in Table 1. The properties of used nanofiller can be seen in Table 2.

Table 1. The polymers and nanofillers used in this study

Material	Trade Name	Company
PA6	B24 N03	BASF AG, Ludwigshafen, Germany
PA6	Domamid PW 6FC NC	Domo Engineering Plastics, Arco, Italy
PE	Evolue-H	Prime Polymer Co. Ltd., Tokyo, Japan
MWCNT	NC7000	Nanocyl S.A., Sambreville, Belgium
CB	Vulcan XC72	Cabot Corp., Billerica, USA
SWCNT	Tuball matrix 816 Beta (90%PE-10% SWCNT)	OCSiAl Ltd., Novosibirsk, Russia

Table 2. The properties of the nanofiller

Properties	MWCNT NC7000	CB Vulcan XC72	SWCNT TUBALL
Morphologie	little rod	pellet	powder
Carbon purity	>90%	-	75%
Diameter	9.5 nm	<0,044 nm	2 nm
Length	1.5 $\mu$ m	<0,044 nm	1-5 $\mu$ m
Surface area	250–300 m <sup>2</sup> /g	254 m <sup>2</sup> /g	331 m <sup>2</sup> /g
Bulk density	0,06 kg/cm <sup>3</sup>	0,264 kg/m <sup>3</sup>	90 kg/m <sup>3</sup>
Volume resistivity	0,001 Ohm.cm	6 Ohm.cm	-

## Methods

### *Laboratory-scale melt spinning of PE/SWCNT nanocomposite fibers*

Laboratory-scale melt spinning of the PE/SWCNT nanocomposite fibers was performed in conical twin-screw micro-extruder DSM15 having a volume of 15 ccm with a winder of the “Micro Fiber Spin Device” (Xplore Instruments BV, Sittard, Netherland).

Melt compounding was performed at a mixing temperature of 180 °C, the rotation speed of 20 rpm and a mixing time of 5 min. After mixing the material was led out as a strand using a 1 mm nozzle and rotations speed lowered to 25 rpm. The extruded strands are not granulated because the winder is carried out with the same system.

SWCNT/PE fiber nanocomposite fibers were spun with SWCNT concentration varying starting from 0.01 wt% to 10 wt%.

### *Pilot-scale melt spinning of PA6/MWCNT-CB nanocomposite fibers*

CNT and CB-modified PA6 nanocomposite fibers were prepared from granular masterbatches containing 5 wt% CNT and 5 wt% CB in PA6. The masterbatches were fabricated in two levels by Nanocyl S.A (Sambreville, Belgium).

For the preparation of the PA6 5 wt% MWCN + 5 wt% CB compound and pure PA6 granules both dried overnight at 85 °C in a vacuum oven. A pilot-scale bicomponent melt spinning line was used for yarn production (Fourné Maschinenbau GmbH, Alfter-Impekoven, Germany) where the core and sheath materials are combined into a monofilament yarn and afterward collected. A polymer mass throughput of up to 2.5 kg/h is possible using this system. Extruder 1 (core extruder) and Extruder 2 (sheath extruder) are held at

270 °C in the final zone. Both metering pumps (core and sheath metering pumps) are held at 270 °C. The melt volume flow rate from the metering pump 1 (core) is varied between 3 and 12 g/min.

## DESCRIPTION OF THE MAIN RESULTS OBTAINED

### **Analysis**

The conductivity of the melt-spun yarns was determined by two-point DC resistance measurements and the yarns were visualized by optical microscopy.

The electrical resistance  $R\Omega$  was on single filaments with a length of about 100 mm using a DT-61 digital multimeter manufactured by MASTECH (Multimeter Warehouse, Walnut, CA, USA). The filaments are fixed between the two fasteners and contacted with two alligator clamps and bound to the multimeter. Optical microscopy was carried out to determine fiber quality using a Leica M205C (Leica Microsystems AG, Heerbrugg, Switzerland).

### **Results and discussion**

#### *PE/SWCNT nanocomposite fibers*

The influence of SWCNT concentration on electrical conductivity can be analyzed for those fibers in which the percolation threshold has been exceeded. The values for electrical conductivity (S/m) for these fibers could be measured PE/WSWCNT melt-spun fibers at the loading of 8 wt% and 10 wt% (Table 3). Concentration below 8 wt% drop so low that measurement with the used device is no possible.

Table 3. Electrical volume conductivities of PE/SWCNT nanocomposite fibers

<b>Con. (wt%)s</b>	<b>Yarn diameter [<math>\mu\text{m}</math>]</b>	<b>Conductivity [S/m]</b>
10	1045	0,91
8	760	0.06

#### *PA6/MWCNT-CB nanocomposite fibers*

Table 4 shows the average conductivity values for PA6/MWCNT-CB (each 5 wt%) nanocomposite filaments that spun with different melt volume flow rates. Increasing the melt volume flow rate from 3 g/min to 6 g/min caused up to drop by half lower conductivity while increasing the melt volume rate from 6 g/min to 9 g/min and 12 g/min, respectively, shows no substantial conductivity change for PA6/MWCNT-CB filaments.

Table 4: Electrical volume conductivities of PA6-5wt%MWCNT+5wt%CB nanocomposite filaments

<b>Sample</b>	<b>Melt volume flow rate (g/min)</b>	<b>Fiber diameter (<math>\mu\text{m}</math>)</b>	<b>Electrical Conductivity (S/m)</b>
V1	3	336,7	3,13E-02
V2	6	486,7	6,72E-03
V3	9	490,0	7,91E-03
V4	12	506,7	6,55E-03

This signifies that there is a change in sample morphology during fiber drawing, which will affect the resistivity. When the fiber morphology is changed, the percolated network structure will change, which then further affects the conductivity. In the filament samples spun with 6 g/min melt flow rate, the solid-state drawing leads possibly to a change in the orientation and the dispersion of the carbon nanotubes.

#### REFERENCES

- [1] Yetisen, A. K.; Qu, H.; Manbachi, A.; Butt, H.; Dokmeci, M. R.; Hinestroza, J. P.; Skorobogatiy, M.; Khademhosseini, A.; Yun, S. H. Nanotechnology in Textiles. *ACS Nano* 2016, 10 (3), 3042–3068.
- [2] Krause, B.; Barbier, C.; Levente, J.; Klaus, M.; Pötschke, P. Screening of Different Carbon Nanotubes in Melt-Mixed Polymer Composites with Different Polymer Matrices for Their Thermoelectrical Properties. *J. Compos. Sci.* 2019, 3 (4), 106.
- [3] Straat Martin, Rigdahl Mikael, H. B. Conducting Bicomponent Fibers Obtained by Melt Spinning of PA6 and Polyolefins Containing High Amounts of Carbonaceous Fillers. *J. Appl. Polym. Sci.* 2010, 123, 936–943
- [4] Bischoff, M.; Köhler, T.; Bandelin, J.; Möhrcke, J.; Jung, R. CNTs in Fibres : Multilayered Polymerfibers for Conductive and Sensoric Applications. 17th World Text. Conf. AUTEX 2017 - Shap. Futur. Text. No. 29-31 May 2017, 032001, 1–7.

#### FUTURE COLLABORATIONS (if applicable)

The obtained data will be considered for submitting as an article. Especially characterization measurements of PE/SWCNT and PA6/MWCNT-CB nanocomposite fibers underlines the need for further and more detailed investigations.