Thermal Properties of PEDOT-*compl*-PSS Sensor Yarns and Textile Reinforced Thermoplastic Composites

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ABSTRACT

Smart textile structures such as sensor yarns provide real possibility for in situ structural health monitoring of textile reinforced thermoplastic composites. In this work thermal properties of E-glass/polypropylene (GF/PP) and E-glass/poly(N,N'-hexamethylene adipamide) (GF/PA66) sensor yarns based on conductive polymer complex [3,4(ethylenedioxy)thiophene]-compl-poly(4-vinylbenzenesulfonic acid) (PEDOT-compl-PSS) and related composites were studied. Thermogravimetric analysis (TGA), microscale combustion calorimetry (MCC) and limiting oxygen index (LOI) methods were used to detect thermal behaviour of these structures and effect of coatings applied. According to TGA, GF/PP sensor yarn started to decompose at higher temperature, 345 °C, and showed higher pyrolysis residue, 28 %, compared to GF/PA66 sensor yarn that started to decompose at 316 °C and had lower pyrolysis residue, 23 % . The MCC showed that Heat Release Rate peaks of GF/PP sensor yarn, 341 W/g, and GF/PA66 sensor yarn, 348 W/g, occurred at similar Heat Release Temperature, ~ 430 °C. The additional peak, 51 W/g, was detected for GF/PP sensor yarn at 493 °C. Finally, LOI 22 and LOI 23 were detected only for GF/PP and GF/PA66 composites with integrated sensor yarns.

KEYWORDS

Smart textile, PEDOT-compl-PSS, textile sensors, textile reinforced composites, thermal properties

INTRODUCTION

Smart textile structures can be made by coating or treating textile yarns, filaments, or fabrics with conductive and semi conductive polymers. They provide real possibility for *in situ* structural health monitoring of textile reinforced composites [1-2]. Commercially very useful conductive polymer complex poly[3,4 (ethylenedioxy)thiophene]-*compl*-poly(4-vinylbenzenesulfonic acid) (PEDOT-*compl*-PSS) produce transparent coatings with high mechanical flexibility, excellent thermal stability and ease of synthesis [3]. Textile reinforced thermoplastic composites can be used for transportation applications due to their high fracture toughness, recycling possibility, damage tolerance, etc. [3-5] Polypropylene (PP) and poly(N,N'-hexamethylene adipamide) (PA66) as polymer matrices have been widely taken for automobile applications [6]. Glass fibres are suitable reinforcements in composites and are characterised by hardness, resistance to chemical agents, insulating properties, etc. [7] Textile materials are very flexible in all directions and sensors used should be able to support mechanical deformations [8]. In this work thermal properties of sensor yarns and composites were studied to detect thermal behaviour of these structures and effect of coatings applied.

EXPERIMENTAL

Materials and Methods

E-glass/polypropylene (GF/PP) and E-glass/poly(N,N'-hexamethylene adipamide) (GF/PA66) commingled yarns by PD Fiberglass group (Glasseiden GmbH, Oschatz, Germany) were used for sensor yarns manufacturing. Fineness of GF/PP is 842 tex (GF/PP mass content of 71%:29%), while fineness of GF/PA66 yarn is 957 tex (GF/PA66 mass content of 65%:35%) [2-3]. A novel piece of laboratory equipment, aluminum roll to roll device and plexiglass bath, was taken to ensure effective and equally distributed coating onto yarn without destruction of textile properties. During the sensor yarns manufacture, two layers of conductive coating based on polymer complex PEDOT-*compl*-PSS were applied. The aqueous dispersion of copolymers of acrylic esters (synthetic latex) was used also as protective coating to join yarn filaments together and protect sensor yarns from abrasion [3].

Finally, sensor yarns based on PEDOT-*compl*-PSS were integrated during weaving 2D fabric (thickness ~2.660 x 10^{-3} m), 4-end satin, in weft direction, using computer controlled hand weaving loom (ARM, Biglen, Switzerland); GF/PP fabric (warp density, 4 ends/cm and weft density, 6 ends/cm) or GF/PA66 fabric (warp density, 5 ends/cm and weft density, 6 ends/cm). Three-layered textile preforms with integrated sensor yarns were consolidated at the Dolouets heating press (Soustons, France) under the strict conditions (Table 1) to develop composites with integrated sensor yarns.

Table 1. Consolidation conditions of 2D textile preforms with integrated sensor yarns

Conditions		
Three-layered 2D fabric for textile preform preparation	GF/PP	GF/PA66
Sensor yarns	GF/PP	GF/PA66
Temperature, T (°C)	185	230
Pressure, P (MPa)	4-5	4-5
Time of cooling at 100 °C (min)	2-3	3-4

Thermogravimetric analysis (TGA), microscale combustion calorimetry (MCC) and limiting oxygen index (LOI) methods (average of three samples per each structure) were used for thermal properties determination of dry films (conductive and protective coatings), non-coated and sensor yarns, and textile reinforced 2D thermoplastic composites with integrated sensor yarns.

TGA (5 mg test samples) was carried out (TGA Q50, TA Instruments, New Castle, DE, USA) in nitrogen atmosphere under the following conditions: flow rate of 50 mL/min and heating rate of 10 °C/min over the temperature range from 50 °C to 600 °C to achieve data of coating effects on the treated yarns, pyrolysis residues and the temperature of sample decompositions.

MCC tests were performed using microscale combustion calorimeter, model MCC-2 (Govmark, Farmingdale, NY, USA) according to the ASTM D 7309 standard. In the MCC test, 5 mg test samples were heated from 75 to 600 °C at the heating rate of 1 °C/s, in an inert gas stream (nitrogen, 1.33 ml/s).

LOI measurements were performed on the LOI instrument (Dynisco, Franklin, MA, USA) according to the ISO 4589-1:1996 and the ISO 4589-2:1996 standards to obtain flammability data of textile reinforced 2D thermoplastic composites with integrated sensor yarns.

RESULTS AND DISCUSSION

Thermogravimetrical data of dry films (conductive and protective coatings), non-coated and sensor yarns are shown in Table 2 and in Figure 1.

Table 2. Thermogravimetric data of dry films, non-coated and sensor yarns

Sample Description	Sample Label	Initial Decomposition Temperature, T_{ign} (°C)	Final Decomposition Temperature T_{endset} (°C)	Yield of Pyrolysis Residue, Y_p (%)
Conductive Dry Film	8 % PEDOT-compl-PSS FET- LApp96100DF	233	480	7
Protective Dry Film	Lapp96100DF	328	447	4
Non-coated Yarn	GF/PP	344	447	72
Non-coated Yarn	GF/PA66	365	482	67
Sensor Yarn	GF/PP-Sy08	345	483	28
Sensor Yarn	GF/PA66-Sy08	316	470	23

Conductive dry film, 8 % PEDOT-*compl*-PSS FET LApp96100DF, started its degradation earlier than protective dry film, LApp96100DF, while its final decomposition temperature was higher. Conductive dry film showed also higher pyrolysis residue. GF/PP yarn started to decompose first, and its degradation ended earlier compared to GF/PA66 yarn.

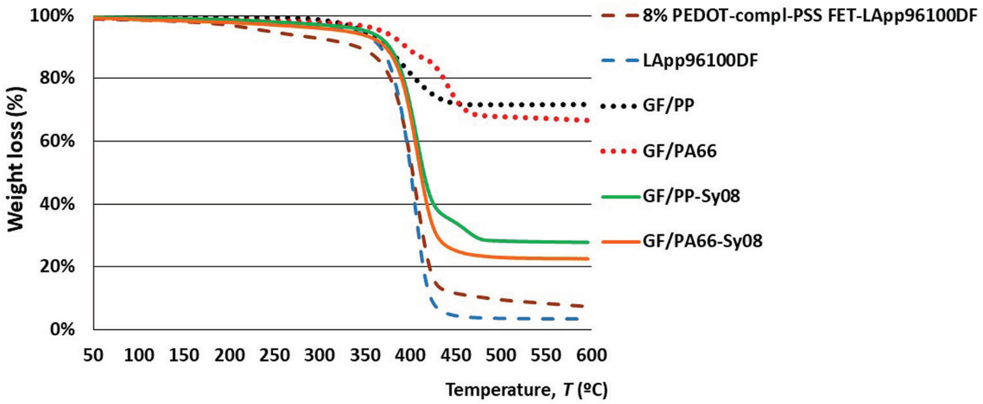

Figure 1. TGA curves of dry films, non-coated and sensor yarns

This can be explained with a lower decomposition "phase" of PP compared to the PA66 thermoplastic polymer. GF/PP sensor yarn started to decompose at higher temperature than GF/PA66 sensor yarn. The cause could be greater coating thickness of GF/PA66 sensor yarn that showed also lower pyrolysis residue. The MCC data of dry films (conductive and protective coating), non-coated and sensor yarns are presented in Table 3. The results of Heat Release Rate, HRR (W/g), in correlation with the maximum temperature, T_{max} (°C) are shown in Figure 2.

Table 3. MCC data of dry films, non-coated and sensor yarns

Sample Description	Sample Label	Heat Release Capacity, H_c (J/G-K)	Maximum Specific Heat Release, Q_{max} (W/G)	Heat Release Temperature, T_{max} (°C)	Yield of Pyrolysis Residue, Yp (%)
Conductive Dry Film	8% PEDOT-compl-PSS FET LApp96100DF	358	362	427	8
Protective Dry Film	LApp96100DF	476	483	430	2
Non-coated Yarn	GF/PP	302	303	485	73
Non-coated Yarn	GF/PA66	233	216	447	61
Sensor Yarn	GF/PP-Sy08	337	341/51	434/493	25
Sensor Yarn	GF/PA66-Sy08	344	348	430	21

Conductive and protective dry films provided high HRR peaks (362 W/g and 483 W/g) and low Yp (8 % and 2 %). Non-coated yarns, GF/PP and GF/PA66, showed lower HRR peaks (303 W/g and 216 W/g) and higher Yp (73 % and 61 %). The HRR peaks of GF/PP (341 W/g) and GF/PA66 (348 W/g) sensor yarns occurred at similar Heat Release Temperature, Tmax (~ 430 °C). The additional peak (51 W/g) was detected for GF/PP sensor yarn at 493 °C. Finally, MCC pyrolysis residues were coherent with the obtained TGA residue values.

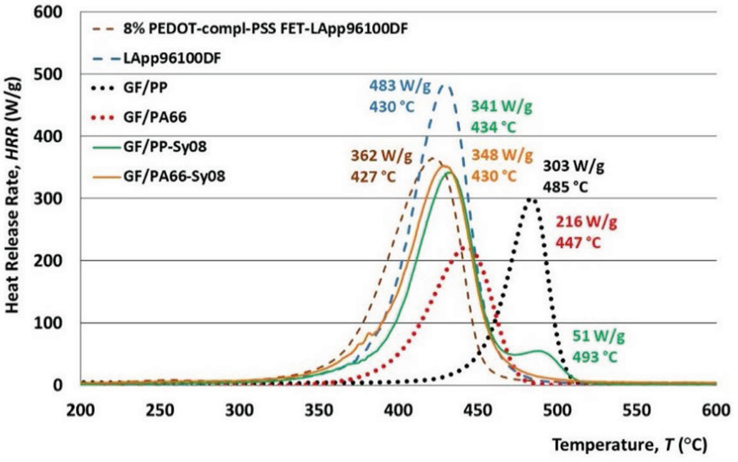

Figure 2. MCC curves of dry films, non-coated and sensor yarns

LOI measurements of textile reinforced 2D thermoplastic composites with integrated sensor yarns are presented in Table 4.

Table 4. LOI of textile reinforced 2D thermoplastic composites with integrated sensor yarns

Sample Description	Sample Label	Time, t (s)	LOI
GF/PP Composite with Integrated GF/PP Sensor Yarn	GF/PPcmp-GF/PP-Sy08	379	22
GF/PA66 Composite with Integrated GF/PA66 Sensor Yarn	GF/PA66cmp-GF/PA66-Sy08	237	23

During heating PP or PA66 polymer softened, melt and dripped. GF/PP composite with integrated GF/PP sensor yarn showed LOI 22 . PP burns rapidly with a smoke-free flame, with no char residue left and can aid in fire propagation. On the other hand, GF fibres, as inorganic material, do not burn or support combustion, but start to deform when the temperature reaches 500 °C or above [9]. GF/PA66 composite with integrated GF/PA66 sensor yarn showed LOI 23 due to different thermal consolidation conditions of 2D textile preform prepared.

CONCLUSION

GF/PP sensor yarn started to decompose at higher temperature than GF/PA66 sensor yarn due to greater coating thickness applied onto the GF/PA66 commingled yarn during sensor yarn manufacture. MCC pyrolysis residues were coherent with the obtained TGA residues. Higher HRR peaks of sensor yarns occurred at slightly lower temperature compared to non-coated yarns. GF/PP and GF/PA66 composites with integrated PEDOT-*compl*-PSS sensor yarns showed low LOI and fire.

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