Solid State Phenomena
ISSN: 1662-9779, Vol. 333, pp 11-20
doi:10.4028/p-9q3n92

Submitted: 2021-10-13
Revised: 2021-12-16
Accepted: 2021-12-16
Online: 2022-06-10

Protective Coating for Electrically Conductive Yarns for the Implementation in Smart Textiles

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Keywords: silver plated, polyamide yarn, embroidery, resistance, mechanical impact, textile sensor

Abstract. The Cluster of Excellence "Centre for Tactile Internet with Human-in-the-Loop (CeTI)" [1] deals with developments and inventions concerning smart devices used in many fields, e.g. industry 4.0, medicine and skill learning. These kind of applications require smart devices, sensors, actors and conductive structures. Textile structures address these applications by meeting requirements such of being flexible, adaptable and wearable.
Within this paper, the development of a protective coating for electrically conductive (EC) yarns is captured. These EC yarns are nowadays often used for smart textile applications. One challenge in their application is the integration into textile structures. Often, the handling and usage of EC yarns leads on the one hand to damages on the surface of the yarn and on the other hand to reduced electromechanically characteristics. This paper aims to characterize these EC yarns in regard to develop a suitable protective coating based on polypropylene (PP). To achieve this development, an extensive characterization of the EC yarns as well as the protective coating itself is important. The surface free energy (SFE), the topographical and the chemical characteristics are necessary for developing a suitable protective coating. However, the yarns are characterized before and after implementation into the textile structure and furthermore after the coating respectively with the developed finish.

Introduction

Smart textiles gain more and more interest and applications in our society. By implementing electronic components into textile garments and technical textiles, these products are created. One application of sensor yarns is the implementation in smart textiles i.e. in sportswear, health care and work safety [2]. Here, they can be used as elastic or stretchable sensors [3]. To make these electrical components combinable with our everyday live and with the handling in production is a big challenge [2]. The electronic components implemented in textiles have different functions and forms. Moreover, they can be classified into sensing and actuating units. Electrically conductive (EC) materials in yarn shape dominate the first group. Here, different materials like stainless steel, core-sheath yarns and wrapped metal yarns are first thought products [2 ,4, 5] .These yarn shaped sensors can be implemented via knitting, weaving or embroidery [2, 6]. Another method to create EC components in or on textiles is a functionalized surface. Methods are e.g. chemical metallization or etching. Further methods are printing [6], coating and lamination of electrical conductive materials [2, 4, 5].

EC materials are metals, polymers (intrinsic or extrinsic) [5], filaments and fibers e. g. carbon fibre and stainless steel yarn [2]. Additionally, the attention of this paper lies on polymeric metal-plated yarns. Despite the following section deals with state of the research of EC materials shaped like yarn or even beeing yarn. Beside silver-plated EC yarns, there are also other yarn-shaped EC structures. Uzun *et al.* developed a cellulose yarn coated with MXene. MXene are two-dimensional transition metal carbides and nitrides, they are highly electronically conductive [7]. Furthermore, Islam *et al.* developed flexible melt spun thermoplastic EC yarns based on polypropylene (PP) and polylactid acid (PLA), coated with polydopamine (PDA) and poly(3,4-ethylenedioxy thiophene): poly(styrenesulfonate) (PEDOT:PSS) [4].

Metallized yarn offer currently the smallest and most flexible EC structures on textile basis. Because of their textile basis they can be more easily integrated by typical textile technologies i.e. knitting, weaving or embroidery. Nevertheless, the protection of the EC yarn or textile structure is challenging. Through the sizing/avivage of the producer and the surrounding textile, the EC yarn has a minimum of protection. However, the used textile technologies and the daily usage of the smart textiles gives high mechanical impact on the EC yarn. The impact causes abrasion and the resistance of the EC yarn increases and electrical conductivity decreases.

To analyse the origin of these damages various research projects have been done. Ismar *et al.* analysed the effect of water and chemical stresses on silver-plated polyamide (PA) yarns. They state that washing of *e*-textiles is one of the major hurdles that have to be faced nowadays. Additionally, it is proven by FTIR-ATR, UV-Vis spectroscopy and electrical resistance measurement that water damages the yarn more than the washing detergent does. The damage is originated in the removal of the silver layer in water and washing detergent solution [8]. Conductive structures, which are available on the market such as silver-coated yarns, do not have enough abrasion resistance for the implementation via knitting in an industrial scale. Furthermore, they are damaged via the usage in smart textiles by wearing, washing and drying [2]. To protect these yarns against the mechanical impact arose by these scenarios a protective coating should be developed. Next to this, research has been done concerning the protection of metal-plated EC yarns. Baribina *et al.* examined a water-repellent coating for PA yarn plated with silver. The analyses are fulfilled with textured and non-textured yarn and the coating should improve the conductive behaviour after several washing cycles to further improve the lifetime of the product. To achieve this, a silicone and nano-coating are used and applicated with a brush directly and discontinuous on the fiber [9]. Within another project a protective coating out of PP for PA yarns plated with silver is developed. Alagirusamy *et al.* used PP staple fibers to wrap around silver-coated yarns. Afterwards the yarns is put in the oven and the PP is melted to an homogenous sheath [10]. Further, Raja *et al.* used poly vinyl pyrrolidone (PVP) for stabilization of silver nanoparticles in watery solutions as well as for the application on textiles. Within this work the used substrates are wool and cotton based and the application is microbial resistance [11]. On the market exists one yarn produced by Statex, which is covered in thermoplastic polyurethane (TPU). However, this protection is made in shape of a loose shell with no mounting on the EC yarn. To increase the protection against mechanical impact via implementation, wearing, washing and drying this paper claims that a direct coating has to be brought onto the fiber. So within this paper a development or rather application of suitable coating should be made, that has good adhesion to TPU or better is directly applicated onto the silver-coated PA and would lead to higher abrasion resistance and with that better processability.

A protective coating has to meet some requirements besides the later on application of the conductive yarn concerning the implementation and usage. The coating should not obstruct the EC characteristics. Moreover, the coating should be temperature stable and glideable or should show less frictional resistance to guarantee good processability in textile processes. Furthermore, the coating should have sufficient elasticity but at the same time sufficient adhesion to the silver layer on the PA yarn. Finally, the coating should be isolating or electrical conductive depending on the use case.

For usage with the silver-coated polymeric yarns, the coating should be non-abrasive, strong but also flexible. For the appliance, the process temperatures for the coating should not be higher than

the glass temperature of the polymeric material of the yarn. Therefore, the coating materials have to be melted underneath this temperature or should be applicable as a solution.

In preliminary studies, various polymeric materials are tested as coating for EC yarns. During these tests, waxes became of major interest in particular as watery emulsions. At first, PP is chosen as basis for the coating due to their excellent characteristics concerning temperature resistance, strength and availability. However, PP does not fulfil all set requirements for the coating. The most disadvantageous characteristic of PP is its inelasticity. To weaken this characteristic in the final coating and to ensure the usability especially in smart textiles a further polymer (polyethylene (PE)) and an additive (glycerine) are added.

To determine the impact of the coating the optical analysis (light and scanning electron microscopy) and resistance measurement are used to analyse the reference yarns in comparison to the coated yarns. Furthermore, the reference yarns are characterized concerning their surface energy with tensiometric analysis and their chemical composition is analysed with Fourier-transformed infrared spectroscopy. In addition, the coating is analysed with these two methods but also with thermogravimetric analysis.

Methods and Materials

Silver-plated yarns. In Table 1 the yarn materials are shortly presented due to their characteristics given by producer. All yarns are PA 6.6 based and plated with silver.

Table 1. Electrically conductive yarns used as basis material

Producer	Name	Abbreviation	Composition	Titer [dtex]	Resistance [Ω·m^{-1}]
Madeira	HC40	HC40	PA 6.6	117x2	< 300
Statex	Shieldex 117/17 dtex 2PLY HC+B	S HC+B	PA 6.6	117/17 x2	< 300
Statex	Shieldex 117/17 dtex 2PLY	S	PA 6.6	117/17 x2	< 1500

Coating materials. The following table shows the used components for the coating.

Table 2. Components used for the coating

Producer	Name	Material composition
Deurex AG	P 3601 W	Water-based emulsion of PP wax
	EO 4001 W	Water-based emulsion of oxidized wax
Carl Roth GmbH + Co KG	Solvagreen	Glycerine, ≥98% water free

Coating process. The coating has a basis of PP wax solved in a watery emulsion combined with a watery emulsion of PE and as slip additive glycerine. The two wax emulsions are each added with an amount of 49 vol.-% while the glycerol is added with 2 vol.-%. The coating is stirred with a magnetic stirrer for 30 minutes at room temperature with a stirring rate of 180 rpm.

With the Lab Foulard HVF from Mathis AG (Oberhasli, Swiss) the yarns are coated with a barrel speed of 2 m·min^{-1} and a barrel pressure of 1 bar. For leading the yarns through the coater, a yarn tube is placed above the barrels. After the yarns went through the barrels, they are winded on cardboard tubes for drying under constant conditions in a drying cabinet at 30 °C for about 30 minutes.

Contact angle measurement. With the contact angle measurement, the surface free energy (SFE) of the reference yarns is calculated and assessed. Later, also the coatings SFE is determined and theoretical compatibility of both, yarn and coating is compared.

The EC yarns are measured using Washburn method also known as capillary method. Here, the yarn is cut into small pieces with a length of about 5 mm. Afterwards these short fibers are filled into a glass and metal sample holder with a filter paper at the bottom. The sample holder then is put into n-hexane (>99%, Carl Roth GmbH + Co KG (Karlsruhe, Germany), surface tension at 23 °C, 18.40 mN·m^{-1}) to determine the capillary constant. Afterwards, the measurement is performed with a minimum two different test liquids. These test fluids are deionized water (surface tension at 23 °C, 72.8 mN·m^{-1}) and diiodomethane (>99%, Sigma-Aldrich Chemie Gmbh (Darmstadt, Germany),

surface tension at 23 °C, 50.8 $mN \cdot m^{-1}$). The performed measurement is static and takes 120 s. Per sample five measurements are performed. As mentioned before the coating is also characterized via tensiometric analysis. The measurement is static and sessile drop method is used. For this, the coating is prepared as a film on a glass objective. The film is dried for 24 hours at room temperature.

The measurement is performed following DIN 55660-2 with a minimum of two test fluids (deionized water and diiodomethane). The method of Owens, Wendt, Rabel and Kaeble (OWRK) is used to determine the polar and disperse part of SFE of the specimen [12,13]. Eq. 1 describes the relation of the contact angle and polar and disperse surface tensions:

$$\frac{(1+\cos\theta)\cdot\sigma_l}{2\cdot\sqrt{\sigma_l^D}} = \sqrt{\sigma_s^P}\cdot\sqrt{\frac{\sigma_l^P}{\sigma_l^D}}. \quad (1)$$

The contact angles (θ) are used to calculate the SFE. Furthermore, the polar (σ_P) and disperse (σ_D) parts of the SFE are determined with this formula. The indices l and s are describing the liquid and solid states, from i.e. the test liquid and the filament surface. Krüss Force Tensiometer K100 (Hamburg, Germany) is used to determine the contact angles between the test liquids and the yarns and Drop Shape Analyzer (DSA) 100 from Krüss is used for the SFE measurement of the coating. The Software Advance from Krüss GmbH (Hamburg, Germany) is used to perform the measurement and to calculate the SFE from the contact angles.

Resistance measurement. The determination of the electrical resistance is used to quantify and assess the conductive behavior of the yarns before and after being coated and as well after being mechanically demanded. The measurement is performed with a self-constructed four-terminal sensing measuring station with a specimen length of 500 mm ten times per specimen. To define the values of the measurements a voltmeter (2) and a constant current source (1) are used. The specimen (3) is contacted via four Kelvin clamps (4). Fig. 1 below shows the circuit diagram of the measuring station.

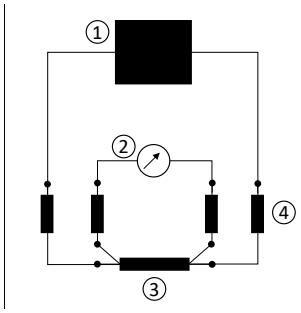


Fig. 1. Four-terminal sensing measuring station

Optical analysis. The evaluation of the yarns surface is determined with the light microscope (LM) Scope A.1 Axio from Carl Zeiss Microscopy Deutschland GmbH (Oberkochen, Germany) with incident light. The measurement of the diameters is performed with the software Zen2Core from Zeiss and five measurements per sample are made. Further, a scanning electron microscope (SEM) Quanta 250 FEG SEM from FEI (Hilsboro Oregon, United States of America) with the magnitudes 500 and 1000 is used.

Fourier-transformed infrared spectroscopy. Fourier-transformed infrared attentuated total reflection (FTIR-ATR) spectroscope Nicolet 6700 from Thermo Fisher Scientific GmbH (Waltham, Massachusetts, United States of America) with a diamond is used for the determination of the yarns composition.

Thermogravimetric Analysis. Thermogravimetric analysis (TGA) is performed with TGA Q500 from TA Instruments (New Castle, Delaware, United States of America). Measurements are done in nitrogen atmosphere from 30 °C to 350 °C in high-resolution mode.

Proof of concept – embroidery. To examine the developed coating a proof of concept was performed. Within this investigation the silver-plated and coated yarn was threaded through all guiding elements of the embroidery plant F-Kopf from ZSK Stickmaschinen GmbH (Krefeld,

Germany). Part of the guiding elements are the preload clamp, main road roller, thread take-up lever, fabric pusher and needle.

Results

Composition. Fig. 2 shows the FTIR-ATR spectra of the yarns references. At approximately 3300 cm^{-1} and 3100 cm^{-1}, the samples of HC40 and Statex show stretching vibration of N-H group. The band round 3000 cm^{-1} hints towards stretching vibrations of C-H groups as well as the band at 2850 cm^{-1}. At approximately 1700 cm^{-1}, the stretching vibration of C=O bond can be found. The bands at 1500 cm^{-1} and 1600 cm^{-1} hint towards C=C bonds. These, in turn, are hinting towards the amide groups of the PA. The band at approximately 1250 cm^{-1} is suggesting again a C-N stretching vibration hinting towards amide groups.

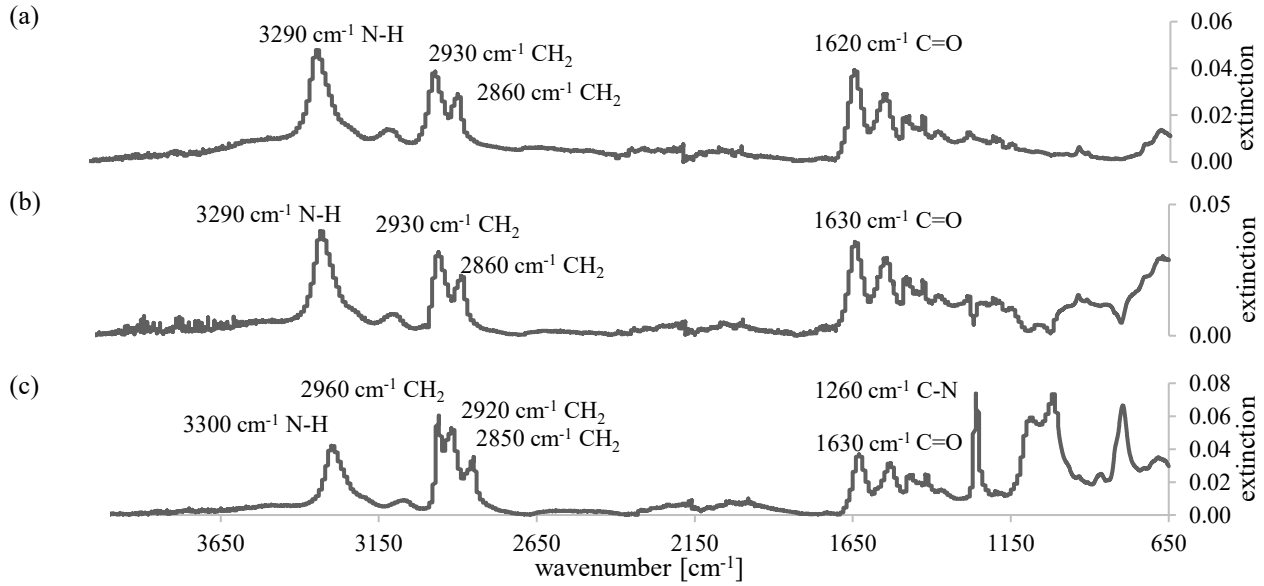


Fig. 2. FTIR-ATR spectroscopy of reference yarns (a) S, (b) S HC+B and (c) HC40

Surface characteristics. Table 3 shows the values determined with the Washburn method via tensiometric analysis for the yarns reference. On the first sight, it is obvious that the SFE of all reference yarns have similar values of about 21 to 23 $mN \cdot m^{-1}$. Furthermore, all samples show small deviations and even polar and disperse parts of the yarns have similar values. Overall, the polar part of the yarns is smaller than the disperse ones.

Table 3. Surface free energy of yarns reference determined with WASHBURN method

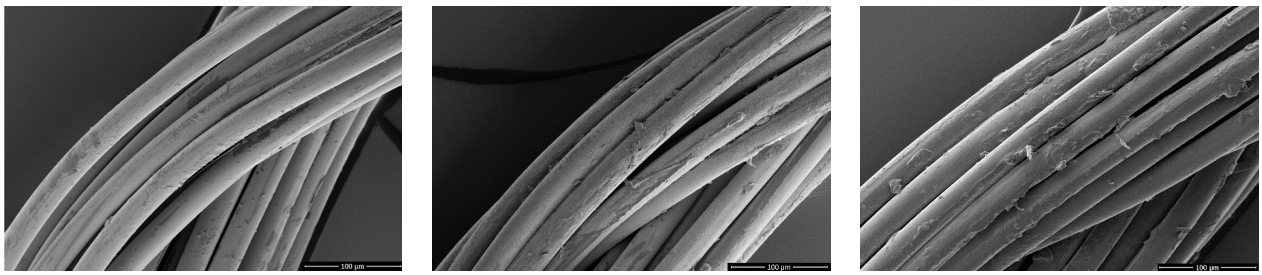
	S	S HC+B	HC 40
SFE, total [$mN \cdot m^{-1}$]	21.96 ± 0.22	22.99 ± 0.68	20.98 ± 0.50
SFE, disperse [$mN \cdot m^{-1}$]	15.58 ± 0.16	16.82 ± 0.45	13.83 ± 0.50
SFE, polar [$mN \cdot m^{-1}$]	6.37 ± 0.07	6.17 ± 0.23	7.15 ± 0.03

Table 4 shows the measured contact angles from the reference yarns to the test liquids (diiodomethane and water). Here, the yarns show similar values concerning the contact angle to water. The contact angles to diiodomethane are slightly smaller than the ones to water. Looking at Table 3 with increasing contact angle to diiodomethane the disperse part of the SFE decreases.

Table 4. Contact angles of yarns reference determined with WASHBURN method

	S	S HC+B	HC 40
water [°]	89.91 ± 0.03	89.23 ± 0.31	89.92 ± 0.03
diiodomethane [°]	83.82 ± 0.32	81.32 ± 0.90	87.50 ± 0.77

Further, the references are characterized optically with LM and SEM. The following images in Fig. 3 are taken with a magnitude of 500. On each of the yarns surfaces particles, small abrasions and damages can be seen. S yarn (Fig. 3 (a)) shows the smoothest surface compared to the other yarns.



(a) (b) (c)

Fig. 3. Scanning electron microscopy images of (a) S, (b) S HC+B and (c) M HC40

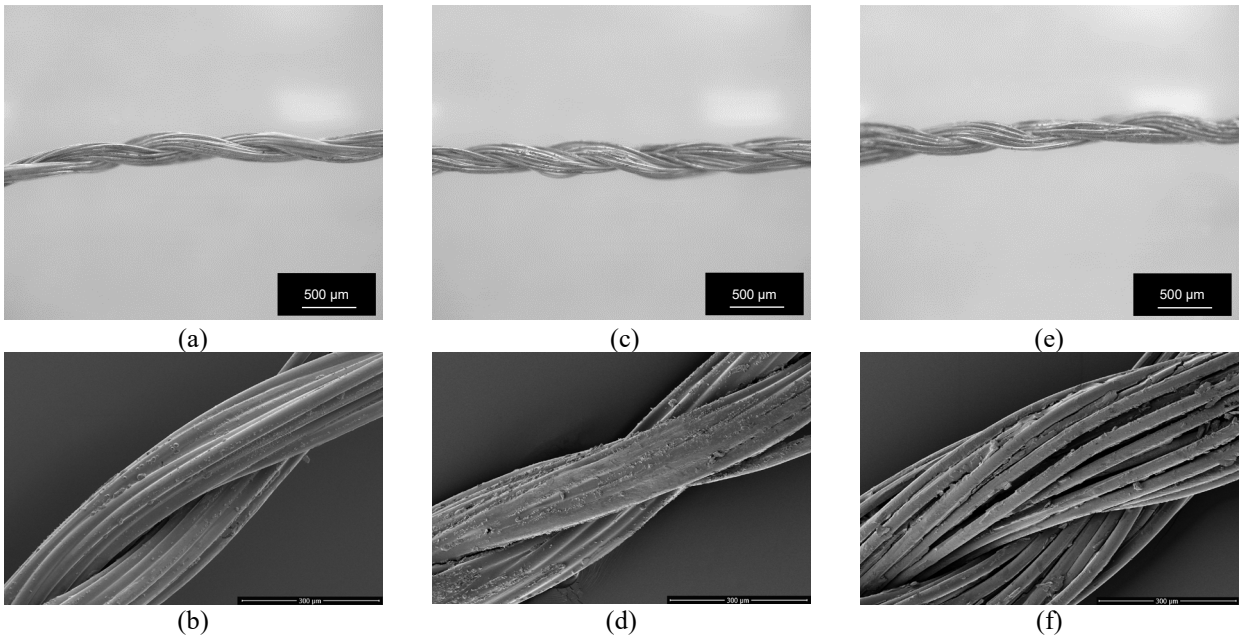
The images are showing no obvious difference concerning the diameter of the filaments comprised in the yarns. Table 5 shows the diameters of the yarns.

Table 5. Filament diameter of reference yarns

Yarn	Diameter of filament [µm]
HC40	28.53 ± 0.82
S HC+B	27.64 ± 0.78
S	28.18 ± 0.73

The surface of the coated yarns is analysed with the LM. The following images (Fig. 4 (a) to (c)) show the coated yarns surface. Because the coating has no opaque colour, the yarns look similar to the reference. Furthermore, the coating is very thin, so the silver-plating is still visible.

Moreover, SEM images with a magnitude of 1000 of the coated yarns are shown in Fig 4 (d) to (f). Here, S yarn shows the smoothest surface with fewest deposits. S HC+B shows some small deposits on the surface but simultaneous the coating builds a closed surface around the filaments of the yarn. In contrast, the coating seems very brittle on the HC40 yarn. It is visible that the yarn is coated but there are gaps between the single filaments of the yarn and deposits on the filaments surface.

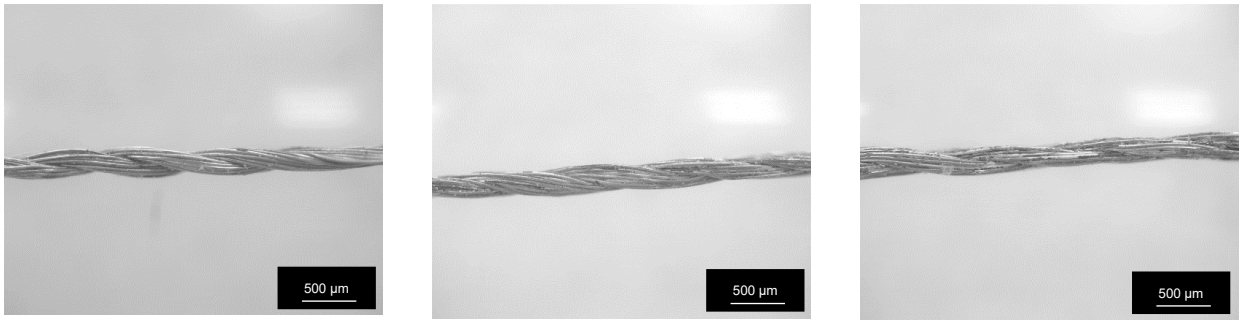


(a) (c) (e)

(b) (d) (f)

Fig. 4. LM and SEM images of coated yarns: (a) and (b) S, (c) and (d) S HC+B and (e) and (f) HC40

Furthermore, the coated and embroidered yarns are also characterized using LM. Fig. 5 shows the images of all three yarns. Both Statex yarns show almost smooth surface while HC40 shows a damaged surface.



(a) (b) (c)

Fig. 5. LM images of coated and embroidered yarns: (a) S, (b) S HC+B and (c) HC40

Electrical characteristics. Table 6 shows the electrical resistance values of the reference, embroidered, coated and embroidered and coated yarns. The second column constitutes the results of the reference yarns. S yarn from Statex shows the highest resistance with a value of $293.20 \pm 13.07\ \Omega \cdot m^{-1}$. The lowest value is represented by S HC+B with a value of $143.98 \pm 3.83\ \Omega \cdot m^{-1}$. HC40 occurs with a resistance of $163.98 \pm 1.96\ \Omega \cdot m^{-1}$. While the two last named yarns show very small deviation the deviation of S yarn is higher, but within the confidence interval with 4.67%.

S yarn after being embroidered exhibits a resistance of $833.00 \pm 237.04\ \Omega \cdot m^{-1}$, which leads to an increase of 284% compared to the reference. S HC+B has a resistance of $215.40 \pm 14.62\ \Omega \cdot m^{-1}$ which is about 150% of the reference. Finally, HC40 has a value of $210.40 \pm 4.80\ \Omega \cdot m^{-1}$ which is about 128% of the reference value (Table).

The resistance of the coated yarns is also determined with the four-terminal sensing measurement set-up (Table 6). S yarn increased about 2.17% to a value of $299.57 \pm 8.52\ \Omega \cdot$m-1 while S HC+B decreased about 20.68% to a value of $114.21 \pm 20.61\ \Omega \cdot$m-1. HC40 increased about 24.36% to a value of $203.79 \pm 20.07\ \Omega \cdot$m-1. In contrast to the values of the reference, the coated yarns show a higher deviation. Just S yarn shows smaller deviation than the reference.

Finally, the resistance of the coated and embroidered yarns is measured with the four-terminal sensing measurement set-up. HC40 shows the lowest resistance with an increase compared to the embroidered reference of about 133%. After S HC+B firstly decreased the resistance now increases compared to the embroidered reference of about 160%. S yarn shows also embroidered the highest value but compared to the embroidered reference it is 56% lower.

Summed up S yarn shows within the references the highest resistance value while S H+B and HC40 are similar in resistance. Furthermore, the resistance of all reference yarns increases after embroidery, headed from S yarn. The coating has little impact on S yarn, decreases the resistance of S HC+B while the resistance of HC40 increases. After being embroidered S yarn shows the lowest increase of resistance followed by HC40 and S HC+B.

Table 6. Resistance of reference, coated yarns and embroidered yarns

Yarn	Resistance [$\Omega \cdot m^{-1}$]			
	Reference	Embroidered	Coated	Coated and Embroidered
S	293.20 ± 13.70	833.00 ± 237.04	299.57 ± 8.52	467.79 ± 79.09
S HC+B	143.98 ± 3.83	215.40 ± 14.62	114.21 ± 20.61	344.94 ± 50.57
HC40	163.87 ± 1.96	210.40 ± 4,80	203.79 ± 20.07	280.77 ± 39.09

Coating – composition. To characterize the chemical composition of the final polyolefin based coating it is analysed with FTIR-ATR spectroscopy. Fig. 6 shows the spectrum of a dried sample of (a) the coating, (b) the PE wax and (c) the PP wax. The band at 2950 cm^{-1} gives a hint on asymmetrical stretching of CH_3 and occurs in Fig. 6 (a) and (c) as the methyl group of PP. Furthermore, the band at approximately 2920 cm^{-1} depicts asymmetric stretching of CH_2 and is part as well in PE and PP. Next to this distinctive peak at 2850 cm^{-1} hints towards symmetric stretching of CH_2 and furthermore towards the methylene group of PE. The band at 1460 cm^{-1} depicts bending deformation caused from the PE and the band at 1380 cm^{-1} depicts symmetrical deformation bending of CH_3 caused by PP

parts. A further band within the finger print area at 837 cm^{-1} points on rocking of C-C bonds evoked by PP.

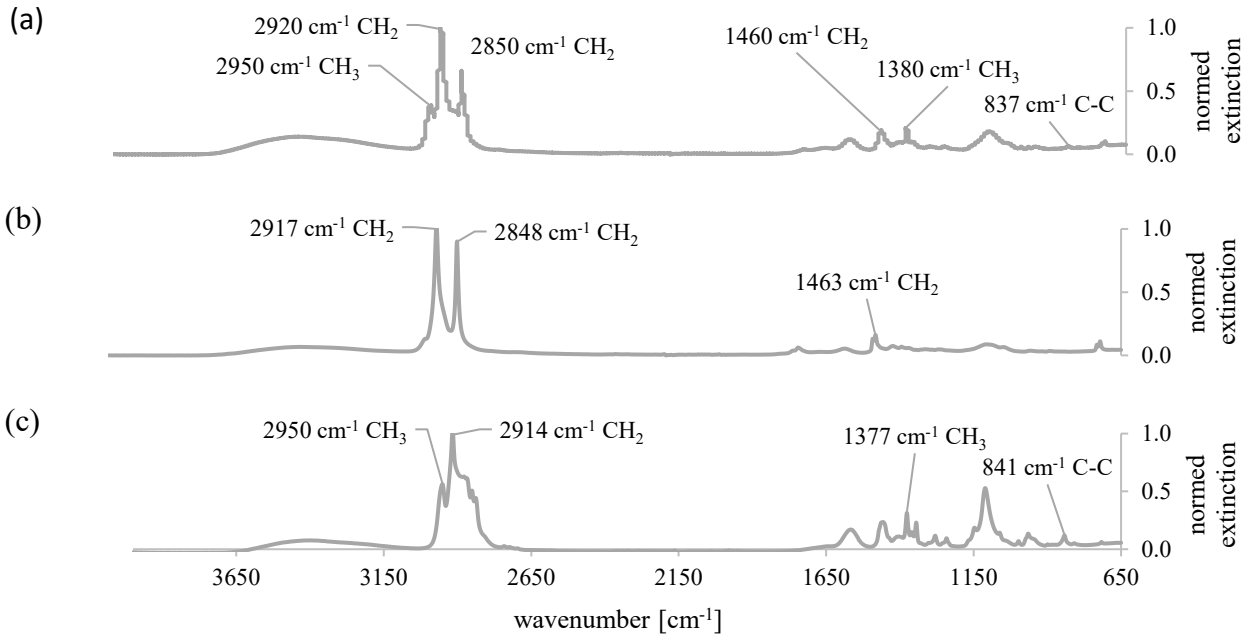


Fig. 6. FTIR-ATR spectrum of the *(a)* developed coating. *(b) E 4001 W and (c) P 3601 W*

Coating. Fig. 7 shows the result of a TGA made of the developed dried coating. At a temperature of 330 °C, there is a residue of 87.08%. The first mass loss emerges with 1.41% ending at 109.99 °C. Further mass loss of 1.38% ends at 159.90 °C. The biggest mass loss occurs with 6.82% ending at 289.97 °C. The last loss of 2.81% ends at 334.39 °C.

To evaluate the results of the TGA the following data concerning the decomposition temperature of the individual components is given. Glycerines decomposition begins at 290 °C, PE decomposition begins at 300 °C and PP decomposition begins at 330 °C. Originated in these values the first mass loss points on the vaporization of water bonded to the coating. This water is a residue from the wax emulsions used as basis for the developed coating.

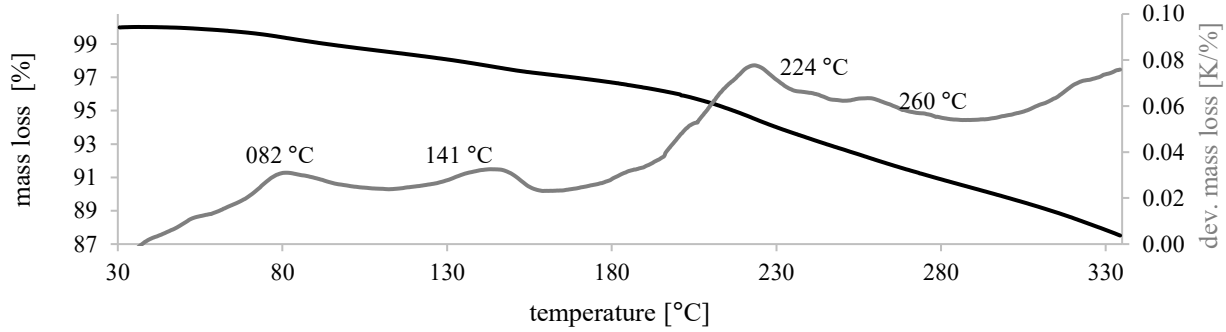


Fig. 7. TGA of coating

Fig. 8 shows the results from sessile drop measurement and the results determined with the Washburn method for the reference yarns. In contrast to the yarns the coating shows a very high SFE with a value of 52.79 ± 4.10 $mN{\cdot}m^{-1}$. The polar part occurs with 32.88 ± 3.13 $mN{\cdot}m^{-1}$ what is about two thirds of the whole value. Rather, the polar part of the yarns is one third of the whole value. Furthermore, the contact angle to water is smaller with a value of 45.78 ± 4.13 $mN{\cdot}m^{-1}$. The contact angle to diiodomethane is similar to the ones of the yarn with 75.40 ± 1.81 $mN{\cdot}m^{-1}$. Moreover, the deviation of the SFE of the coating are higher with a maximum of 9.02% concerning the contact angle of water.

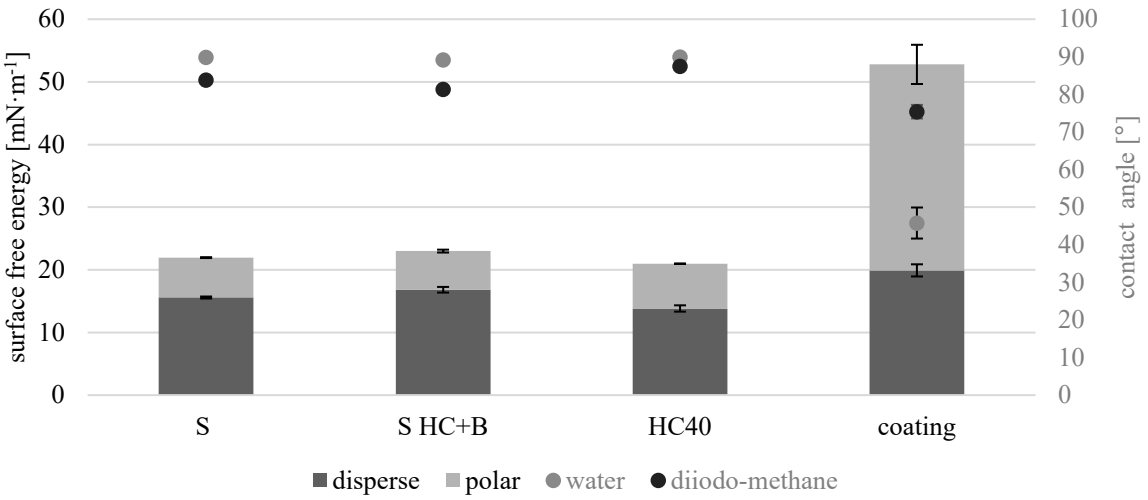


Fig. 8. Surface free energy and contact angle of coating and reference yarns

Discussion

The results of the performed studies state that the coating process itself does not have a negative effect of the characteristics of the yarn. Shieldex Yarn 117/17 dtex 2PLY HC+B (S HC+B) even shows a decrease in electrical resistance after being coated. Further, the developed coating shows better optical results on Statex yarns than on HC40. Nevertheless, HC40 shows the best electrical resistance after being coated and embroidered even if it shows the most damages compared to the other two yarns. The cause of these damages could be the non-sufficient adhesion between HC40 and the developed coating. An adaption of the composition of the coating is desirable for HC40 also regarding the electrical resistance. Shieldex 117/17 dtex 2PLY (S) shows the effect of the protective coating. Compared to the embroidered reference it shows half of the resistance. In consideration of the SEM images of the coated yarns the Statex yarns are covered fully without gaps while the HC40 is not completely covered with the coating and gaps are depicted between the filaments of the yarn. This leads to the need of a thicker coating concerning the HC40.

Summary

Summed up the coating protects the one out of three EC yarn against mechanical stress implemented via textile processing with an embroidery plant. The Shieldex 117/17 dtex 2PLY (S) could be protected while the effect of the coating is not detectable concerning Shieldex 11717 dtex 2PLY C+B (S HC+B) and HC40. This can be remedied with the adjustment of coatings composition to increase the adhesion between the protective coating and EC yarn. Furthermore, the thickness of the coating should be increased to create further protecting potential. The thickness can be increased by using other coating processes and parameters. Additionally, the coating could be applied on other EC yarn-shaped structures to analyze the adhesion and protective effect on them. Also the implementation with other textile processing methods like knitting could be performed to detect differences or similarities in the protective behavior of the coating. Finally further characterization of the coated yarns could be performed, e.g. washing stability and temperature stability.

Acknowledgement

Funded by the German Research Foundation (DFG, Deutsche Forschungsgemeinschaft) as part of Germany's Excellence Strategy – EXC 2050/1 – Project ID 390696704 – Cluster of Excellence "Centre for Tactile Internet with Human-in-the-Loop" (CeTI) of Technische Universität Dresden

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