#### Journal of Fashion Business Vol.21, No.6

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ISSN 1229-3350(Print) ISSN 2288-1867(Online)

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J. fash. bus. Vol. 21, No. 6:16-30, December. 2017 https://doi.org/ 10.12940/jfb.2017.21.6.16

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# A Study on the E-textiles Dip-Coated with Electrically Conductive Hybrid Nano-Structures

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## **Keywords**

silver nanowire, graphene flake, resistance, extension, nylon, spandex.

#### **Abstract**

Currently, e-textile market is rapidly expanding and the emerging area of e-textiles requires electrically conductive threads for diverse applications, including wearable innovative e-textiles that can transmit/receive and display data with a variety of functions. This study introduces hybrid nano-structures which may help increase the conductivity of the textile threads for use in wearable and flexible smart apparels. For this aim, Ag was selected as a conductive material, and yarn treatment was implemented where silver nanowire (AgNW) and graphene flake (GF) hybrid structures overcome the limitations of the AgNW alone. The yarn treatment includes several treatment conditions, e.g., annealing temperature, annealing time, binder material such as polyurethane (PU), coating time, in order to search for the optimum method to form stable conductive nano-scale composite materials as thin film on the surface of textile yarns. Treatedyarns showed improved electrical resistance readings. The functionality of the spandex yarn as a stretchable conductive thread was also demonstrated. When the yarn specimens were treated with colloid of AgNW/GF, relatively good electrical conductivity value was obtained. During the extension and recovery cycles of the treated yarns, the initial resistance values did not deteriorate significantly, since the network of nanowire structure with the support of GF and polyurethane stayed flexible and stable. Through this research, it was found that when one-dimensional structure of AgNW and two-dimensional structure of GF were mixed as colloids and treated on the surface of textile yarns, flexible and stretchable electrical conductor could be formed.

This paper is a part of Ph.D. dissertation.

#### I. Introduction

Recently, studies on the wearable e-textiles and smart textiles, as results of the amalgamation of fashion materials and electronic technologies, are steadily increasing. Along with the traditional conductive materials made of Cu and Ag, there is a strong demand for flexible, conductive, yet wearable, stretchable and durable textile materials for the e-textiles.

Human body is mostly curvilinear and the skin is soft, stretchable and elastic within the elongation range of 3~55% (Hammock, Chortos, Tee, Tok, & Bao, 2013). The e-textiles are developed to exhibit appropriate electrical conductivity level allowing the transmission of electricity or data (Kaltenbrunner, Sekitani, Reeder, Yokota, Kuribara, Tokuhara, Drack, Schwödiauer, Graz, & Bauer-Gogonea, 2013). At the same time, they should possess extensibility and flexibility similar to the human skin, and lightness for the comfort feel by the users.

Currently, commercially available conductive yarns often comprise fine metal wires, made of copper, or polymer-based yarns coated or plated with metal (Tang & Stylios, 2006). The coatings are prepared via evaporative deposition, sputtering, or electroless plating (Meoli & May-Plumlee, 2002). Conductive yarn preparation by employing metallic nanoparticles, such as gold or silver, often shows relatively low conductivity due to their small particle size, resulting in many inter-particle junctions, thereby higher junction resistance values. In contrast to the use of nanoparticles, the use of nanowires for coating textile yarns allows better conductivity at a lower particle concentration with less particle junctions, establishing random network of metallic wires on the surface of the yarns. This also provides better mechanical flexibility to the product.

Current rapid developments in the nano-materials manufacturing sectors including R&D sectors have made the mass production system of the raw materials possible. This study aims to propose a method of

hybridization of conducting coating materials for textile yarns to supplement the electrical property failures in the conducting materials, which are major factors to degrade the electrical conductivity during tensile or bending deformation/recovery cycles of the electrically conductive threads (ECT).

Hybrid colloid of nano-conductive materials (Akter & Kim, 2012) were prepared and treated on the surface of various yarns to analyze the conductivity of the conductive yarns. Synthesized AgNW (Li, Wang, & Yan, 2011) may be dispersed in distilled water or ethyl alcohol for further treatment. Graphene flake (Lee, Lee, Kim, Lee, Park, Choi, Kim, Kim, Lee, & Nam, 2013) may be prepared from pure graphite, and dispersed in water for applications. In order for the graphene flakes to be adhered on the specimens, polyurethane-based finishing agent was employed as a binder.

In this study, as textile specimens, commercially available electrically conductive thread (ECT), nylon sewing thread, polyester sewing thread, and spandex filament yarn specimens were employed for conductive colloid treatments and resistance measurements.

# II. Experiments

Experimental flow diagram is given in Figure 1. Preparation procedure of the nano-conductive colloid, comprising AgNW, GF, and PU, was implemented, and treatment parameters on the ECT in order to select optimum conditions include annealing temperature, time, PU concentration, composition ratio of the colloids, and number of coatings on the yarn specimens.

#### 1. Materials

# 1) Colloidal Reagents

Reagents for the experiments are used as received from the manufacturers without further purification processes. AgNW(Silver Nano Wire): AgNW's are mostly used as dispersed state either in ethyl alcohol, methyl alcohol, or purified water. Colloidal AgNW product (Ditto Technology Co., Ltd., Korea) with diameter of

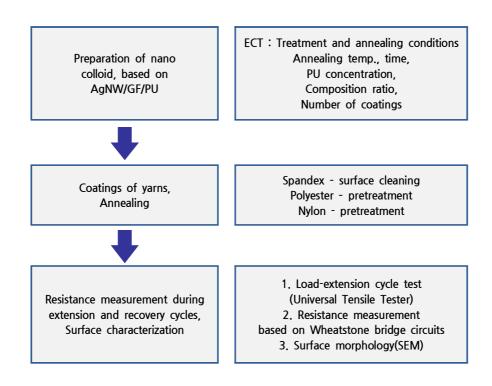


Figure 1. Flow Diagram of Experiments

 $25\pm10$ nm, and length of  $15\pm10\,\mu$ m, concentration of 1 wt%, dispersed in distilled water, is employed.

GF(Graphene Flake) : Graphene flake used in the study is water–dispersible graphene paste (Mexplorer Co., Ltd., Korea) with 0.5wt% in distilled water, thickness  $\langle 5\text{nm}, \text{ and lateral size} \rangle 2 \sim 3 \,\mu\,\text{m}$ . The company reports that the resistivity of PET film specimen, coated with the graphene flake, dried at  $120^{\circ}\text{C}$  for 60min., is around  $5\text{x}10^{-5}\mathcal{Q}\times\text{m}$ .

Polyurethane: Water-dispersible polyurethane is used as a binder for the graphene flake and the textile specimens. PU specifications are as follows: average size,  $0.1\sim60$ nm, molecular weight,  $50,000\sim159,000$ , thermoplastic water-dispersible type.

### 2) Yarn Specimens

Electrically Conductive Thread (ECT): One of the purposes of this study is to investigate the changes in

physical/mechanical and electrical properties by the coating of the AgNW/GF on the commercially available ECT. The ECT specimen, a nylon 66 thread with silver plating (Shieldex, U.S.A.), is known to be within the manufacturer's specification of the resistance per length less than 5Q/cm.(Table 1)

Yarn Specimens for Dip-Coating Treatment: Yarn specimens for dip-coating treatment using colloidal reagents described above include nylon 6, polyester, and spandex specimens.

The properties of the specimens are listed in Table 2. Since untwisted filament specimens have shown poor stability during conductivity test under deformation in a previous study(Lee, 2016), probably due to separation of the coated filament yarns having little or no twists, sewing thread type nylon 6 and polyester specimens with high twists are selected for the coating treatment, except for spandex specimen.

General Usage	Description	Linear Resistance (Ω/cm)	Tenacity (cN/tex)	Elongation (%)	Denier
Electomag. shield, Embroidery, ArtTextiles	99% pure silver plated nylon 66 yarn	< 5Ω/cm	37	27	255

Table 1, Properties of Silver Plated Nylon 66 Thread (from Shieldex Co.)

Table 2, Physical Properties of Yarn Specimens for Treatment with Conductive Materials

Yarn	Fineness (Denier / 's)	Range of Elongation (%)	General Description
Polyester	21's	15~20	Spun Yarn Type Sewing Thread
Nylon 6	439 den.	26~35	Filament Yarn Type Sewing Thread
Spandex	840 den.	500~600	Filament Yarn Type(Bare Yarn)

#### 2. Pretreatment and surface treatment

All the specimens were cleaned in distilled water, and air-dried at room temperature, before dip-coating (Figure 2).

In the case of ECT, it was cleaned in ethyl alcohol, and air-dried at room temperature.

Specimens with hydrophobic properties, such as nylon 6 and polyester yarns, need further chemical treatment to improve adhesive properties, since the AgNW solution does not show good affinity with nylon and polyester.

Polyester yarns were submerged in an aqueous solution of 20wt% NaOH for 1min. at 75°C, rinsed in distilled water, and air-dried at room temperature (Atwa, Maheshwari, & Goldthorpe, 2015)

#### 3. Annealing Conditions

In the preceding studies, for annealing temperature of AgNW materials,150°C(Atwa et al., 2015)and160°C(Lu & Chou, 2010)conditions have been reported, since higher annealing temperature may deteriorate the

performance and stability of the AgNW. When AgNW colloid is hybridized with GF, together with PU, which is stable at higher temperature, the hybrid colloid treatments have been reported to allow the specimen to be stable even at 180°C annealing (Krantz, Richter, Spallek, Spiecker, & Brabec, 2011; Lee, Lee, Jung, Byun, Yi, Lee, & Kim, 2013; Nickel, Haas, Wegner, Bahro, Salehin, Kraft, Gruber, & Colsmann, 2014; Song, You, Lim, Park, Jung, Kim, Kim, Kim, & Park, 2013). In this study, annealing temperature range of 150, 160, and 170°C, and annealing time of 20, 30, and 40min, were chosen for the treatment.

# 4. PU Concentration

Even if AgNW is readily oxidized in air, water, and ethyl alcohol media, the oxidization problem may partly be overcome by employing PU in the colloid. The use of highly concentrated PU colloid, however, may deteriorate the electrical conductivity, since the PU does not exhibit conductivity.

PU concentrations employed in this experiment were 0%, 0.3%, 0.5%, 0.7%, 1%, and 1.3%.

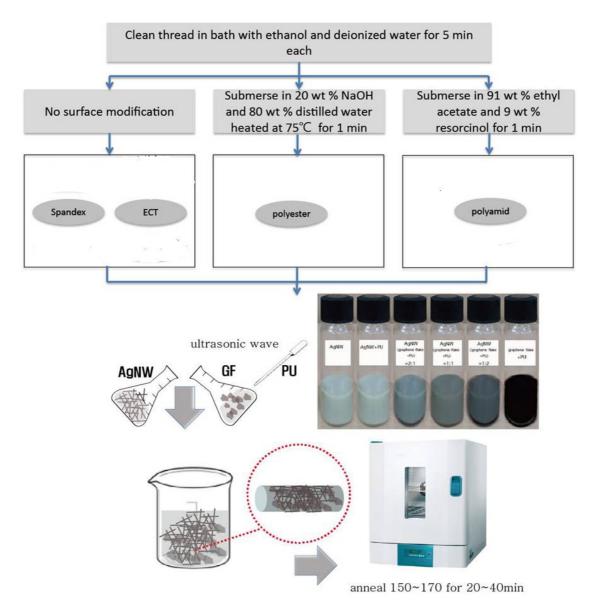


Figure 2. Pretreatment and Dip-Coating Procedure (Atwa et al., 2015, p.3909)

# Composition ratio of hybrid conductive materials with PU binder

In a hybrid conductive materials, the metal component exhibits most of the electrical conductivity of the hybrid, even though the binder also affect the hybrid conductivity partially. The effect of composition ratio on the conductivity of the hybrid material was investigated by changing the ratio of the compositions, including AgNW, GF, and PU binder. The specific ratio is listed in Table 3.

An ultrasonic stirrer was used to mix the GF and PU colloid first, for 2 hours, before preparing hybrid colloid with the Ag NW.

Colloid Number	Colloid Composition  AgNW Colloid (0.1wt%), ml		GF Colloid (0.5wt%), ml	PU binder solution (0.3wt%), ml
1	AgNW	20	0	0
2	AgNW/(PU)	20	0	0.6
3	AgNW_2/GF_1/(PU)*1	20	20	1.2
4	AgNW_1/GF_1/(PU)* <sup>2</sup>	10	20	0.9
5	AgNW_1/GF_2/(PU)*3	10	40	1.5
6	GF/(PU)	0	40	1.2

Table 3, Description of the AgNW/GF/PU Colloid

# Electrical resistivity/load measurements during extension and recovery cycles

By using a universal tensile tester (Testometric, Micro350, U.K.), the resistivity changes, together with the load signals, during tensile deformation and recovery of the conductive yarn specimen are recorded. (Figure 3) Effective specimen length, or gauge length, is set to 40mm. The maximum tensile elongation of the specimen is 15% (6mm). The extended specimen is then recovered to its initial gauge length. Extension/recovery speed is kept at 12mm/min. The cover plates of the grip faces to hold the conductive yarn specimens are modified by using flat copper plates to ensure good electrical contact.

During the first and second deformation and recovery cycles, the resistivity and load signals are digitized via an A/D converter and recorded through a data acquisition software system. The resistivity value is acquired via a Wheatstone bridge circuit under an applied voltage of 5.0vdc (Baccar, Levi, Dallet, & Barbara, 2013; Martinez, Stauffer, Adagunodo, Forro, Vörös, & Larmagnac, 2015; Park, You, Shin, & Jeong, 2015; Silva, Gonçalves, & Carvalho, 2013; Wu, Xu, Xiong, & Wang, 2012).

### III. Results and Discussion

# Resistance change of ECT specimen during extension and recovery cycles

Changes in the electrical resistance against extension and recoveycles are analyzed in Table 4. Graph of the resistance changes with time (sec) or extension (mm) during extension and recovery cycles is shown in Figure 4.

The mid-point (\*a) value is representative of the hysteresis at the mid-point of the 2<sup>nd</sup>cycle. When this value is small, the hysteresis of the specimen resistance is small, meaning that the resistance recovery during the tensile deformation/recovery cycle test is good.

If the conducting material on the surface of the yarn specimen is not flexible, the mid-point (\*a) resistance value will become large, probably due to the joint breakage of the nano-wire mesh structure, in case the AgNW is coated, or breakage or cracks in the plated conductive metal film, in case untreated ECT specimen is extended,

Initial resistance per length of ECT is  $2.47 \, \Omega/\text{cm}$ , and the resistance at the first peak, shown in the vicinity of 30 seconds, equivalent to 6mm extension of the specimen, is  $3.51 \, \Omega/\text{cm}$ .

<sup>\*1</sup> AgNW\_2/GF\_1/(PU)\*; Colloid of AgNW 2: GF 1 with PU binder,

<sup>\*2</sup> AgNW\_1/GF\_1/(PU);Colloid of AgNW 1:GF1with PUbinder,

<sup>\*3</sup> AgNW\_1/GF\_2/(PU);Colloid of AGNW 1:GF2with PUbinder.

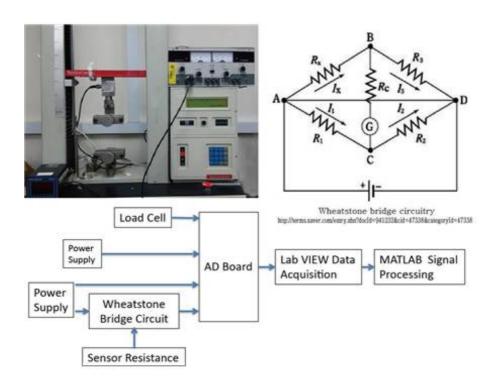


Figure 3, Experimental Setup for Resistance-Extension Measurement during Extension/Recovery Cycles of Yarn Specimens

Table 4. Resistance Values of Curves during Extension-Recovery Cycles of Yarn Specimen, ECT(untreated)

Specimen	Resistance per cm, Ω/cm							
ECT	Initial	1st peak	2nd peak	Mid-point *a	Mean *b	Slope,% *c		
(untreated)	2.47	3.51	3.45	0.30	3.50	7.07		

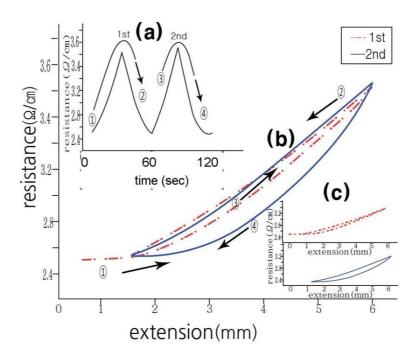
<sup>\*</sup>a: resistance difference between the resistance values of extension and recovery of 2<sup>nd</sup>cycle at the 3mm extension.(=Hysteresis o f2<sup>nd</sup> cycle curve at mid-point)

\*c, slope (%)=
$$\frac{(b-initial resistance)}{15}X100$$

long with the first recovery cycle, the resistance values decrease toward almost the initial value. The resistance value, however, might not recover to the initial value depending on the properties of the specimens, including the tensile properties of the yarn specimens and the nature of conductive materials coated on the specimen surface.

In the case of the treated ECT specimens, the resistance value at the recovered state, at the end of the recovery cycle, is  $2.45\,\Omega$ /cm, which is almost identical to the original value of  $2.47\,\Omega$ /cm. The values of the two peaks, first and second peaks, are almost the same as shown in the table and figure, the difference being  $0.06\,\Omega$ /cm.

<sup>\*</sup>b: mean resistance values of 1stand 2nd peaks.



- (a) Resistance Curve with Time (in sec., x-axis), 2 Cycles.
  (b) Hysteresis Curves of the Resistance with Extension (in mm, x-axis).
  - (c) Separated Hysteresis Curves: 1st and 2<sup>nd</sup> in red and blue.

Figure 4. Resistance as a Function of Extension (max, extension= 6 mm)

The shapes of the extension and recovery cycles were also compared. The differences between the extension and recovery curves in the first and second cycles became larger with the number of repetition. Therefore, the analysis of the hysteresis is important in the evaluation of the conductive yarns. As indices of these properties, the midpoint and mean values were also calculated in the table.

As shown in the case of the untreated ECT (commercial product, as received), the resistance value analysis during the extension and recovery cycles is important as indices to evaluate the performance of the conductive yarns by AgNW and GF treatment with binder such as PU.

It is suggested, therefore, in this study, to employ such indices useful for developing conductive colloid compositions, based on nano-structure materials, for coating of several types of textile yarns. 1) Resistance changes according to annealing conditions: annealing temperature, time

One of the major objectives is to investigate the effect of annealing conditions, such as annealing temperature, time and number of coatings under a composition of the hybrid colloid of conductive nano materials, such as AgNW and GF, with the PU as a binding material for coating.

Table 5 shows the indices of resistance curve during the extension and recovery cycles of the treated ECT specimens.

Compared to the untreated control specimen, ECT, the initial resistance values of the treated specimens with different annealing temperatures, 150, 160, and 170°C, all show lower resistance values by at least 1 Q/cm. Therefore, the coating of hybrid colloid of AgNW and GF is found to be effective in decreasing the resistance of the untreated ECT specimen.

Annealing Condition			Resistance per cm, <b>Q/</b> cm					
Temp.,℃	Time, min.	No. of coatings	Initial	1 <sup>st</sup> peak	2nd peak	Mid- point	Mean	Slope, %
ECT (control, untreated)	-	-	2.47	3.51	3.45	0.30	3.50	7.07
150	30	2	1.56	2.23	2.20	0.24	2.22	4.37
160	30	2	1.37	2.17	2.29	0.12	2.23	5.73
170	30	2	1.37	2.16	2.35	0.19	2.26	5.90
160	20	2	1.25	1.81	1.80	0.20	1.81	3.70
160	40	2	1.44	1.70	1.89	0.23	1.79	2.37

Table 5. Analysis of Resistance-Extension Curve according to the Changes in Annealing Conditions: Temperature, and Time

Annealing time showed some effect on the resistance values. Even if the overall resistance values of specimens given by the treatment of annealing time 30min, may not seem much better than those of 20min, or 40min, the midpoint value of the treatment of 30min, shows better than the other two, which is one index of hysteresis of the resistance curves. Among the treatment conditions, treatment temperature 160°C and 30 minutes gave the best overall resistance values, including lower mid point value, suggesting low hysteresis during the deformation and recovery cycles.

Therefore, the treatment temperature of 160°C and time of 30 minutes will have positive effect on the resistance and hysteresis behavior.

2) Resistance changes by annealing conditions: colloid materials composition and number of coatings

Table 6a shows the resistance changes according to the colloid materials composition and number of coatings. Among the three compositions, AgNW, AgNW/GF/(PU), and GF/(PU), the composition AgNW/GF/(PU) gave the best overall resistance values throughout the number of coatings(1~5). The use of GF(/PU) resulted in the least favorable resistance values, especially the specimen with only one coating gave 'Range over' resistance value. The specimen with two

coatings of GF(/PU) also gave resistance value of  $20,750 \,\Omega$ /cm.

Considering the resistance values of the results, the number of coatings 2 would be enough for AgNW/GF/(PU) composition, in case the maximum extension and recovery conditions are similar to the experiment in this study.

Table 6b shows the analysis results of the resistance–extension curve according to the composition ratio of AgNW and GF colloids. Among the three composition ratio of AgNW/GF 2:1, 1:1, and 1:2, the ratio 2:1 gave the best initial resistance, 1.37 g/cm, the second, 1:1, 1.72 g/cm, and the last, 1:2, 1.76 g/cm. The trend of resistance values at 1st and 2nd peak are also similar to those of initial resistance. Therefore, the ratio of AgNW:GF=2:1 gave the best resistance results.

# Resistance change of yarn specimens by coating of hybrid nano-conductive materials -Polyester, nylon, and spandex

Three different types of yarn specimens, polyester, nylon, and spandex, were treated with conductive colloid, and the resistance change during the extension and recovery cycles. The polyester fiber is stiffer than the other two fibers due to the structural characteristics.

<sup>\*)</sup> Colloid composition, AgNW:GF =2:1, 0.3wt% PU.

**Annealing Condition** Resistance per cm, Ω/cm Temp., ℃ Time, min. No. of Coatings **AgNW** AgNW/GF/(PU) GF/(PU) 160 30 3.95 3.07 Range over 160 30 2 1.38 1.12 20,750 160 30 3 1.37 1.27 780 4 160 30 1,22 1.07 490 5 160 30 1.05 1.12 94 5

Table 6a, Resistance Changes according to the Colloid Materials Composition and Number of Coatings

Table 6b. Analysis of Resistance-Extension Curve according to the Changes in Treatment Conditions: Composition Ratio

Composition		Slope,				
AgNW:GF	Initial	1st peak	2nd peak	Mid-point	Mean	%
2:1	1.37	2.17	2.29	0.12	2.23	5.73
1:1	1.72	2.51	2.52	0.13	2.51	5.30
1:2	1.76	2.44	2.46	0.09	2.45	4.60

<sup>\*)</sup> Colloid composition: 0.3wt% PU.

Nylon fiber is a pliable and extensible. The spandex fiber is the most stretchable and shows the best recovery-related properties.

### 1) Polyester

Polyester sewing thread was pretreated by using NaOH solution.

Table 7 shows the resistance change by coating different colloid of conductive materials. The number of coatings were also changed, 1 through 5, at annealing temperature 160°C, 30min. Colloid GF/(PU) did not improve the resistance values of treated polyester, in comparison to those of AgNW or AgNW/GF/(PU) colloids.

The surface states of the annealed conductive polyester threads were so brittle that resistance measurement during tensile extension/recovery cycles was not possible, due to the small cracks initiated by the pressure exerted on the specimen during the

clamping of the polyester thread between the clamps, leading to higher resistance readings of more than several mega ohms per cm.

The resistance shown in Table 7 were measured right after the annealing of the treated polyester. Careful handling of the specimen was necessary to avoid the initiation of the cracks on the surface of the specimen, especially for GF/(PU) colloid treatment, since the GF coating is quite brittle. Even a small curvature given to the specimen during the resistance measurement procedure would gave quite high resistance readings. Resistance values of the treated specimens, after five coatings annealed at 160°C for 30min., were 5.9, 6.5, and 210 9/cm for AgNW, AgNW/GF/(PU), and GF/(PU) treatment, respectively. Since all the treated polyester specimens showed very poor performance in measuring the resistance values during extension/recovery cycle test, the result was not tabulated.

Table 7. Resistance Values according to the Colloid Materials Composition and Number of Coatings: Polyester

Annealing Condition			Resistance per cm, Q/cm			
temp., ℃	time, min.	no, of coating	AgNW	AgNW/GF/(PU)	GF/(PU)	
160	30	1	36.0	35.0	64,000	
160	30	2	12.5	11.7	18,000	
160	30	3	7.8	8.1	340	
160	30	4	7.8	8.1	287	
160	30	5	5.9	6.5	210	

Table 8a, Resistance Changes according to the Colloid Materials Composition and Number of Coatings, Nylon 6 Sewing Thread

	Annealing Condition	1	Resistance per cm, Ω/cm			
temp., ℃	time, min.	no, of coating	AgNW	AgNW/GF/(PU)	GF/(PU)	
160	30	1	58.0	57.0	61,000	
160	30	2	22.5	21.7	17,100	
160	30	3	7.80	6.10	328	
160	30	4	7.80	6.30	271	
160	30	5	7.90	7.20	42.8	

Table 8b. Analysis of Resistance-Extension Curve according to Annealing Conditions, Nylon 6 Sewing Thread

Composition		Resistance per cm, Q/cm					
AgNW/GF(/PU)	Initial	1st peak	2nd peak	mid point	mean	slope(%)	
2:1	3.66	17.32	21.16	1.70	19.24	103.87	

Annealing condition: 160℃, 30min, 2 coatings

## 2) Nylon 6

Table 8a shows the resistance changes according to the colloid materials composition and number of coating treatments on nylon 6 sewing thread specimens. It shows the resistance change by coating different colloid of conductive materials. The number of coatings were also changed, 1 through 5, at annealing temperature 160°C, 30min. The colloid GF/(PU) treatment performed poor in the resistance values of treated nylon 6, in comparison to those of AgNW or AgNW/GF/(PU) colloids.

Among a number of nylon 6 specimens treated with AgNW/GF/(PU) colloids, one specimen with good resistance value, 3.66  $\Omega$ /cm, was selected for the subsequent resistance–extension cycle measurement. The nylon 6 specimen showed 17.32, and 21.16  $\Omega$ /cm, at 1st and 2nd peaks. (Table 8b) In terms of resistance–extension cycle measurement results, the nylon6 specimens performed better than the polyester specimens. The performance difference may possibly be related to the fact that the polyester specimen is a staple yarn sewing thread type, while the nylon

specimen is a continuous filament sewing thread type, resulting in more crack formations on the coated surface of the specimens due to a number of protruding free fiber ends of the polyester specimen together with the differences in polymer properties.

### 3) Spandex

The spandex specimens, dip-coated with the conductive colloids without pretreatment, showed low resistance values. With 15% extension of the treated specimen, however, the resistance values increased considerably to tens of thousands  $\Omega$ /cm, and the original resistance value was not resumed well even after the extended specimen remained relaxed for more than an hour. After 24 hours relaxation, the resistance value of the relaxed spandex specimen decreased to 50% higher than that of the original resistance value.

Considering the stretchability of the spandex specimen, the spandex specimen was stretched to 50, 75, and 100% of the original length before the colloid dip-coating. By maintaining the stretched state, the

spandex specimens were annealed at given condition. The results are given in Tables 9a and 9b.

The treated spandex specimen showed 5.02, and  $64.31\,\mathrm{g}/\mathrm{cm}$ , at  $1^{\mathrm{st}}$  and  $2^{\mathrm{nd}}$  peaks. The functionality of the treated spandex yarn as a highly stretchable conductive material was also demonstrated. As a result, compared to other thread specimens, the spandex showed low stress to repeated strain recovery process, and the slope level of 221.57% is achieved in a rather wide range of  $1\sim50\,\mathrm{g}/\mathrm{cm}$ .

Figure 5 shows the surface layer cracks developed during the extension and recovery cycles of the spandex specimens, treated with conductive colloid at extended conditions. Specimens treated under 100% extension exhibited much less crack development on the surface of the treated yarn.

#### IV. Conclusion

Conductive hybrid nano-structures based on AgNW and GF colloid materials, which may increase the

Table 9a, Resistance Changes according to the Colloid Materials Composition and Number of Coatings, Spandex

Annealing Condition			Resistance per cm, Q/cm			
temp., ℃	time, min.	no, of coating	AgNW	AgNW/GF/(PU)	GF/(PU)	
160	30	1	128	117	64,000	
160	30	2	35.50	21.70	18,000	
160	30	3	7.80	6.10	18,000	
160	30	4	3.80	3.30	340	
160	30	5	1.90	1.50	287	

Annealing condition: AgNW/GF/(PU)=2:1, 100% extended state.

Table 9b. Analysis of Resistance-Extension Curve according to Annealing Conditions, Spandex

Composition		Resistance per cm, Ω/cm						
AgNW/GF(/PU)	Initial	1st peak	2nd peak	mid point	mean	slope(%)		
2:1	1.43	5.02	64.31	3.50	34.67	221.57		

Annealing condition: 160°C, 30min, 2 coatings, 100% extended state.

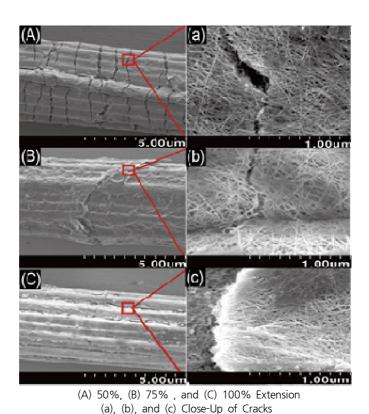


Figure 5. SEM Images of Spandex Filament Specimens treated with Conductive Colloid (AgNW:GF=2:1) under stretched State

conductivity of the textile threads for use in wearable and flexible smart apparels, were studied. For this aim, yarn treatment was implemented where silver nanowire (AgNW) and graphene flake (GF) hybrid structures overcome the limitations of the Ag alone. Through the pre-conditioning process and treatment, the conductive yarns showed improved electrical resistance values.

Optimum treatment conditions of the ECT found in this study, providing improved resistance values, were 1) colloid composition; AgNW:GF=2:1 with 0.3wt% PU, 2) annealing condition; 160°C, 30min 3) number of coatings; 2 or more.

Resistance values of the treated polyester specimens, after five coatings annealed at 160°C for 30min., were 5.9, 6.5, and 210 9 /cm for AgNW, AgNW/GF/(PU), and GF/(PU) treatment, respectively. During the extension–recovery cycles, the nylon 6 specimen showed

17.32, and 21.16 Q/cm, at 1<sup>st</sup> and 2<sup>nd</sup> peaks. In terms of resistance–extension cycle measurement results, the nylon 6 specimens performed better than the polyester specimens. The performance difference may possibly be related to the differences in type of yarn construction factors: twisted spun yarn type versus twisted continuous filament sewing yarn.

The treated spandex specimen showed 5.02, and 64.31  $\,^{\circ}$  /cm, at  $\,^{\circ}$  and  $\,^{\circ}$  and  $\,^{\circ}$  peaks. The functionality of the treated spandex yarn as a highly stretchable conductive material was also demonstrated. As a result, compared to other thread specimens, the spandex showed low stress to repeated strain recovery process, and the slope level of 221.57% is achieved in a rather wide range of  $\,^{\circ}$  1~50  $\,^{\circ}$  /cm.

During the extension and recovery cycles of the treated yarns, the initial resistance values did not

deteriorate significantly, since the network of nanowire structure with the support of GF and polyurethane stayed flexible and stable. Through this research, it was found that when one-dimensional structure of AgNW and two-dimensional structure of GF were mixed as colloids and treated on the surface of textile yarns, flexible and stretchable electrical conductor could be formed.

#### References

- Akter, T., & Kim, W. S. (2012). Reversibly stretchable transparent conductive coatings of spray-deposited silver nanowires. *ACS Applied Materials & Interfaces*, *4*(4), 1855–1859.
- Atwa, Y., Maheshwari, N., & Goldthorpe, I. A. (2015). Silver nanowire coated threads for electrically conductive textiles. *Journal of Materials Chemistry C*, *3*(16), 3908–3912.
- Baccar, S., Levi, T., Dallet, D., & Barbara, F. (2013). Modeling and simulation of a wheatstone bridge pressure sensor in high temperature with VHDL-AMS. *Instrumentation Viewpoint*, (14), 55–55.
- Hammock, M. L., Chortos, A., Tee, B. C., Tok, J. B., & Bao, Z. (2013). 25th anniversary article: The evolution of electronic skin (E-Skin): A brief history, design considerations, and recent progress. Advanced Materials, 25(42), 5997–6038.
- Kaltenbrunner, M., Sekitani, T., Reeder, J., Yokota, T., Kuribara, K., Tokuhara, T., Drack, M., Schwödiauer, R., Graz, I., & Bauer–Gogonea, S. (2013). An ultra–lightweight design for imperceptible plastic electronics. *Nature*, 499(7459), 458–463.
- Krantz, J., Richter, M., Spallek, S., Spiecker, E., & Brabec, C. J. (2011). Solution-processed metallic nanowire electrodes as indium tin oxide replacement for thin-film solar cells. Advanced Functional Materials, 21(24), 4784–4787.
- Lee, E. A. (2016). Study on the e-textile for strain sensor dip-coated with AgNW/graphene flake electrically conductive nano structure hybrid

- (Unpublished doctoral dissertation). Ewha Womans University, Seoul, Korea.
- Lee, M., Lee, K., Kim, S., Lee, H., Park, J., Choi, K., Kim, H., Kim, D., Lee, D., & Nam, S. (2013). High-performance, transparent, and stretchable electrodes using graphene-metal nanowire hybrid structures. *Nano Letters*, *13*(6), 2814–2821.
- Lee, W., Lee, J. U., Jung, B. M., Byun, J., Yi, J., Lee, S., & Kim, B. (2013). Simultaneous enhancement of mechanical, electrical and thermal properties of graphene oxide paper by embedding dopamine. *Carbon*, 65, 296–304.
- Li, X., Wang, L., & Yan, G. (2011). Review: Recent research progress on preparation of silver nanowires by soft solution method and their applications. *Crystal Research and Technology*, *46*(5), 427–438.
- Lu, Y., & Chou, K. (2010). Tailoring of silver wires and their performance as transparent conductive coatings. *Nanotechnology*, *21*(21), 215707.
- Martinez, V., Stauffer, F., Adagunodo, M. O., Forro, C., Vörös, J., & Larmagnac, A. (2015). Stretchable silver Nanowire – Elastomer composite microelectrodes with tailored electrical properties. ACS Applied Materials & Interfaces, 7(24), 13467–13475.
- Meoli, D., & May-Plumlee, T. (2002), Interactive electronic textile development, *Journal of Textile and Apparel, Technology and Management, 2*(2), 1–12.
- Nickel, F., Haas, T., Wegner, E., Bahro, D., Salehin, S., Kraft, O., Gruber, P. A., & Colsmann, A. (2014). Mechanically robust, ITO–free, 4.8% efficient, all–solution processed organic solar cells on flexible PET foil. Solar Energy Materials and Solar Cells., 130, 317–321.
- Park, J., You, I., Shin, S., & Jeong, U. (2015). Material approaches to stretchable strain sensors. *Chem. Phys. Chem.*, 16(6), 1155–1163.
- Silva, N. L., Gonçalves, L., & Carvalho, H. (2013). Deposition of conductive materials on textile and polymeric flexible substrates. *Journal of Materials Science: Materials in Electronics*, 24(2), 635–643.
- Song, M., You, D. S., Lim, K., Park, S., Jung, S., Kim, C. S., Kim, D., Kim, D., Kim, J., & Park, J. (2013).

Highly efficient and bendable organic solar cells with solution-processed silver nanowire electrodes. *Advanced Functional Materials*, *23*(34), 4177–4184. Tang, S. L., & Stylios, G. K. (2006). An overview of smart technology for clothing design and engineering. *Int. J. Clothing Sci. Tech.*, *18*(2), 108–128.

Wu, G., Xu, D., Xiong, B., & Wang, Y. (2012). Wheatstone bridge piezoresistive sensing for bulk-mode micromechanical resonator. *Applied Physics Letters*, *101*(19), 193505.

Received (July 13, 2017) Revised (September 5, 2017) Accepted (September 21, 2017)