Silver nanowire coated threads for electrically conductive textiles

by

Yahya Atwa

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AUTHOR'S DECLARATION

I hereby declare that I am the sole author of this thesis. This is a true copy of the thesis, including any required final revisions, as accepted by my examiners.

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Abstract

The emerging area of e-textiles requires electrically conductive threads. In this thesis it is demonstrated that nylon, polyester, and cotton threads can be made conductive by coating their surfaces with random networks of solution-synthesized silver nanowires. A chemical pre-treatment was used on the nylon and polyester threads to improve the adhesion of the nanowire coating. A resistance per unit length of 0.8 $\Omega \cdot cm^{-1}$ was achieved and can be varied through the density of the nanowire coating. Because the nanowires are 35 nm in diameter, and the mesh structure does not cover the entire surface like a thin-film, less metal is used compared to conventional silver-coated conductive threads. This leads to a much lower weight and mechanically flexible coating. The resistance of the nanowire-coated thread did not degrade after washing. The functionality of the thread as a heater is also demonstrated.

Acknowledgements

First and foremost, all my thankfulness is to Allah who helped and guided me to finish this thesis. I would like to thank Professor Irene Goldthorpe for her valuable suggestions, support, encouragement, and patience during my master's program.

Also, I would like to express my thanks to all the members of my research group and a special thanks to Hadi, Jianjin and Arash for their recommendations and friendship.

Moreover, I would like to thank to Prof. and for being readers of my thesis.

I am especially grateful to my parents, my wife and my son for their patience, prayers, understanding and for believing in me. They have given me much care, love and support throughout the research.

Dedication

This thesis is dedicated to my lovely parents, my wife, and my son who will be happy to see my graduation.

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Chapter 1
Introduction

1.1 Electronic textiles

1.1.1 Definition

Electronic textiles, or e-textiles, are fabrics that have electronic devices integrated into them. They are also known as smart textiles or intelligent textiles. They can have abilities such as sensing, power transmission, and communications, while maintaining the feel and mechanical flexibility associated with traditional fabrics.

Figure 1.1: Health monitoring sensors integrated into baby clothing (photo taken from Wikipedia)

E-textiles can be generally divided into two types. The first is where classic electronic devices such as light emitting diodes, integrated circuits, and traditional batteries are essentially attached on the surface of a traditional textile. This type of e-textile tends to be less desirable because it is less 'textile-like'. That is, properties such as breathability, flexibility, and lightweight that are associated with textiles can be lost. In the other type, electronic devices are integrated directly into the textile fibers. Either inactive electronics such as pure metal wires, or more sophisticated active electronics such as transistors, diodes and solar cells (figure 1.2), can be contained in modern e textiles [1].

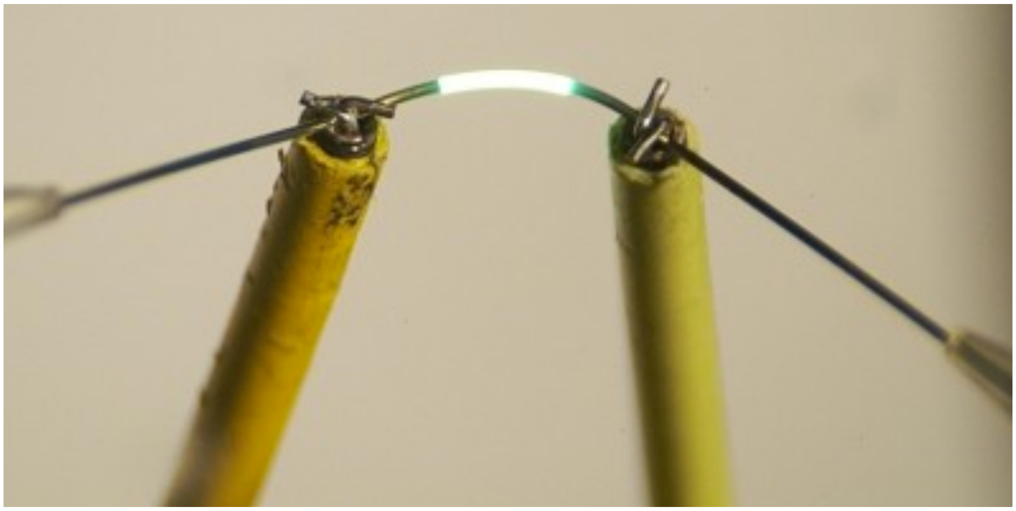


Figure 1.2: Organic light emitting diode directly integrated into a fiber. (image taken from laserfocusworld)

1.1.2 Applications of e-textiles

Fashion is one application of e-textiles. These consist of lighting and activated decorative components on clothing. They can also serve entertainment purposes. Devices such as cellular phones, personal stereos, and computers can be integrated into apparel. For medical applications, e-textiles can be used to monitor vital signs such as heart rate or blood pressure. For example, socks can have pressure sensors that alert the wearer to put his feet up when high blood pressure is detected. Because e-textiles can measure body motion parameters, they also have applications in apparel for exercising. Bio-feedback can track a person's vital signs to improve endurance and overall health. In addition, e-textiles can be used in military purposes. Enemies can be detected by environment sensing [2]. Finally, solar cells are an exciting application for e-textiles. Solar cell textiles can generate power for electronic devices, such as mobile phones and tablets, by collecting sunlight with nano-based chemicals [2].

1.2 Electrically conductive threads

1.2.1 Overview

Commonly used textiles such as cotton, nylon, and polyester are all electrical insulators. However, conductivity is required to transfer electrical power and signals to or from a textile-integrated device. Furthermore, conductive threads and fabric on their own are needed for electromagnetic interference (EMI) shielding [3] and wearable antennas [4].

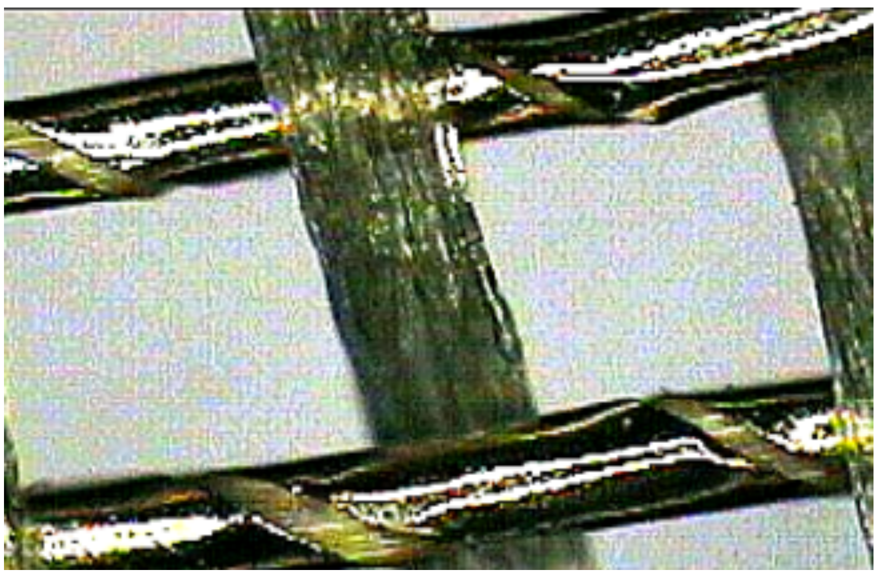


Figure 1.3: wrapping a non-conductive thread with a metal [5].

Commercially available conductive thread is typically either a solid metal wire, such as copper or stainless steel, or a non-conductive thread coated with a ~1 μm thick metal film, usually silver.

1.2.2 Solid metal wires

Solid metal wires are single, usually cylindrical, flexible elements or rods of metal such as silver, gold or stainless steel. They can be in the form of a solid core or braided strands, and woven into traditional fabric. The problem with these types of wires is that they tend to be stiff and brittle, which causes problems both during weaving and in end applications. Furthermore, they can breakdown (resistance greatly increases) after repeated bends [6], and can add significant weight to the textile if a high density of conductive threads is required.

1.2.3 Metal-coated wires

Conventional textile threads can be made electrically conductive by coating their surfaces with a thin-film of metal. These coatings can be used on several textile types (eg. nylon, yarns, polyester). Common textile coating techniques include electroless plating [7], sputtering [8], and dip coating [9].

Electroless plating involves immersing the textile in an electroless plating solution, in which metal ions attach to the textile substrate through chemical reactions. The most common metals used for electroless plating are nickel and copper. The advantages of electroless plating are the production of uniformly thick films and attaining coatings that have desirable mechanical, magnetic, and chemical properties. However, the fabrication process of electroless plating can be expensive [7].

Deposition of a metal film by sputtering needs to be done in a vacuum chamber. The coating metal is emitted atom by atom and is transferred to the surface of the textile, making a thin coating. This method can achieve a uniform coating with good adhesion to the substrate. However, it is very slow [8].

The dip coating process is an easy way to coat textiles. It is done by immersing the textile in the solution of the coating metal. The textile has to remain inside the solution for a period of time and then pulled up. A thin layer bonds itself on the surface of the textile while it is pulled up [9]. However, it is difficult to control deposition thickness, resulting in non-uniformity and depositing more metal than desired.

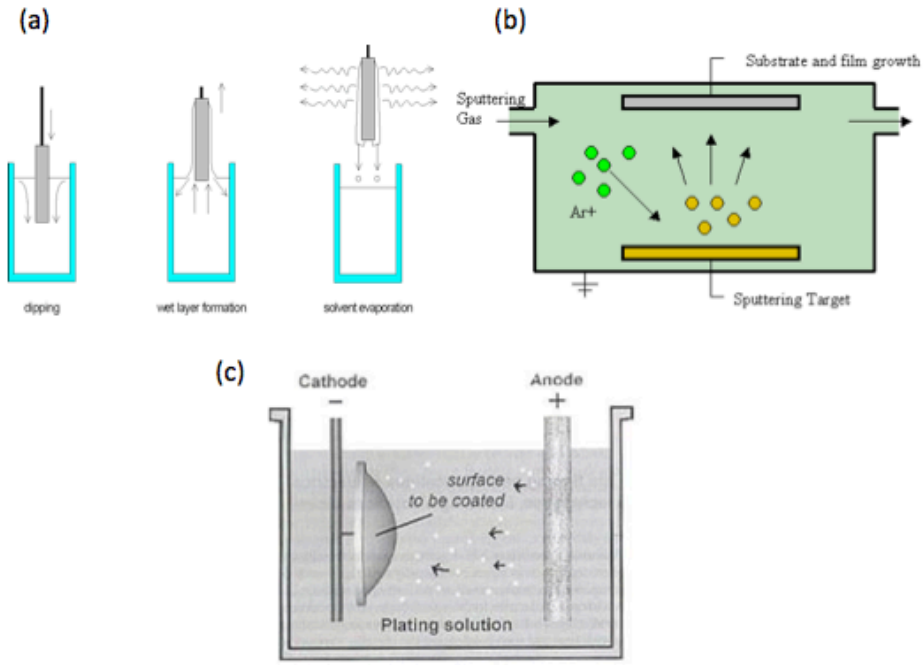


Figure 1.4: (a) dip coating, (b) sputtering, and (c) electroless plating. (diagram taken from Wikipedia)

1.3 Recent alternative conductive threads

Because of the rise of e-textiles, the demand for electrically conductive textiles has increased significantly in recent years. As mentioned above, existing commercially-available conductive threads have issues such as cost and physical and mechanical problems. Therefore, several research groups have been working on alternatives. Demonstrated solutions include thread made from carbon-based materials, and coating non-conductive thread with films of carbon nanotubes [10], graphene flakes [11], and conductive polymers [12]. These various technologies are briefly explained in the following subsections.

1.3.1 Carbon nanotube threads and nanotube coated threads

A carbon nanotube (CNT) is a tube-shaped structure made from carbon which has a diameter of nanometer scale. A CNT has a length-to-diameter ratio of up to 132,000,000:1. Many fields of materials science and technology, such as nanotechnology, electronics and optics, can have the opportunity to engage CNTs due to their unusual properties [13]. The electron mobility of a single carbon nanotube can be higher than 100 000 $cm^2/V \cdot s$ [14]. However, although the resistance of individual carbon nanotubes is extremely low, the junction resistance between two overlapping nanotubes is very high, in the range of 200 kΩ - 20 MΩ. This leads to low conductivity of a connected CNT network which would coat the surface of a thread. Secondly, CNTs can be either metallic or semiconducting, with the metallic tubes having much higher conductivity than the semiconductor ones [14]. Researchers have not been able to synthesize batches of metal-only CNTs and instead only mixes of metal and semiconductor nanotubes. The semiconductors nanotubes have relatively high resistance, and create even higher junction resistances than metal-metal junctions.

Conductive CNT thread can be made either from CNTs exclusively or from coating the surface of thread with a film or network CNTs. Motta and his group have created a conductive yarn directly made of pure carbon nanotube fibers by chemical vapor deposition [15]. Dalton et al. also fabricated carbon nanotube textiles, which were used for supercapacitors and electronic textiles. They used solution spinning to fabricate the fiber. The threads were very strong, with the energy required to break these fibers being four times higher than spider silk and twenty times higher than steel wire [16]. Xue et al. obtained CNT-coated threads with two different methods, wet-spinning a thread made exclusively of CNTs, and coating the surface of non-conductive threads with carbon nanotubes. The conductivity of the thread obtained by the wet spinning process was 10 kΩ/cm which is very high. As for the CNT-coated threads, the electrical conductivity varied with the type of thread used. Table 1 shows the resistance of various threads coated with CNTs. Because of the low density and small diameter of the yarn, CNT-coated polyester, polypropylene, and silk have much better conductivity than the other coated threads. Shim et al have also created conductive yarn using a polyelectrolyte-based coating using CNTs. Furthermore, they found that albumin, the key protein of blood, could be detected by CNT-coated threads with high sensitivity and selectivity [17].

The main problem of conductive threads employing CNTs is their high resistances, as can be seen in Table 1.1. This is caused by a few factors, most importantly by the reasons stated above – high junction resistances and the inability to obtain metal-only nanotubes. However, on top of this, wet-spinning and coating processes tend to use a small quantity of CNTs, and coated threads have contaminations which reduce electrical conductivity [18]. Furthermore, carbon nanotubes can have stability issues in air [19], and evidence exists that carbon nanotubes may be toxic upon skin exposure [20].

Table 1.1: Conductivity of various conductive threads coated with CNTs

Material	Resistance (kΩ)
Cotton	2.87
Silk	1.17
Wool/nylon	48.67
Polyester	0.25
Polypropylene	0.54

1.3.2 Graphene coated fabrics and threads

Graphene is a thin layer of carbon bonded together in a hexagonal honeycomb lattice. Graphene has very high electrical conductivity when external dopants are added. Samad et al have discovered that a range of textiles could be coated with graphene through a facile cladding technique to produce electrical conductivity. Electrical conductivities of about 5 S/cm and 50 mS/cm were attained for otherwise electrically insulating nylon and cotton threads, respectively. Wool and glass fibers were made to have conductivities of 10 mS/cm and 0.6 S/cm, respectively. The coating is uniformly distributed, and cannot be destroyed by washing or sonication [21] (Fig 1.5). Yazdanshenas also fabricated graphene-coated cotton textiles and investigated its conductivity. They used a dip and dry method to freeze graphene oxide (GO) on the surface of a cotton textile. In order to convert the GO to graphene, they immersed the textile into an aqueous solution of reducing agents, which were $NaBH_4$, N_2H_4, $C_6H_8O_6$, $Ba_2S_2O_4$ and NaOH. The resultant resistances are shown in Table1. 2 [22].

Table 1.2: Surface resistance of the GO–cotton textiles.

Sample	Surface resistance ($k\Omega cm^{-1}$)
$NaBH_4$–GO–cotton	34.600
NaOH–GO–cotton	23.300
N_2H_4–GO–cotton	62.7
$C_6H_8O_6$–GO–cotton	31.2
$Ba_2S_2O_4$–GO–cotton	19.4

Liu and his group have also created grapheme nanocomposite sheets (GNSs)-cotton fabric, which showed good electrical conductivity. They used a simple brush-coating and drying technique for the fabrication. The fiber have shown good flexibility, and good adhesion between GNSs and cotton fibers [23]. Xiang et al. have coated Kevlar fibers with graphene nanoribbons through a spray-coating technique. The fiber had conductivity of 20 S/cm [24].

Like carbon nanotubes, graphene flakes have a high contact resistance between overlapping flakes. Thus these type of graphene coatings result in low conductivity and thus cannot be used in most applications. Graphene can be deposited in larger single sheets instead of flakes using, for example, chemical vapour deposition. These graphene sheets would result in higher conductivity, but the deposition costs are too high.

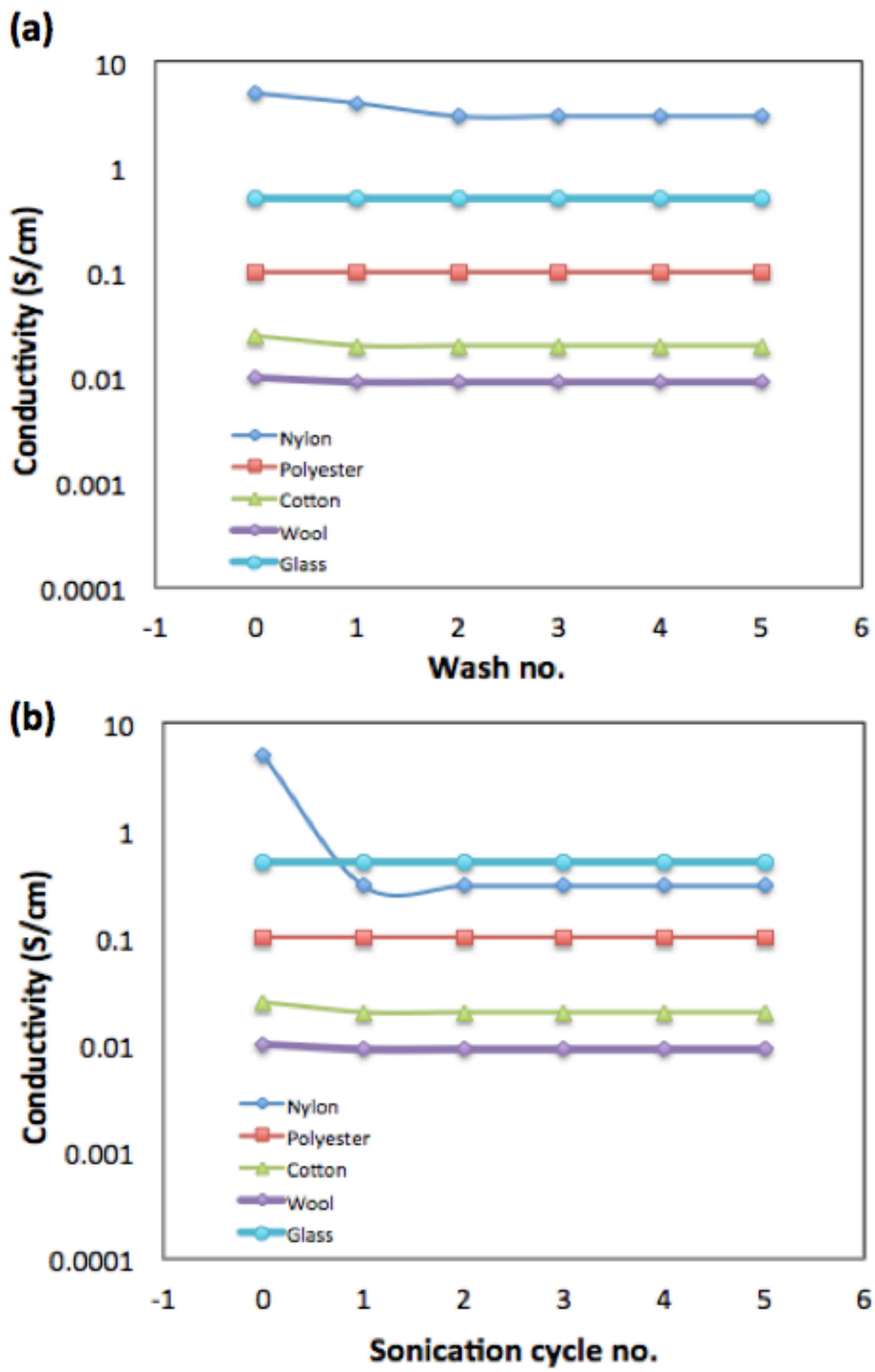


Figure 1.5: (a) Electrical conductivity vs. wash number of graphene coated threads and (b) vs. sonication cycle number. (Reproduced with permission from [21])

1.3.3 Conductive polymer coated threads

In order to create electronic devices with novel properties, conductive polymers have been used since the early 19th century [25]. Devices such as organic solar cells (OSCs) [26] and organic light emitting diodes (OLEDs) [27] are low-cost, mechanically flexible and lightweight. A simple solution-based dip coating process can be used to coat a textile with a conductive polymer to render it conductive [25].

Irwin and his group fabricated a conductive non-metallic textile and shown its successful function. They have coated silk fibers with A double-layered graphene/poly(3,4-ethylenedioxythiophene):poly(styrenesulfonate) (PEDOT:PSS) via a dip-coating method. They found that PEDOT:PSS is an appropriate polymer for the application and the conductivity was 8.5 S/cm. Additionally, Irwin et al. discovered that PEDOT:PSS had no negative influence on the mechanical properties of the conductive textile. They tested the conductive textile as an interconnect in circuit and it worked well [25].

Although conductive polymer coatings are cheap, light-weight, and easy to deposit, they have drawbacks. One of the main problems of conducting polymers is that they are unstable in air because of the absorption of oxygen and moisture [28]. Additionally, the conductivity of polymer-coated thread is low [29] for most applications.

1.4 Silver nanowires

A nanowire is a nanostructure with a cylindrical shape, where the diameter is ~100 nm or less and the length is hundreds of nanometers or more. It is considered to be a 1D nanostructure. There are several different types of nanowires that have been synthesized: superconducting, metallic, semiconducting, and insulating. Nanowires can be synthesized using either top-down or bottom-up methods. In top-down methods, a material is reduced from the macroscale down to the nanoscale. In bottom-up methods, atoms are self-assembled to synthesize the nanowire.

Silver is a good material choice for creating conductive threads because it is the most conductive of all metals, is more cost effective than gold or platinum, and is relatively stable in air. Simple and inexpensive methods exist to fabricate them. Silver nanowires have been used in many applications such as transparent electrodes. For example, Hu and his group have fabricated silver nanowires transparent electrode by using Meyer rod coating technique [30]. Silver nanowires can be synthesized in solution at relatively low temperatures by the now well-known polyol method [31]. In a common implementation of this method, a mixture of $AgNO_3$ and ethylene glycol (EG) is regularly added to a solution containing EG with polyvinylpyrrolidone (PVP) and NaCl and is heated to 170 ◦C. Ag nanoparticles seeds are first formed, which grown upon the continual adding of the silver nitrate. The PVP is a polymeric capping agent and is the key to the creation of 1D nanostructures, as they allow the silver to grow only along one direction. A slight variation of this process is be shown in figure 1.6 (platinum seeds are used to catalyze the growth of the NWs instead of first having to create silver seeds).

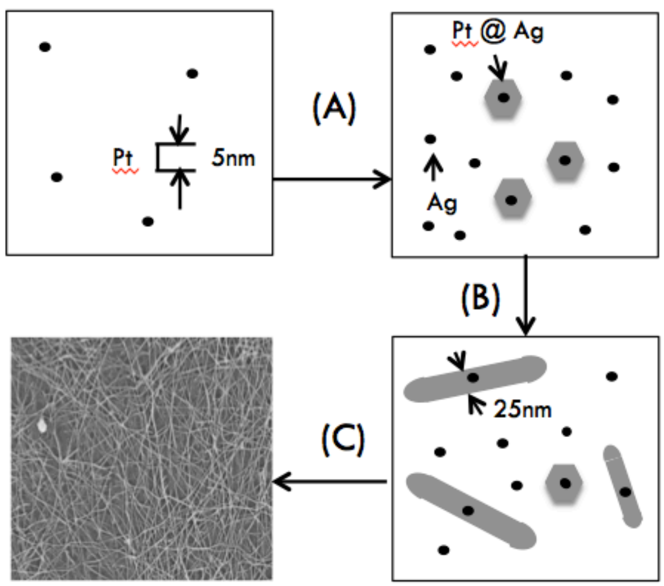


Figure 1.6:: The synthesis process of the silver nanowires: A) Creation of silver nanoparticles. B) Growth of the elongated-shape silver nanostructure. C) Evolution of the silver nanorods into wires. .(Reproduced with permission from [32])

The resulting nanowires are crystalline with five twin planes running along the longitudinal axis as shown in figure 1.7.

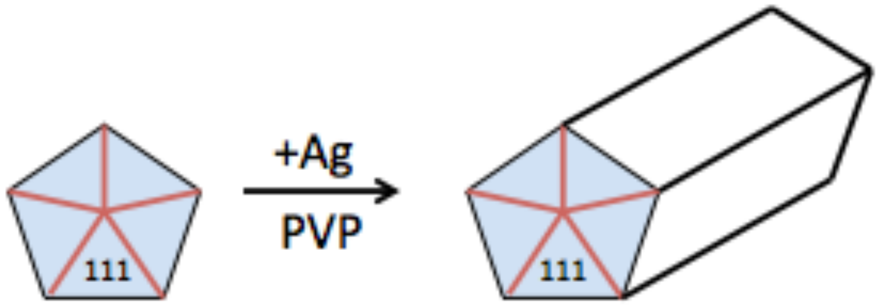


Figure 1.7: Cross-sectional image of a silver nanowire (Reproduced with permission from [31])

1.5 Metal nanowire coated thread

The use of metallic nanowires in textiles has only been briefly mentioned in the literature with little accompanying data. Cui and his group have created electrically conductive textiles by combining silver nanowires and fabrics together using a dipping-drying technique [33]. They showed that electrically conductive textiles were formed by adhering the silver nanowires not only onto the surface of the textile but also into the gaps and spaces in the textile. The textile was made of cellulosic material (100 mm x 100 mm x 250 μm). They obtained a low resistivity (0.0047－0.0091 Ω) and good stretchability (strains of 0%－190%).

Schoen and his group have fabricated a high-speed electrical sterilization of water using silver nanowires, carbon nanotubes, and cotton to kill highly concentrated bacteria [34]. Nateghi et al. have created multifunctional cotton fabric by depositing silver nanowires on the surface of cotton. A dip and dry coating method was used by immersing the cotton fabric in a silver nanowire solution. Their fabric was capable of killing the bacteria and had an average surface electrical conductivity of 27.4 Ω/sq. The conductivity did not vary after repeated bending [35].

In all the above, it was entire textiles rather than threads that were coated with nanowires. Furthermore, the papers focused on applications rather than the investigation of the deposition process, characterization of various textile properties such as washability and current-voltage curves, cost analysis, the dependence of resistance on nanowire density, deposition on different types of fabrics, etc. There do exist papers where silver nanoparticles, with spherical shapes, are used to coat threads to make them conductive [36]. However, the conductivity of metallic-nanoparticle coated threads is low because their small size leads to many inter-particle junctions. Unlike silver nanoparticles, the elongated shape of a nanowire allows for a conductive film to be achieved at a far lower particle density and therefore there are less junctions. Other benefits of obtaining conductive threads using nanowires are that metal junctions can be sintered to greatly reduce junction resistance, unlike carbon-based materials. Nanowire coatings can be deposited as a mesh rather than a continuous film, and the nanowires are less than 100 nm thick. Therefore, much less metal is used compared to conventional conductive threads where a metal film coating or a solid wire is used. This can lead to a lower material cost, lower weight, and thinner thread, as well as greater mechanical flexibility. The coating can be simply deposited as a dye with no vacuum or complex processes required.

1.6 Organization of this thesis

In this thesis I show that nylon, polyester, and cotton can be coated with a thin metallic mesh made up of a random network of silver nanowires.

The coating process is discussed in Chapter 2, including the procedure developed, coating materials used, and the improvement of nanowire adhesion to synthetic threads. Characterization of the properties of nanowire-coated threads is discussed in Chapter 3. This includes I-V measurements, bending tests, washing tests and using the conductive thread as a heater. Lastly, in Chapter 4 a comparison to other conductive thread technologies is outlined and future work is presented.

Chapter 2
Synthesis of nanowire-coated threads

This chapter explains a process for fabricating silver nanowire-coated threads. Improving nanowire adhesion to synthetic threads is discussed. Cost and density calculations are also explained.

2.1 Overview of coating materials and procedure

Polyester, nylon, and cotton threads were purchased commercially. The cotton and polyester threads were multifilament with diameters of 200 µm and 300 µm, respectively. The nylon thread was monofilament with a diameter of 700 µm. The silver nanowires used in this work were supplied in ethanol from Blue Nano Inc (Charlotte, North Carolina). Their average diameters and lengths were 35 nm and 15 µm, respectively. The silver nanowire concentration as-received was 20 mg/ml and was diluted further with ethanol to vary the density of the nanowires.

Deposition of nanowire films around threads was achieved through dip-coating. The procedure is illustrated in figure 2.1. All types of thread were cleaned in an ultrasonic bath using ethanol, alcohol, and deionized water for 5 min each. The NW solution was put in a vial and then a piece of thread was submersed in it. Generally, thread lengths of 3 cm were used. The vial was then shaken for 5 minutes. Then, the thread was left in air to dry for 5 minutes. After that the thread was re-dipped if multiple dips were desired to achieve a denser nanowire coating.

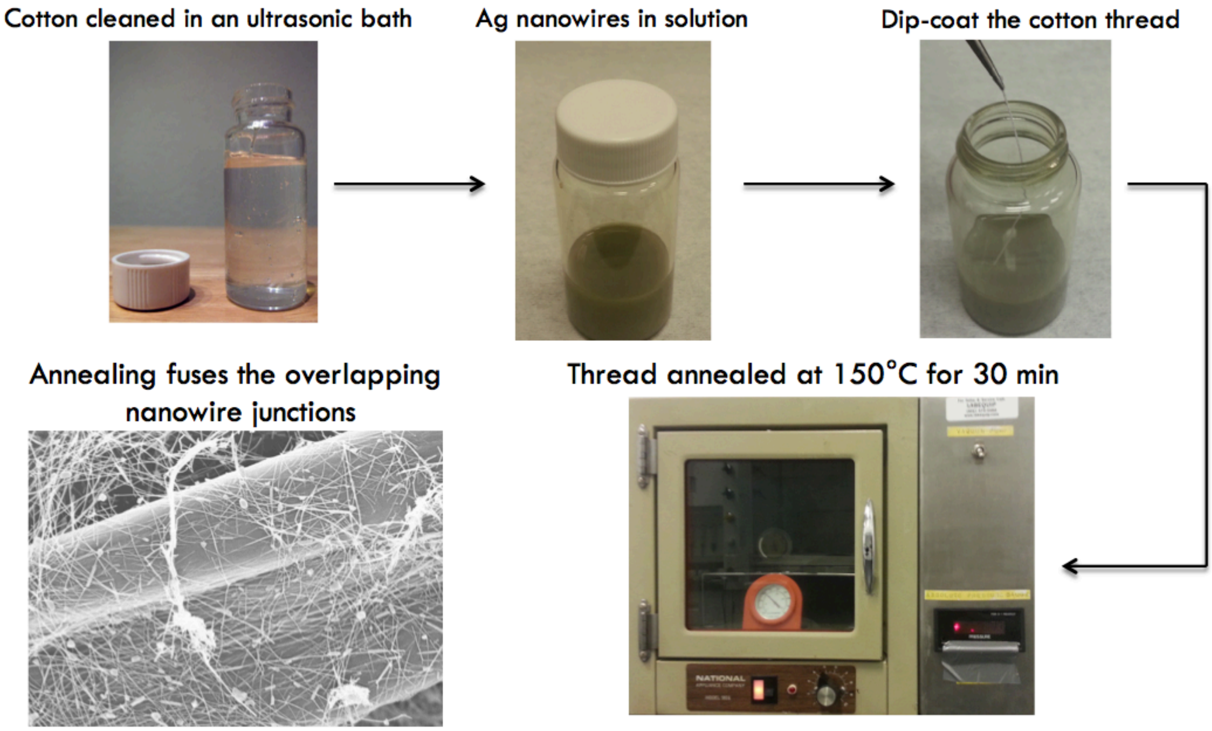


Figure 2.1: Procedure for depositing nanowire films around threads

The nanowire solution adhered well to the surface of the cotton thread, and the thread was conductive. This was not the case with polyester and nylon. These latter two synthetic textiles required a chemical pre-treatment before they were coated with nanowires. After cleaning, the polyester threads were submersed for 6 minutes in a solution consisting of 20 wt. % NaOH and 80 wt. % distilled water heated at 75 °C, then dried in hot air. The nylon threads were submersed in a solution consisting of 91 wt. % ethyl acetate and 9 wt. % resorcinol for 1 minute and then dried in air. These chemical treatments will be discussed in further detail later in this chapter.

In all the cotton, polyester and nylon cases, the density of the deposited nanowire film was varied through the concentration of the nanowires in the coating solution and through the number of dipping steps. After deposition the threads were annealed in air at 150 °C for 30 min. The annealing was done in atmosphere in a laboratory oven, as pictured in figure 2.1. This annealing step is required because without it, the resistance of the nanowire coating is very high because overlapping nanowires do not make good contact with one another. Furthermore, after the polyol synthesis of the silver nanowires, they have a layer of polymer (PVP) on their surfaces. This PVP layer is an insulator. Annealing both partially decomposes the PVP layer and fuses the nanowire junctions through sintering. Various annealing temperatures and times were investigated, as will be discussed in the following subsections, with 150°C and 30 min giving results of lowest resistance.

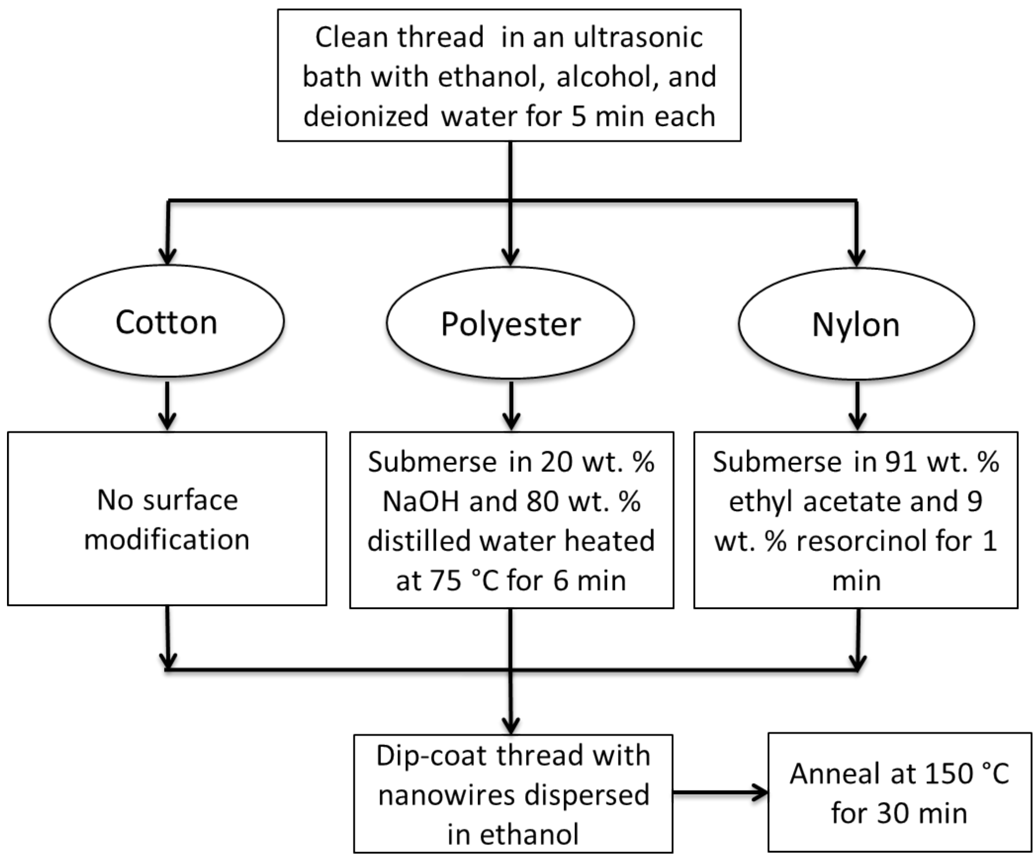


Figure 2.2: Coating procedure of the conductive thread

Figure 2.2 shows the full procedure I developed to prepare conductive threads. In the following subsections, I present data from which this final procedure above was decided upon, as well as discuss in more detail the process conditions and variations of the procedure. SEM images of the various conductive threads can be seen in figure 2.3.

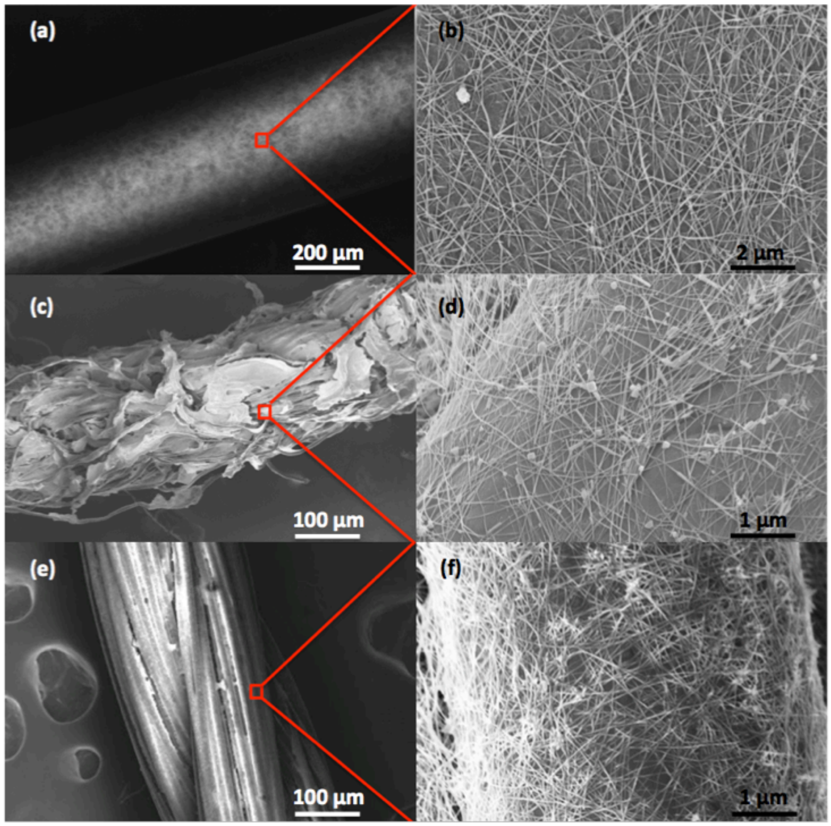


Figure 2.3: SEM images of silver-nanowire-coated (a),(b) nylon thread, (c),(d) cotton thread and (e),(f) polyester thread.

2.1.1 Fabrication of silver nanowire-coated cotton thread

The effect of number of dip-cycles and annealing temperature on the resistance of silver nanowire-coated cotton thread was investigated and displayed in Table 2.1. Extended annealing times were also tested and discovered to have little or no positive consequence on resistance minimization. Accordingly, annealing time was set at 30 min. Thread dipping time was five minutes, as extended dipping times had no effect.

Table 2.1: Resistance data of ($\boldsymbol{\Omega/cm}$) for dip cycles vs annealing temperature. Annealing time was 30 minutes.

Dip cycles	Annealing temperature (^oC)				
	100	**125**	**150**	**175**	**200**
1	43	42	41	42	43
2	32	31	30	31	32
3	24	23	22	23	23
4	13	12	11	12	13
5	7	6	5	6	7

Looking at the table, the annealing temperature that led to the lowest resistance was 150 ^oC, and thus for subsequent experiments, this was the annealing temperature chosen. Temperatures lower than 150 ºC may not be sufficient to fuse nanowire junctions and decompose PVP. For annealing temperatures above 150 °C, the resistance was higher due to the melting of nanowires into disconnected segments.

A cotton thread before coating with nanowires was imaged by SEM (Figure 2.4) with low magnification. Figure 2.5 shows cotton thread after coating with 1 dip and 5 dips. As expected, the density of the nanowires on the thread is higher with the increased number of dips. This is consistent with the data in Table 2.1, which shows that resistance decreases with the number of dip cycles. A high number of nanowires that are coated means more conductive pathways.

The cotton threads were washed in ethanol, alcohol, and deionized water before nanowire deposition to eliminate contaminants. As seen in Table 2.2, this pre-treatment slightly lowered the obtained final resistance. After one dip cycle, an 11.7 % decrease in resistance is detected with the pre-treatment compare to no treatment, and the effect reduces to a 7.5 % decrease in resistance after 5 dip cycles. Pre-treatment solvents methanol, isopropanol, acetone, and toluene all produced similar results.

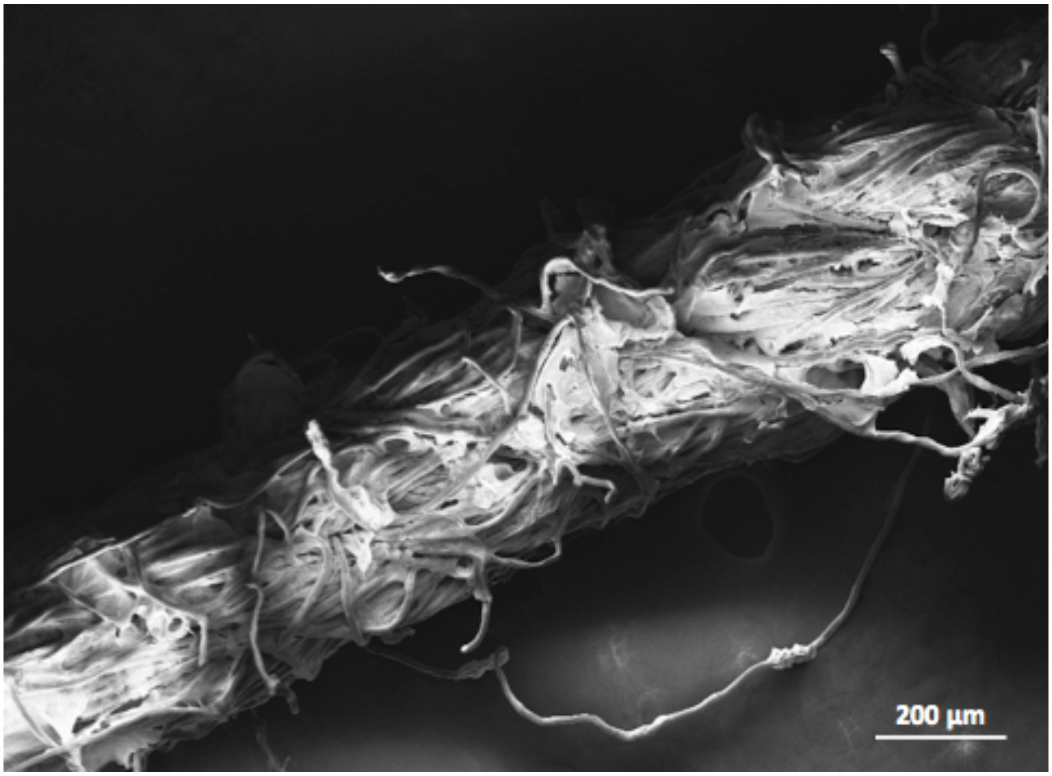


Figure 2.4: SEM image of cotton coated thread with low magnification

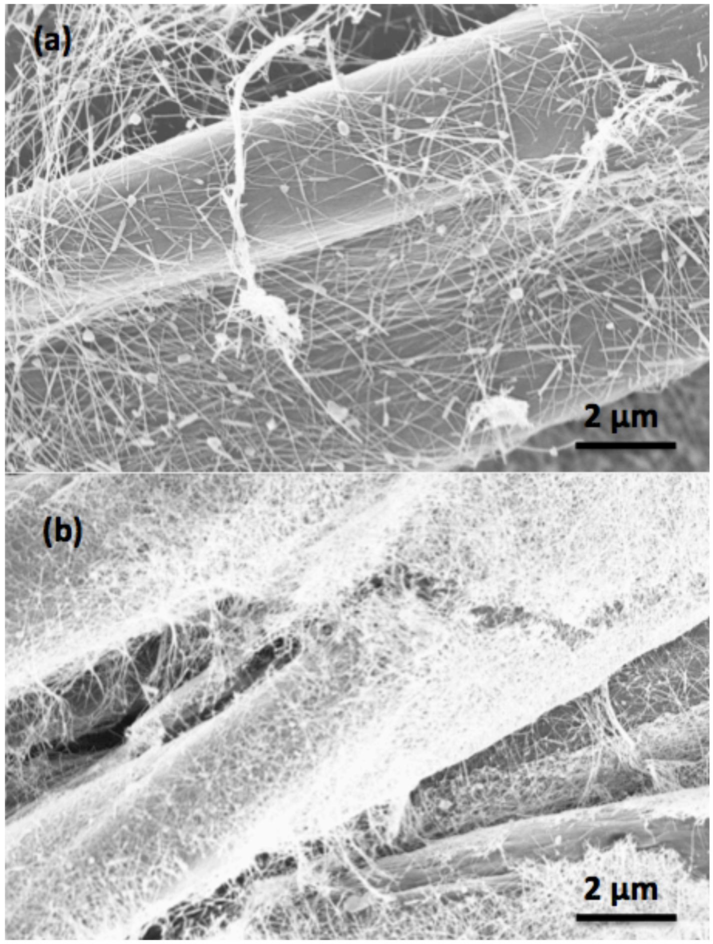


Figure 2.5: SEM images of a) 1-dip cotton coated thread b) 5-dip cotton coated thread.

Table 2.2: Comparison of threads washed with ethanol, alcohol, and deionized water compared with untreated ones

Dip cycles	**1**	**2**	**3**	**4**	**5**
Resistance untreated ($\boldsymbol{\Omega/cm}$)	41.2	30.1	22	11.5	5.3
Resistance pre-treated ($\boldsymbol{\Omega/cm}$)	36.4	28.5	20.3	10.4	4.9

2.1.2 Mixing the nanowire coating with cellulose

Deposition of nanowire films around threads was achieved through dip coating, drop casting, or both together combined. As mentioned earlier, when the nylon thread was dipped in the NW solution, the solution did not stick and no conductivity could be measured. Before trying chemical pre-treatments, I first tried using cellulose to thicken the coating solution and improve adhesion. The density of the nanowire film deposited was varied through the concentration of the nanowires in the coating solution and through the number of dipping steps.

The first coating method tried was dip-coating the nylon thread in the cellulose/nanowire solution. Table 2.3 shows the effect of cellulose concentration and number of dip cycles on the thread resistance. The silver nanowire solution concentration was 10 mg/ml.

Table 2.3: Resistance data of ($\boldsymbol{\Omega/cm}$) vs dip cycles and the concentration of cellulose used using the dip method.

Dip cycles	Concentration of cellulose			
	0.05 g/ml	**0.10 g/ml**	**0.15 g/ml**	**0.2 g/ml**
2	60	42	25	-
3	30	25	14	-
4	15	12	7	130
5	5	4.5	1.5	100

SEM images were taken of some samples in the above table (figures 2.6, 2.7).

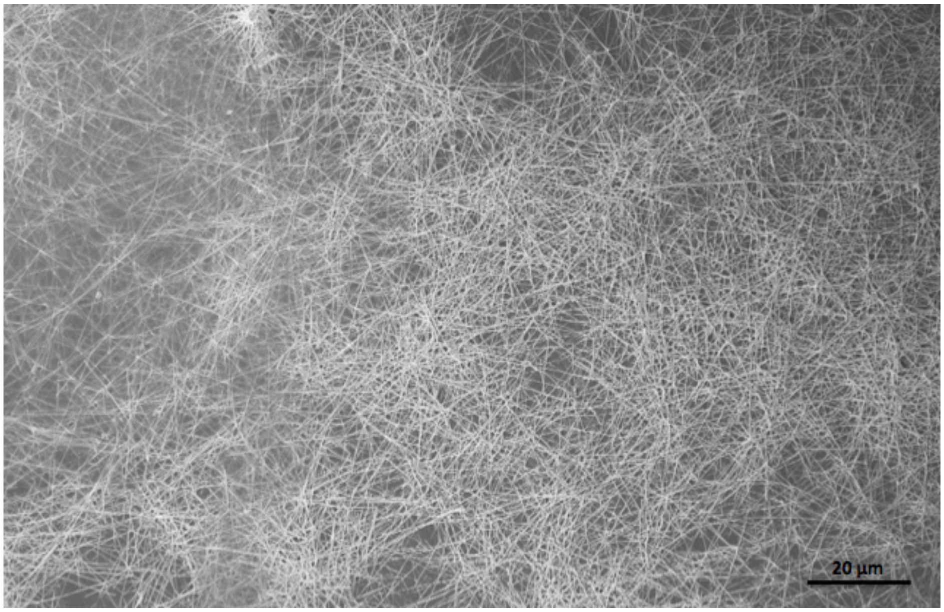


Figure 2.6: The surface of nylon thread fabricated with 5 dips, with a cellulose concentration of 0.10 g/mL.

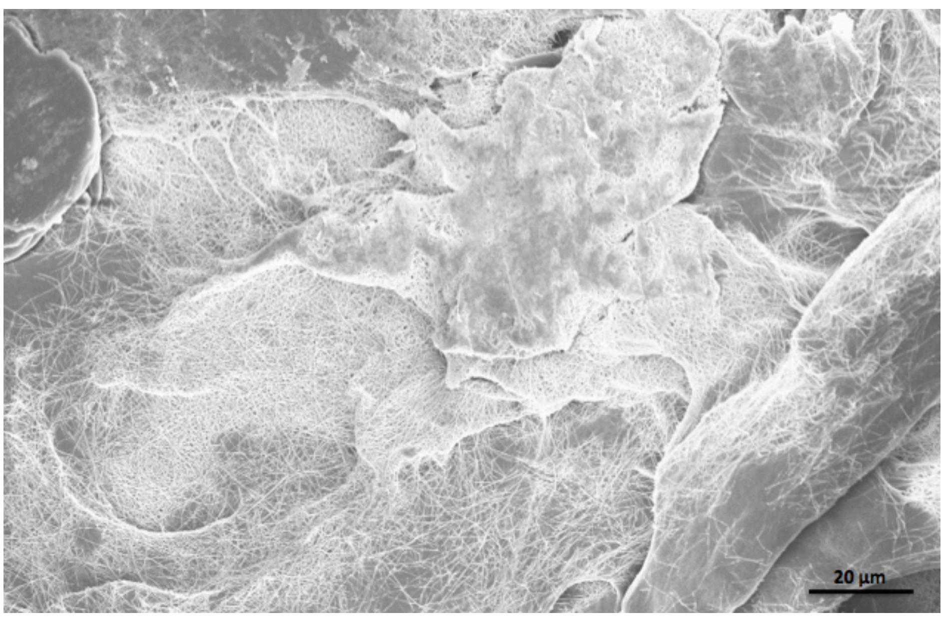


Figure 2.7: The surface of nylon thread fabricated with 5 dips, with a cellulose concentration of 0.15 g/mL.

With the use of cellulose, it was obvious by eye that the NW solution was thickened and that the solution now adhered to the nylon thread. Furthermore, it was now possible to measure a resistance. From the table we can see that the increase of cellulose concentration decreased the resistance until a concentration of 0.15 g/ml. For 0.2 g/ml and higher, the resistance increases with the increase of the cellulose because the silver nanowires are too far from each other and there will be less connections between them (i.e. the cellulose gets between the nanowires). Although mixing cellulose with the nanowire solution was successful in getting the nanowire coating to stick to the nylon thread surface to achieve conductivity, the silver nanowires coating was not uniform, as can be seen in Figures 2.6 and 2.7.

To improve uniform distribution of the nanowire coating around the thread, the second deposition method tried was drop casting using a pipette. The pipette was used in order to put the solution of the top surface of the thread. The thread then was flipped over and solution was pipetted on the other side. The solution was left on the thread for 5 min on each side to dry, then the entire thread was annealed as usual. Table 2.4 shows samples coated using solutions with various nanowire concentrations.

Table 2.4: Resistance data vs dip cycles and the concentration of cellulose using a drop casting method

Dip cycles	Concentration of cellulose				
	0.025 g/ml	**0.02 g/ml**	**0.017 g/ml**	**0.014 g/ml**	**0.0125 g/ml**
1	312	-	-	-	-
2	200	240	-	-	-
3	46	50	120	-	-
4	30	34	65	115	146
5	16	21	35	70	100

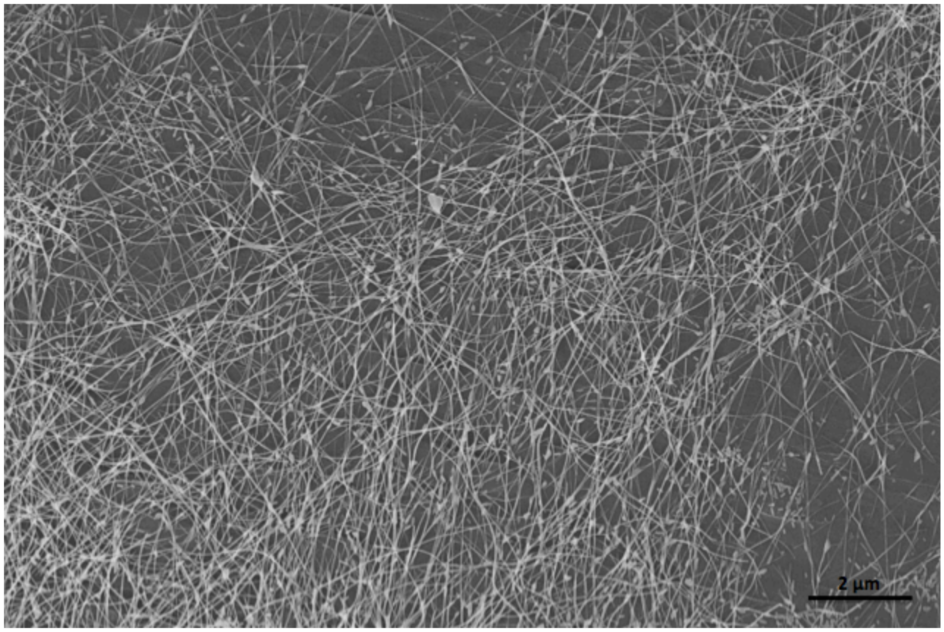


Figure 2.8: Nylon thread coated with a solution that has a cellulose concentration of 0.0.025 g/mL.

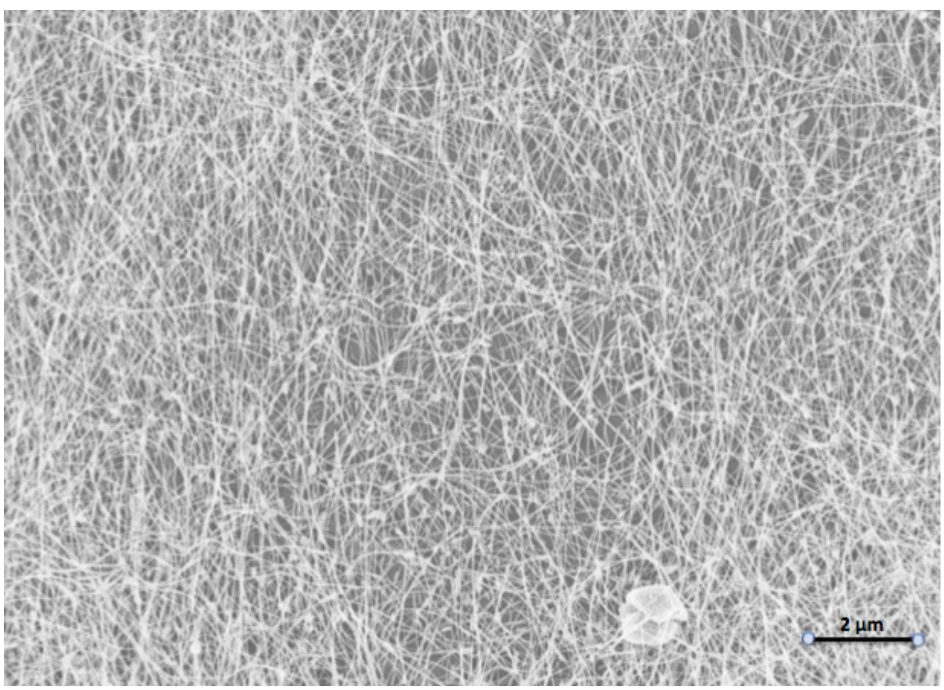


Figure 2.9: Nylon thread coated with a solution that has a cellulose concentration of 0.0.017 g/mL.

As can be seen in Figures 2.8 and 2.9, the uniformity of the nanowire coating was improved. However, with drop casting, the silver nanowires could not be coated on all sides of the thread but rather only on two sides.

The third method was the combination of the previous two methods, which the thread was dipped first into the solution and then the silver nanowires was drop casted. Table 2.5 displays some samples that were fabricated using this method.

Table 2.5: Resistance data vs dip cycles and concentration of cellulose using dip coating and drop casting combined.

Dip cycles	Concentration of cellulose		
	0.017 g/ml	**0.014 g/ml**	**0.0125 g/ml**
1	50	70	-
2	36	45	65
3	27	37	43
4	19	28	35
5	16	20	29

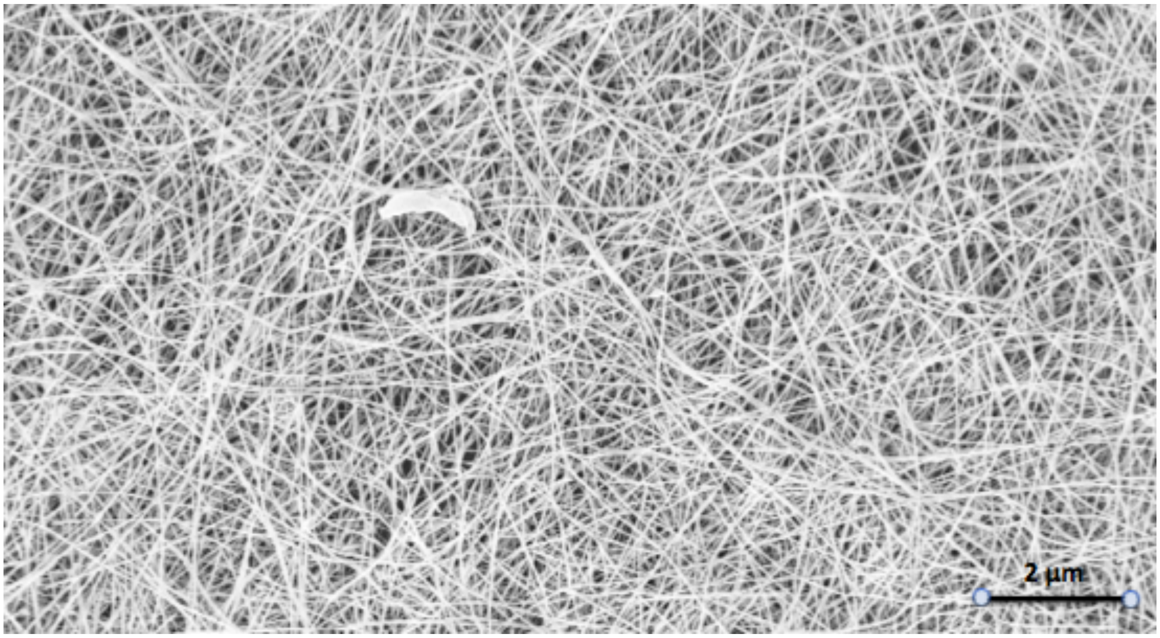


Figure 2.10: The surface of nylon thread coated using the combination method of both dip coating and drop casting. This sample was dip-coated 3 times.

The uniformity of this method is good, as shown in figure 2.10, but the problem is that the silver nanowires is very dense due to the multiple deposition steps.

2.1.3 Surface modification of textiles

Because thickening the nanowire solution with cellulose to achieve coating adhesion was problematic, I then investigated chemically modifying the surface of the threads prior to nanowire coating. Silver nanowires synthesized by the polyol process have a 1 – 3 nm thick PVP layer on their sidewalls. This polymer coating is hydrophilic. Because cotton is also hydrophilic, surface modification was not required for nanowire adhesion. Nylon and polyester, on the other hand, are hydrophobic.

2.1.4 Coating silver nanowires on nylon thread using surface modification

Nylon contains (-CO-NH-) groups, which are hydrophobic. Attaching polar groups to the surface increases the hydrophilicity. The nylon threads were submersed in a solution consisting of 91 wt. % ethyl acetate and 9 wt. % resorcinol for 1 minute and then dried in air. This is a common pre-treatment for planar nylon films. Resorcinol is a dihydroxybenzene with the formula $C_6H_4(OH)_2$. After immersing the nylon in resorcinol, polar hydroxyl groups are created on the surface of nylon.

The nylon thread was immersed in the ethyl acetate/resorcinol solution for different times: 8, 30, 45, and 60 seconds. SEM images were taken in order to see the whether the solution noticeably etched the thread surface (figure 2.11). As can be seen in the figure, the chemical pre-treatment did not noticeably effect the smoothness of the thread surface, and longer immersion times actually helped in removing contamination on the surface. Threads were coated with the nanowire solution (without cellulose) after each pre-treatment immersion time and the resistance decreased with increasing the time of modification. Since 60 seconds gave the results, this amount of time was chosen for our final coating procedure.

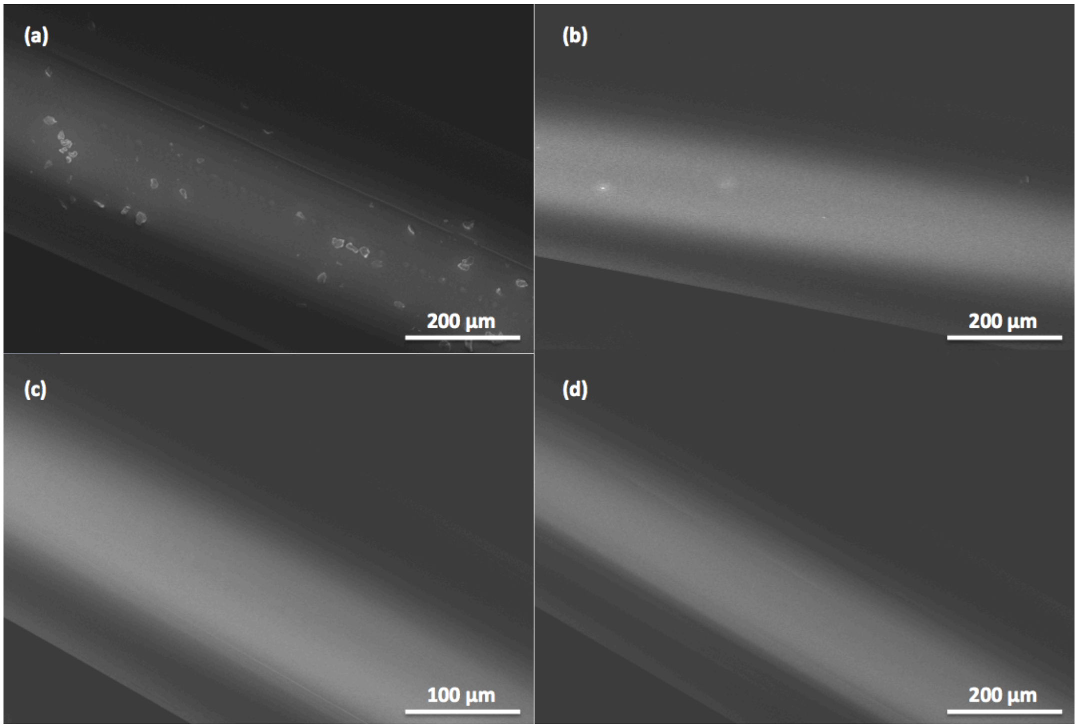


Figure 2.11: SEM images of nylon thread immersed in resorcinol solution for a) 8 s, b) 30 s, c) 45 s, and d) 60s

Using the resorcinol pre-treatment for 60 s, I collected data on how the concentration of silver nanowires in the dipping solution and the number of dips affected the resistance of the thread, to see the possible ranges of resistances that are obtainable. Because resistances can be measured at all, the pre-treatment was clearly successful in increasing the adhesion of the nylon. Compared to cellulose, the advantage of using a chemical-pre-treatment is that the uniformity of the nanowires mesh is superior, as shown in figure 2.12, and at the same time the nanowire density is more controllable.

Table 2.6: Resistance data vs. dip cycles and silver nanowire concentration using surface modification

Dip cycles	Silver nanowire concentration (mg/ml)				
	10	**5**	**4**	**3.3**	**2.8**
1	33.4	614	-	-	-
2	12	42	146	-	-
3	8.4	16	41	75	-
4	4	10	24	45	50
5	3	6	14	20	35

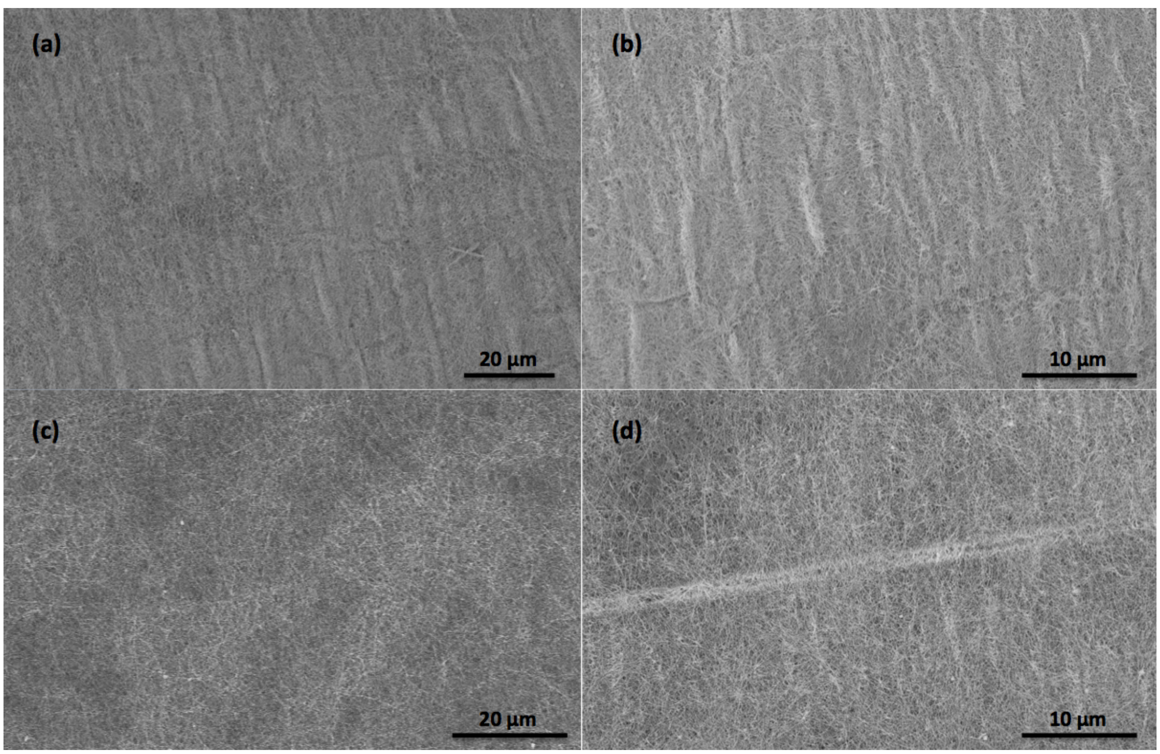


Figure 2.12: SEM images of the surface of nylon thread dipped 3 times in a nanowire solution with a concentration of a) 10 mg/ml , b) 5 mg/ml, c) 2.5 mg / ml, and d) 2.8mg/ml.

2.1.5 Fabrication of silver nanowire-coated polyester threads using surface modification

Because the surface of silver nanowires are hydrophilic, dipping polyester thread in the nanowire solution resulted in a resistance per unit length of > 300 Ω/cm (post-annealing) and the nylon thread did not have any measureable conductivity. The polyester threads were submersed for 6 minutes in a solution consisting of 20 wt. % NaOH and 80 wt. % distilled water heated at 75 °C, then dried in hot air. NaOH is commonly used in the textile industry for scouring, which removes impurities, and is also known to render polyester more hydrophilic [37]. The NaOH produces polar hydroxyl groups along the polyester chains which can then form hydrogen bonds with the carbonyl groups of PVP, thus resulting in nanowire adhesion.

As in the last section, the dependence or resistance on the silver nanowire solution concentration and the number of dips was tabulated (Table 2.7). The low resistances achieved indicate that the NaOH pre-treatment worked well. SEM images of a couple sample from this table are shown in Figures 2.13 and 2.14.

Table 2.7: Resistance data of ($\boldsymbol{\Omega/cm}$) vs. dip cycles and the silver nanowires concentration of polyester thread using surface modification

Dip cycles	silver nanowire concentration (mg/ml)				
	10	**5**	**4**	**3.3**	**2.8**
1	110	-	-	-	-
2	20	90	-	-	-
3	15.4	31	60.5	-	-
4	4.3	15.7	23.6	57.2	73
5	3.7	11	14.5	25	51

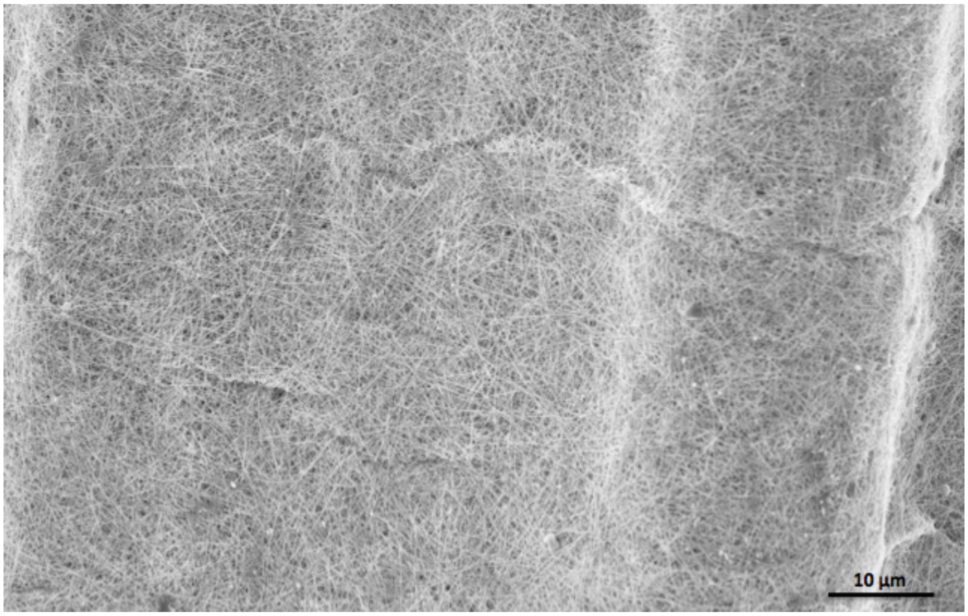


Figure 2.13: Polyester thread pre-treated with NaOH, then immersed for 4 dips in a solution having a nanowire concentration of 10 mg/ml.

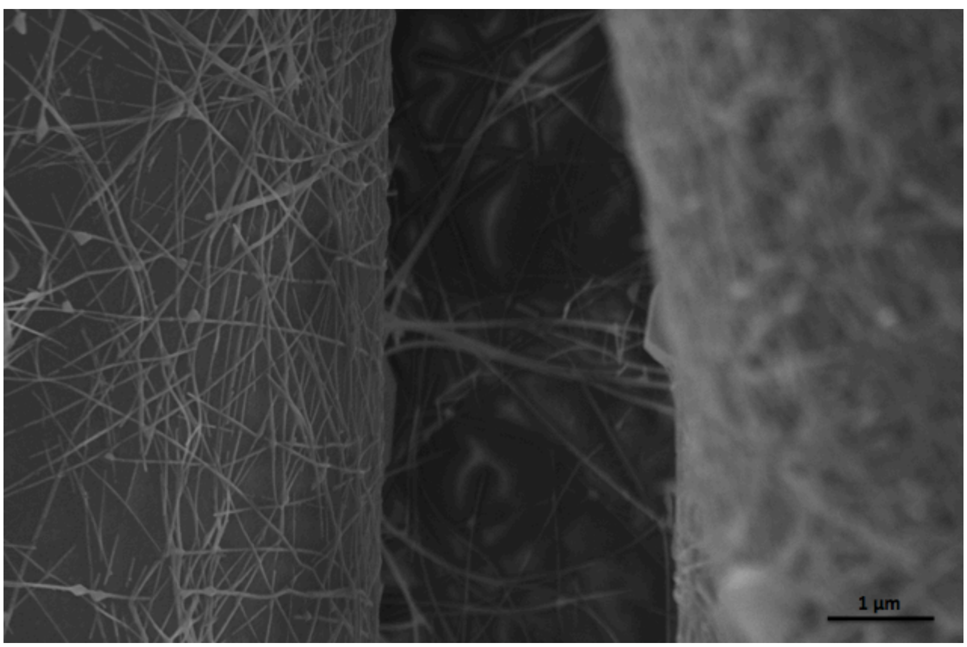


Figure 2.14: Polyester thread pre-treated with NaOH, then immersed for 4 dips in a solution having a nanowire concentration of 2.8 mg/ml.

2.2 Cost and density calculations

The dependence of resistance on density, as well the material costs of the nanowires [38], are tabulated in Table 1 for the nylon threads. A resistance as low as 0.8 Ω/cm was achieved. 0.8 Ω/cm in this case is equivalent to a sheet resistance of 0.18 Ω/square, and the conductivity of the composite (using the cross-sectional area of the nylon thread) is 81 S/cm. For comparison, nylon thread coated with the conductive polymer PEDOT:PSS has been reported to be 40 Ω/cm [39], and nylon coated with carbon nanotubes has been reported to be 49 kΩ/cm [40].

Although silver is an expensive material, so little of it is used so costs are low (Table 2.8). The density of nanowires was determined from SEM images and Image J. The brightness of the images was first reduced to reduce the amount of grey pixels, then the threshold was adjusted to convert all pixels into either black or white. The white pixels are the nanowires. The number of white pixels was divided by the number of total pixels to find the areal coverage of nanowires. By knowing the dimensions of the image, the areal coverage of nanowires per micrometer square could be calculated. By knowing that the nanowires are cylindrical, and knowing that their diameter is 35 nm, this number could then be converted into a volume of nanowires per area. Multiplying the volume by the density of silver nanowires converts this number into a mass per area. Dividing by the circumference of the thread gives mass per length of thread.

The above technique worked well for images with a low density of nanowires. But for higher densities, it was less accurate since there were too many areas with overlapping nanowires and this method would underestimate the density. Instead, for threads dipped multiple times in the nanowire coating, I first calculated the density after 1 dip, and then calculated the density after two dips. The density of the final coating was calculated using = (Density after dip 2) + (total # of dips – 2)*(density after dip 2 – density after dip 1).

In regards to weight, around 1 mg/m or less is added to the thread. If the coating were instead a typical 1 μm thick silver film the coating would weigh 23 mg/m, so the nanowire coating is less than 5% the weight of a solid film.

Table 2.8: The dependence of resistance and material cost on the coating density of the nanowires on nylon thread.

Metal density (mg/m)	Resistance per length (Ω/cm)	Nanowire cost ($/m)
0.24	12	0.008
0.52	2.5	0.017
1.07	0.8	0.035

To calculate costs, the metal density was multiplied by the cost of nanowires per mg. The cost to make silver nanowires is more than the cost of silver alone because added reactants, such as ethylene glycol, are essential to create silver nanowires. The cost of silver nanowire production is $32.58/g (table 2.9) if the nanowire solution is created in the lab, and therefore, the material cost of the thread would be 0.035 $/m for a 0.8 Ω/cm thread, and less for threads with higher resistances.

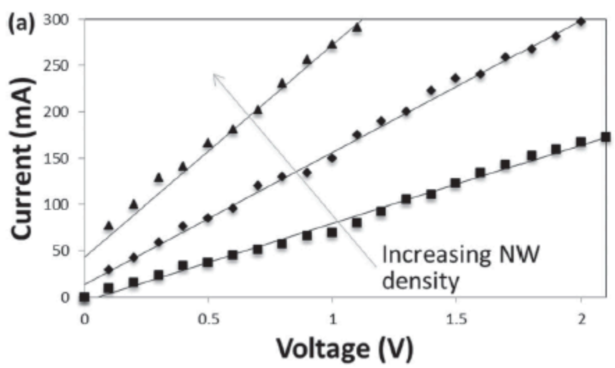
Table 2.9: Reactants used to create the silver nanowires an their cost [31]:

Reactants	$/g Ag nanowires
Ethylene glycol	27.00
Silver nitrate	5.02
PVP	0.56
Ag nanowires	$32.58/g

Chapter 3
Characterizing the properties of nanowire-coated threads

3.1 I-V measurement

To measure resistivity, copper tape was affixed to each end of the nanowire-coated thread and connected to a multimeter using alligator clips. As is typical for conductive threads, the resistance of the threads will be stated in resistance per unit length of thread, or Ω/cm. The current–voltage relationship was obtained using a multi-meter and a direct current (DC) power supply. Figure 3.1a shows the I-V relationship of nylon thread coated with three different nanowire densities. The curves are linear and thus conduction behaves like a metal. In Figure 3.1b, nanowire-coated thread is used in a circuit to power an LED.



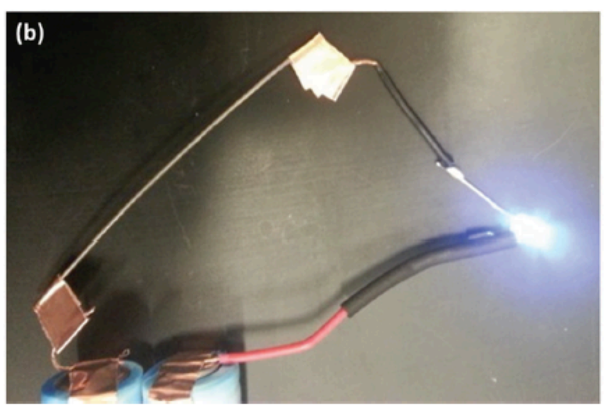


Figure 3.1: (a) Current-voltage curves of nylon thread coated with different nanowire densities. (b) Passing current through nanowire-coated nylon thread (top left of image) to power an LED

Silver nanowires are known to react with sulfur containing gases in the atmosphere and corrode over time [41]. To test the stability of the conductive threads over time, silver nanowire coated nylon thread was left out in air and its resistance was measured every day for one week. As can be seen in Figure 3.2 the resistance increased over time, and after 7 days the resistance increased by 24%, from 1.7 Ω/cm to 2.1 Ω/cm. The shape of the curve suggests that the resistance would continue to increase over time. Therefore, for application purposes, a passivation layer should be deposited over the nanowire coating to prevent degradation over time.

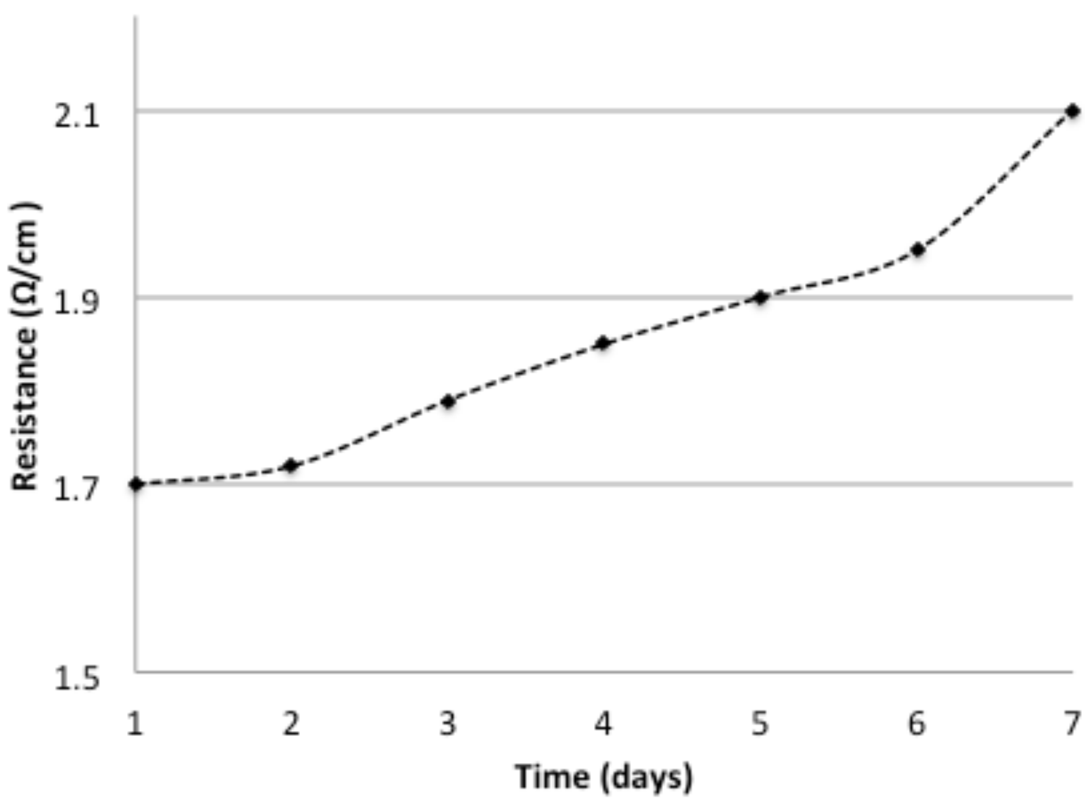


Figure 3.2: Change of resistance of a nanowire-coated nylon thread left in air for one week.

Repetitive dipping cycles increased the conductivity in a quasi-linear way (Figure 3.3). The concentration of nanowires in the coating solution was 10 mg/ml. After 6 dipping cycles, however, the conductivity no longer increased much with subsequent dips. Furthermore, because conductive material was obviously flaking from the surface of the thread at and beyond 10 dipping cycles, no further dipping cycles were done. The maximum conductivity achieved through this procedure was 45 S/cm.

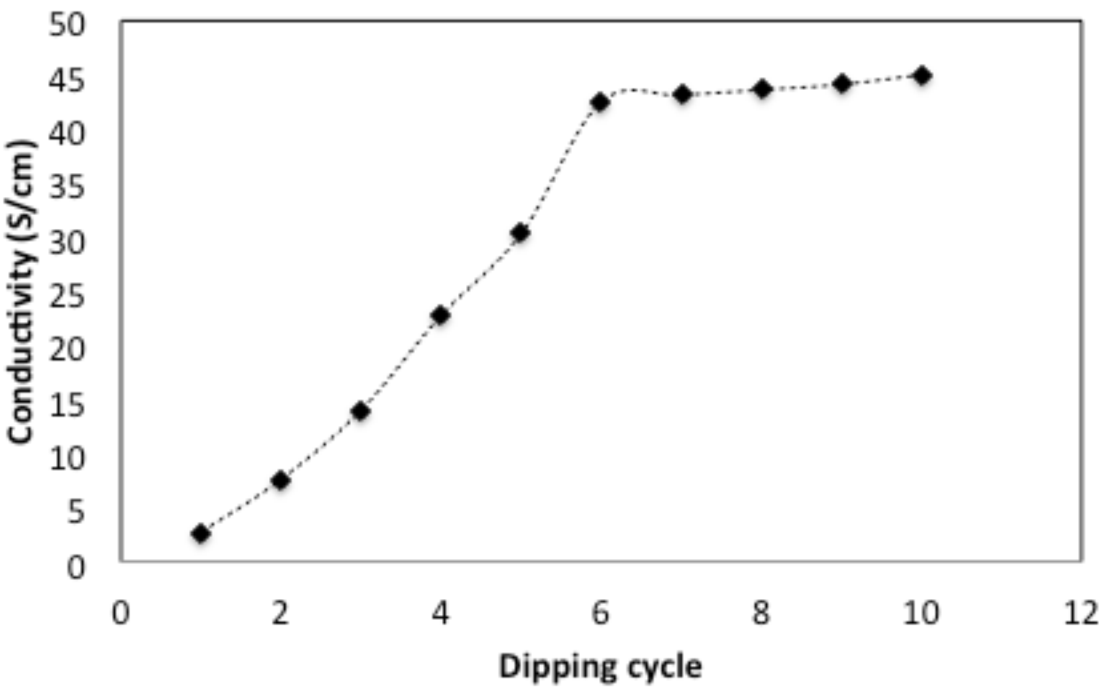


Figure 3.3: The conductivity increase for silver nanowire coated thread across multiple dipping cycles**.**

3.2 Mechanical properties

Conductive thread must be mechanically flexible and its resistance must not degrade significantly after repeated bends, especially when used in applications such as clothing-integrated sensors. A commercially available conductive thread was purchased from Shieldex. The commercial thread was multifilament nylon where the filaments were coated with a thin-film of silver. The variation of resistance with repeated bending for both the commercial conductive thread and a nanowire-coated nylon thread with the same initial resistance is plotted in Fig. 3.4. The resistance of the commercial thread increased by more than 4 times, from 2.8 Ω/cm to 12.2 Ω/cm after being bent 200 times. Silver thin-films are brittle and can crack after repeated bends [42], thus increasing its resistance. Films of silver nanowires, on the other hand, are much more flexible[43]; the cylindrical geometry and nanosized diameter of nanowires is known to make them stronger and more flexible than their bulk-like counterparts [44], and they can endure higher elastic strains [45]. The resistance of the nanowire-coated thread only increased 16% after 200 bends, from 2.8 Ω/cm to 3.2 Ω/cm. Furthermore, when the resistance of nanowire-coated threads was measured when the thread was bent (as opposed to measuring the resistance after returning the thread flat as was done to collect the data in Fig. 3.4), the resistance was actually lower than an unbent thread. For example, when an initially 2.8 Ω/cm nanowire-coated nylon thread was bent around a rod with a radius of 6 mm, its resistance decreased to 2.3 Ω/cm. This may be because bending causes mechanical forces that improve the connections between overlapping nanowires and thus lower their junction resistances. The resistance of the commercial thread, on the other hand, increased from 2.8 Ω/cm to 4.4 Ω/cm when bent.

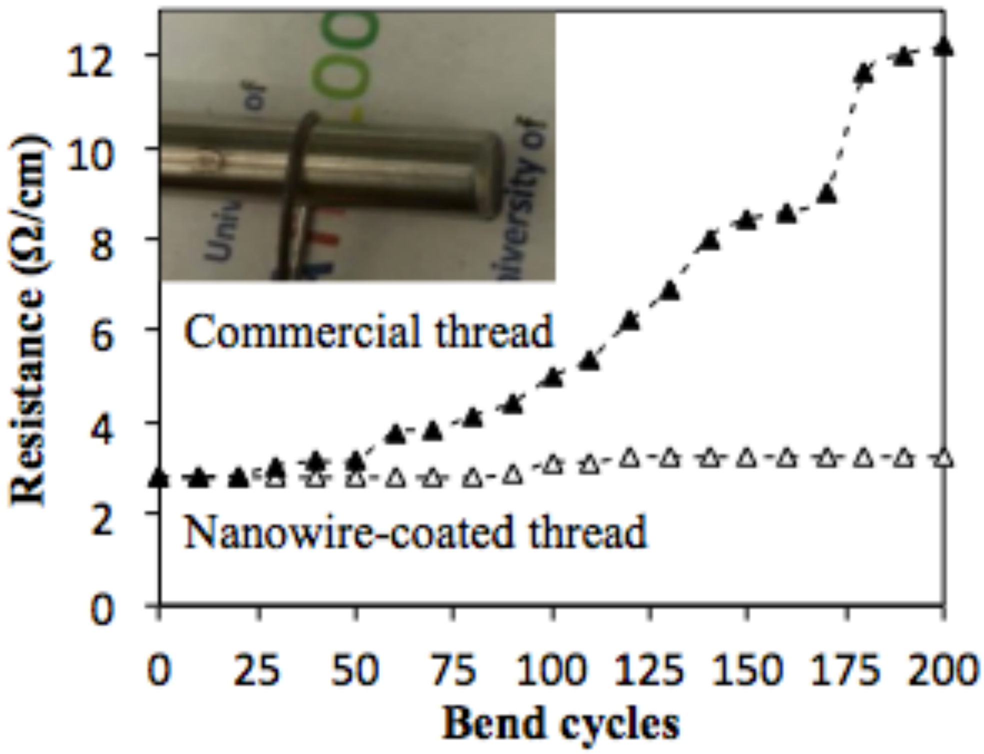


Figure 3.4: The resistance of the nanowire-coated nylon thread and a commercially available conductive thread after repeated bends to a 6 mm radius of curvature

3.3 Washing tests

Adhesion of the nanowire coating to the thread is another important parameter. Two kind of washing tests were done to investigate the adhesion of the nanowire coating to the nylon thread. First, a solution of distilled water and detergent was put in a vial and then the nylon conductive thread was immersed inside and shaken for 5 min, was allowed to settle for 5 minutes, and the shaking was repeated 4 more times. The resistance did not change as can be seen in figure 3.5.

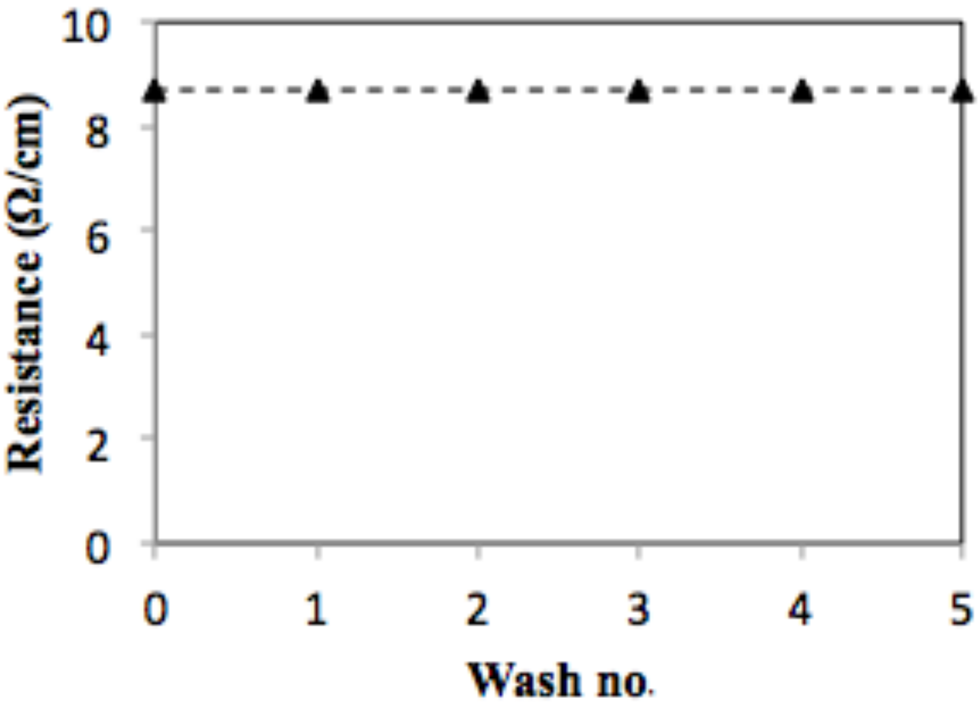


Figure 3.5: The resistance of the nanowire-coated nylon thread after shaking it in detergent

The second method of washing was immersing the nanowire-coated nylon in water plus detergent with a stir bar mixing the liquid. A magnetic stir bar, which is operated using a hot plate was set to 500 rpm and the water/detergent was heated to 25 ºC. The resistance of the thread was measured every 5 minutes over a period of 30 mins. Also, the commercial thread was washed with the same method. As can be seen in Fig. 3.6, the resistance of the nanowire-coated nylon thread did not change after five repeated washings in liquid detergent. This indicates that the coating did not come off or degrade and that the chemical pre-treatment of the nylon provides good adhesion. The resistance of the commercial thread also maintained its conductivity after the same washing procedure.

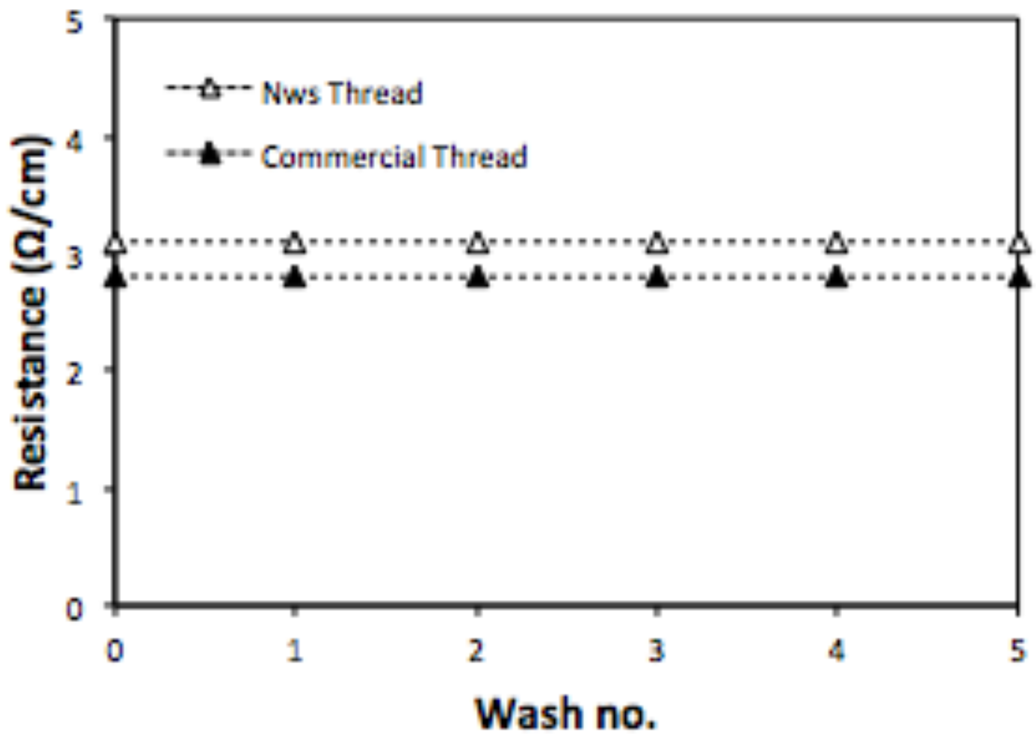


Figure 3.6: The resistance of the nanowire-coated nylon thread and a commercially available conductive thread after washing in a solution of detergent and water mixed with a stir bar.

3.4 Thread heater

Conductive threads can be used as a heating element, where electricity is converted to heat through Joule heating. Thread heaters can have applications in areas such as car seats, heating of clothing and as an anti-freezing material. In the past, the following were used as heating materials in clothing: metallic heating elements, conductive rubbers, and graphite elements. They have many limitations such as increased the mass of clothing, rigidity of systems, and limited extraction of sweat. The nanowire coating, on the other hand, is lightweight and flexible. Because the nanowires are only 35 nm, the coating is also very thin. This means that there will still be space between adjacent threads in a fabric where vapour (sweat) can pass.

Conductive threads with various resistances were fabricated using nylon or polyester threads, as described in Chapter 2. A DC power supply was used to apply a constant voltage for 300 seconds across the thread which generated a flowing current. The surface temperature was measured using a thermal couple. Temperature data was collected every second by connecting the multimeter to a computer as shown in figure 3.7. Then the cooling trend was also plotted after the voltage was disconnected from the thread.

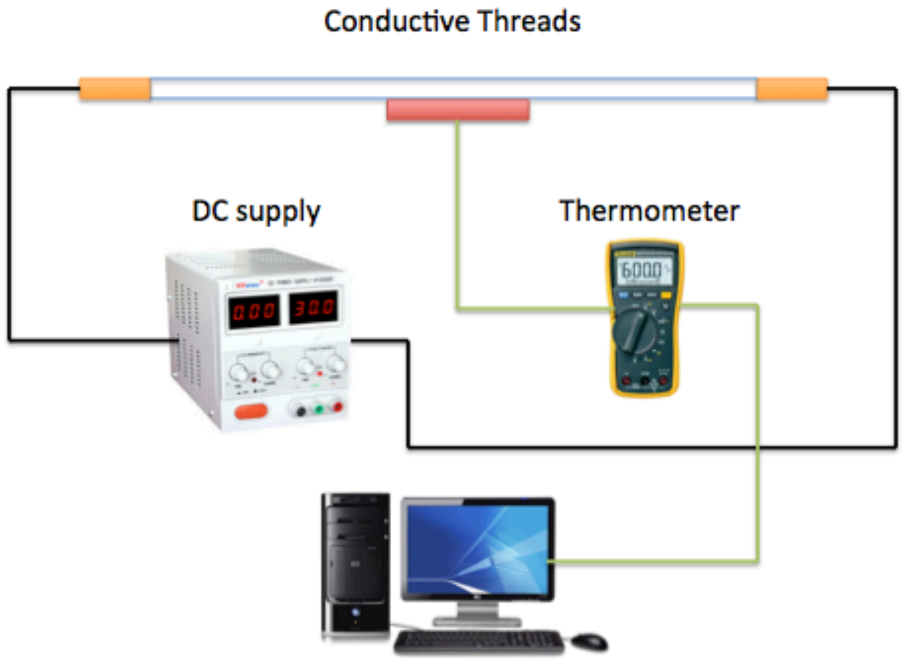


Figure 3.7: Operating and measuring the temperature of the thread heaters.

The temperature response of a nanowire-coated nylon thread of 10 Ω/cm to three different applied voltages is plotted in Figure 3.8. When a bias of 1.8 V was applied across a 1.4 cm long section of thread, a temperature above 50 °C was achieved, demonstrating its functionality at low voltages. Furthermore, for all magnitudes of voltage used, the steady-state temperature was achieved within 60 s, confirming a fast response time of the thread heater.

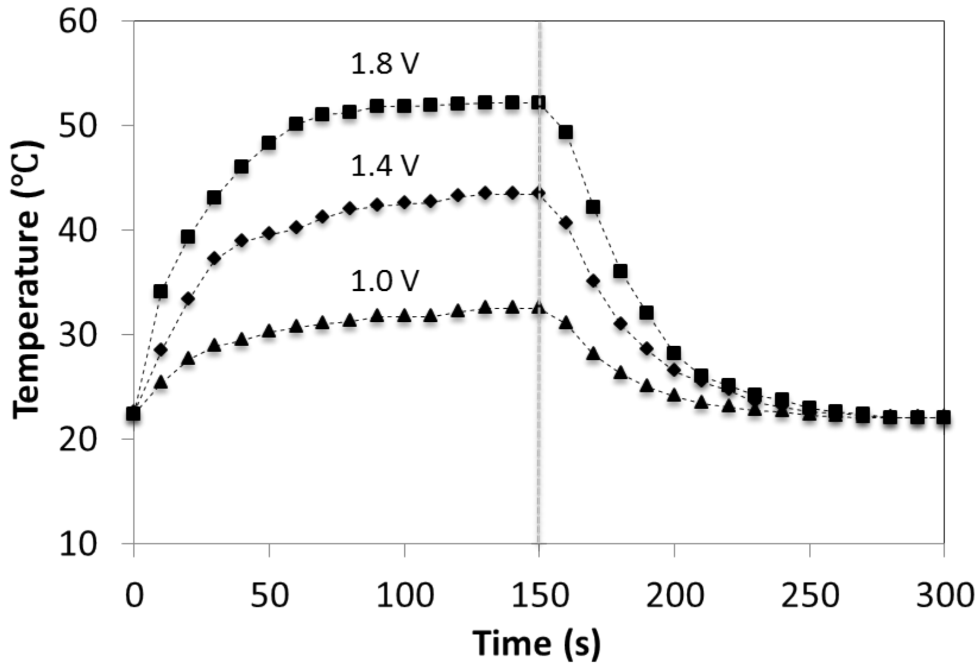


Figure 3.8: Temperature profiles of the thread heater at different input voltages. Voltages were applied across a 1.4 cm long section of thread for 150 s.

Figure 3.9 displays the time-dependent temperature profiles of the thread heater with respect to the resistance of the conductive thread. Under the application of the input voltage (1.4 V), the maximum temperature at steady state increases as the resistance of the thread decreases, which shows that the resistance of the conductive thread should be around or less than 4.3 Ω/cm to provide a maximum temperature above 50 °C at an input voltage of 1.4 V. For conductive threads with R= 8 Ω/cm, an input voltage of at least 1.6 V is required for the temperature to reach 50 °C. In the case of the conductive thread with R = 12Ω/cm, input voltages more than 2 V are needed to reach this temperature.

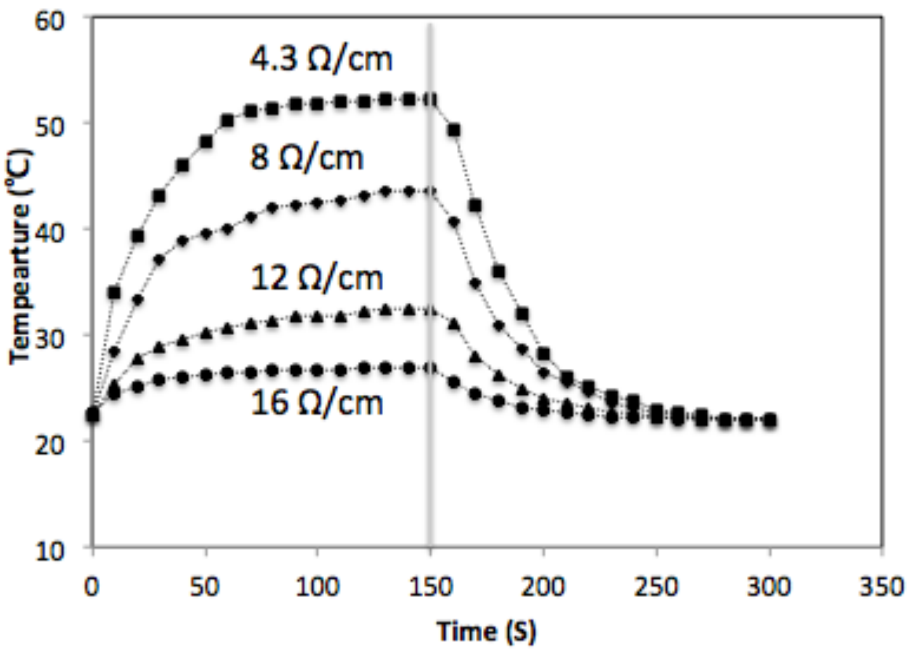


Figure 3.9: Time-dependent temperature profiles of thread heaters with different resistances.

Figure 3.10 shows the variation in temperature of a thread heater under repeated bending cycles. The resistance of the thread was 10 Ω/cm and the applied voltage was 1.8 V. The temperature was taken when the thread was bent which displays no obvious increase or decrease in the temperature. The conductive thread was bent to a curvature of 6 mm.

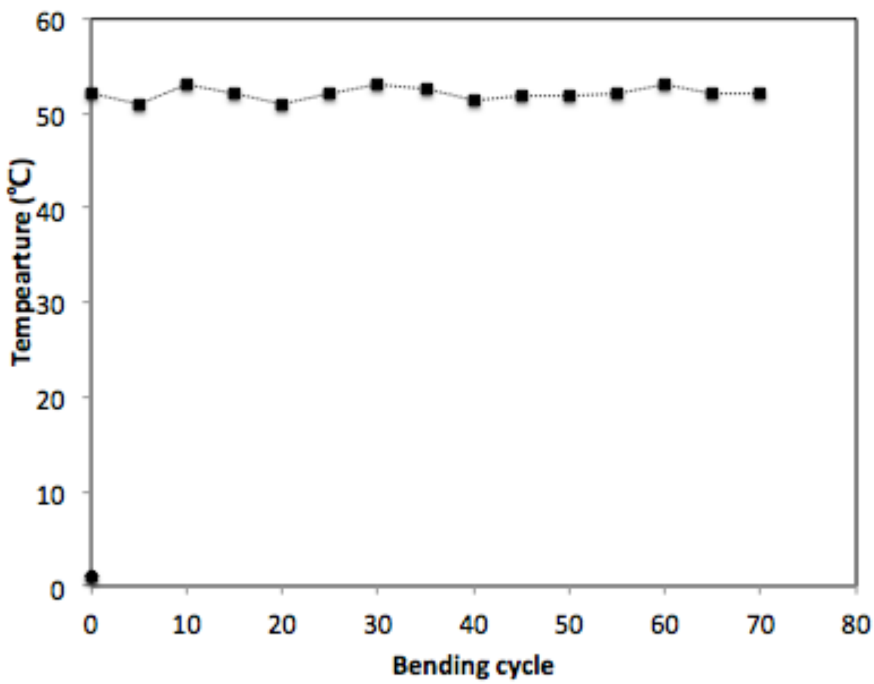


Figure 3.10: Variation in temperature of a thread heater under repeated bending cycles.

In order to examine the long-term performance of the silver nanowire thread heaters, an input voltage of 1.8 V was constantly applied to a thread for two days and the surface temperature of the thread were measured continuously. The resistance of the thread was 4.3 Ω/cm. The temperature was steady throughout the two days of the examination as shown in figure 3.11.

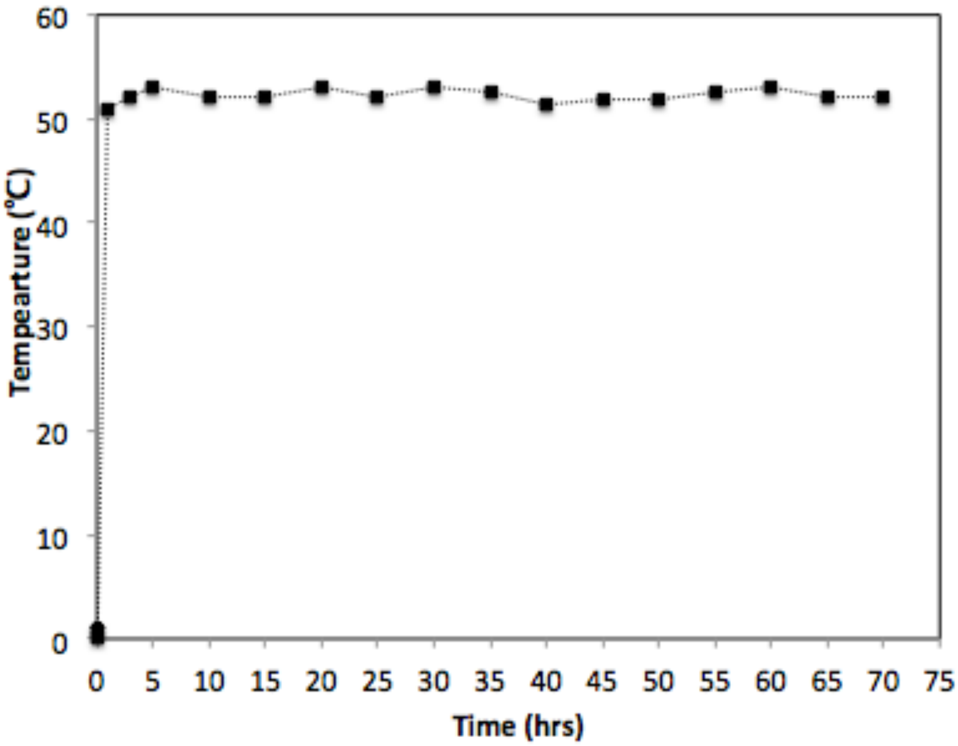


Figure 3.11: The temperature variation of 4.3 Ω/cm thread heater.

Chapter 4
Conclusions and future work

4.1 Summary and conclusion

In this thesis, cotton, polyester and nylon threads were successfully made electrically conductive by coating their surfaces with meshes of silver nanowires. The materials and coating procedure were developed and discussed, and a surface modification technique was used to improve nanowire adhesion to nylon and polyester threads. I-V measurements, bending tests, and washing tests were completed. I found that the nanowire coating is less than 5% the weight of typical silver thin-film coatings used in commercial conductive thread. The nanowire-coated thread is also more mechanically flexible, with its resistance increasing only 14% after 200 bending cycles while the resistance of the commercial conductive thread increased by more than 300%. The ability of the thread to function as a heater was demonstrated.

Silver nanoparticles already exist in commercially available consumer products, including textiles. Fortunately, silver nanowires have low cytotoxicity (toxicity to cells), and in the form of a connected film their toxicity is even less [46]. However, as with any nanomaterial, their health and environmental effects should be considered including their impact during their manufacture and disposal.

Overall, this work demonstrates a simple, economical, and functional conductive thread for e-textiles. Furthermore, these nanowire-coated threads may enable additional applications. For example, because silver nanoparticles have antimicrobial properties [47], these threads could be used in antibacterial dressings and clothing. And reports have also used silver nanowires in biosensors [48],[49], which opens the possibility of textile-integrated sensors.

4.2 Future work

4.2.1 Using nanowire coatings as a transparent electrode for solar cell threads

Solar cells convert sunlight into electrical power. Integrating a solar cell with thread would be very useful because they could sewn into ‘regular’ textiles and be used to generate energy in clothing to power personal electronics. They also could allow for integration of solar cells into tents and blinds.

Solar cells require two electrodes. In a thread configuration, the outermost electrode must be optically transparent so that light can enter the solar cell. In planar solar cells, this transparent electrode is made from indium tin oxide (ITO). ITO, however, is brittle. When bent, cracks form in the ITO and its conductivity degrades. ITO has been used as a transparent electrode in solar cell thread [50], but these threads lack flexibility which is a particular problem for textiles. Other groups have used alternative materials as the transparent electrode in solar cell thread [51], [52] but these threads had low energy conversion efficiencies due to the low transparency of the electrode material. Because light can pass through the open spaces of the mesh, nanowire coatings can be used as a transparent electrode for solar cell thread. Nanowire films have already shown high promise as a transparent electrode for planar solar cells [53–55]. To make solar cell thread, polymer solar cell materials will be coated around thread, with the outermost layer being the metal nanowire mesh which both allows photons to enter the device and the generated electricity to be collected (figure 4.1). I have already shown that nanowire coatings are threads are mechanically flexible. Furthermore, other groups have already shown that efficient planar solar cells can be fabricated using silver nanowire electrodes. Therefore, our solar cell thread should be both flexible and efficient, and may pave the way for textiles such as curtains and clothing to generate power from the sun.

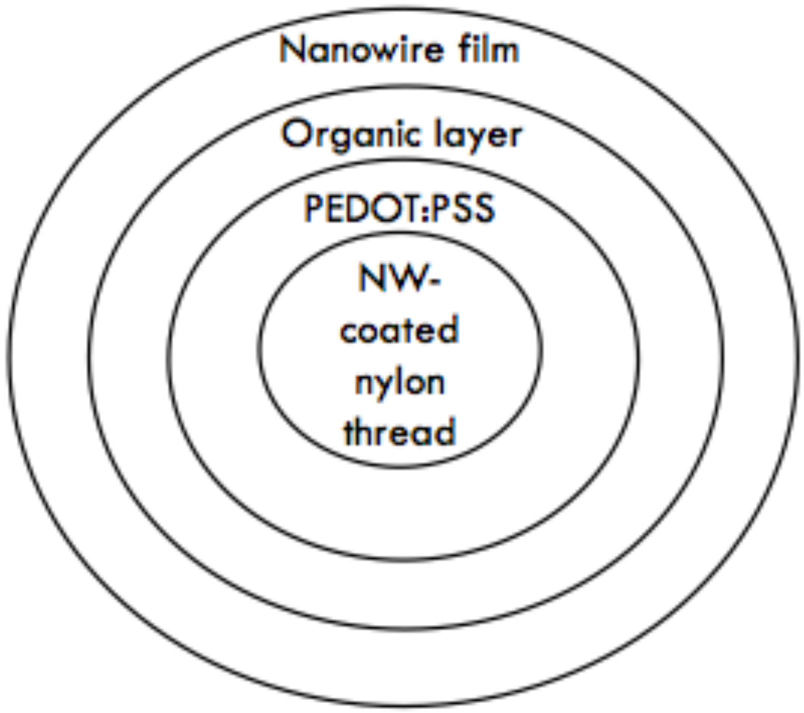


Figure 4.1: Schematic diagram of s solar cell thread using silver nanowires as both the inner electrode and the outer transparent electrode

4.2.2 Evaluating the antimicrobial properties of the thread

The antimicrobial properties of silver (Ag) ion or salts are well known. The silver nanowire-coated threads developed in this thesis may thus have anti-microbial properties. The silver nanowire coated thread will improve some items that need protection from microbes such as hospital furnishings, muscle supports, and outdoor gear. Furthermore, sportswear industrialists wanting to provide odor-free and antimicrobial footwear is also a possible market. Therefore, investigating the anti-microbial properties of silver nanowire-coated thread would be an interesting and perhaps very useful endeavor.

4.3 Papers based on this thesis

Portions of this work was published in the Journal of Materials Chemistry C [56]. A conference paper and a talk based on this work was also presented at IEEE NANO 2014 [57].

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