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High durability conductive textile using MWCNT for motion sensing

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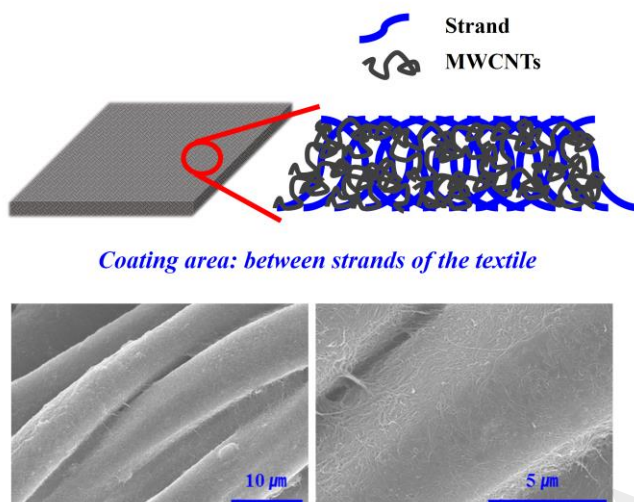
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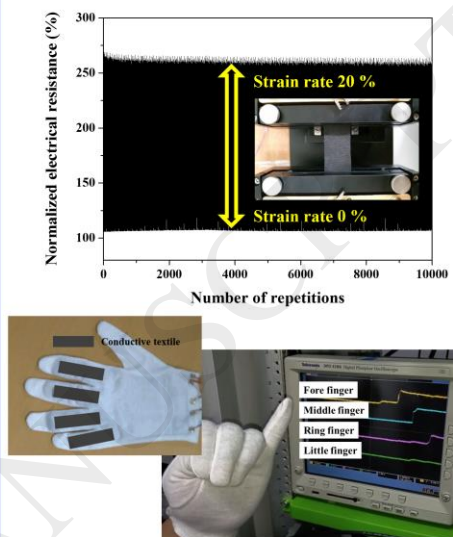
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Graphical Abstract:

Conductive textile fabricated by vacuum filtration of MWCNT ink



Evaluation and application for motion sensing



Highlights:

- A conductive textile was fabricated by vacuum-filtration of water-based MWCNTs ink.
- The conductive textile showed resistance variations of less than $\pm 3\%$ after 10,000 cycles of stretching, and the pulse of the resistance variation at a strain rate of 0%–20% remained uniform during the stretching cycles.
- A motion-sensing glove fabricated with the conductive textile showed that the pulse of the oscilloscope changed accurately with movements of the fingers.

Abstract

A conductive textile was fabricated by vacuum-filtration using conductive ink prepared from multi-walled carbon nanotubes. The fabricated conductive textile was evaluated as its

resistance varied while it was subjected to repeat stretching at strain rates of 0% to 20%. The textile samples showed resistance variations of less than $\pm 3\%$ after 10,000 cycles of stretching, and the pulse of the resistance variation at a strain rate of 0% – 20% remained uniform during the stretching cycles. A motion-sensing glove fabricated with the conductive textile showed that the pulse of the oscilloscope changed accurately with movements of the fingers. These results show that the conductive textile prepared in this study can be applied to motion-sensing products.

Keyword: conductive textile; MWCNT; vacuum-filtration; resistance variation; motion sensing

1. Introduction

The demand for wearable devices has increased exponentially in recent years. So far, most of the commercialized wearable devices have been limited to accessories such as watches, bands, and glasses. To improve existing products, more flexible and biocompatible wearable parts have been studied [1-4]. In addition, there have been many research studies on integrating clothing and smart devices [5-7], which makes it necessary to maximize the flexibility of smart device parts. Stretchable conductive textiles are also an important element in smart clothing. Conductive textiles can be used as sensors for monitoring human motion through changes in the electrical resistance to varying strain rates. Smart clothing for sensing motion would be useful in various fields such as sports,

games, and medical care. In particular, smart clothing is expected to be useful in the medical field due to increasing life expectancy. As the aging population increases worldwide, smart clothing will be increasingly adopted as routine elements of preventative medicine, monitoring and treatment of chronic diseases [8-10].

Low-cost home healthcare monitoring systems, which include smart clothing based on textile sensors, flexible circuits, and wireless communication [11-12], have attracted the attention of researchers and medics [13]. Smart clothing for health monitoring will help to improve the quality of life and decrease medical expenses for disease management by providing long-term sensing of bio-signals with minimal intrusion in the daily activities of patients. Smart clothing that can sense bio-signals of electrocardiograms, electromyograms, respiration, body movement, and skin temperature is already under development [14].

Conductive textiles with excellent durability and reliability are essential to the development of smart clothing for health monitoring. Many studies have been conducted to fabricate conductive textiles according to these demands. Previous studies have focused on methods of coating conductive materials on flexible polymer substrates [15-17]. Because this method can change the basic properties of the textiles, the feeling of fit may be reduced when applied to smart clothing. In addition, methods of mixing conductive materials with polymers such as PDMS or PMMA [18], dispensing silver nanowires [19], printing dry-spun CNTs [20], and aligning CNTs on polymer substrate have been introduced [21]. These manufacturing methods are not suitable for mass-production due to the high cost of materials or complicated processes. Transparent conductive films [22] and conductive cotton textile [23] have been studied using CNTs ink and dip coating process. Dip coating is simpler than any other manufacturing methods, and therefore it can be advantageous in

terms of manufacturing speed and cost when applied to mass-production. However, there are some considerations when applying the dip coating method to textile. First, the wettability differs depending on the type of textiles, and when a textile having a low wettability is used, the manufacturing time may increase and the uniformity of the coating state may be lowered. Dip coating have no parameters that can be controlled except CNTs concentration and immersion time. To compensate for this, additional factor is needed to apply external forces on the dip coating base. Second, dip coating is based on coating the entire area of the textile. Performance is the most important in the manufacture of smart textiles, but the design must also be considered for commercialization. In order to implement various designs, patterning of the coating area is occasionally required, which should be considered when applying dip coating.

To improve these problems, we propose a water-based MWCNTs (multi-walled carbon nanotubes) ink and a vacuum-filtration process. MWCNTs have excellent electrical conductivity and mechanical strength and is cheaper than other metallic materials. Because of these advantages, MWCNTs have been applied to various devices [24-25]. The vacuum-filtration process is simpler and cheaper than other manufacturing methods. In addition, the vacuum-filtration process can complement the dip coating method by designing the pump capacity and the structure of the equipment. The purpose of this study is to examine the possibility of mass production through fabricating and evaluating conductive textile using existing technologies. This study suggests a method of coating conductive materials directly on all types of textiles using simple technologies. This approach differs from the manufacturing method of printing on a flexible polymer substrate and has advantages in terms of maintaining the basic characteristics of the textiles. In general, screen-printing is

the most common method for printing on textiles; however, it has limitations in achieving good durability for stretching because it is only suitable for printing certain materials on the surface of textiles. To circumvent this limitation, a water-based MWCNT ink was coated on the textile by vacuum-filtration method to bond the MWCNTs between textile strands. To compare these differences, we fabricated conductive textiles using screen-printing and used commercial MWCNTs paste. From the viewpoint that the material should be prepared in a suitable form according to the process method, ink and paste based on MWCNTs were used for vacuum-filtration and screen-printing, respectively. Fig. 1 shows a schematic of the two coating methods for the preparation of conductive textiles.

2. Experimental procedure

The manufacturing process of the conductive ink for the vacuum-filtration method proceeded as follows. MWCNTs (Kumho Petrochemical, Korea) were used as the conductive material, and deionized (DI) water was used as the solution to disperse the MWCNT powder. HNO_3 (70%, Sigma-Aldrich, USA) was used for surface treatment of the MWCNT powder: the MWCNTs were oxidized to increase their distribution in DI water. The oxidation of the MWCNTs was carried out by mixing 1 g of MWCNTs in 200 mL of HNO_3 in a three-neck flask. The flask was connected to a reflux-cooling device, and the contents were stirred on a heating mantle for 8 hours at 150 rpm and 120 °C. The oxidized MWCNTs were centrifuged, and after the supernatant was discarded, DI water was added to the precipitate to neutralize the MWCNTs until a final pH of 5 was obtained. The oxidized and neutralized MWCNTs were then extracted by vacuum filtration of the

solution and placed in a petri dish, which was later dried in an oven at 70 °C for 24 hours.

Next, 500 mg of oxidized MWCNTs were added to 500 mL of DI water and were dispersed using a sonicator (VCX-500, Sonics, USA). The sonication was carried out for 20 minutes at a power of 400 W and a frequency of 20 kHz; the probe of the sonicator was 25 mm in diameter.

The textile used to prepare the conductive textile consisted of 93% polyester and 7% polyurethane. Polyester is a suitable substrate for motion sensing because of its excellent elasticity. Conductive threads (Amogreentech, Korea) were used to form the electrical contact points for measuring the electrical resistance. The conductive threads were stitched onto the textile at intervals of 8 cm. The MWCNTs ink was coated on the stitched textile using the vacuum-filtration process. The area coated with MWCNTs was approximately 100 cm² (10 cm × 10 cm) and was cut to a size of 10.5 cm × 2.5 cm. After vacuum-filtration, the wetted textiles were dried at 70 °C for 1 hour. Fig. 2 shows a manufacturing process for conductive textile by vacuum-filtration of MWCNTs ink. Samples of the same size were also fabricated by the screen-printing process to compare with the samples prepared by the vacuum-filtration process. Commercial MWCNTs paste (CP-01, Daewha Alloytech, Korea) was used in the screen-printing process. After screen-printing, the samples were dried at 150 °C for 30 minutes.

The conductive textile prepared by the vacuum-filtration method was attached to the finger joints of a glove to form a motion-sensing glove. The conductive textile was attached to the joints of four fingers except for the thumb so that the finger movements could be

confirmed by the changes in resistance of the conductive textile. A four-channel oscilloscope (DPO4104, Tektronix, USA) was used to observe the four fingers' movements in real time.

Thermogravimetric gravimetric analysis (TGA; SDT Q600, TA Instruments, USA) was performed to investigate the thermal stability of the MWCNTs before and after oxidation. The defect variations in the MWCNTs before and after oxidation were confirmed by Raman spectroscopy (LabRam HR, Horiba, Japan), and field-emission scanning electron microscopy (FESEM; S-4200, Hitachi, Japan) was used to examine the morphology of the conductive textile samples. A stretch evaluation system was constructed to measure the variations in electrical resistance according to changes in the length of the conductive textile; the stretch evaluation system consisted of a bending machine (Sciencetown, Korea) and a digital multimeter (2400 SourceMeter, Keithley, USA).

3. Results and discussion

Fig. 3 presents the thermal stability of the pristine and oxidized MWCNTs. The oxidized MWCNTs were more damaged and exhibited more defects than the pristine MWCNTs, which resulted in degradation of their thermal stability. Fig. 3 shows that this tendency appeared dramatically at temperatures of ≥ 300 °C.

The Raman spectra in Fig. 4 illustrate the defect rate in the oxidized MWCNTs. The D peak indicates the disorder in the sample and the G peak represents the graphite peak that is commonly observed in the Raman spectra of carbon-based materials. The intensity of the D peak increased after oxidation of the MWCNTs, and the ratio between the intensities of the

D and G peaks, I_D/I_G , was found to be 0.82 and 1.0 for the pristine MWCNTs and oxidized MWCNTs, respectively. Therefore, the I_D/I_G value is a measure of the defect rate, and a higher value indicates a higher defect rate. As shown in Fig. 4, the defect rate of the oxidized MWCNTs was higher, indicating improvement in the particle distribution. Fig. 4(b) shows the oxidized MWCNTs solution dispersed in DI water, and it was much better dispersed than the pristine MWCNTs solution shown in Fig. 4(a) even though the dispersion occurred under the same sonication condition. The pristine MWCNTs solution showed some dispersibility immediately after sonication, but aggregation of the MWCNTs was quite severe after some time. Friend and co-workers discussed the effect of this acid treatment of the MWCNTs, which formed carboxylic acid groups on the surface of the MWCNTs; these surface chemical groups disrupted the π -conjugation and introduced surface dipole moments, leading to high dispersibility in the solution [26].

Surface images of the conductive textiles are shown in Fig. 5. In Fig. 5(a) and 5(b), the surface images of the conductive textile manufactured by vacuum-filtration indicate that the MWCNTs were well distributed, and they formed a network for electrical connection between strands of the textile. Fig. 5(c) and 5(d) present surface images of the sample manufactured by screen-printing. Comparing with the sample prepared by vacuum filtration, the shape of the screen-printed textile strands could not be observed and only the film formed by the MWCNTs paste could be seen. The obvious difference between these two samples was due to difference in the processing method, as illustrated in Fig. 1.

Fig. 6 presents variations in resistance as functions of the strain rates of the conductive textiles prepared by the two processes. As shown in Fig. 6(a), the difference in resistance variation was small among the samples fabricated by vacuum-filtration, and linear plots

were obtained. This shows that when the vacuum-filtration method was used, the electrical properties were uniform within the coating area. Fig. 6(b) shows the variations in resistance according to the strain rate of three samples prepared by screen-printing, and the samples presented a tendency to be different from that exhibited in the vacuum-filtration samples. The difference in resistance variation is large among the samples, and the linearity of the plots is poor. The scale of the y-axis ($\Delta R/R_0$) in Fig. 6(b) is about 3,000 times that in Fig. 6(a). The large variations in resistance at the same strain rate are advantageous when fabricating a sensor using resistance displacement, but they should maintain a similar level in repeated stretching. These results are explained below.

Fig. 7(a) shows the variation in the resistance as functions of the strain rates of vacuum-filtration sample1 according to repetitive stretching. The variations in resistance were measured three times at a strain rate of 20%, and the sample maintained a constant resistance variation. Fig. 7(b) shows the corresponding result for screen-printing sample1'. The variation in the resistance of sample1' were measured in the same method, but the result was poor compared to that of vacuum-filtration sample1. After the first stretching, the resistance variation decreased dramatically; the range of $\Delta R/R_0$ was reduced to about 1/400 of that of the first stretching, and the plot showed poor linearity. Based on this result, serious damage to sample1' was expected after the first stretching. The photograph in Fig. 7 (b) presents shape of sample1' after first stretching at a strain rate of 20%. This is an important result associated with the topic of this study. When a water-based MWCNTs solution is coated on a textile by a vacuum-filtration process, the MWCNTs are uniformly adhered to each other to provide mutual electrical contact. This conductive textile has excellent durability up to a certain strain rate due to the characteristics of MWCNTs having

excellent mechanical strength and electrical network formation. In contrast, in a screen-printing process in which a paste containing a binder is generally used, the conductive material is coated only on the surface of the textile, and its mechanical strength depends on the adhesive strength of the binder. That is, electrical resistance of the conductive textile manufactured by screen-printing does not recover after forcing exceeding the tensile strength of the binder. This phenomenon would be strongly influenced by the type of binder. Fig. 7(c) shows only the results for the second and third stretching of the same sample. The variation in resistance gradually decreased according to increasing number of stretching, and the resistance did not change until the strain rate of 10%.

The results of 10,000 cycle's tensile test (20% strain rate) are shown in Fig. 8 for the samples prepared by vacuum-filtration. All results were obtained at the restored state after stretching. The resistance was measured every 1000 cycles, and the results of each samples were uniform. All samples showed resistance changes of less than $\pm 3\%$ after 10,000 cycles of stretching, demonstrating the excellent durability of the conductive textile prepared by vacuum filtration.

Fig. 9(a) shows the pulsed data of 10,000 cycle's tensile test (20% strain rate) for the sample fabricated by vacuum-filtration, and the pulse of the resistance variation was uniform. Based on this result, this sample can be applied to a uniform and reliable resistance displacement sensor. Fig. 9(b) shows a photograph of the sample after 10,000 cycle's tensile test. After repetitive stretching, the surface of the sample remained in good condition, indicating that the vacuum-filtration method is suitable for fabricating durable conductive textile.

Fig. 10 shows the motion-sensing glove fabricated with the conductive textile prepared

by vacuum filtration. A four-channel oscilloscope was used to observe the four fingers' movements in real time. Fig. 10(a) shows the structure of the glove and the position at which the conductive textile was attached. Fig. 10(b) shows the oscilloscope pulse in the normal state, and Fig. 10(c–f) show the oscilloscope pulses corresponding to motions of the fingers. The pulse of the oscilloscope was accurate according to the movement of each finger, and the operation was performed correctly even in repetitive motion. These results demonstrate that the conductive textile prepared by vacuum filtration can be applied to motion sensing of actual products [27].

4. Conclusions

In this study, we fabricated a conductive textile for motion sensing through resistance displacement. The conductive textile was fabricated by vacuum filtration of water-based MWCNTs ink. The subject of this study is to examine the possibility of mass production through fabricating and evaluating conductive textile using existing simple technologies. The conductive textile prepared by vacuum filtration was evaluated for resistance variations as it was subjected 10,000 cycles of repeated stretching at strain rates of 0% to 20%. The samples showed good results with resistance changes of less than $\pm 3\%$ after 10,000 cycles of stretching. This result demonstrates that the conductive textile prepared by the vacuum filtration had excellent durability. The pulse of the resistance variation was uniform, which means that this textile can be applied as a reliable resistance displacement sensor. A motion-sensing glove was fabricated with the conductive textile prepared by vacuum filtration. The pulse of the oscilloscope changed accurately according to the movements of the fingers, demonstrating that the conductive textile prepared in this study can be applied

to motion sensing in real products.

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Figure caption

Fig. 1. Schematic of conductive textiles prepared by (a) vacuum-filtration and (b) screen-printing.

Fig. 2. Manufacturing process for conductive textile by vacuum-filtration of MWCNTs ink.

Fig. 3. TGA data of (a) pristine MWCNTs and (b) oxidized MWCNTs.

Fig. 4. Raman spectra of (a) pristine MWCNTs and (b) oxidized MWCNTs.

Fig. 5. SEM images of conductive textiles prepared by vacuum-filtration at magnifications of (a) $\times 3,000$ and (b) $\times 10,000$ and that prepared by screen-printing at magnifications of (c) $\times 3,000$ and (d) $\times 10,000$.

Fig. 6. Variations in resistance as functions of the strain rates of samples prepared by (a) vacuum-filtration and (b) screen-printing.

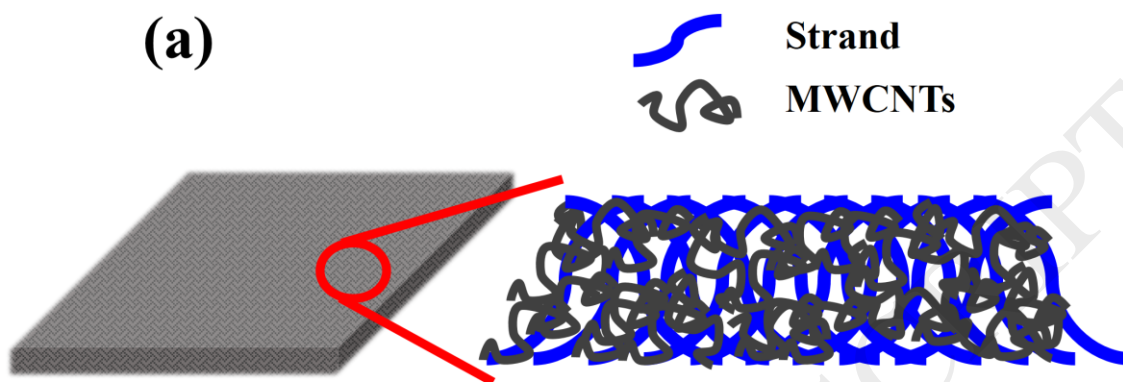
Fig. 7. Variations in resistance as functions of the strain rates of (a) sample1 prepared by vacuum filtration and (b, c) sample1' prepared by screen-printing during repetitive stretching.

Fig. 8. Results of 10,000 cycles of tensile test (20% strain rate) for conductive textile samples prepared by vacuum filtration.

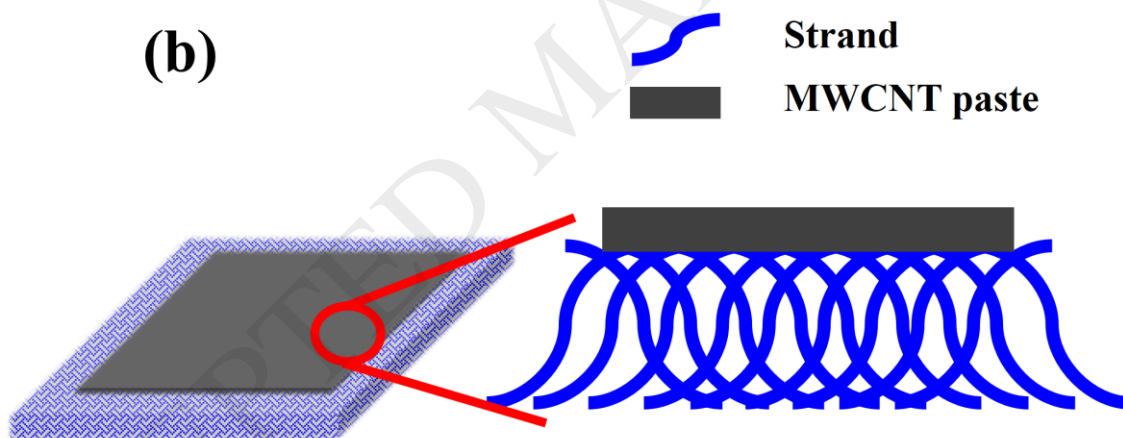
Fig. 9. Results of 10,000 cycles of tensile test (20% strain rate) for sample1 prepared by vacuum filtration: (a) pulse data of resistance according to strain rate; (b) photograph of the sample1 after 10,000 cycles of tensile test.

Fig. 10. Motion-sensing glove fabricated with conductive textile prepared by vacuum filtration: (a) structure of the glove; (b) oscilloscope pulse in normal state; (c–f) oscilloscope pulses resulting from different types of finger motion.

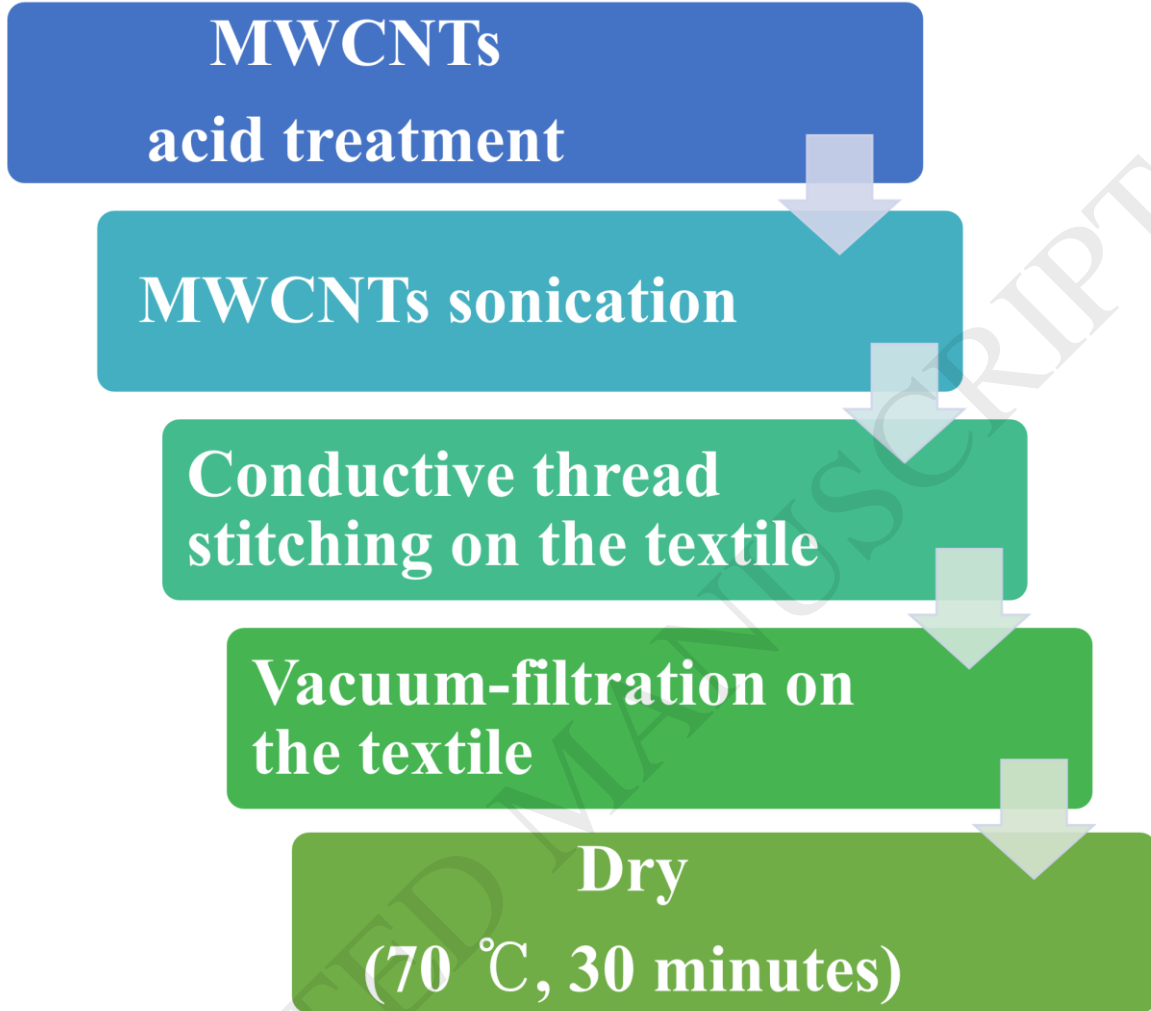
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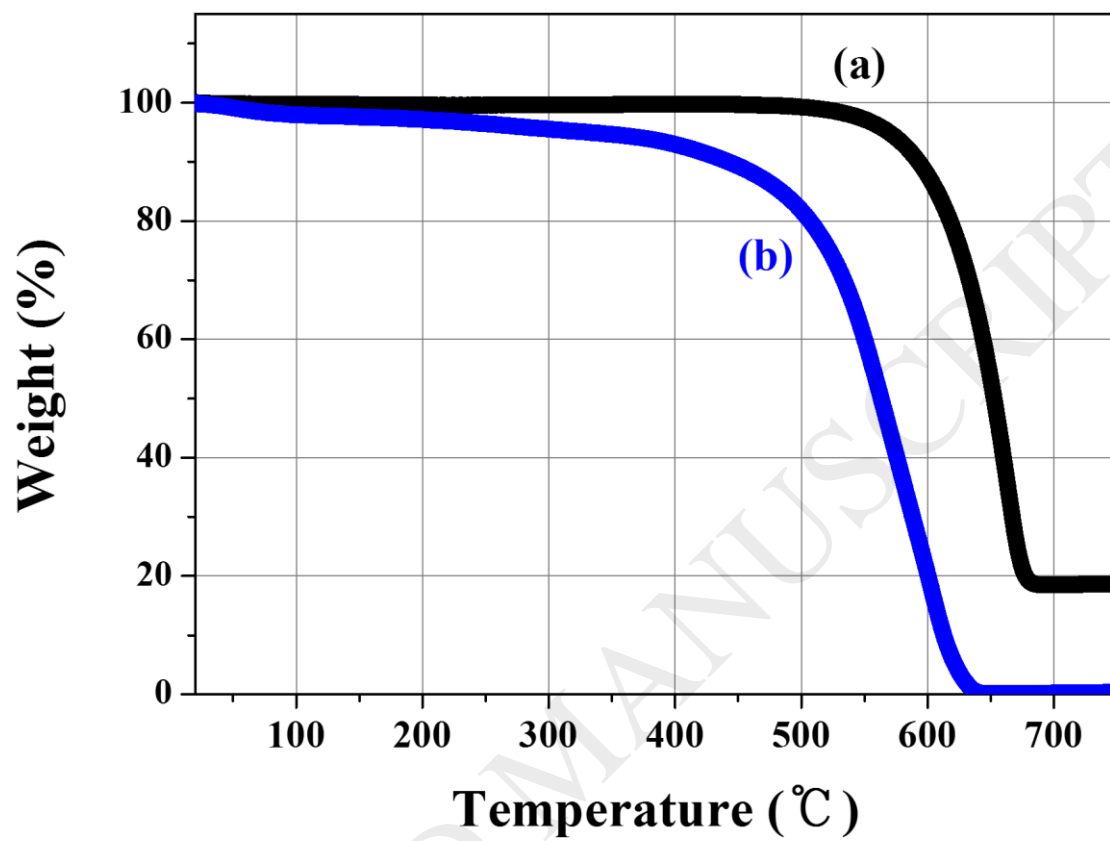


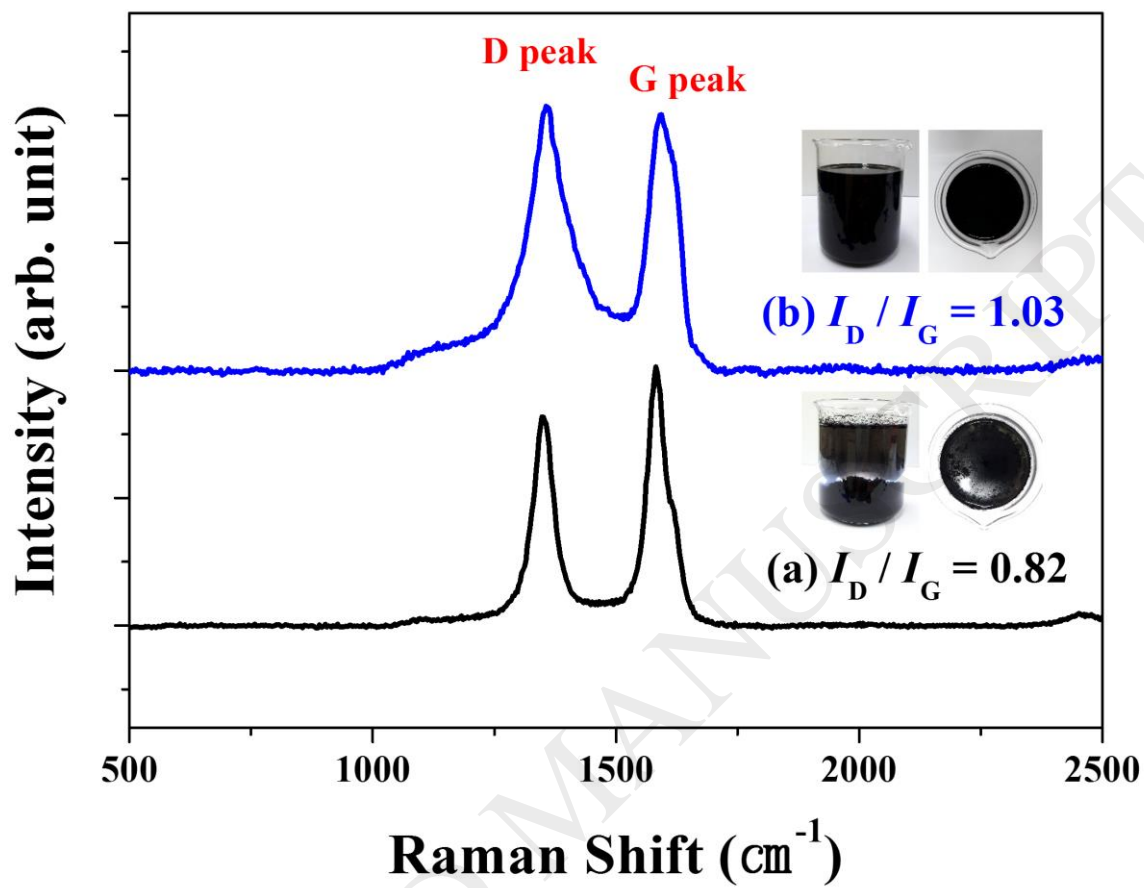
Coating area: between strands of the textile

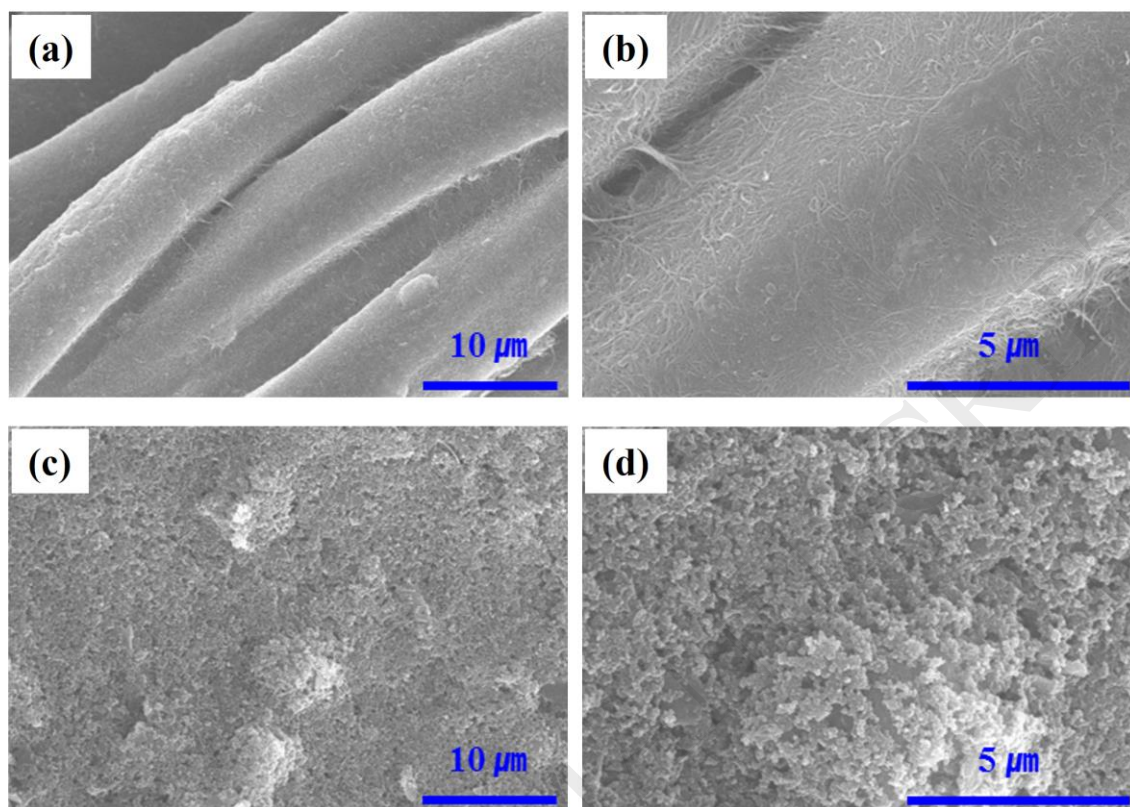


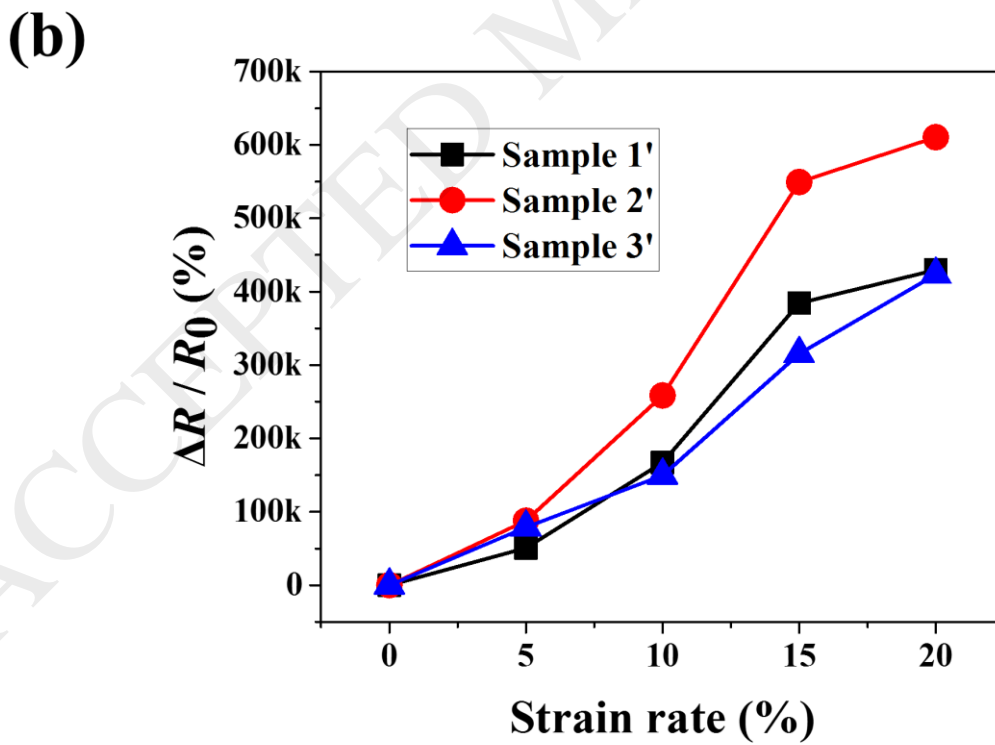
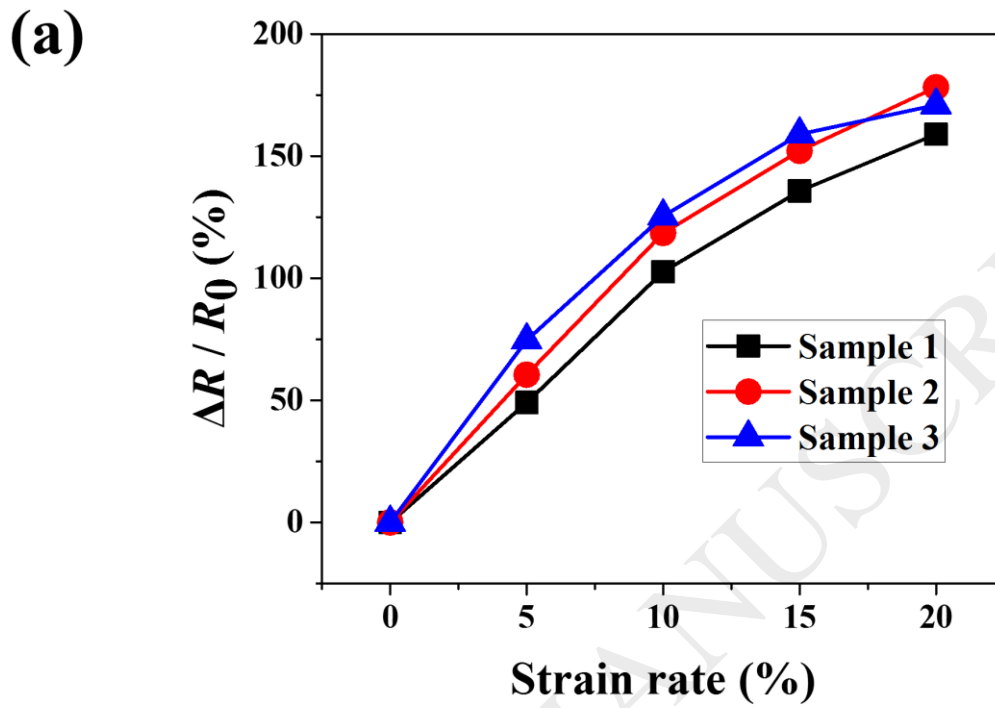
Coating area: only surface of the textile



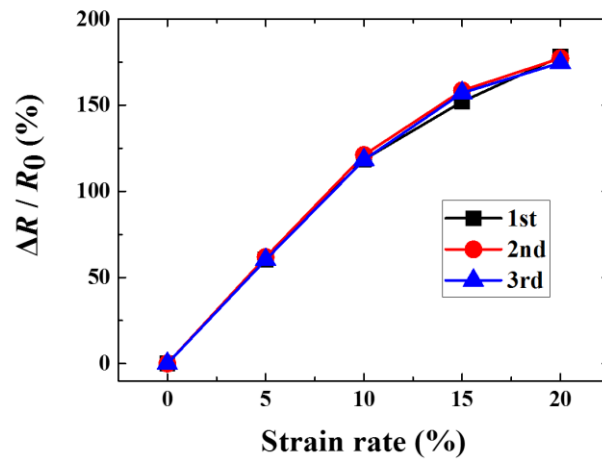




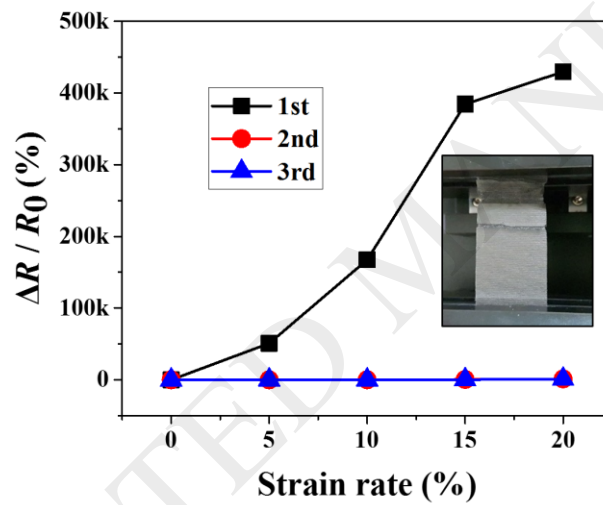




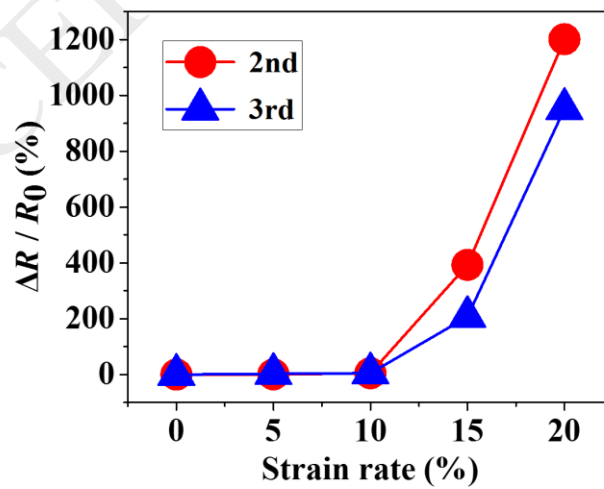
(a)

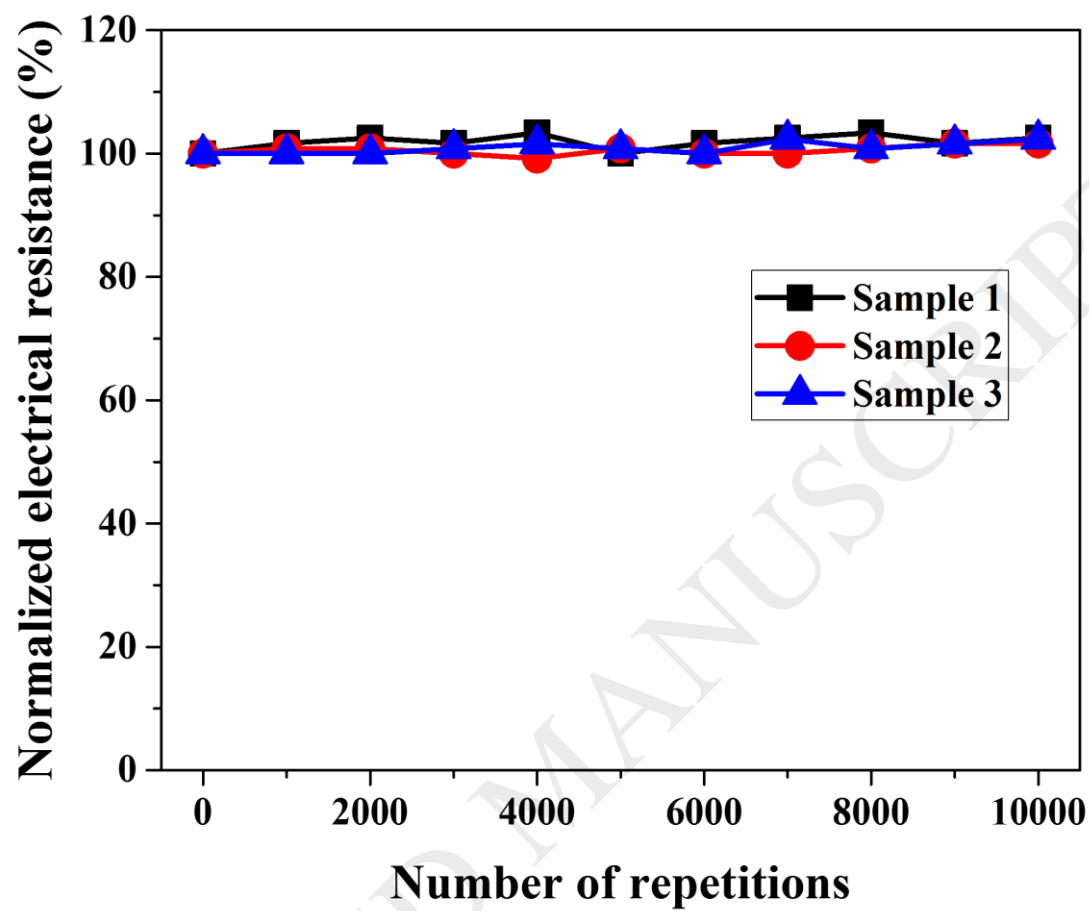


(b)



(c)





(a)