Silk fibroin and ultra-long silver nanowire based transparent, flexible and conductive composite film and its Temperature-Dependent resistance

Xiangzheng Qin\textsuperscript{a}, Yu Peng\textsuperscript{a}, Piaopiao Li\textsuperscript{a}, Kai Cheng\textsuperscript{a}, Zhenzhong Wei\textsuperscript{a}, Ping Liu\textsuperscript{b}, Ning Cao\textsuperscript{a}, Junyi Huang\textsuperscript{b}, Jinjun Rao\textsuperscript{a}, Jinbo Chen\textsuperscript{a}, Tao Wang\textsuperscript{a}, Xiaomao Li\textsuperscript{a}, and Mei Liu\textsuperscript{a}

\textsuperscript{a}Shanghai Key Laboratory of Intelligent Manufacturing and Robotics, School of Mechatronic Engineering and Automation, Shanghai University, Shanghai, China; \textsuperscript{b}School of Life Sciences, Shanghai University, Shanghai, China

ABSTRACT
A transparent, conductive, smooth, and temperature sensitive thin films was fabricated and characterized in this paper. Silk fibroin could be processed into transparent thin films, which can act as ideal opto-electronic substrates. As pure silk fibroin film is nonconductive, ultra-long silver nanowires coating and platinum sputtering were used to strengthen its conductivity. Ultra-long nanowires were used to reduce the junctions between wires, and platinum was to improve the conductivity of the film. The new nanowire-metal-organic composite film possesses excellent conductivity and good transmittance. The composite films containing different silver nanowires exhibit conductivities of as low as 6.9 $\Omega$/sq, and transmittance of 60--80% in the visible light range. The films also showed potentials in practical applications as their resistance is almost linearly temperature-dependent. It also can transfer power to electrical devices. The new composite films could be expected to function in wearable electronics or implantable devices and sensors.

1. Introduction
Transparent conductive films (TCFs) are key elements to many modern technologies and devices, such as touch panels, smart phones, organic light-emitting diodes, and wearable devices. Numerous materials are implemented as TCFs substrates in optoelectronic devices, for example, glass, plastic (PET, PVP, PVA, PEN), paper, and textiles.\cite{1-5} Highly flexible transparent electrodes are especially desired in flexible optoelectronic devices.

Plastic is a commercially popular substrate made from nonrenewable resources, but its limitations have become increasingly problematic. A widely used material for TCFs is indium tin oxide (ITO), an n-type semiconductor. It is transparent in the visible spectral region and reflective in the infrared region. ITO can also achieve relatively low electrical resistivity. However, high-performance ITO films require a high deposition vacuum and also require high deposition or annealing temperature. It is brittle by nature and has limited global supply restricting its
sustainable applications. Researchers keep on developing alternatives to ITO, for example, layers of carbon nanotubes (CNTs), graphene, and metal nanowires. Single layer graphene is usually synthesized by chemical vapor deposition (CVD) with controlled growth parameters and large area production. However, it inevitably requires a long processing time, high processing temperature up to 1000 °C, and low output, resulting in high power consumption for mass production. In addition, it is difficult to remove the grapheme from the metal substrate. Reduced graphene oxide (rGO) is also an alternative material to fabricate TCFs. A flexible transparent conducting thin film composed of rGO and SF was reported, whose measured surface resistance is approximately 1000 Ω/sq, much higher than ITO. Some researchers combined rGO and silver nanowires (AgNWs) and generated more conductive and more transparent films on PET or glass slides. Its biocompatibility has yet to be proven.

AgNWs are particularly popular for its ability to create a nanotrough network, which was able to form a foldable, conductive, reliable, and transparent conducting electrode layer that can be adsorbed on or be buried into surfaces of various substrates. However, there are several serious challenges on the applications AgNWs-based TCFs to optoelectronic devices. One of them is that the high resistance of the AgNW films arises at the network junctions of wires. In addition, the films have large non-contacted areas in the partially percolated network, which result in high electrical resistance. Furthermore, the poor adhesion between the AgNW films and the plastic substrates also restrict their applications into commercial devices. Silk fibroin (SF), a natural macromolecular fibrous protein derived from silkworm cocoons, displays very good mechanical and optical properties. For example, as mentioned in Qi’s paper, the breaking stress of the SF film was measured to be more than 70 MPa, superior to PET. Pure SF films also displayed excellent visible light transmittance of 90%, comparable to the transmittance of glass, PET, synthetic polymers, and other clear substrates. It can also be conveniently processed into various forms, such as gels, strands, sponges, blocks, foams and films, which is superior to other materials, such as ITO, PET, PVP, PVA, PEN, as related to biocompatibility, biodegradability, and implantability. SF film is thus also a good candidate for TCFs substrate for silver nanowires.

Qi et al. used SF as substrates in places of glass, synthetic polymers, and flexible bases for AgNWs. The AgNWs-SF film’s conductivity was strengthened with platinum sputtering, which eliminated the non-contacted areas in the partially percolated network mentioned above. The composite films exhibited satisfactory conductivity and transparency in the visible light range, comparable to commercial ITO substrates. In addition, the composite films retained conductivity after 100 times cyclic loading, presenting much better flexibility than other polymeric substrates. In their paper, short AgNWs (1–2 μm) were used, and quite a few of them were redundant and separated from the NW network, giving no contribution to film conductivity.

To achieve better performance (i.e., better conductivity and transmittance), AgNWs must be defect-free, have conductivities close to their values in bulk, be as long as possible to minimize the electrical resistance between the NWs, and have uniform deposition and distribution on the substrate.

**Nomenclature**

| Abbreviation | Full Form |
|--------------|-----------|
| TCF(s)       | Transparent conductive film(s) |
| PET          | Polyethylene terephthalate |
| PVP          | Polyvinyl pyrrolidone |
| PVA          | Polyvinyl alcohol |
| PEN          | Polynaphthalene |
| ITO          | Indium tin oxide |
| CNT(s)       | Carbon nanotube(s) |
| CVD          | Chemical vapor deposition |
| rGO          | Reduced graphene oxide |
| AgNW(s)      | Silver nanowire(s) |
| SF           | Silk fibroin |
| NW(s)        | Nanowire(s) |
| LED(s)       | Light emitting diode(s) |
| UV-VIS       | Ultraviolet-visible |
| DC           | Direct current |
| SEM          | Scanning electron microscope |
| Pt           | Platinum |
| TCR          | Temperature coefficient of resistance |
| SFPtAg       | The composite film |
the number of wire-to-wire junctions, and exhibit small junction resistance.[1] In this paper, SF films embedded with ultra-long AgNWs were prepared to optimize the composite structure. Platinum sputtering and annealing were adopted to reduce non-contacted areas between AgNWs network and SF film, ultra-long NWs were used to reduce junction numbers, and also electrical resistance, accordingly. Compared to short NWs, ultra-long NWs also takes less space and allows more lights to pass. The composite films containing different AgNWs exhibit good conductivities and transmittance in the visible light range. The films also showed potentials in practical applications, such as a conduit to light emitting diodes (LEDs) and temperature sensors.

2. Experiments

2.1. Structure of the composite film

In the composite film, simplified as SFPtAg film, SF film is used as the supporting substrate which is transparent, a layer of Pt works as the buffer and bonding agent between SF film and network of AgNWs, a layer of AgNWs works as the electrical conductive nanowire network, as shown in Figure 1(a,b).

As illustrated in Figure 1(c,d), for the same long electric circuit, compared with short NWs, ultra-long NWs exhibit fewer wire-wire junctions, which are key resistance sources; and also, ultra-long NWs generate fewer redundant branches, leading to better optical transmittance.

2.2. Preparation of SF solution

SF solution was made from *Bombyx mori* raw silkworm cocoons, which were purchased from JD.com, China, as shown in Figure 2. Briefly, the preparation of SF solution requires degumming, dissolving, dialysis, etc.[24,25,30]

2.3. Fabrication of composite thin film

Original AgNWs solution (Concentration: 20 mg/mL, diameter: 50 nm, length: 100–200 μm, solvent: water, Nanjing XFNANO Materials Tech Co., Ltd, China) was diluted to different concentrations
(1 mg/mL, 3 mg/mL, 5 mg/mL, 10 mg/mL) in ethanol. The diluted AgNWs solutions were respectively spin coated on polished silicon wafers. Spinning speed was set to 3000 rpm and spinning time was 60 s. The AgNWs covered wafers were annealed at 100 °C for 1 h and then sputtered with platinum in ion sputtering apparatus for 30 s. A 3 nm Pt layer will be deposited. SF solution was dropped onto each AgNWs-platinum-coated wafer in a Petri dish, which was put in a climatic chamber (20 °C, humidity: 65%). SF PtAg composite films formed on the wafers after water evaporation, which were carefully peeled off the substrate as generated, shown in Figure 2.

SF-AgNWs films with no Pt sputtering were also prepared with AgNWs solution of each concentration, for comparison.

2.4. Measurement and analysis instruments

Pt layer thickness was measured by a fluorescence measuring instrument (Fischerscope X-Ray XAN250, Fischer, Germany). The morphology of the SF PtAg composite films was examined using a field emission scanning electron microscope (SU3500, Hitachi, Japan). Film transmittance spectrum was measured using a UV-VIS spectrophotometer (UV2600, Shimadzu, Japan). Film sheet resistance was tested by a four-point probe method. The relationship between the resistance and temperature of the film was tested by a hotplate and a resistance detector.

To test the ability of the composite film on transferring power to other optoelectronic devices, commercial LED chips were connected to the SF PtAg films strips to form a conductive network circuit. Since the pin of a single LED is too small to be in good contact with the SF PtAg film, the
pin of each LED was soldered to a section of wire. Note that the welded wires on different LEDs cannot touch each other when sticking to the films. Then, a DC power was applied into the two ends of the strip.

3. Results and discussion

3.1. Morphology and characterization

SEM images indicated that AgNWs were quite long and curved (Figure 3(a)). As firmly anchored and buried into the SF surfaces, as shown in Figure 3(b,c), the previously fragile AgNWs network became mechanically robust. Most NWs were connected with each other, different from previous publications, where quite a few NWs were not connected with any other NWs due its limited length. And, as the thickness of the SFPtAg films were in the range of 100 μm (Figure 3(d)) while the diameter of the NWs is ~100 nm, the Pt layer thickness is 3 nm, AgNWs and Pt layer both have negligible effects on film thickness. There might be AgNWs locating away within 1 nm. However, as the conducting web is a good conductor, tunneling effect will only happen when there is no direct path, which why we did not consider the tunneling effect.

3.2. Transmittance of the composite films

As shown in Figure 4(a,b), pure SF films have displayed a visible light transmittance of around 90%, which is comparable to the transmittance of other clear substrates, such as ITO, PET, and
PET-ITO, making SF film a viable transparent substrates alternative. Without Pt sputtering, the SFPtAg films displayed a transmittance of \( \frac{80\%}{C24} \) in the visible light range, which is weaker than the transmittance of pure SF films. This is due to the shielding effect caused by the AgNWs network absorbing and reflecting light. There is an obvious absorption peak around 375 nm, due to the natural plasmonic effects of the AgNWs networks caused by light trapping.[30,31] However, in the visible spectrum, the transparency of the composite film is similar to that of ITO. With Pt sputtering of 30 s, the film seems darker and transmittance of films decreases inevitably, as shown in Figure 4(c,d). This is due to the shielding of the Pt film. Also, the adsorption peak disappears as the plasmonic effects disappear with AgNWs embedded in the Pt film.

### 3.3. Conductivity of the composite film

A comparison of the resistances of all SFPtAg films is shown in Figure 5. Apparently, with Pt sputtering, the sheet resistances of the SFPtAg films demonstrate a significant decrease, as shown in Figure 5(a), which is because sputtered Pt is also a good conducting material. And, as the AgNWs concentration increased, which means more conductors in the film, the resistances will also decrease; the effect of Pt sputtering weakens simultaneously, as shown in Figure 5(a). For example, for 1 mg/mL AgNWs solution, Pt sputtering exerts 96.86 \( \Omega/sq \) decrease while for 10 mg/mL AgNWs solution, Pt sputtering only plays 23.8 \( \Omega/sq \) decrease, which is because high
concentration AgNWs network has a very good conductivity and the whole conductivity is less dependent on Pt film (Ag: $1.586 \times 10^{-8} \ \Omega \cdot \text{mm}$, Pt: $10.6 \times 10^{-8} \ \Omega \cdot \text{mm}$, $20^\circ \text{C}$).

Figure 5(b) shows the comparison of the sheet resistance of two specific SFPtAg films with better electrical conductivity (concentration-sputtering time: $5 \ \text{mg/mL-30 s}$ and $10 \ \text{mg/mL-30 s}$) with that of two commonly used conductive thin films (ITO, PET-ITO). The sheet resistance of $10 \ \text{mg/mL-30 s}$ SFPtAg film ($6.9 \ \Omega/\text{sq}$) is better than that of ITO ($8.1 \ \Omega/\text{sq}$) and PET-ITO ($9.4 \ \Omega/\text{sq}$), but the resistance of $5 \ \text{mg/mL-30 s}$ film ($12.8 \ \Omega/\text{sq}$) is relatively high. The resistance is also comparable to or even better than previous publications, where the film resistance is $15 \ \Omega/\text{sq}$ (AgNWs concentration: $0.5 \ \text{mg/mL}$, diameter: $30–40 \ \text{nm}$, length: $1–2 \ \mu\text{m}$).[24] One possible reason is that ultra-long AgNWs were used in the composite film, and wire-wire junctions are much fewer.

Another noteworthy issue is that transmittance and conductivity are two conflicting factors. As AgNWs concentration increases, the film transmittance will decrease, but the conductivity will be better; as AgNWs concentration decreases, the film transmittance will increase, but the conductivity will decrease, as shown in Figure 5(c). It is also true with Pt sputtering: it improves conductivity, meanwhile undermines transmittance. So a balance should be tactfully picked depending on applications.

Silver and platinum are both temperature sensitive resistors, and platinum is a commonly used temperature sensitive material. Their film phase should also be temperature sensitive. The dependence of film resistance on temperature ($3 \ \text{mg/mL-30 s}$ and $10 \ \text{mg/mL-30 s}$) was also tested, as shown in Figure 6. It can be seen from Figure 6(a,b), with same Pt sputtering time (30 s), the sheet resistance of the SFPtAg films almost increases with temperature linearly, proving its potential as a transparent and conductive temperature sensor. The TCR

![Figure 5](image-url)

Figure 5. (a) Sheet resistance of different composite films with/without Pt sputtering. (b) Sheet resistance of composite film in comparison with other type of films. (c) Sheet resistance versus optical transmission (at 550 nm) of different composite film.
(temperature coefficient of resistance) of the two films in Figure 6 are 0.00216/°C and 0.0014/°C respectively, on the same scale with its block phase (TCR of platinum: 0.00374/°C, 0–60 °C; silver, 0.0038/°C, 20 °C).

Due to its high electrical conductivity, the 10 mg/mL-30 s film was also used as a conductor in LED strips, as introduced in Section 2.4. It can successfully light the LED, indicating that it had the ability to conduct electricity for other electronic components in certain circumstances, as shown in Figure 7.

Though several studies suggest that AgNWs might not be totally harmless for biological environments,[32,33] the SFPtAg film was aimed for applications that require biocompatibility, biodegradability, and implantability. In selection of suitable AgNWs, its toxicity to human should be taken into account. As the conductive layer does not have to be in direct contact with the human body, it could be enveloped inside biocompatible SF film to avoid direct contact between AgNWs and human body. Its biocompatibility, biodegradability, and implantability will also be tested in future work. It may also find applications in super capacitor materials, solar cells, piezoelectric materials, and organic field effect transistors.

![Figure 6](image-url) **Figure 6.** (a) Resistance temperature dependence of the 3 mg/mL-30 s film. (b) Resistance temperature dependence of the 10 mg/mL-30 s film.

![Figure 7](image-url) **Figure 7.** SFPtAg films help lighting LEDs.
4. Conclusion

In summary, this research has demonstrated a spin-coating method combining metal sputtering to prepare SFPtAg composite films. To reduce wire-wire junction numbers and redundant nanowires, ultra-long AgNWs were applied. Pt sputtering was implemented for improving the conductivity and also temperature-dependence. These organic composite films displayed quite satisfactory transmittance and conductivity. In addition, it also possesses almost linear conductivity-temperature correlation. The films could be a good candidate in further development of wearable textile and conducting devices. It can not only be used as a conducting transparent material but it also may be used as a temperature sensor.

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