공학석사학위논문

Highly Conductive and Stretchable Composite Using Ultra-long Silver Nanowires, Elastomer, and Fluorosurfactant

매우 긴 은 나노선과 탄성체, 불소계 계면활성제를 이용한 고 전도성 및 신장성 혼합물

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Abstract

Highly Conductive and Stretchable Composite Using Ultra-long Silver Nanowires, Elastomer, and Fluorosurfactant

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The stretchable conductor which has higher conductivity and stretchability are needed with recent development of wearable device and robot industry. Here, the highly conductive and stretchable composite was developed using ultra-long silver nanowires, styrene-butadiene-styrene, and zonyl, a fluorosurfactant. Silver nanowire is considered a fascinating candidate as conductive filler for stretchable conductor composite due to its high intrinsic conductivity and high-aspect-ratio, which enables the conductivity to maintain stably under deformation of the composite. The ultra-long silver nanowires were used in this study, and consequently the merits of silver nanowire could be enlarged. The ligand-exchange reaction was applied to the ultra-long silver nanowires and the ligand-exchanged ultra-long silver nanowires formed homogeneous mixture with styrene-butadiene-styrene. This ultra-long silver nanowires/SBS composite showed high conductivity of over 30,000 S/cm and stretchability of ~45% without introduction of structure for stretchability. In addition, Zonyl was added to the composite and enhanced the stretchability of the composite by separating into conductive and stretchable regions and making micro-porous structure. As a consequence, the composites which has stretchability of ~450% with initial conductivity of ~20,000 S/cm and stretchability of ~100% with initial conductivity of ~29,000 S/cm were achieved. The simple application as stretchable interconnects using the composite was demonstrated and the composite showed stable performance under stretched condition.

Keywords : stretchable conductor composite, stretchable electrode, ultra-long silver nanowires, fluorosurfactant

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1. Introduction

With recent development of wearable devices, stretchable conductors have been intensively researched. Several remarkable researches of stretchable conductors with various materials and fabrication methods have reported such as PU composite containing self-assembly of gold nanoparticles [1], rubber fiber wrapped by CNT sheets [2], structured copper nanowire embedding into PDMS [3], fiber composite fabricated by wet spinning with silver particles/silver decorated-CNTs [4] and silver nanowires [5], and fiber composite of PU/cotton/silver nanowires/PDMS [6].

Although all of these composites have their own specialties, they have insufficient conductivity and/or stretchability to be utilized as interconnects. In order to widen the applications of stretchable conductor composites, especially as interconnects, the researches about composites that have higher conductivity and stretchability are required.

Among the conductive fillers used in these composites, silver nanowire is a promising candidate for highly conductive and stretchable composite. Silver is known as the material that has the highest conductivity. The high-aspect-ratio of silver nanowire enables silver nanowires to form electrical percolation network well in the elastomer matrix [7]. These characteristics of silver nanowire make the composites using silver nanowire as conductive filler have higher conductivity and maintains more stable electrical performance under large deformation than the composites using other materials.

Zonyl, which is a fluorine surfactant, has been used in stretchable conductor composites for various purposes [8], [9], [10]. One of the purposes is to improve the conductivity and stretchability of the composite. The addition of zonyl in the composites causes region separation into conductive and stretchable parts. The separated conductive and stretchable regions take charge of the conductivity and stretchability respectively and the conductivity and stretchability are enhanced [8].

In this paper, I presented the highly conductive and stretchable composites using ultra-long silver nanowires (ultra-long Ag NWs, ul-Ag NWs), styrene-butadiene-styrene (SBS), and fluorosurfactant. The ultra-long length of silver nanowire enlarges the effects of silver nanowire on the stretchable conductor composite. The introduction of ligand-exchange reaction to ultra-long Ag NWs allows ultra-long Ag NWs to be homogeneously mixed with SBS. The addition of surfactant improves stretchability of the composites by separating of ultra-long Ag NWs and SBS region and building micro-porous structure. The composite was prepared as solution mixture and manufactured by evaporating the solvent. Therefore, the composite can possess processability. The application as interconnects was demonstrated and the composite showed stable performance under stretched situation.

2. Ultra-long silver nanowires as conductive filler

2. 1. Synthesis and Purification of ultra-long Ag NWs

To produce ultra-long Ag NWs, a modified synthesis method of ultra-long Ag NWs was used [11]. In the method used in this paper, there are several modifications to raise productivity compared with the original method. First, the synthesis was scaled up twice. Second, the PVP solution was poured instead of injection. Third, the $AgNO_3$ solution was sonicated before injection to reduce agglomeration on the synthesis solution. The schematic image of synthesis process and photograph of synthesis solution are presented in **Figure 1**. The details for the process are written in Experimental Section.

After synthesizing, the solution was purified by ethanol instead of acetone, centrifuged 3 times, and dispersed in dimethylformamide (DMF). There are several advantages of using ethanol instead of acetone as solvent of purification step. First, as-synthesized ultra-long Ag NWs do not aggregate in ethanol, while they aggregate unusably in acetone. Second, purification using ethanol efficiently removes nanoparticles that are byproduct of the synthesis. Lastly, it also removes necessity of drying step for following process.

The SEM image of ultra-long Ag NWs that were achieved through this modified synthesis method and purification is presented in **Figure 2**.

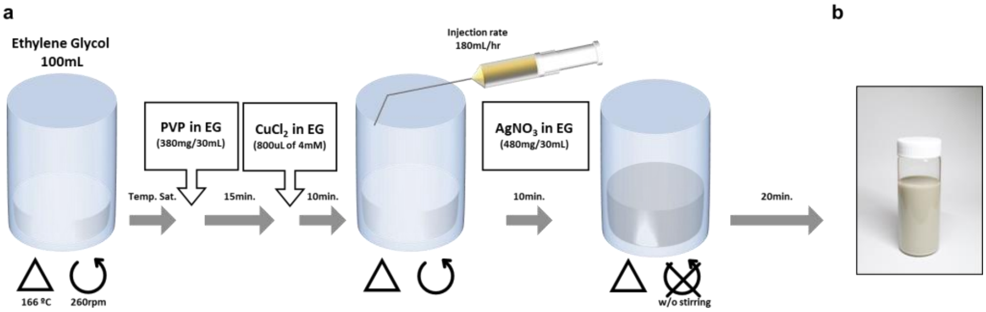

Figure 1. **Synthesis of ultra-long Ag NWs**

(a) Schematic image of synthesis process of ultra-long Ag NWs. EG; Ethylene glycol, Temp. Sat.; Temperature Saturation, w/o; without

(b) Photograph of synthesized ultra-long Ag NWs dispersed in ethylene glycol.

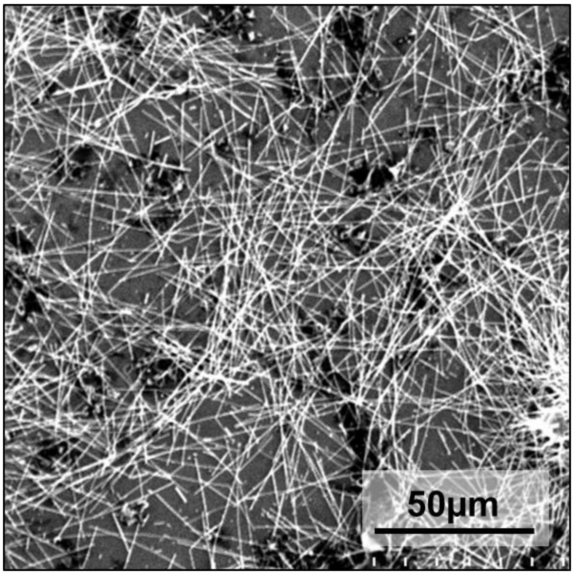

Figure 2. **SEM image of synthesized ultra-long Ag NWs**

2. 2. Characteristics of ultra-long Ag NWs

The length of nanowires is key factor for conductivity of the composite. The nanowires which have longer length form electrical percolation easier and have higher conductivity [12].

The lengths of synthesized ultra-long Ag NWs are compared with normal silver nanowire (normal Ag NWs, n-Ag NWs) that used our previous work [7]. The histograms showing length distribution of ultra-long Ag NWs and normal Ag NWs are presented in **Figure 3**. The average length of ultra-long Ag NWs was about 80 um and it was 2.5 times longer than the average length of normal Ag NWs.

The conductivities of ultra-long Ag NWs and normal Ag NWs are compared in **Figure 4**. The average conductivity of ultra-long Ag NWs was 14,000 S/cm and it was almost twice higher than the average conductivity of normal Ag NWs.

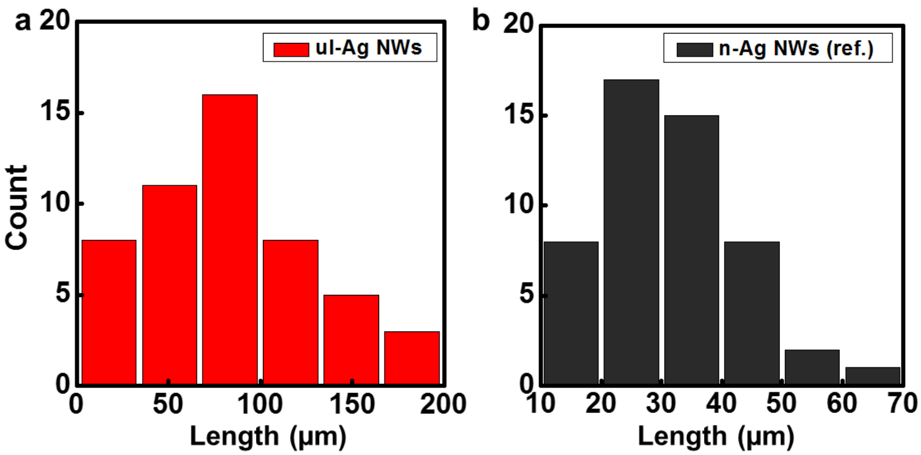

Figure 3. **Length of Ag NWs**

Histograms showing length distribution of (a) ultra-long Ag NWs and (b) normal Ag NWs. The average length of ultra-long Ag NWs is 83.76um and the average length of normal Ag NWs is 30.91um.

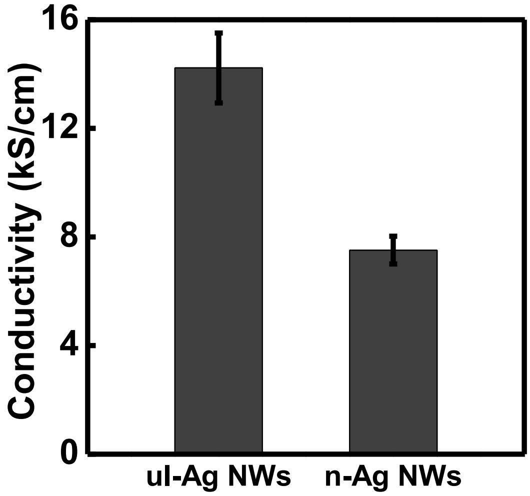

Figure 4. **Conductivity of ultra-long Ag NWs and normal Ag NWs**

Bar graph showing conductivity of ultra-long Ag NWs and normal Ag NWs annealed at 100°C. The average conductivity of ultra-long Ag NWs is 14,225 S/cm and the average conductivity of normal Ag NWs is 7,515 S/cm. Error bar; STDEVA. (n=11 for ultra-long Ag NWs, n=12 for normal Ag NWs)

3. Highly conductive and stretchable composite by using ligand-exchanged ultra-long Ag NWs, elastomer and surfactant

3. 1. Components of the highly conductive and stretchable composite

The highly conductive and stretchable composite was developed using ultra-long Ag NWs as conductive filler, styrene-butadiene-styrene (SBS) as elastomeric matrix, and a fluorine surfactant, zonyl® FS-300 (zonyl).

The synthesized ultra-long Ag NWs was initially dispersed in DMF, hydrophilic solvent while SBS could not be dissolved in hydrophilic solvent. The ligand of ultra-long Ag NWs, polyvinylpyrrolidone (PVP) was exchanged to Hexylamine (HAm) using $NOBF_4$. The schematic image of ligand-exchanged ultra-long Ag NWs is present in **Figure 5a**. The ligand-exchange reaction allows ultra-long Ag NWs to be dispersed in toluene and consequently, enables homogeneous mixing with SBS. In addition, the ligand-exchange reaction from PVP to HAm improves the conductivity of ultra-long Ag NWs due to shorter chain length of HAm than PVP [7].

In order to enhance stretchability, SBS was used as elastomeric matrix and zonyl was used as surfactant. The structure formulas of SBS and zonyl are presented in **Figure 5b and 5c**, respectively. SBS, which is the physical cross-linking elastomer, could contain large amount of ultra-long Ag NWs and therefore, the conductivity of composite could be maximized. The addition of zonyl which is the fluorine surfactant, improved the stretchability of the composite considerably. The mechanism of zonyl in the composite will be discussed in-depth in chapter 4.

The highly conductive and stretchable composite was prepared in solution mixture and the final film-type composite could be obtained in desired shape by evaporating the solvent. There was no need of repetitive fabrication process as well. In the other words, the composite has processability.

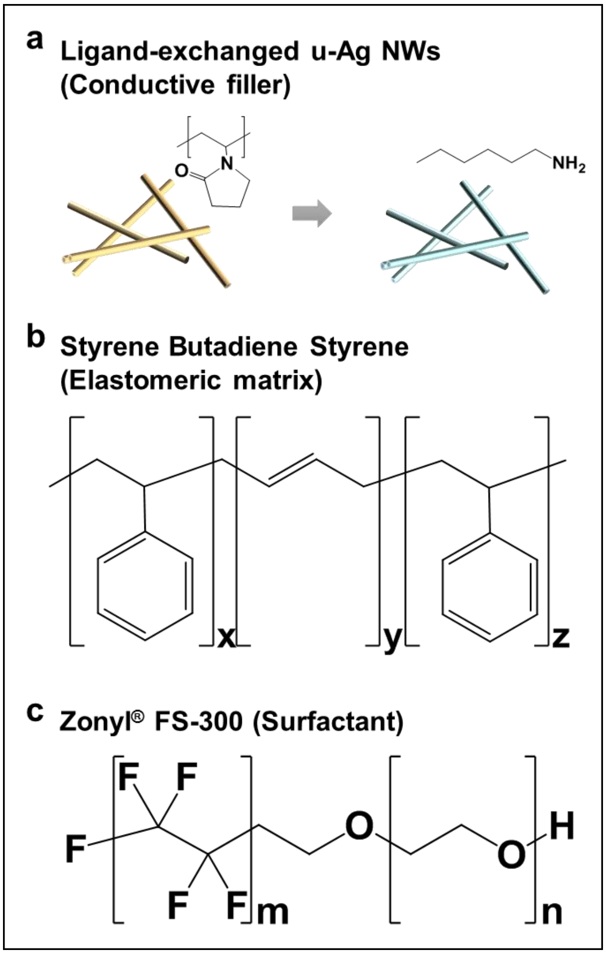

Figure 5. **Components of the highly conductive and stretchable composites**

(a) Schematic image of ligand-exchanged ultra-long Ag NWs as conductive filler

Ligand-exchanged reaction from PVP to hexylamine

(b) Structure formula of Styrene-Butadiene-Styrene (SBS) as elastomeric matrix

(c) Structure formula of Zonyl® FS-300 (zonyl) as surfactant

3. 2. Characteristics of the highly conductive and stretchable composite

The conductivities of ultra-long Ag NWs/SBS composite were measured depending on the ultra-long Ag NWs concentration (**Figure 6a**). The conductivity seemed to be saturated above 18 vol% ultra-long Ag NWs concentration and the maximum conductivity was over 30,000 S/cm. Compared with the conductivity of normal Ag NWs/SBS composite in our previous work [7], the ultra-long Ag NWs/SBS composite had 3 times higher conductivity than the normal Ag NWs/SBS composite at the same Ag NWs concentration.

The stretchability test was performed with film-type composites at 7, 12, 15.5, and 21.5 vol% of ultra-long Ag NWs concentrations (**Figure 6b**). Above 12 vol% of ultra-long Ag NWs concentration, the composite had ~45% stretchability. The composite at the low ultra-long Ag NWs concentration had worse stretchability and showed larger relative resistance (*R/R0*) with increased strain.

In order to elevate such inadequate stretchability of the ultra-long Ag NWs/SBS composite, zonyl was added to the composite. The conductivity and stretchability was measured at 18/82 vol% ultra-long Ag NWs/SBS composite depending on the zonyl concentration and the data are presented in **Figure 7**. As the zonyl concentration increased, the conductivity of the ultra-long Ag NWs/zonyl/SBS composite was decreased, but the stretchability of the composite was increased. As a result, the composite has stretchability of ~100% with initial conductivity of

~29,000 S/cm at 3 wt% zonyl concentration and has stretchability of ~450% with initial conductivity of ~20,000 S/cm at 10 wt% zonyl concentration.

The durability test of the composite was performed at 3 wt% zonyl concentration (**Figure 8**). 3000 repetitive cycles of 10% stretched/released states were applied to the composite and the composite maintained stable resistance during the cycles.

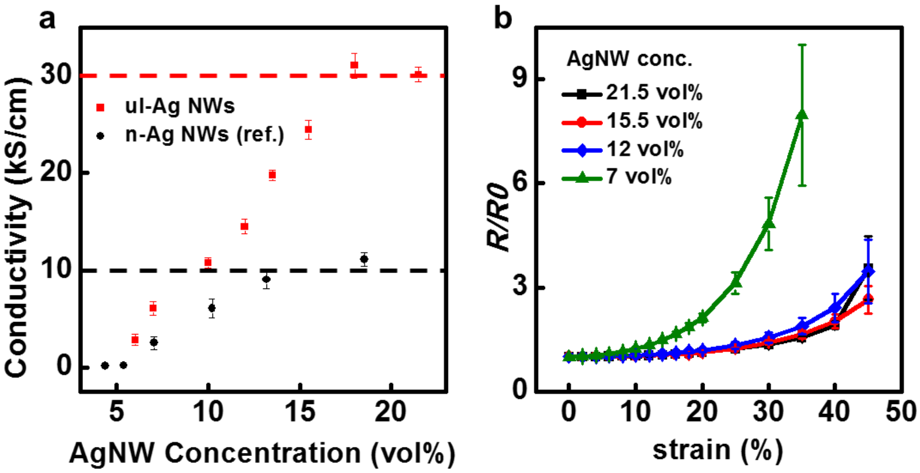

Figure 6. **Properties of the ultra-long Ag NWs/SBS composites**

(a) Conductivity of the ultra-long Ag NWs/SBS composite depending on ultra-long Ag NWs concentration. Red dash line indicates 30 kS/cm and black dash line indicates 10 kS/cm. Error bar; STDEVA (n=3)

(b) *R/R0*-strain data of ultra-long Ag NWs/SBS film-type composite (W:L=3:1) depending on ultra-long Ag NWs concentration. Error bar; STDEVA (n=3)

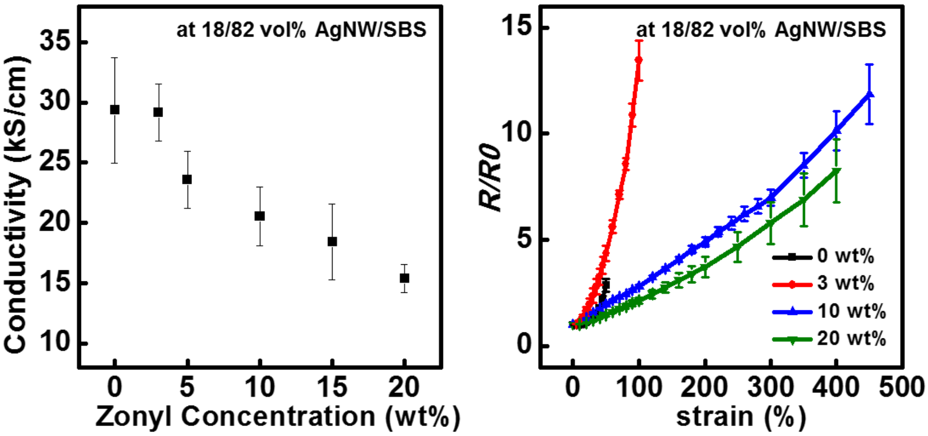

Figure 7. **Properties of the ultra-long Ag NWs/zonyl/SBS composites**

(a) Conductivity of ultra-long Ag NWs/zonyl/SBS composite at 18/82 vol% of ultra-long Ag NWs/SBS concentration depending on zonyl concentration. Error bar; STDEVA (n=5 for 0 wt%, n=6 for the others)

(b) *R/R0*-strain data of the ultra-long Ag NWs/zonyl/SBS film-type composite (W:L=3:1) at 18/82 vol% of ultra-long Ag NWs/SBS concentration depending on zonyl concentration. Error bar; STDEVA (n=3)

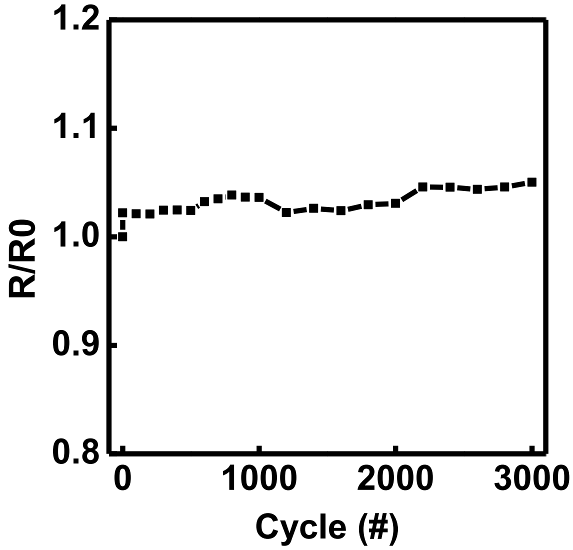

Figure 8. **Durability test**

10% stretched/released test for 3000 cycles was performed with the ultra-long Ag NWs/zonyl/SBS film-type composite at 3 wt% zonyl concentration. The resistance data were measured at released states.

4. Mechanism of surfactant effects in the composite

4. 1. Interphase action of zonyl : Region separation

Zonyl is a fluorine surfactant, so it has both hydrophilic and hydrophobic parts in the molecule. Owing to this features, the addition of zonyl to the composite cause separated region of ultra-long Ag NWs (hydrophilic) and SBS (hydrophobic). The separated ultra-long Ag NWs and SBS regions take charge of the conductivity and stretchability respectively and the conductivity and stretchability are enhanced [8]. In case of the conductivity, however, the conductivity showed decrease tendency with increased zonyl concentration over certain concentration of zonyl because zonyl is insulator materials itself [9].

These region separation of ultra-long Ag NWs and SBS are observed at SEM top-surface images of the composites. The ultra-long Ag NWs were randomly and homogeneously located in the SBS matrix without zonyl (**Figure 9a**). On the other hand, the ultra-long Ag NWs were gathered together with zonyl and the tendency of gathering were increased with increased zonyl concentration. (**Figure 9b, c, and d**).

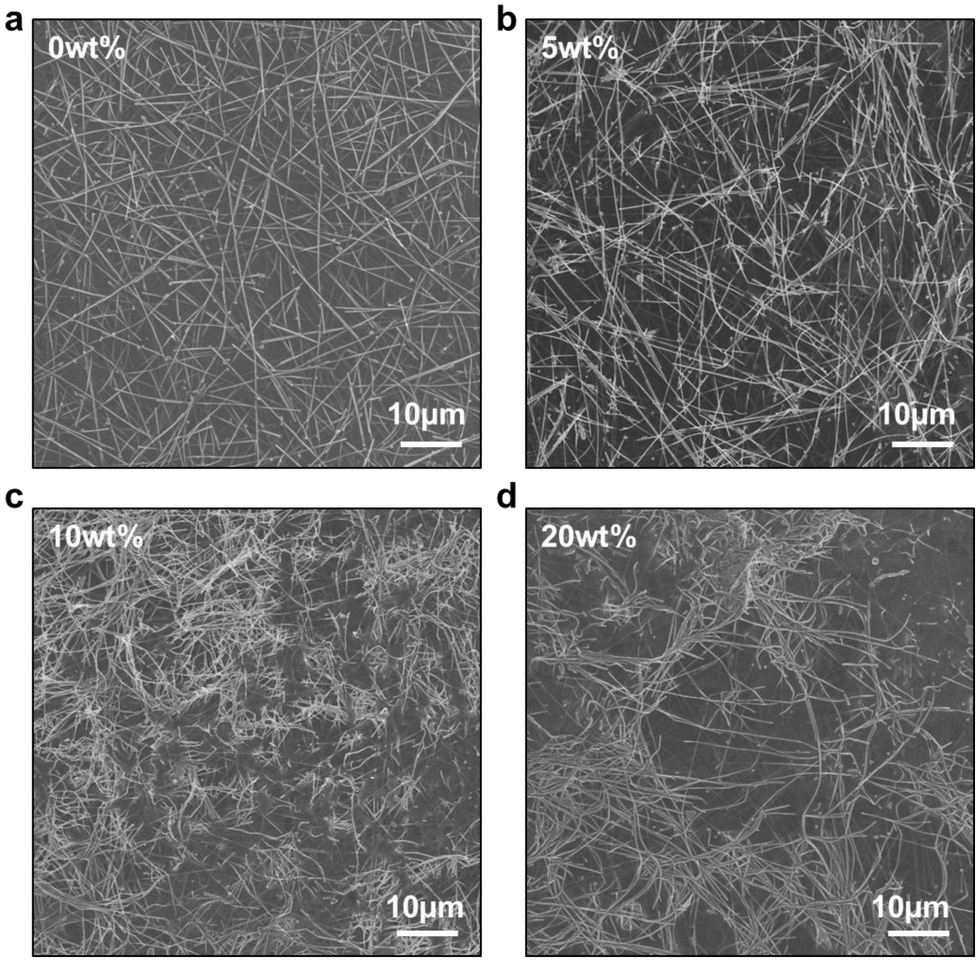

Figure 9. **SEM top-surface images showing interphase action of zonyl**

SEM top-surface images of ultra-long Ag NWs/zonyl/SBS composite at various zonyl concentration of (a) 0 wt%, (b) 5 wt%, (c) 10 wt%, and (d) 20 wt%

4. 2. Micro-porous structure produced by zonyl

Surfactants have feature to form reverse micelles in organic solvent. In our composite in which ethanol and toluene are used as the solvent, micro-porous structure is built by reverse micelles that formed by zonyl, the fluorine surfactant. This micro-porous structure in the composite generates hidden serpentine, which is an essential element of the stretchability. The schematic image about the reverse micelles and hidden serpentine is presented in **Figure 10**.

The modulus data were measure with the composite of zonyl concentrations of 0, 5, 10, and 20 wt%. The average modulus of composites were 61.44 MPa at 0 wt%, 51.72 MPa at 5 wt%, 35.86 MPa at 10 wt%, and 11.76 MPa at 20 wt%. The modulus data presented decrease tendency with increased zonyl concentration (**Figure 11**). The micro-porous structure of the composite was confirmed indirectly from this result.

To prove the presence of the micro-porous structure obviously, SEM cross-sectional images were obtained. While the flat cross-section was observed in the composite without zonyl (0 wt%, **Figure 12a**), the micro-porous structure was observed with zonyl and the porosity of the composite seemed to increase with increased zonyl concentration (**Figure 12b, c, and d**).

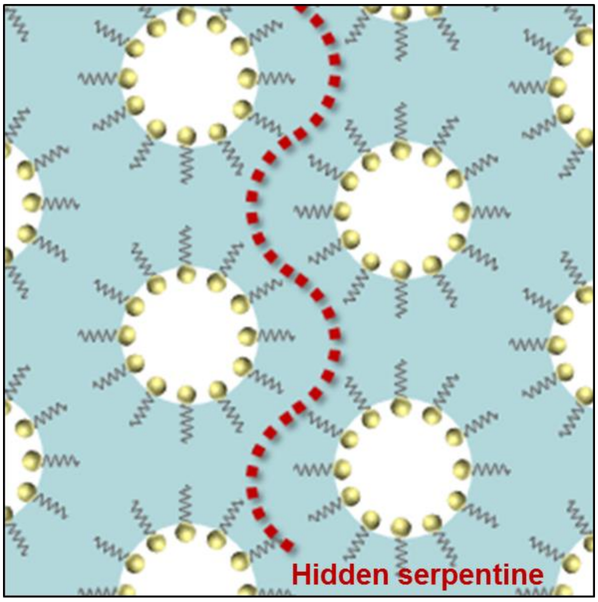

Figure 10. **Schematic image of micro-porous structure**

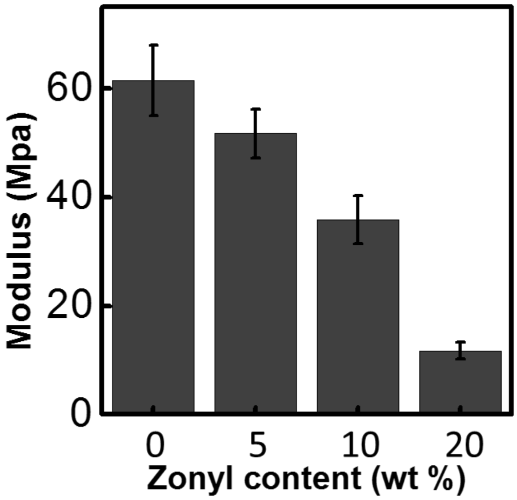

Figure 11. **Modulus test of the ultra-long Ag NWs/zonyl/SBS composite depending on zonyl concentration.**

The average modulus of composites is 61.44 MPa at 0 wt%, 51.72 MPa at 5 wt%, 35.86 MPa at 10 wt%, and 11.76 MPa at 20 wt%. Error bar; STDEVA.

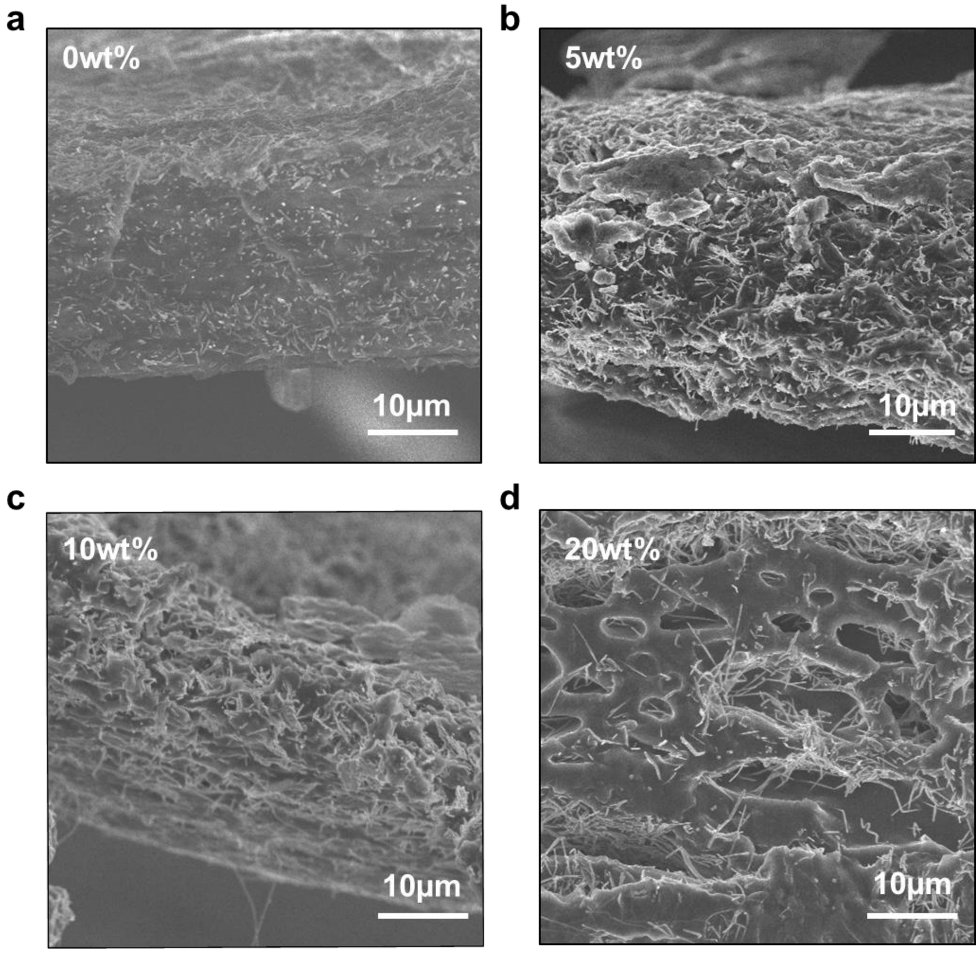

Figure 12. **SEM cross-section images showing micro-porous structure**

SEM cross-section images of ultra-long Ag NWs/zonyl/SBS composite at various zonyl concentration of (a) 0 wt%, (b) 5 wt% (c) 10 wt%, and (d) 20 wt%

5. Application as stretchable interconnects for LEDs

The application as stretchable conductor was demonstrated. The film-type composite of 10 wt% zonyl with 18/82 vol% ultra-long Ag NWs/SBS concentration was used as stretchable interconnects for LEDs. The composite was used as interconnects between power supplier and LED. The LED gave out light stably even under ~300% stretched composite. Photographs of this application are presented in **Figure 13**.

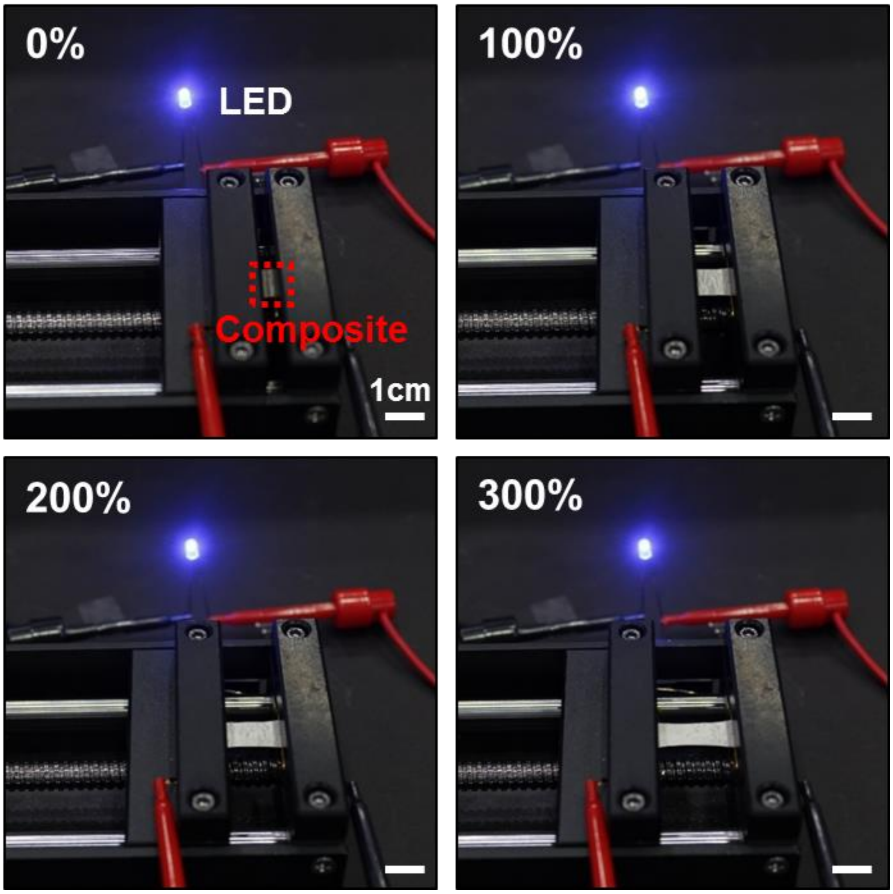

Figure 13. **Application as stretchable interconnects for LEDs**

6. Experimental Section

Ultra-long Ag NWs Synthesis and Purification

A modified synthesis method of ultra-long Ag NWs was used. 100mL of ethylene glycol was heated in an 166°C oil bath with 260rpm stirring. As temperature was saturated, 380mg of PVP (average molecular weight, 360k; Aldrich) dissolved in 30mL ethylene glycol was added. After 15 minutes, 800μL of a 4mM copper chloride solution ($CuCl_2 \cdot 2H_2O$; Aldrich) was added. After 10 minutes, 480mg of $AgNO_3$ ($AgNO_3$; > 99% purity, Strem Chemicals, Inc.) dissolved in 30mL ethylene glycol by shaking and sonicating for ~8 minutes at 60°C was injected for 10 minutes (180mL/hr). The stirring was stopped when injection was finished and the reaction lasted 20 minutes more. As-synthesized ultra-long Ag NWs solution washed with ethanol 3 times to remove ethylene glycol and byproducts such as nanoparticles.

Length distribution counting

The length data of Ag NWs were obtained from SEM images using the image processing program (Image J, NIH, Maryland).

Ligand-exchange reaction of ultra-long Ag NWs

200mg of as-synthesized ultra-long Ag NWs was dispersed in 38mL of dimethylformamide (DMF). First, 2mL of 0.1M $NOBF_4$ in DMF was added and gently shaked. Second, 40mL of hexane was added. Third, 0.33mL of hexanoic acid and 0.33mL of hexylamine was added and gently shaked. After ligand-exchange reaction, the solution was purified by ethanol, centrifuged 3 times, and dispersed in toluene:ethanol (volume ratio of 5:2)

Preparation of the ultra-long Ag NWs/SBS and the ultra-long Ag NWs/zonyl/SBS composite

The ultra-long Ag NWs were dispersed in toluene/ethanol with concentration of 40mg/mL. SBS was dissolved in toluene and Zonyl® FS-300 ($R_fCH_2CH_2O(CH_2CH_2O)_xH$; Aldrich) was diluted with tetrahydrofuran. These solutions were mixed with proper ratio by voltexing and the solvent was evaporated to produce the film-type composite.

Conductivity measurement

The sample was made by dropping of Ag NWs solution on SiO_2 wafer. The solvent was slowly evaporated. Sample was annealed at 100°C for 1 hour. Sheet resistance of sample was measured using the automatic resistivity meter (CMT-100MP, AiT, Republic of Korea). Thickness of sample was measured from SEM cross-section images. The SEM images were obtained by a scanning electron microscope (SEM; JSM-6701F, JEOL, Japan). Conductivity was calculated based on sheet resistance and thickness of sample.

Conductivity [S/cm] = 1 / (sheet resistance [ohm] * thickness [cm])

Conductivity of the composite was measured in the same way, but the sample was made by dropping of the composite solution on PDMS.

Stretchability test

Stretchability test was performed with film-type composite. (Width : Length=3:1, initial L = 5mm) As the film-type composite was stretched by using an automatic uniaxial stretching stage (Jaeil Optical System, Republic of Korea), the resistance data were measured with a Source Measure Unit (Series 2400, Keithley, USA).

Durability test

Durability test was performed along the same lines as the stretchability test, but the applying and releasing of strain were repeated. The resistance data were measured at released state and taken at an interval of 100 cycles from 0 to 1000 cycles and at an interval of 200 cycles from 1000 to 3000 cycles.

Modulus test

The modulus data was obtained from the stress-strain curve. Stress-strain curve was recorded with the film-type composite using a tensile mechanical testing system (ESM301, Mark-10, USA).

Application as interconnects

The composite of 3 wt% zonyl concentration with 18/82 vol% Ag NWs/SBS concentration was used for the application. The film-type composite was welded on the SBS film. The commercial micro-sized LEDs were attached on the both ends of the composite. The stretching of the composite was performed with manual uniaxial stretching stage under applied 5V using Triple output DC Power Supply (U8031A, Keysight, USA)

7. Conclusion

In summary, I developed the highly conductive and stretchable composite by using ultra-long silver nanowires (ultra-long Ag NWs), styrene-butadiene-styrene (SBS), and surfactant. The ultra-long Ag NWs/SBS composite has high conductivity of ~30,000 S/cm and stretchability of ~45% without introduction of structure for stretchability. Furthermore, the composites that have better stretchability were achieved by adding surfactant. Depending on the concentration of the surfactant, the composites with various specifications were obtained. The composite has stretchability of ~450% with initial conductivity of ~20,000 S/cm at high surfactant concentration and has stretchability of ~100% with initial conductivity of ~29,000 S/cm at low surfactant concentration. These effects are based on region separation and micro-porous structure caused by zonyl. The composite was prepared as solution mixture and fabricated by evaporating the solvent. Thus, the composite could have processability. The application as stretchable interconnects was demonstrated and the composite showed stable performance under stretched condition.

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요약 (국문 초록)

매우 긴 은 나노선과 탄성체, 불소계 계면활성제를 이용한 고 전도성 및 신장성 혼합물

서울대학교 공과 대학원

화학생물공학부

현원지

최근 웨어러블 디바이스와 로봇 산업의 발전에 따라, 더 높은 전도성과 신장성을 가지는 신장성 전도체가 요구되고 있다. 본 논문에서는 매우 긴 은 나노선과 탄성체 (스타이렌-부타디엔-스타이렌), 불소계 계면활성제 를 이용하여 높은 전도성과 신장성을 가지는 혼합물을 구현하였다. 은 나노선은 높은 고유 전도도와 함께 높은 직경 대비 길이 비를 통해 변형 상태에서도 안정적인 전도성을 유지하는 특성을 가지는 물질로, 신장성 전도체의 전도성 충전제로서 주목 받고 있다. 이

연구에서는 매우 긴 길이를 가지는 은 나노선을 이용하였고, 그 결과, 은 나노선의 전도성 충전제로서의 우수성을 극대화할 수 있었다. 매우 긴 길이의 은 나노선에 표면처리를 도입하여, 탄성체와 균일한 혼합물을 형성하였다. 이러한 혼합물은 30,000 S/cm 이상의 높은 전도성을 보였으며, 신장성을 향상시키기 위한 별도의 구조 없이 ~ 45%의 신장성을 보였다. 추가로, 혼합물에 불소계 계면활성제(zonyl)를 첨가하였고, 이는 혼합물의 구획화와 미세 다공성 구조의 형성을 통해 혼합물의 신장성을 향상시켰다. 이를 통해 20,000 S/cm의 초기 전도도에서 ~450%의 신장성을 가지는 혼합물과 29,000 S/cm의 초기 전도도에서 ~100%의 신장성을 가지는 혼합물을 얻어낼 수 있었다. 이러한 혼합물을 이용하여 신장성 인터커넥트로서의 간단한 응용을 시도하였고, 혼합물이 늘어난 상태에서도 안정적인 성능을 보이는 것을 확인할 수 있었다.

주요어 : 신장성 전도체 혼합물, 신장성 전극, 매우 긴 은 나노선, 불소계 계면활성제

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