공학석사학위 논문

Fabrication of silver nanowire based low haze transparent conducting film through optimization of synthesis protocol

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은나노와이어 합성 최적화를 통한 낮은 흐림도를 가지는 투명전극 제작에 대한 연구

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문 현 진

Abstract

The need for next generation transparent conducting films that can replace indium doped tin oxide film has consistently come up. To do this, over the last decade, researchers explored the possibility of using nanomaterials such as CNT, graphene, and metal nanowires. Amongst, since silver nanowire based transparent conducting films have shown higher transmittance and electrical conductivity compared to those of ITO films, the electronic industry has recognized them as promising substitutes. However, due to higher haze value of silver nanowire based transparent conductor compared to ITO films, poor resolution is observed when silver nanowire based films are applied to touch screen panels. The poor visibility is attributed to light scattering by the silver nanowires, and this haze value can be dramatically reduced by using thin silver nanowires. In this research, to synthesize thin silver nanowires with high length to diameter ratio, we first identified the roles of important synthesis parameters such as molar concentration of $AgNO_3$, NaCl and molecular weight of PVP. Based on the findings, we were able to synthesize silver nanowire solution with high aspect ratios. After that, we fabricated transparent conducting films using synthesized silver nanowires, which showed 2% of haze value as well as both higher transmittance and electrical conductivity compared to ITO films.

Keyword : Silver Nanowire, Transparent Conducting Film, Haze

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molecular weight of 1,300,000. Many nano and micro sized particles were produced.

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Figure 8. Doubling the amount of chloride reduced the diameter of synthesized AgNWs from 90-120 nm to 70-90 nm.

Figure 9. (a) SEM image of thick nanowires whose thickness is around 100 nm, and length distribution ranges from 80 – 100 μm. (b) SEM image of thin nanowires. Thickness and length are approximately 48 nm and 24 μm, respectively. (c) Transmittance spectra of TCFs fabricated using thick and thin nanwires. (d) Transmittance and haze values of sample number 1 to 4. Inset images show relative light scattering of films with thick and thin silver nanowires.

Figure 10. (a), (b) Demonstration of Touch screen panel fabricated by thin silver nanowires. (b), (c) Clarity difference between transparent films composed of thin and thick nanowires.

Chapter 1. Introduction

1.1. Study Background

Cell phones, tablet PCs, and solar cells all have something in common: These devices have used materials that are transparent to visible light, while conductive as an essential part of the technology stack. Today, the dominant material used for this purpose is indium-doped tin oxide (ITO)[1]. However, problems have emerged with this material, mainly due to lack of supply and the natural properties of ceramics[2]. These issues with ITO as transparent conductors are being addressed by developing next-generation transparent materials such as conducting polymers, carbon nanomaterials (e.g., CNT and graphene), and metal nanostructures (e.g., metal thin film, metal grids, and nanowires)[3-6]. Due to poor conductivity compared to metal, conducting polymer has been used as an intermediate layer rather than the transparent current collector layer[7]. Solution-processable CNT have suffered from dispersion stability in solution and separation between single and multi-wall structures[8]. Although single-layer graphene has demonstrated superior performance, expensive equipment and processes are still required[9]. In this regard, silver nanowire (AgNW) is getting attention as an alternative transparent electrode material. AgNWs are very conductive materials due to their free-electron density, and their network structure can be highly transparent, while maintaining good electrical conductivity in the visible wavelength range. Moreover, large-scale, high-yield synthesis of AgNW has been achieved through the polyol process.

Using AgNWs with a high-aspect ratio is fundamental to the fabrication of highly transparent and conductive AgNW films. To control the aspect ratio of AgNW, a series of research efforts concerning the molar ratio of reaction agents, temperature, reaction time, stirring rate, and the control agent have been conducted for the past few decades[10-14]. Based on these results, some have modified synthesis protocols and found a way to synthesize very long AgNWs (>100μm), showing higher conductivity and lower sheet resistance compared to previous efforts[15, 16]. In terms of performance, much previous research has disregarded the haze factor compared to ITO[17-19]. However, industry has required at least similar or superior alternative results. But from the previous research, AgNW network electrodes have shown high haze values (over 5%). With these values, mainly due to AgNW's thickness, it is hard to see more clear images compared to those from ITO electrodes. Therefore, in order to produce AgNW mesh electrodes comparable to ITO electrodes in terms of transmittance, sheet resistance and haze, it is essential to synthesize AgNWs having thin diameters (~50 nm) as well as a high aspect ratio**.** In terms of process, a relatively high-temperature annealing process has been regarded as a disadvantage of AgNW's percolation network[20]. Even though hybrid approaches and pressing process have reduced contact resistance of the junction between AgNWs and allowed room temperature soldering, using only intact metal NW networks is more advantageous than the aforementioned approaches in terms of transmittance[21, 22].

1.2. Purpose of Research

In this study, we found how $AgNO_3$, NaCl molar concentration, and polyvinylpyrrolidone (PVP) molecular weights affect the diameter and length of nanowires, and we eventually synthesized thin and long AgNWs by applying the findings obtained from the parametric studies to other synthesis methods. Based on optimized chemicals' molar concentrations, thin (~48 nm) AgNWs with a high aspect ratio (~500) were successfully synthesized. Furthermore, we have achieved a low-haze and annealing-free process and the fabricated electrodes maintain superior properties (~96%, 100 ohm/sq, 2% haze) after solvent evaporation at room temperature. Finally, we have fabricated a flexible and highly transparent touchscreen with the optimized AgNW percolation network. Fabricated transparent electrodes have shown better visibility by reducing light scattering on the surface with thin AgNWs.

Chapter 2. Experiment

2.1. Synthesis Method

In a typical experiment, 50 mL of 0.2 M Glycerol solution of PVP was prepared at 160℃ and 5 mg of NaCl was added into the solution. After that, 600 μL of DI water containing 0.56 g of $AgNO_3$ was injected into the flask. The solution was under continuous magnetic stirring at a rate of 225 rpm during the whole process. Upon the completion of the synthesis, the nanowire solution was air-cooled down to room temperature and some portion of the solution was diluted with methanol in a ratio of 1:9 and centrifuged three times at 5,000 rpm for 30 minutes. For a parametric study, $AgNO_3$ molar concentration (in a range of 0.022-0.0132 M), NaCl molar concentration (in a range of 0.34-10.2 mM), and PVP molecular weight (in a range of 29,000-1,300,000) were modulated, with other parameters fixed.

2.2. Transparent Conductor Fabrication

To confirm the effect of diameter size on the haze of transparent conductors, two different types of transparent conductors composed of thin or thick AgNWs were fabricated through the Mayer rod coating method. The cleaned thin or thick AgNWs were dispersed in isopropyl alcohol solution at a concentration of 0.003 mg/mL. The mixed solution was deposited onto a glass substrate by pipetting and coating by a Mayer rod. The transmittance and sheet resistance of electrodes were adjusted by repeating the coating steps.

2.3. Characterization

AgNWs dispersed in DI water solution were dropped on bare silicon substrates and the morphology of the nanowires were examined using scanning electron microscopy (SEM). The average lengths and diameters of AgNWs were determined by examining two hundred nanowires using image measurement software. The sheet resistances of transparent conductors were determined with a four-point probe apparatus (M4P 302-System) by measuring the diverse points on the surface of the transparent conductor. Both transmittance and haze were measured simultaneously with the JCH-200S system.

Chapter 3. Results & Discussion

3.1. Effect of $AgNO_3$ Molar Concentration

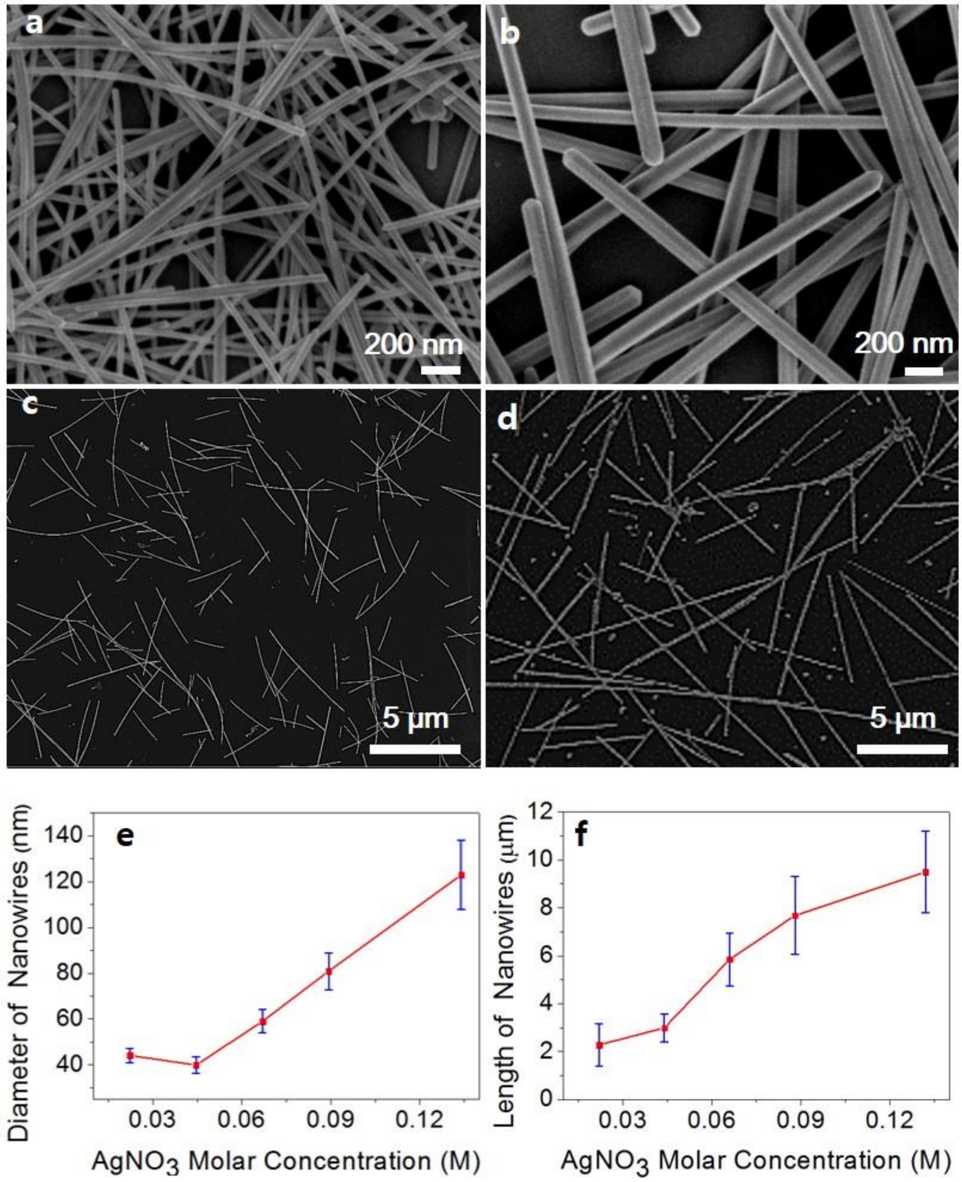

Figure 1. The effect of molar concentration of $AgNO_3$. (a), (c) SEM images of nanowire product made by 0.044 M of $AgNO_3$. (b), (d) SEM images of nanowire product made by 0.134 M of $AgNO_3$. (e), (f) Higher molar concentrations of

$AgNO_3$ lead to the formation of thicker nanowire with longer lengths.

To investigate the effect of $AgNO_3$ molar concentration on the morphology of AgNW, we injected several different amounts of $AgNO_3$ (0.18-1.12 mol), with other parameters fixed. As illustrated in **Figure 1-e**, we found that $AgNO_3$ molar concentration affects both the diameter and length of nanowires. An increase in

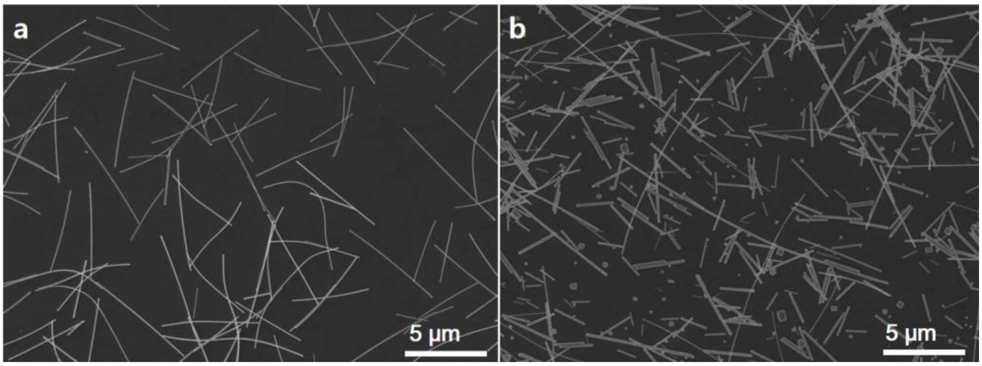

Figure 2. (a), (b) SEM images of nanowire product by 0.088 M and 0.17 M of $AgNO_3$.

$AgNO_3$ molar concentration leads to an increase in the average diameter of AgNWs. We believe that the diameter size is related to the amount of free silver cations (Ag^+) at the initial stage. In AgNW polyol synthesis, multiply-twinned particles (MTPs) are formed when nuclei grow to certain sizes, depending on the solution environment. Put simply, the larger MTPs are shaped by more Ag^+ sources at the initial stage, which leads to a larger average diameter[23]. However, injection of excessive amounts of $AgNO_3$ (0.17 mol) create many thick but irregular-sized nanowires and particles. This is due to a relatively insufficient amount of PVP, which coordinates with the surfaces of AgNWs (**Figure 2**). We also found that nanowire length was affected by $AgNO_3$ concentration (**Figure 1-f**). Namely, an increase in $AgNO_3$ concentration results in nanowires with larger lengths. As in the case of the growth of diameter, we believe that enough free Ag^+ in the solution facilitates growth in length.

3.2. Effect of NaCl Molar Concentration

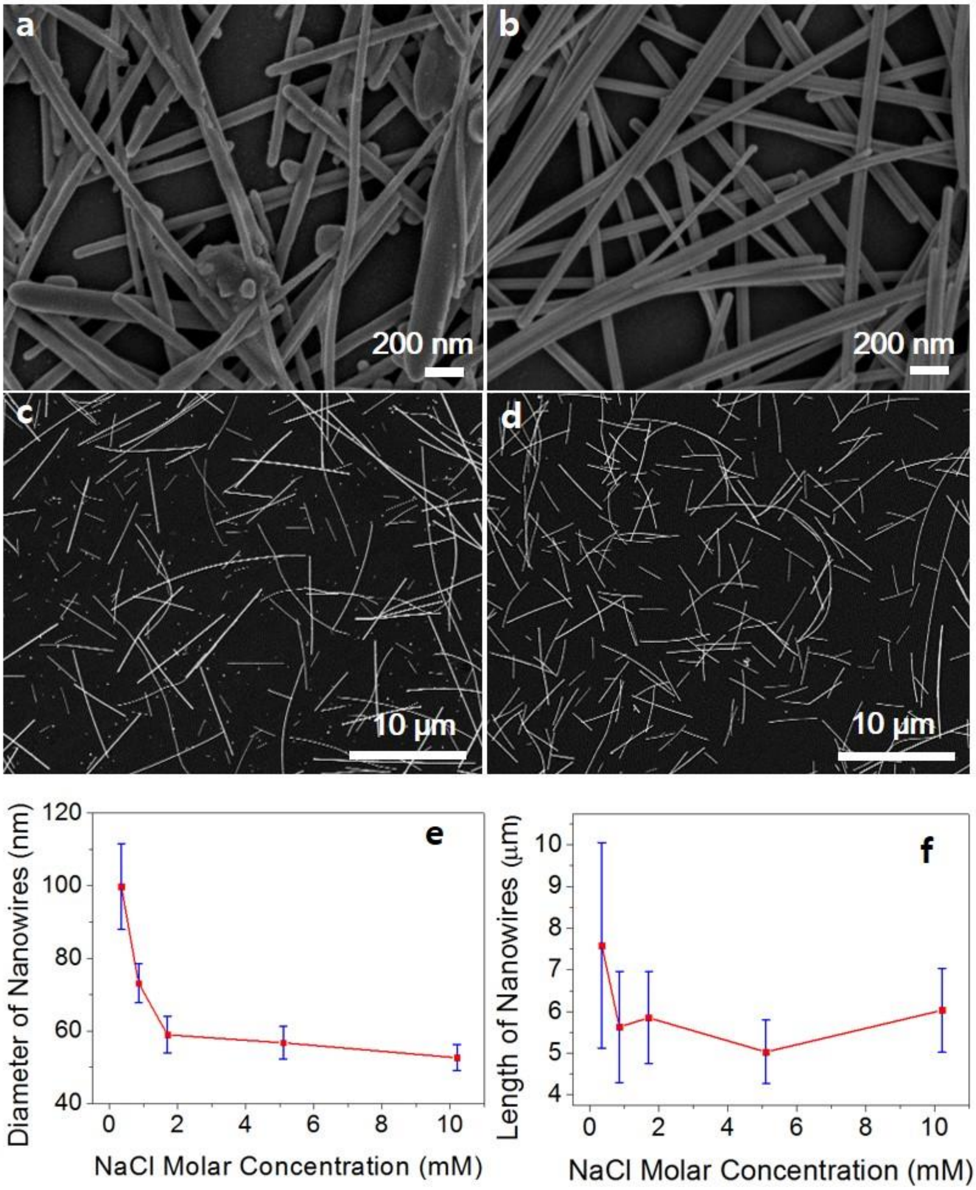

Figure 3. The effect of NaCl molar concentration. (a), (c) SEM images of nanowires produced by 0.34 mM of NaCl. (b), (d) SEM images of nanowires produced by 0.85 mM of NaCl. (eLarger amounts of NaCl lead to smaller diameter. (f) Size distribution is determined by the amount of added NaCl.

There have been a few reports on the effects of chloride (Cl^-) in the polyol process. Kylee E. Korte et al. have suggested that, due to the low solubility product (K_{sp}) value of silver chloride (AgCl) compared to that of $AgNO_3$, there is a decrease in free Ag^+ during initial Ag seed formation and subsequent slow release of Ag^+, mimicking the use of a syringe pump[24]. Sahin Coskun et al. have found an optimized NaCl concentration for their experimental setting to minimize micrometer-sized particles and AgCl particles, because Cl^- concentration controls the kinetics of the reduction process of $AgNO_3$[13]. Similarly, in our experimental setting, we found that not only does Cl^- molar concentration affect the quality of the AgNW solution, but the diameter of AgNWs as well. According to our results, the more NaCl that was injected, the thinner AgNWs that were synthesized (**Figure 3-e**). Our explanation for this is as follows. If more NaCl is injected into the solution with the $AgNO_3$ molar concentration fixed, more AgCl colloids are formed, leading to a decrease in free Ag^+. Because of the reduction in free Ag^+, when nucleates grow to MTPs, smaller MTPs are made, which results in a smaller AgNW diameter. In addition, because of the larger amount of AgCl colloids, a low concentration of Ag^+ is maintained over the course of the synthesis. When we consider the fact that modulating the quantity of Cl^- plays a similar role with changing the injection rate of the syringe pump, it is reasonable to conclude that Cl^- molar concentration is one of the major factors that control the size of AgNW diameter. With respect to length, when 0.34 mmol of NaCl was added, the standard deviation was largest in our experimental setting, and a SEM image of nanowires with insufficient NaCl is provided in **Figure 4**. Except for the first solution (0.34 mmol of NaCl), the other four nanowire solutions showed similar lengths and high quality.

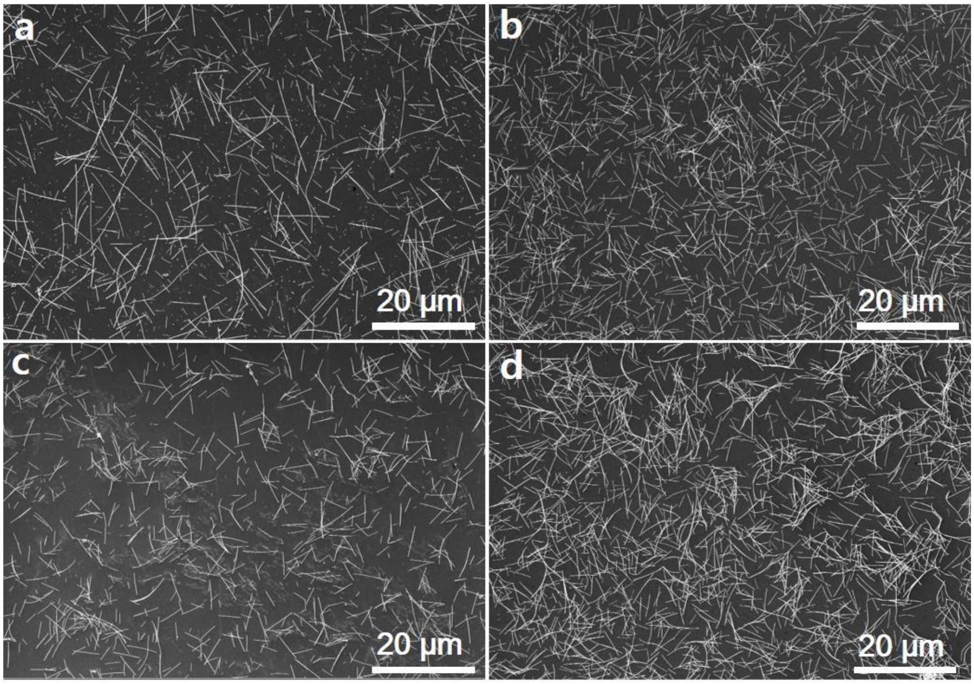

Figure 4. (a), (b), (c) SEM images of nanowires produced by 0.34, 0.85 5.1 and 10.2 mM of NaCl with other parameters fixed.

3.3. Effect of PVP Molecular Weight

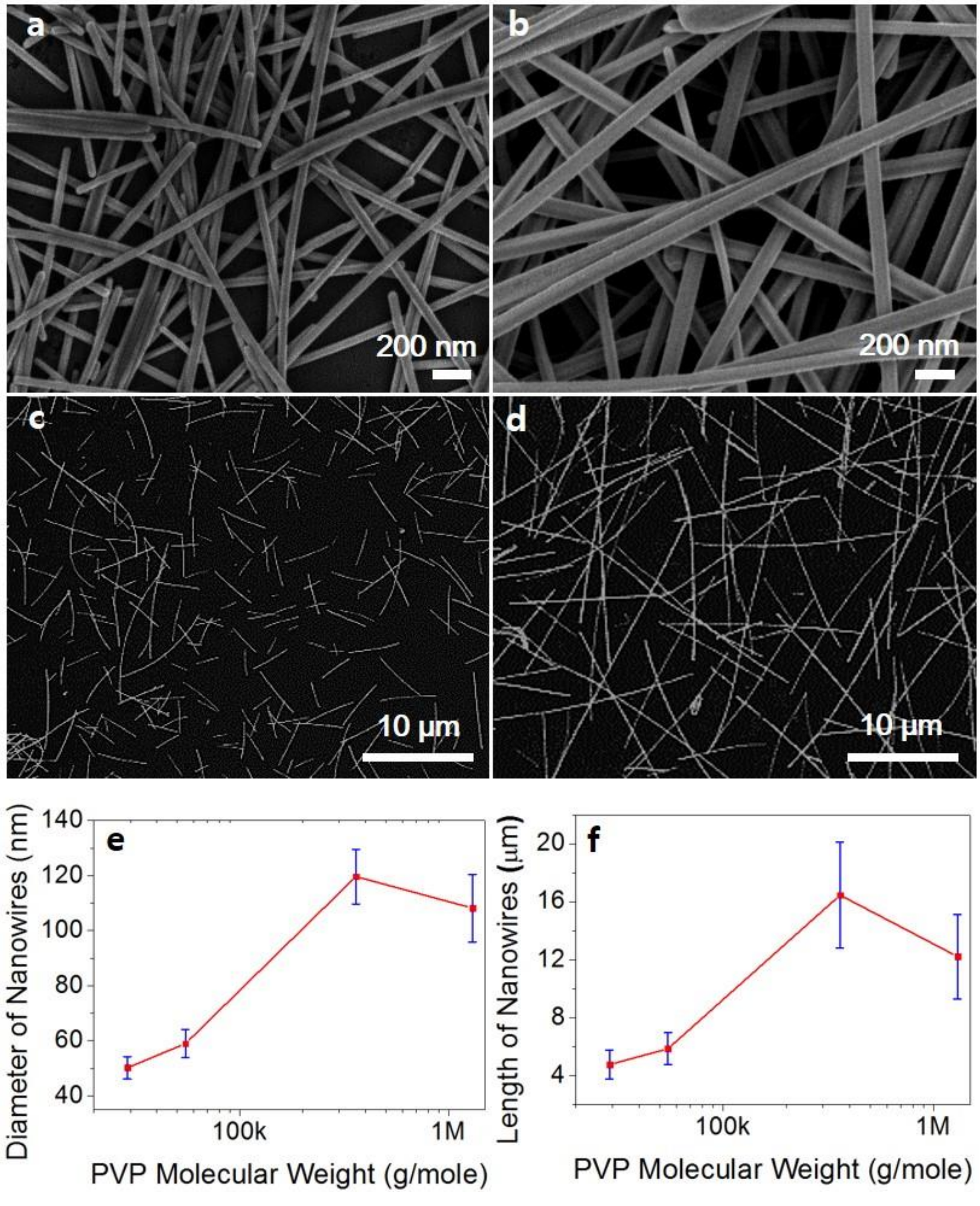

Figure 5. The effect of molecular weights of PVP. (a), (c) SEM images of nanowires produced by MW of 55,000. (b), (d) SEM images of nanowires produced by MW of 360,000. (e), (f) Longer chains of PVP result in longer and thicker nanowires.

The AgNW length and diameter at the end of the polyol process were strongly dependent on the molecular weights of PVP. In the polyol synthesis, the oxygen atoms in PVP are adsorbed on the (100) side of Ag surfaces through nonbonding electrons, and then hinder the growth in the [110] direction, consequentially facilitating 1D growth through the reduction of free Ag^+ on the (111) facet[24]. Yugan Sun et al. reported that using different molecular weights of PVP results in different morphologies of Ag nanostructures[25]. According to the paper, using PVP with a relatively low molecular weight (~10,000) produced Ag nanoparticles with irregular shapes. By contrast, AgNWs were successfully synthesized when a PVP molecular weight of 55,000 was used. The role of the chain length of polymers was recently reported by Jie-Jun Zhu et al, who suggested that PVP with higher molecular weights can more easily induce the formation of AgNWs with high aspect ratios due to more carbonyl groups in PVP molecules and more Ag^+ coordinated[26]. To further investigate the influence of molecular weights on the diameter and length, we used PVP with different molecular weights, while fixing other parameters. As shown in the **Figure 5-c**, long nanowires (~16μm) were synthesized from PVP with high molecular weight (360,000). The reason for the increased diameters as the molecular weight of PVP increases can be inferred from the fact that PVP forms a random coil[27]. According to a previous study, the PVP coil dimensions (average end-to-end distance) are 1-100 nm, depending on the molecular weights, ranging from 1,000 to 1,000,000[28]. In other words, there are many inner carbonyl groups that barely interact with Ag atoms, since PVP with long chains shows larger spherical shapes. In fact, since the PVP molecules with short chains have smaller-sized random coils, more carbonyl groups interact with (100) facets of AgNWs, resulting in smaller diameters. Using PVP of 1,300,000 led to long and thick nanowires, as well as a hundred nanometer-sized particles (**Figure 6**). The formation of many nanometer-sized particles is believed to be due to an unbalanced ratio of each reagent.

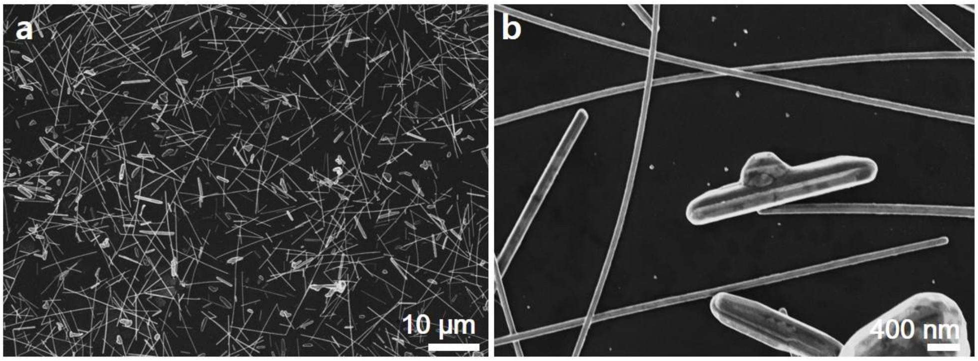

Figure 6. (a), (b) SEM images of silver nanowires produced by PVP having molecular weight of 1,300,000. Many nano and micro sized particles were produced.

3.4. Synthesis of Thin Silver Nanowire

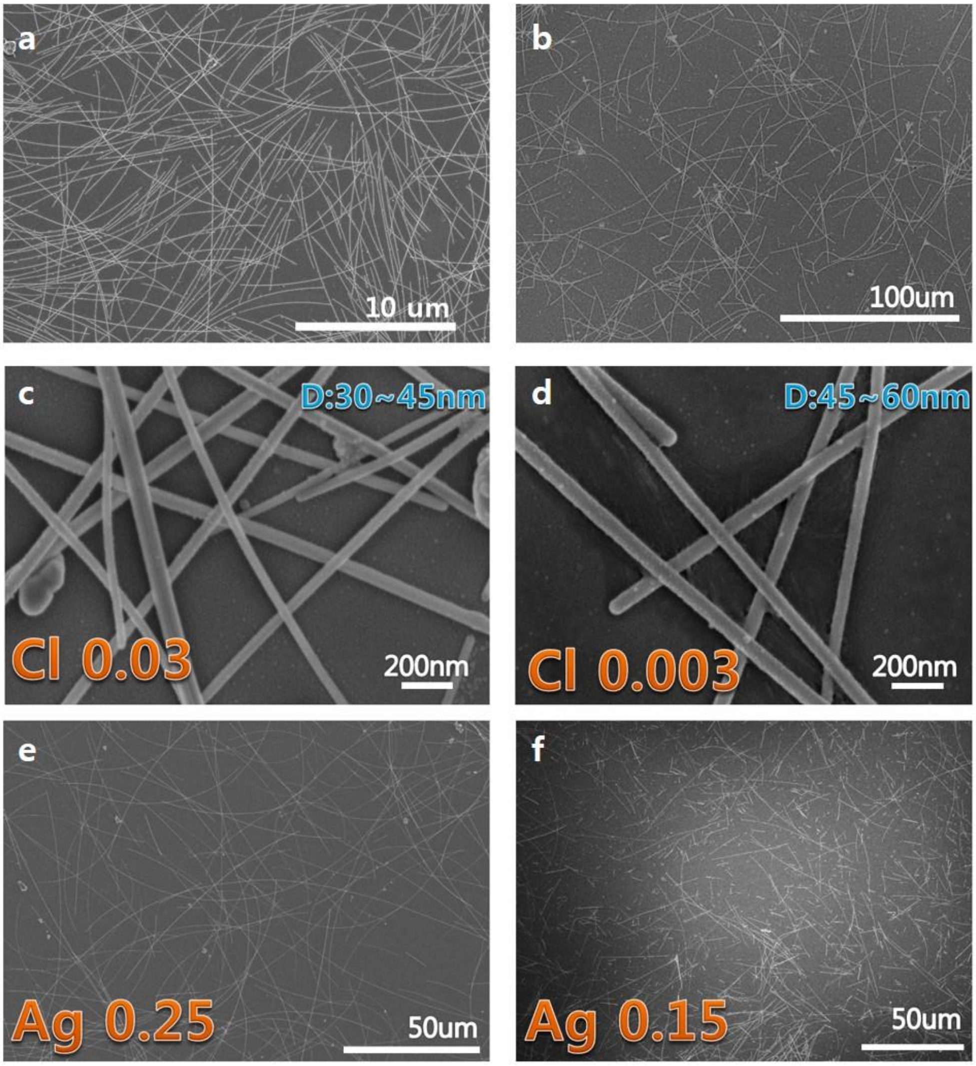

Figure 7. (a), (b) SEM images of nanowires produced by glycerol-based polyol method with the addition of higher molecular weight of PVP. (c), (d) The nanowire thickness was controlled by the amount of chloride in glycerol-based polyol method. (e), (f) The lengths of silver nanowire was controlled by the amount of silver ion.

Previous research results that are related to parametric study and process optimization have focused on one method to modify their result. In contrast, we have applied important findings that obtained from parametric study to other AgNW synthesis processes. One popular method to synthesize AgNWs at a large scale is through using glycerol-based polyol method. In this process, NWs have been synthesized with a diameter of 30-50 nm and a length of 5-20 µm. We applied PVPs with higher molecular weight to this method. As shown in **Figures 7-a, b**, although standard NWs have a diameter of 30-40 nm and a length of 5-20 µm, the modified process yielded a length of over 100 µm. This result again showed that higher molecular weight has an effect on increasing the length of AgNWs. In addition, the initial diameter also increased from 30-50 nm to 200 nm. **Figures 7-c**, d have shown the effects of chloride on the glycerol-based method. The diameter of synthesized AgNWs have increased about 10-20% by decreasing chloride by a factor of 10. In addition, another synthesis method also has been analyzed with chloride. Recently, one of the modified polyol methods was reported by J. Jiu et. al[28]. In this study, researchers synthesized AgNWs with a length of around 100 µm by reducing stirring speed and temperature. We have applied the chloride effect to this result to reduce the diameter of long AgNWs. As shown in **Figure 8**, the diameter of synthesized AgNWs has been reduced from 90-120 nm to 70-90 nm by doubling chloride quantity.

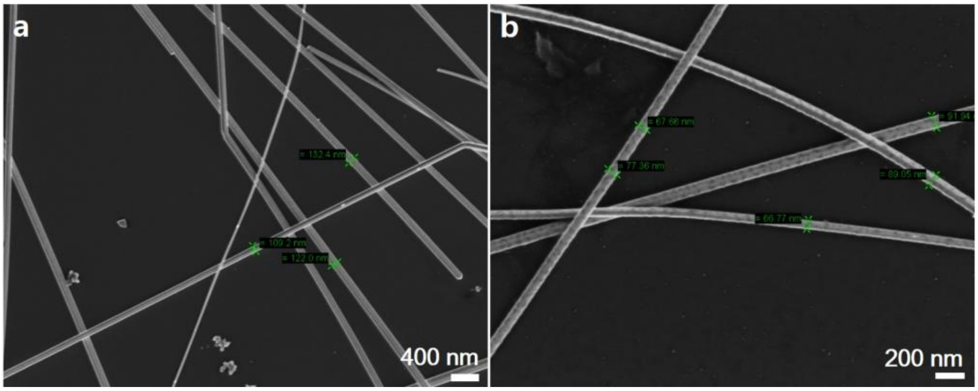

Figure 8. Doubling the amount of chloride reduced the diameter of synthesized AgNWs from 90-120 nm to 70-90 nm.

As indicated by our result in **Figure 1**, reduced silver source enabled thinner and shorter NWs. We also applied this result to the previous synthesis method. As shown in **Figures 7-e, f**, the length of AgNWs has been decreased by reducing $AgNO_3$ quantity, and length distribution has been widened because of the reduced amount of free Ag^+ at the initial stage. Based on these characteristics, in order to reduce nanowire diameter and maintain the quality of nanowire solution, we selected 0.035 M and 1.4 mM as the molar concentration for $AgNO_3$ and NaCl, respectively. In addition, we mixed different PVP molecules, whose molecular weights are 55,000 and 360,000, respectively, in a ratio of 4:1 to synthesize thin AgNWs with a high aspect ratio. By synthesizing nanowires in these conditions, we obtained thin nanowires (~48 nm) that still showed high length-to-diameter ratios (L: ~24 μm, D: ~48 nm), as shown in **Figure 9-b**. These synthesized thin AgNWs appeared relatively dim in the SEM image compared to thick AgNWs (**Figures 9-a, b**) because of the reduced surface scattering.

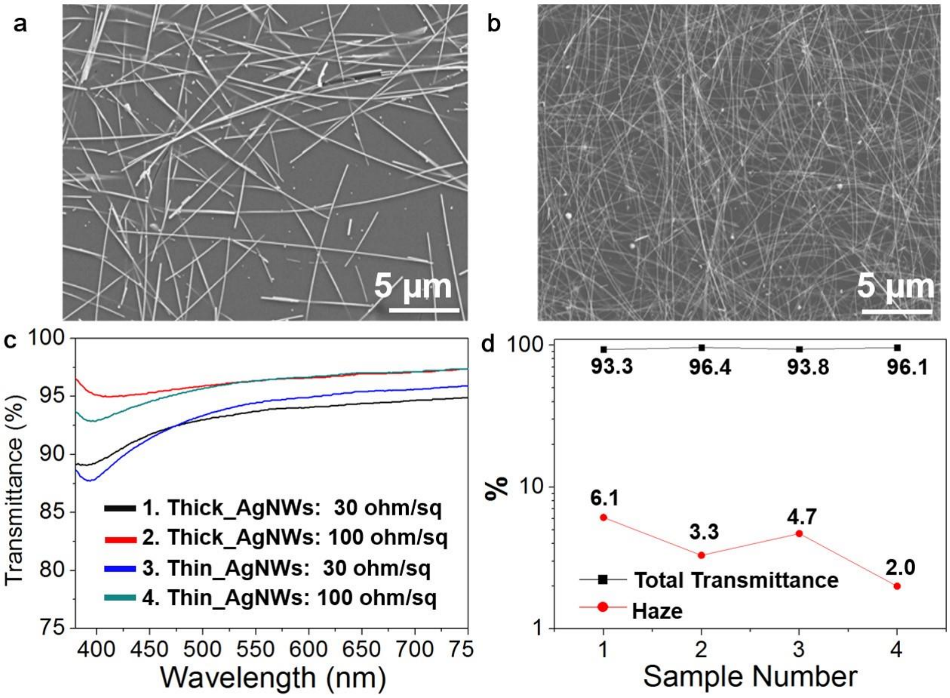

Figure 9. (a) SEM image of thick nanowires whose thickness is around 100 nm, and length distribution ranges from 80 – 100 µm. (b) SEM image of thin nanowires. Thickness and length are approximately 48 nm and 24 µm, respectively. (c) Transmittance spectra of TCFs fabricated using thick and thin nanwires. (d) Transmittance and haze values of sample number 1 to 4. Inset images show relative light scattering of films with thick and thin silver nanowires.

3.5. TCF Characterization and Application to Touch Screen Panels

To compare the performance of synthesized thin AgNWs with thick AgNWs for transparent conductors, we fabricated AgNW percolation networks on PET films using thin and thick NWs, respectively. Representative transmittances are addressed in **Figure 9-c**. Black and red lines in **Figure 9-c** indicate the transmittance and sheet resistance related to our previous result with thick AgNWs (L: ~90 μm, D: ~100 nm). Based on percolation theory, longer lengths of 1-dimensional structure show better performance in transmittance because fewer objectives can be used to make electrical paths than short structures. However, we have demonstrated almost the same or superior performance using the thin AgNWs in this research, even as they are shorter than thick AgNWs, as shown in **Figure 9-c**. We strongly believe that the high aspect ratio with a diameter of under 50 nm play important roles in terms of transmittance. Through the synthetic study, as we demonstrated above, we have found optimal conditions for various spectrums for lengths and diameters. Moreover, we have achieved a low haze that is comparable to that of ITO electrodes using thin and high-aspect-ratio AgNWs. As shown in **Figure 9-d**, only 2% of haze has been achieved, even as its transmittance is over 95% in visible wavelength. This haze value is very meaningful for the AgNW-based electrodes, which is one of the candidates to replace ITO. The haze property can be recognized directly by exposure of the light source on fabricated electrodes. As shown in the insets of **Figure 9-d**, incident light spread more on the film composed of thick AgNWs than the film with thin AgNWs. This phenomenon occurs by wide-angle light scattering on rough surfaces and insufficient finishing. Furthermore, these amazing results have been demonstrated without a post-anneal process. Flexible displays based on polymer substrate require a low-temperature process because of deformation by thermal treatment. In this manner, many approaches, such as cold pressing and hybrid structures, have been introduced to reduce the contact resistance at room temperature. In short, the electrical path can be made easily by just adding more structures without post-annealing, because the greater amount of objects over the theoretical percolation critical number increase the probability of ohmic contacts. In this context, we can assume that the annealing-free process can be achieved using many thin structures, while simultaneously maintaining high transparency of electrodes. From this result, we have applied optimized thin AgNWs to flexible touchscreens to compare the performance with thick AgNW-based touchscreens.

As shown in **Figure 10-a**, flexible touchscreens have been successfully demonstrated with a fabricated electrode composed of thin AgNWs. In addition, **Figures 10-b, c** clearly show that the diameter size of AgNWs have an effect on the haze that can been seen with the naked eye. The touchscreen based on AgNWs with reduced thickness and optimized length led to higher clarity for the fabricated electrode (**Figure 10-b**).

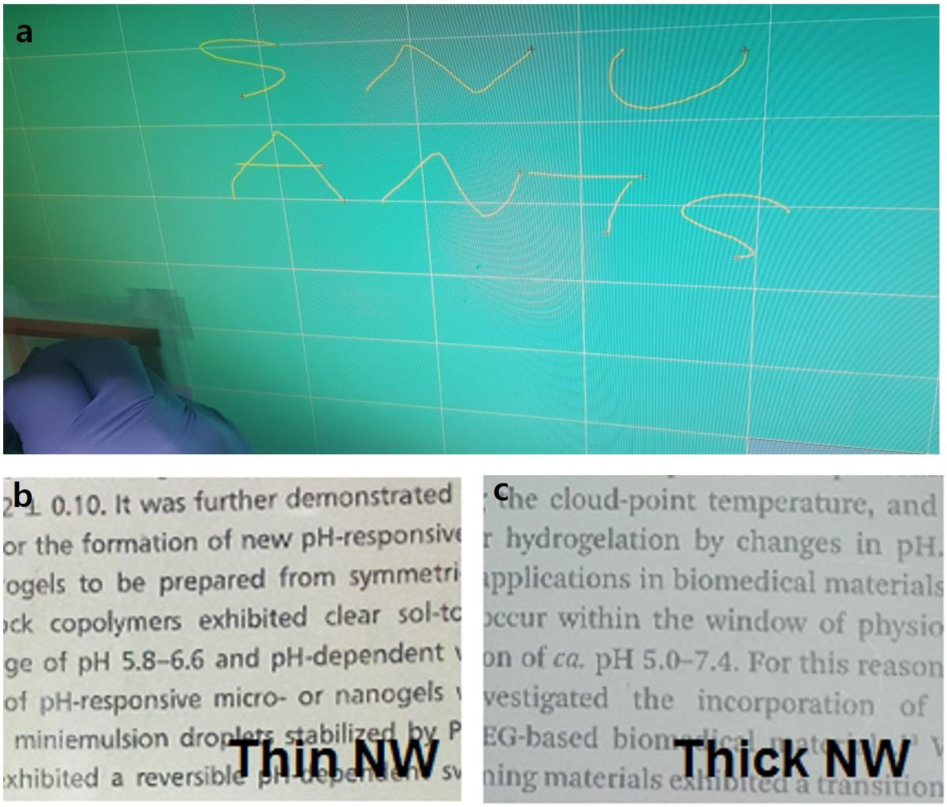

Figure 10. (a), (b) Demonstration of Touch screen panel fabricated by thin silver nanowires. (b), (c) Clarity difference between transparent films composed of thin and thick nanowires.

Chapter 4. Conclusion

We explored several synthesis factors to increase the controllability of the AgNW synthesis method. In particular, we studied the effects of molar concentration of $AgNO_3$ and NaCl, and the molecular weight of PVP, on the diameter and length of AgNWs. By applying the findings obtained from parametric study, it is confirmed that our results are applicable to various AgNW synthesis methods. Ultimately, we synthesized thin nanowires with a high aspect ratio by mixing PVP with short and long chains, choosing a low $AgNO_3$ molar concentration, and gradually increasing NaCl molar concentration. By using synthesized thin AgNWs with a high aspect ratio, we were able to fabricate low-haze transparent conductors by minimizing diffusive light transmittance. In addition, the transparent conductor showed both high transmittance and low sheet resistance as well (96%, 100 ohm/sq), which are comparable to conventional ITO conductors. More importantly, direct deposition of AgNWs allows fabrication of flexible transparent conductors without any post-treatment compared to previously reported fabrication methods. Finally, touchscreen panels were fabricated to demonstrate the possibility of thin AgNW-based transparent conductors as replacements for ITO conductors. We believe that our research suggests the useful insights for the fabrication of low-haze transparent conductors.

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초록

기존의 ITO 필름을 대체할 수 있는 투명전극 개발에 대한 연구 필요성이 꾸준히 제기되어왔다. 이를 위해 지난 10여년 동안 연구자들은 탄소나노튜브, 그래핀, 그리고 금속나노와이어를 이용한 투명전극 개발을 시도해왔다. 이 중에서 은나노와이어를 이용한 투명전극은 ITO 필름보다 높은 투명성 및 전기전도성을 보이기에 산업적으로 유망한 대체제로 인식되고 있다. 하지만 은나노와이어 기반의 투명전극은 ITO 필름에 비해 높은 흐림도 값을 보이기 때문에 터치스크린 패널에 적용될 선명도가 떨어진다. 이는 빛이 은나노와이어를 통과할 시 두께에 의한 빛의 산란 때문이다. 따라서 이러한 흐림도 값은 얇은 은나노와이어를 사용함으로써 획기적으로 줄일 수 있다. 본 연구에서는 은나노와이어 합성시의 중요한 요인들이 합성시에 미치는 영향을 파악하고, 이로부터 얇고 종횡비가 높은 은나노와이어를 합성하였다. 얻어진 은나노와이어를 이용하여 투명전극을 제작하였으며, 본 전극은 2%의 흐림도를 나타냈고, 또한 측정된 투명도, 전기전도도가 일반적인 ITO 필름보다 높음을 확인할 수 있었다.

Key word : 은나노와이어, 투명전극, 흐림도

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