
Optimization of Conditions for the Synthesis of Silver Nanowire

Anand Habib

The University of Texas at Dallas

2006

Abstract

This paper discusses an approach employing a Pluronic 123 (P123) surfactant that acts as a reductant of silver at critical concentrations, while also investigating both a hydrothermal treatment at autogenous pressure in autoclave and one atmosphere reflux at approximately 100 °C. As the key issue of reproducibility becomes the cornerstone of research, the effects of changes in different variables such as the ratio between P123 and silver nitrate, the concentration of reactants, the temperature at which the reaction takes place, and the type of solvent employed were examined. Though an SEM was unavailable to analyze the morphology of the nanomaterial, X-ray diffraction confirmed that silver (01-089-3722) was produced with crystal sizes up to 60 nanometers. Additionally, experiments were run concerning the optimal concentration of polyvinylpyrrolidone (PVP) when using ethylene glycol to reduce silver. After using concentrations of 0.02 M, 0.2 M, and 2.0 M PVP, it was discovered that the most favorable concentration was slightly less than 0.2 M using intensities of peaks from X-ray diffraction. Though problems with replication have not been resolved, the use of reflux techniques at lower temperatures resulted in promising results. Still, further testing must be carried out in order to pinpoint the reaction conditions between P123 and silver nitrate and to design a technique that is easily reproduced.

Optimization of Conditions for the Synthesis of Silver Nanowire

Introduction

Silver, element 47 on the periodic table, is a white lustrous metal. Its inherent properties as the best electrical and thermal conductor among all metals make silver the prime target of nanotech research. The development of electronic devices to manipulate nanomaterial at the atomic level makes the field of nanotechnology not only promising but also heightens the need to develop nanowires capable of widespread implementation as interconnects in microelectronics, optical, electronic, and magnetic devices. [1-7]

The intrinsic problem with the manufacturing of silver nanowires is the inability to effectively reproduce nanowires. The problem becomes twofold – a) selection of proper reactants in the correct quantities and b) the technique with which one executes such a procedure. Several prominent scientists have offered methods, but the hunt for a less expensive, straightforward procedure continues with each successive approach leading to silver nanowires with higher aspect ratios with greater lengths and smaller diameters.

In 2001 S. Liu, *et. al.* [7] reported that the formation of silver nanowires could not be completed without a template. Their investigations led them to believe that 1D silver nanostructures needed a physical template such as carbon nanotubes or zeolites to define the desired morphology of the nanowire. Their approach led them to use AgBr crystals and a developer containing AgNO₃ along with a gelatinous template. Using X-ray diffraction and transmission electron microscopy, they obtained nanoparticles and nanowires (30% yield by weight) with diameters of 80 nm and lengths of 9 μm. They theorized that the gelatin provided the reduced silver a preferential growth direction leading to anisotropic growth. Interestingly, they discovered that the silver comprising the nanowire originated from the silver nitrate and not the silver bromide crystals.

In 2002 Y. Sun, *et. al.* [6] described a soft, solution-phase approach that would obviate the need to dissolve and disintegrate hard templates that could potentially lead to diminished results. In their experiment they reduced silver nitrate with ethylene glycol (EG) at 160 °C and subsequently added solutions of silver nitrate and polyvinylpyrrolidone (PVP) to the solution leading to nanowires with diameters of 30-40 nm and lengths up to 50 μm. The formation of silver nanowires centered around preformed seeds from the initial reduction of AgNO₃ in EG which served as both a reducing agent and solvent. PVP served to control the growth faces of crystalline faces, thus its concentration was critical to morphology. Too high of a concentration might lead to isotropic growth, but also the order in which the PVP and AgNO₃ solutions were added also mattered. Additionally, they reported that seeding with Pt or Ag led to nanowires with larger diameters while longer reaction times enhanced the morphology as well. Finally, they singled out three variables which affected silver growth. First, the ratio between PVP and AgNO₃ was critical. As previously mentioned, a high ratio between these two components would lead to isotropic growth while a low ratio would lead to

incomplete coverage of the faces of the nanowire. Temperatures greater than 100 °C were necessary to dissolve smaller nanoparticles and to diffuse silver atoms onto the faces of the nanowires; high temperatures (~185 °C) led to low aspect ratios. Lastly, the concentration, size, distribution, and structure of seeds were found to be critical.

In 2003 Z. Hu, *et. al.*, noted that silver could be used in chemical and electrical catalysis by virtue of its structure and high surface activation [5]. Returning back to physical templates, Hu implemented an anodized aluminum physical template (self-seeding) along with acetaldehyde reduction of silver. They noted that once the template was dissolved away, the silver nanowires agglomerated due to the high surface energy of the nanowires. They were successful in creating nanowires of 30 μm length and 60 nm diameters. Finally, they noted that the XRD patterns of the silver nanowires produced confirmed the face-centered cubic crystallinity of silver. Y. Gao, *et. al.* [2] used a procedure similar to Sun [6] in that they used a reflux technique to reduce silver nitrate with ethylene glycol in the presence of PVP. Through SEM analysis, it was determined that the silver had a fivefold twinned structure with pentagonal faces. In addition, they reported that silver nanowires preferentially aligned along a silicon substrate when in an EG atmosphere. The strong interaction between PVP and Ag along with the nonvolatility and high viscosity of EG led to this unique ability of the silver nanowires to self-assemble, a characteristic that could be key in the development of commercial applications involving silver.

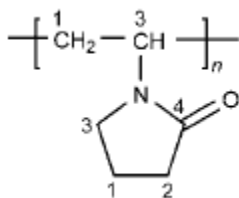
In 2004 J. Hu, *et. al.* [4] discussed the disadvantages of hard template synthesis and espoused a soft-template solution-phase approach involving surfactants that could form protective micelles in order to confine growth. These surfactants, they supposed, could obviate the need for silver seeding as long as a critical surfactant concentration could be met. They employed tri-sodium citrate in the presence of a capping agent, sodium dodecylsulfonate (SDSN), to reduce silver nitrate, noting that controllable diameters and high aspect ratios depended on the reducing agent concentration. Again, the SDSN concentration was deemed to be critical in that concentrations that were too low led to aggregation, while concentrations that were too high resulted in the formation of primarily nanoparticles. SDSN, though, plays only an assistant role in synthesis as its absence does not inhibit the growth of silver nanowires altogether, but its adsorption and desorption from the growth faces is key to good crystallinity. Ultimately, they concluded that the selection of an appropriate reductant and appropriate surfactant were significant in the resultant morphology. P. Jiang, *et. al.* [1] discouraged the use of physical templates for the reason that they limited the quantity and therefore the yield of silver nanowires produced. They encouraged a poly-ol reduction of silver with ethylene glycol in the presence of PVP resulting in nanowires with length from 50-100 μm and diameter in the range of 150-200 nm. They confirmed their previous results of the ability of silver to self-assemble while also noting that it had the peculiar ability to bend back on itself with angles of 60°, 90°, and 120° leading to outstanding mechanical stability.

In 2005 Z. Wang, *et. al.* [3] presented a “clean” approach to synthesis of silver nanowires without the use of organic solvents such as ethylene glycol. Interestingly, they implemented glucose to reduce the silver in temperatures which they noted had to be greater than 140°C. Additionally, increases in glucose concentration led to direct

increases in yield of the final product. Additionally, they proposed that it was advantageous to use only slightly soluble silver salts such as AgBr, AgCl, and AgOH rather than AgNO₃ since a delayed reaction would support anisotropic rather than isotropic growth.

In the Balkus lab at UT Dallas, Fern Edwards and Chunrong Xiong have attempted to synthesize silver nanowire with some success. Their methods have involved the use of P123, a surfactant from BASF Corporation, to reduce Ag⁺ to Ag and ultimately lead to wire morphology in an autoclave at 135 °C. Thus far they have been able to produce nanowire and nanofibers; however, they hit a stumbling block when it came to reproducibility and finding the proper technique.

Polyvinylpyrrolidone (PVP), (C₆H₉NO)_n, according to the Sigma-Aldrich database is a component of Denhardt's solution. Throughout the literature, it is employed as a surfactant assisting in the formation of silver nanomaterial. As a surfactant, it has the ability to form micelles whose size is dictated by concentration and confines and directs nanowire growth. PVP has a polyvinyl skeleton with polar groups. The lone pairs of electrons from the nitrogen and oxygen atoms can be used to reduce Ag⁺ and form intra- and interchain interactions between the PVP and Ag [1]. The PVP implemented in this paper has a molecular weight of 40,000, while the monomer or subunit of the polymer has a formula weight of 111.



Scheme 1. Structure of the PVP monomer. [1]

Pluronic 123 (P123) is also a surfactant capable of forming micelles to aid in the growth of silver nanowires. In this paper, P123 acted as both a surfactant/capping agent as well as a reductant by the method described in Hu's publication [4]. According to the material safety data sheet (MSDS) provided by BASF, it is a difunctional block copolymer surfactant terminating in primary hydroxyl groups that are nonionic and 100% active. It has a formula weight of 5750 and is relatively soluble in aqueous solutions which was necessary in the experimentation conducted.

Two methods were implemented to create silver nanomaterial – reflux and autoclave. Reflux involves the heating of reactants in a round bottom flask attached to a Liebig condenser which allows for the cooling of solvent vapors so that reactant volume and concentration remain nearly constant. An autoclave enables the scientist to heat a solution well above a solvent's boiling point since the high pressures found within the vessel lower the vapor pressure enough to prevent successful vaporization of a solution.

My main research goal involved devising a method that would ameliorate the difficulties faced by previous researchers at UT Dallas in reproducibility of experiments. The

approach involved singling out unique variables – ratio of P123 to AgNO_3 , temperature, volume of deionized water, presence of a silver seed, solvent used – and subjecting a general procedure to controlled experimentation. Additionally, devising a method involving reduction of silver ions at lower temperatures was seen as an objective. Commercially this would be more net beneficial as higher temperatures require ovens to use greater amounts of energy to produce relatively low yields of nanowires and this would also be a novel approach in contrast with the much higher temperatures thought necessary by mainstream publications. Finally, the utilization of the reflux technique would enable the researcher to view the progress of silver reduction, would require only one atmosphere of pressure in contrast with the autoclave, and had thus far been investigated by only a handful of scientists working with silver nanomaterial.

Procedure

Chemicals.

Pluronic P 123 surfactant ($\text{EO}_{20}\text{PO}_{70}\text{EO}_{20}$) was received as a gift from BASF corporation (Mt. Olive, NJ). Silver nitrate (99.9999%), AgNO_3 , was purchased from Sigma-Aldrich (Milwaukee, WI). Dodecane (anhydrous $\geq 99\%$), $\text{CH}_3(\text{CH}_2)_{10}\text{CH}_3$, was purchased from Sigma-Aldrich (Milwaukee, WI). Cyclohexane ($\text{CH}_2(\text{CH}_2)_4\text{CH}_2$) was purchased from EMD Chemicals Inc. (formerly EM Science), an affiliate of Merck KGaA (Darmstadt, Germany). Polyvinylpyrrolidone (PVP 40-T, MW = 40,000) was purchased from Sigma Inc. (St. Louis, MO). Acetone was purchased from EMD Chemicals Inc., an affiliate of Merck KGaA (Darmstadt, Germany). Ethylene glycol (MW = 62.07), $\text{HOCH}_2\text{CH}_2\text{OH}$, was purchased from Fisher Scientific (Fair Lawn, NJ).

Solution Preparation for Autoclave.

First, obtain a 50 mL glass beaker, place it on a gravimetric balance, and tare (zero) the balance. Using two Fisher 5 $\frac{3}{4}$ inch disposable pasteur glass pipets, extract x grams of Pluronic 123 (P123) surfactant and carefully place the allotted amount on the bottom of the 50 mL beaker. On wax weigh paper, measure out x grams of silver nitrate (AgNO_3) and place it into a 20 mL glass scintillation vial. Then, measure out x mL of deionized (DI) water and pour the DI water into the vial. The vial should then be closed and gently shaken in order to dissolve the salt. Using a glass pipet and a pipet bulb, transfer the silver nitrate solution to the 50 mL beaker. Place a 0.75” magnetic stir rod into the beaker and place the beaker on a hot plate stirrer for approximately 20 minutes or until the P123 gel has dissolved in the solution. The solution should become translucent and pale yellow once the P123 enters solution. Under a vent hood, add x mL of cyclohexane via a 10 mL graduated cylinder and continue to stir on low for an additional 10 minutes as the cyclohexane and the P123/ AgNO_3 solution will initially be immiscible. Once the beaker is removed from the stirrer, immediately pour the reactant solution into a clean Teflon-coated autoclave container but take care not to overflow the container. Cover the container with its proper lid and place it into a metal autoclave. Add corrosion and rupture disks before screwing on the final top of the autoclave. Tighten the autoclave using a vise and a lever handle. Place the autoclave into the 135 °C oven using metallic tongs. Ensure the oven is at the correct temperature by checking the attached thermocouple and by adjusting the sensitive knob on the oven.

Solution Preparation for Reflux.

First, obtain a 100 mL 3- or 4-necked round bottom (RB) flask as well as a Celsius thermometer. Place the RB flask onto a cork platform and place the flask on a gravimetric balance and zero the balance. Using two glass pipets, place x grams of P123 onto the bottom of the flask being careful not to allow any P123 to stick to the upper sides of the flask. On wax weigh paper, measure out x grams of silver nitrate (AgNO_3) and place the silver nitrate crystals into the RB flask with the P123. Using a 10 mL graduated cylinder, pour x mL of DI water into the flask. Place a $\frac{3}{4}$ inch stir rod into the flask and stir on a Fisher Scientific stirring hot plate on level 6 for 15-20 minutes. The solution should start to turn murky and pale yellow as it did in the autoclave procedure. Using a BD 20G 1.5" needle and a Norm Ject 10 mL syringe, extract x mL of dodecane and inject into the round bottom flask. The solution should turn milky white. Secure the round bottom flask to the condenser using plastic clamps. Place the Celsius thermometer into the solution and hold it there using a screw-type glass adapter or a rubber stopper. Place a $\frac{3}{4}$ inch magnetic stir rod into the flask if the procedure calls for it. Close up the remaining necks of the flask using glass stoppers. Lower the flask into the oil bath until the solution is fully immersed. Turn on the heat to level 3 and monitor the temperature using the thermometer. If using the sand bath, remove some sand and lower the flask into the apparatus and finally fill the apparatus until the silver solution inside the flask is completely covered. Use a variable autotransformer to monitor and regulate the temperature setting on the sand bath. Turn on the stirrer to 3 or 4 as needed but make sure the oil does not drip out of its container.

Method Proposed by P. Jiang, et. al. [1].

In a 100 mL round bottom flask, add 10 mL of ethylene glycol (EG) using a 10-mL graduated cylinder. Begin heating the flask to 160°C without stirring. On wax weigh paper, measure out x grams of silver nitrate and place the crystals into a 20 mL glass beaker. Measure out 10 mL of EG and pour it into the 20 mL beaker. Stir on a stirring hot plate for 5 minutes with a $\frac{1}{2}$ inch stir bar until all of the AgNO_3 is dissolved. Using a metal spatula, weigh out x grams of PVP on weigh paper. Pour 10 mL of EG into another 20 mL glass beaker. Begin stirring with a $\frac{1}{2}$ inch stir bar and slowly add small amounts of PVP until all of the powder dissolves. Cover both beakers with parafilm. After two hours of heating, begin adding 5 mL both PVP and silver nitrate solutions drop wise using 2, 5mL Norm Ject syringes and BD 20G 1.5" needles at a rate of 0.2 mL/minute (over about 25 minutes). Continue heating for another hour at 160°C . After turning off the reflux, allow it to cool for a few hours. Pipet the solution (20 mL total volume) into a 50 mL conical test tube and refrigerate at $0\text{-}10^\circ\text{C}$ overnight to allow the precipitate to settle.

Processing the Autoclave/Reflux Material.

Autoclave –

After the sample is finished heating in either the autoclave, remove the autoclave using metal tongs so as to avoid burning oneself. Allow the autoclave container to cool overnight to room temperature ($\sim 24^\circ\text{C}$). The following morning, tighten the autoclave within the vise and use the lever handle to unscrew the top to the container. Once

sufficiently loosened, begin to unscrew by hand making sure to keep the autoclave facing away from one's body as the pressure inside may cause the autoclave to pop open. Remove the Teflon-coated container from the autoclave. Using a glass pipet, siphon the clear organic solvent from the top of the precipitate, which should have settled to the bottom. Using the same pipet, transfer the precipitate and any residual liquid to a Nunc 15 mL polypropylene conical test tube. Rinse the autoclave using acetone from a wash bottle in order to ensure that all the precipitate makes it into the test tube.

Reflux –

Allow the round bottom flask to cool down to room temperature. This should take 3-4 hours or allow it to sit overnight. Remove the round bottom flask from any clamps and place it on a cork bottom. Carefully pour any organic solvent (from the top liquid portion) into a 50 mL glass beaker. Use a glass pipet to remove as much solvent from the precipitate without losing any silver material (dark grey). Using a glass pipet and pipet bulb, extract the precipitate and transfer it to a Falcon 50 mL polypropylene conical test tube. Wash the sides and bottom of the flask with acetone and stir for about 30 seconds to try to loosen any precipitate that may be sticking to the walls of the flask. Transfer this solution to the test tube as well.

Place the test tube into the Damon/IEC 15 mL (autoclave) or 50 mL (reflux) clinical centrifuge along with a test tube of equal volume as a counterbalance. Spin the test tubes at level 5 for approximately 30 minutes. After 30 minutes, remove the supernatant from the test tube with the silver material using a glass pipet. If the supernatant is clear, then discard it in an organic waste bottle. However, if the supernatant is yellow or remains turbid, save the yellow supernatant for later centrifugation. Rinse the pellet in the test tube with 5 mL of acetone and shake the test tube to release the pellet from the bottom. Again, centrifuge the test tube at level 5 for 20 minutes. Repeat the procedure again – remove the supernatant, wash the pellet and centrifuge again at level 4 for 15 minutes. After this third centrifugation, remove any acetone and discard, rewash with a few milliliters of new acetone, shake, and pipet out into a 20 mL glass scintillation vial. Place the glass vial into a 90°C oven for a few hours or until the acetone evaporates leaving only a dry precipitate on the bottom or sides of the vial.

Preparation for X-Ray Diffraction (XRD).

Remove the glass scintillation vial from the 90°C oven. Using a metal spatula, scrape off what should be a grey precipitate from the bottom of the vial. Using the same metal spatula, place a small amount of the powdered precipitate onto a VWR 1 ounce micro cover glass. Place a 1 or 2 drops of XRD glue (glue and acetone) onto the powder and allow 2 minutes for it to dry.

Instrumentation.

All samples were analyzed using X-ray Powder Diffraction (XRD) in order to determine the chemical composition and degree of crystallinity of each precipitate. X-ray Pattern Diffractograms were obtained by using a Rigaku (Woodlands, TX) Ultima III Diffractometer with CuK_α radiation. Images were recovered using a FEI Nova 200 (Hillsboro, Oregon) Focused Ion Beam (FIB) microscope. A Fisher-Scientific (Hampton,

NH) A-160 gravimetric balance was used to measure the mass of all reactants. Finally, 15 and 50 mL Damon/IEC (Needham, MA) Clinical Centrifuges were used to prepare samples for analysis with the X-ray diffractometer. Scanning electron microscopy (SEM) was then used to determine the physical morphology of the nanomaterial made. SEM images were obtained using a Leo 1530VP Field-Emission microscope.

Results and Conclusions

After analysis using the Rigaku X-Ray Diffractometer, nearly all of the brownish grey precipitates obtained were indexed to silver. The intensity of the primary peak usually at 38° and the width of that peak can be used in the Scherrer formula to calculate the degree of crystallinity of each of the samples with the general trend being that a thinner width of the primary peak entails a greater degree of crystallinity. Figures 1-6 are X-ray diffractograms of the samples analyzed.

Figure 1 represents the results from the first attempt at silver nanowire synthesis using the reflux at one atmospheric pressure at 100°C . Of the 6 peaks that appeared on the X-ray diffractogram, four appear in the powder diffraction file (PDF) entry for silver 01-089-3722. After completing two autoclaves and two refluxes, the degree of crystallinity for this first reflux appeared to be most promising and hence this recipe was repeated in refluxes 3R (Figure 3) and 4R.

Figure 2 shows Autoclave #3 which was a replication of Xiong's Procedure A with a modification in the volume of DI water. According to the publication by Sun [6] and Hu [4], the concentration of the reductant or surfactant is critical to the ultimate morphology of the silver nanomaterial. Thus, rather than modifying the individual concentration of P123, the final molarity of each reactant was halved in order to determine the effect that a concentration change would have. The results appear to be promising in that the X-ray diffractogram showed the greatest crystalline size of 60 nm.

Figure 3 shows the result of a reflux using the procedure uncovered from the first reflux. The key difference however was the lack of stirring of the solution which allowed a slow emulsion of silver in the presence of P123 without agitation. This could ultimately lead to thicker and longer nanowires due to the lack of disruption of the inter- and intrachain interactions described by P. Jiang, et. al. [1].

Figure 4 represents the result of synthesis of silver nanomaterial using Xiong's Procedure A with additional modifications in the volume of DI water and thus the concentrations of reactants. As described above, changes in the concentrations of reactants can easily lead to alterations in the morphology of the nanomaterial, and thus finding a critical ratio and concentration for the initial reactants is key to determining a reproducible method for the synthesis of silver nanorods that are less susceptible to capricious technical errors. In addition, an S70 seed that Chunrong Xiong had produced in the past year was used to act as a physical template and an initiator of the reaction between P123 and silver nanowires. It was thought that the seed could induce a certain morphology for the silver nanoparticles and hence lead to aggregation in the form of nanowires rather than

nanospheres. The crystalline size of 51 nm which is comparable to the crystalline sizes resulting from similar procedures (as seen in Table 1) reinforces the initial hypothesis that concentration of reactants plays a critical part in the product yielded.

Figure 5 demonstrates an attempt to reproduce the results of a method described by P. Jiang, et. al. [1]. The reason for such an attempt stemmed from the need to check the reproducibility of accepted methodologies. Reproducibility was the cornerstone of the research that was conducted and thus it was to be necessary to see if it was possible to reproduce results from mainstream literature and use the lessons learned from these attempts as a basis for modifying existing attempts at UT Dallas. Graph 1 summarizes the results from three refluxes using three different concentrations of PVP – 0.02 M, 0.2 M, and 2.0 M. The crystalline size from all three experiments were comparable, however, the intensity of the peaks resulting from X-ray diffractogram analysis showed that the optimal PVP concentration would be slightly less than 0.2 M, the concentration espoused by P. Jiang [1]. One could suppose as well that the sample resulting from the 0.2 M PVP would have the highest aspect ratio (long wires, small diameters).

Figure 6 represents the X-ray diffraction pattern from the second autoclave effort. Though there appear to several anomalous peaks indexed to insoluble silver chloride (AgCl), the most intense peaks results from silver 01-089-3722. A limited number of initial samples were analyzed using a Focused Ion Beam (FIB) microscope (as seen in Figure 7), which showed that the ratio modification of Fern's Procedure 83 B in an autoclave resulted in possible nanorods similar to those that Fern synthesized in Figure 11B. This is promising in that replication of at least one trial seems to have been achieved.

Images in figures 8 through 11 demonstrate the final goals of this project. Based on these SEM images, it was determined that these procedures would be the basis of the experiments conducted. The hope is that the samples that were created during this five week period will show positive results similar to the SEM images provided in the appendix. Due to the repairs that needed to be made to the scanning electron microscope (Leo 1530 VP), the morphology of the nearly 15 samples synthesized could not be verified, meaning that only educated assumptions, rather than valid conclusions, can be made. However, many of the results show potential regarding the production of silver nanowires with high aspect ratios. The synthesis of nanomaterial using the reflux technique at 100°C was successful in producing clean X-ray diffractograms and in fact resulted in several silver crystals of sizes greater than 40 nanometers. Due to time limitations, several variables remain when it comes to reproducibility. Ideally, once SEM images are recovered, further experimentation should be conducted using those procedures whose results were most promising in an effort to design a technique that can be replicated every time.

As discussed in the introduction, the singular characteristics of silver – its conductivity and its mechanical stability – mean that any breakthrough in the field of nanotechnology could enhance the ability of cutting edge technology and research to move forward. T. Yajima, et. al. [8] described the potential significance of mass replication of silver

nanowires in the medical industry with relation to the reduction of electrical interference in patients with pacemakers. But first and most importantly, problems with reproducibility must be resolved through continued systematic elimination of variables both in technique and types and amounts of reactants used.

Works Cited

1. P. Jiang, S. Li, S. Xie, Y. Gao, L. Song, *Chemistry – A European Journal* 10 (2004) 4817-4821.
2. Y. Gao, P. Jiang, D.F. Liu, H.J. Yuan, X.Q. Yan, Z.P. Zhou, J.X. Wang, L. Song, L.F. Liu, W.Y. Zhou, G. Wang, C.Y. Wang, S.S. Xie, *Chemical Physics Letters* 380 (2003) 146-149.
3. Z. Wang, J. Liu, X. Chen, J. Wan, Y. Qian, *Chemistry – A European Journal* 11 (2005) 160-163.
4. J. Hu, Q. Chen, Z. Xie, G. Han, R. Wang, B. Ren, Y. Zhang, Z. Yang, Z. Tian, *Advanced Functional Materials* 14 (2004) 183-189.
5. Z. Hu, T. Xu, R. Liu, H. Li, *Materials Science & Engineering A* 371 (2004) 236-240.
6. Y. Sun, Y. Yin, B. Mayers, T. Herricks, Y. Xia, *Chemistry of Materials* 14 (2002) 4736-4745.
7. S. Liu, J. Yue, A. Gedanken, *Advanced Materials* 13 (2001) 656-658.
8. T. Yajima, K. Yamada, S. Tanaka, *The Japanese Society for Artificial Organs* 5 (2002) 175-178.

Acknowledgments

First and foremost, I would like to thank the Robert A. Welch Foundation for providing the funding for such a worthwhile and enjoyable experience in a lab and at a college campus such as UT Dallas. Additionally, I would like to thank Dr. Paul Pantano for conducting such an organized program from the transportation to the apartments to the daily checkups to make sure that we were all doing fine. I must also thank Dr. D.J. Yang, Fern Edwards, and Chunrong Xiong. Dr. Yang provided me with an intriguing project that stimulated me and challenged me to think outside the box. He always reminded me to think for myself and to never lose hope even when I was confronted with setbacks. Fern and Xiong guided me throughout the process whether it was explaining how to use various pieces of equipment in the lab or laying the foundation for my work or simply answering my everyday questions. They allowed me to work rather independently, compelled me to dictate the direction of my research, and pushed me to reach my goals. Everyone else in the lab – Harvey, José, Nadia, Rita, and especially Minedys – provided me with a nurturing atmosphere, advised me when Xiong and Fern were not in the lab,

and allowed me to borrow their glassware. I would like to express my gratitude to Erling Beck for accompanying us on the weekends. This program would not have been nearly as fun had it not been for the other Welch Scholars – Andrew, Amy, Jenny, Robert, and Janice. Their quirks, jokes, and company made this experience truly once in a lifetime. I will always cherish the times and memories that we all had together. I would truly have not been prepared for this program had it not been for Mr. Joe Wilkins. I thank him for nominating me to participate in the Welch Summer Scholars Program. Last, but certainly not least, I have to thank my parents and my sister for putting up with me throughout the years and for not only endowing their wisdom upon me, but also for opening my eyes to a vast number of opportunities.

Optimization of Conditions for the Synthesis of Silver Nanowire

Table 1. Experiments conducted during the Welch Program.

Date	Sample Designation	Temp. (°C)	P123 (FW = 5750)	AgNO ₃ (FW = 169.9)	H ₂ O	organic solvent	Time	XRD Results (crystallite size)
14-Jun	Fern's Procedure 78 in autoclave	135	0.1995 g 3.46957×10^{-5} mol 1.573 mM	0.4017 g 0.002364 mol 0.1075 M	2 mL	20 mL cyclohexane	6/14 - 6/19 5 days	n/a
15-Jun	Xiong's Procedure A in autoclave	135	0.3498 g 6.08348×10^{-5} mol 2.821 mM	0.3003 g 0.001768 mol 0.08204 M	1.55 mL	20 mL cyclohexane	6/15 - 6/19 4 days	27 nm
#1A								
20-Jun	Xiong's Procedure A in reflux	~100	0.4052 g 7.0470×10^{-5} mol 1.6 mM	0.7013 g 0.004128 mol 0.09382 M	4 mL	40 mL dodecane	6/20 - 6/21 1 day	28 nm
#1R								
20-Jun	Fern's Procedure 83B in autoclave ratio modification	135	0.4037 g 7.0209×10^{-5} mol 3.191 mM	0.6012 g 0.003539 mol 0.1609 M	2 mL	20 mL cyclohexane	6/20 - 6/24 4 days	21 nm
#2A								
22-Jun	Xiong's Procedure A in reflux mod. H2O (reactant concentration)	~100	0.7000 g 1.2174×10^{-4} mol 2.535 mM	0.6016 g 0.003541 mol 0.07377 M	8 mL	40 mL dodecane	6/22 - 6/24 2 days	25 nm
#2R								
23-Jun	Xiong's Procedure A in autoclave mod. H2O (reactant concentration)	135	0.3522 g 6.1252×10^{-5} mol 2.545 mM	0.2986 g 0.001758 mol 0.07310 M	4.05 mL	20 mL cyclohexane	6/23 - 6/27 4 days	60 nm
#3A								
29-Jun	Reflux of Silver w/o stirring	~100	0.4018 g 6.9878×10^{-5} mol 1.5881 mM	0.6995 g 0.004117 mol 0.09357 M	4 mL	40 mL dodecane	6/29 - 6/30 1 day	52 nm
#3R								
29-Jun	Silver Reflux w/ Xiong's S70 seed	~100	0.4011 g 6.9757×10^{-5} mol 1.5854 mM	0.7027 g 0.004136 mol 0.0940 M	4 mL	40 mL dodecane	6/29 - 7/3 4 days	38 nm
#4R								41 nm
3-Jul	Replication of Reflux from publication (0.2 M PVP) subunit FW = 111, overall = 40,000	~160	0.2216 g PVP 0.001996 mol 0.20 M	0.1700 g 0.0010 mol 0.100 M	n/a	20 mL ethylene glyco	7/3 - 7/3 3.5 hours	
#5R								
3-Jul	Autoclave using H ₂ O modified S.A. with S70 seed	135	0.3515 g 6.113×10^{-5} mol 2.547 mM	0.3017 g 0.00178 mol 0.07399 M	4 mL	20 mL cyclohexane	7/3 - 7/7 4 days	51 nm
#4A								
5-Jul	Fern's Procedure 83B in reflux ratio modification	120	0.5983 g 1.04×10^{-4} mol 3.152 mM	0.9009 g 0.005303 mol 0.1607 M	3 mL	30 mL dodecane	7/5 - 7/7 2 days	35 nm
#6R								
6-Jul	Fern's Procedure 83B in A/C ratio modification	135	0.4027 g 7.0035×10^{-5} mol 3.1834 mM	0.6022 g 0.003544 mol 0.1611 M	2 mL	20 mL cyclohexane	7/6 - 7/10 4 days	41 nm
#5A								
6-Jul	Xiong's Procedure A in reflux mod. H2O (reactant concentration)	115	1.3990 g 2.4330×10^{-4} mol 2.829 mM	1.2010 g 0.007069 mol 0.0822 M	16 mL	80 mL dodecane	7/6 - 7/10 4 days	33 nm
#7R								
7-Jul	Replication of Reflux from publication (2.0 M PVP)	160	1.1112 g PVP 0.01001 mol 2.002 M	0.0860 g 5.062 mol 0.1012 M	n/a	20 mL ethylene glyco	7/7 - 7/7 3.5 hours	42 nm
#8R								
7-Jul	Replication of Reflux from publication (0.02 M PVP)	160	0.0227 g PVP 2.0450 mol 0.0205 M	0.1708 g 0.00101 mol 0.101 M	n/a	20 mL ethylene glyco	7/7 - 7/7 3.5 hours	44 nm
#9R								

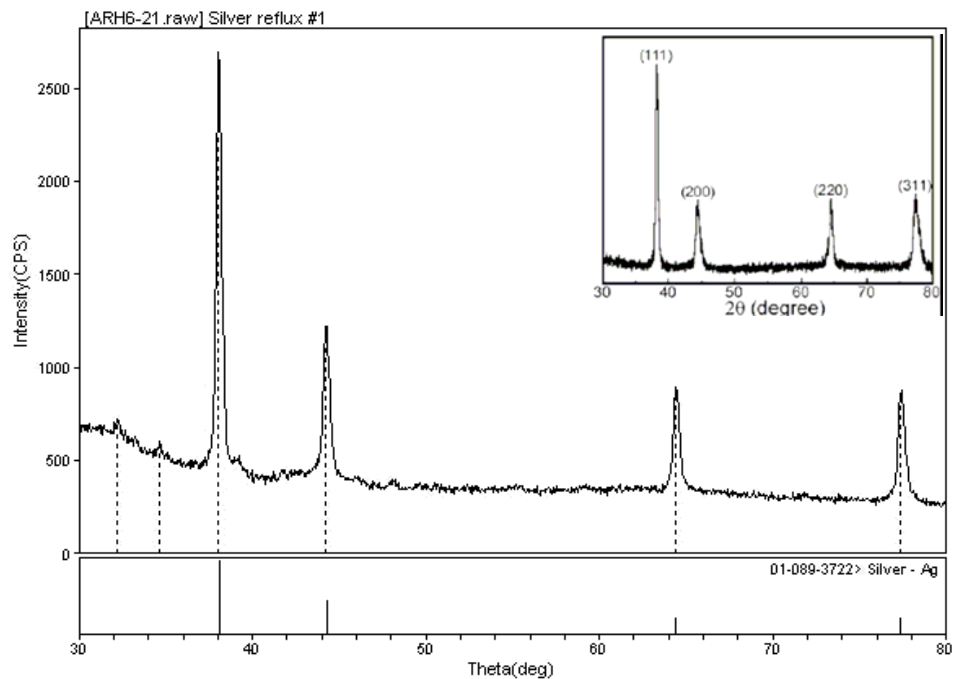


Figure 1. An X-ray diffractogram of the silver nanomaterial synthesized using Xiong's Procedure A with reflux at 100°C. The peaks are indexed to silver (01-089-3722) with the primary peak at 38.054°. The average crystalline size according to Scherrer formula analysis is 28 nm. The inset represents an X-ray diffractogram of silver nanowire from Sun's publication [6].

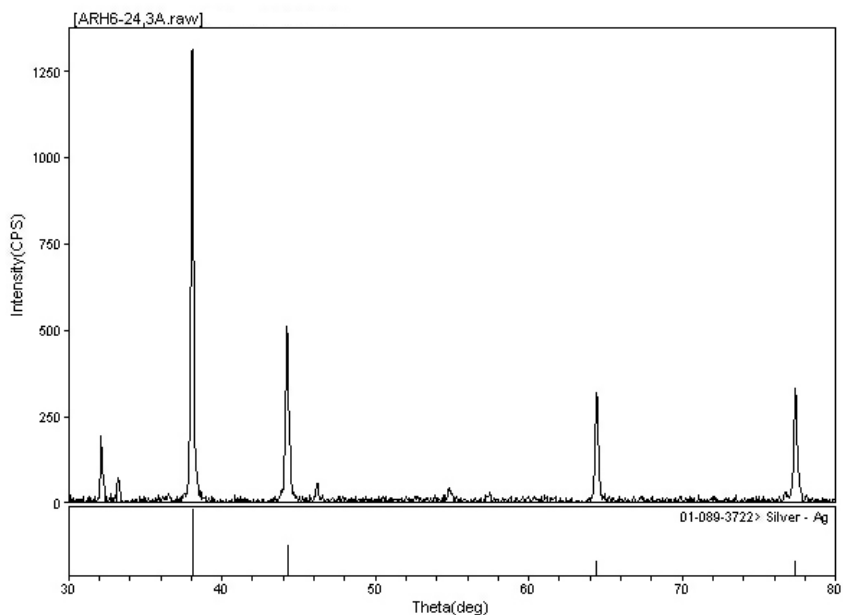


Figure 2. An X-ray diffractogram of the silver nanomaterial synthesized using a modification in the volume of DI water used with Xiong's Procedure A in autoclave at 135°C. The peaks are indexed to silver (01-089-3722) with the primary peak at 38.089°. The average crystalline size according to Scherrer formula analysis is 60 nm.

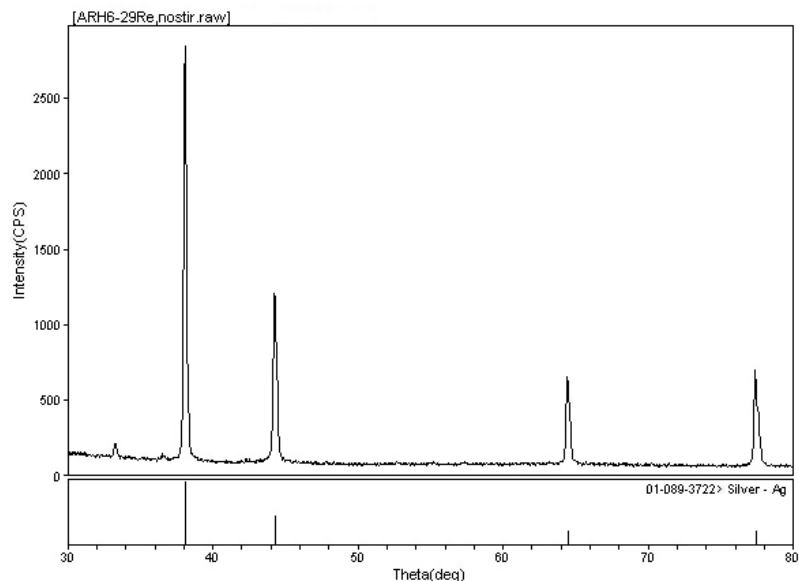


Figure 3. An X-ray diffractogram of a reflux of silver using the same reactant values as in Figure 2, however, the solution was not stirred while heating at 100°C. The peaks are indexed to silver (01-089-3722) with the primary peak at 38.098°. The average crystalline size according to Scherrer formula analysis is 52 nm.

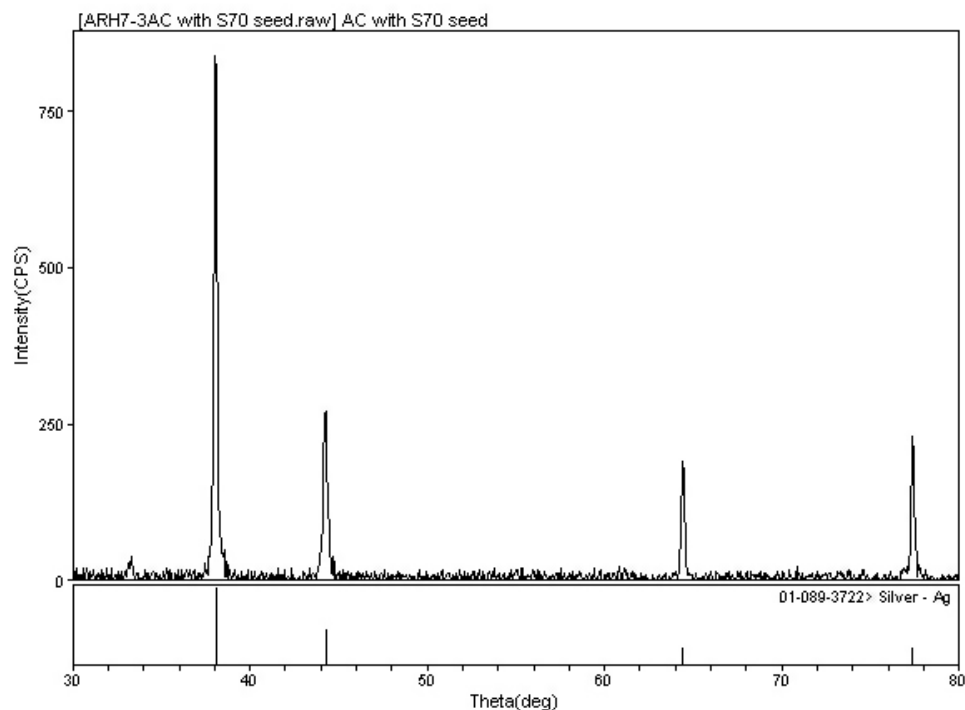


Figure 4. An X-ray diffractogram of autoclaved material prepared following Xiong's Procedure A at 135°C with modification in the volume of DI water along with an S70 seed synthesized by Xiong. The peaks are indexed to silver (01-089-3722) with the primary peak at 38.061°. The average crystalline size according to Scherrer formula analysis is 51 nm.

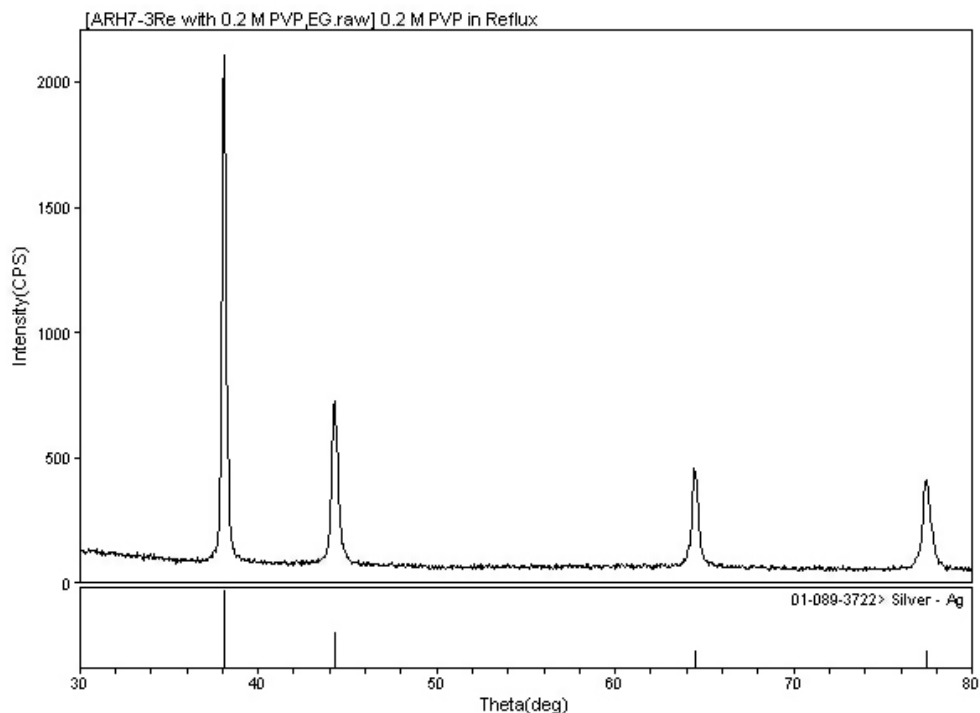
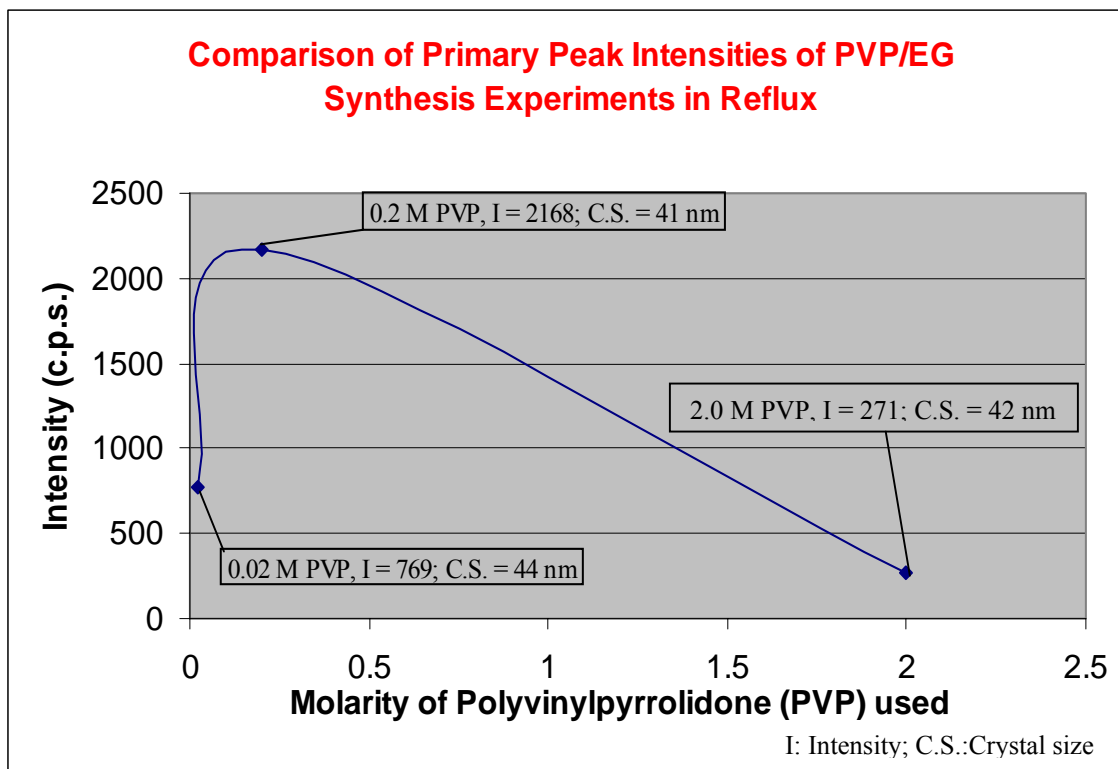


Figure 5. An X-ray diffractogram of refluxed material prepared following the procedure as presented in P. Jiang, et. al.'s publication [1] using PVP and EG at 160 °C. The peaks are indexed to silver (01-089-3722) with the primary peak at 38.101°. The average crystalline size according to Scherrer formula analysis is 41 nm.



Graph 1. Optimization of the molarity of PVP using P. Jiang, et. al.'s procedure. [1]

Optimization of Conditions for the Synthesis of Silver Nanowire

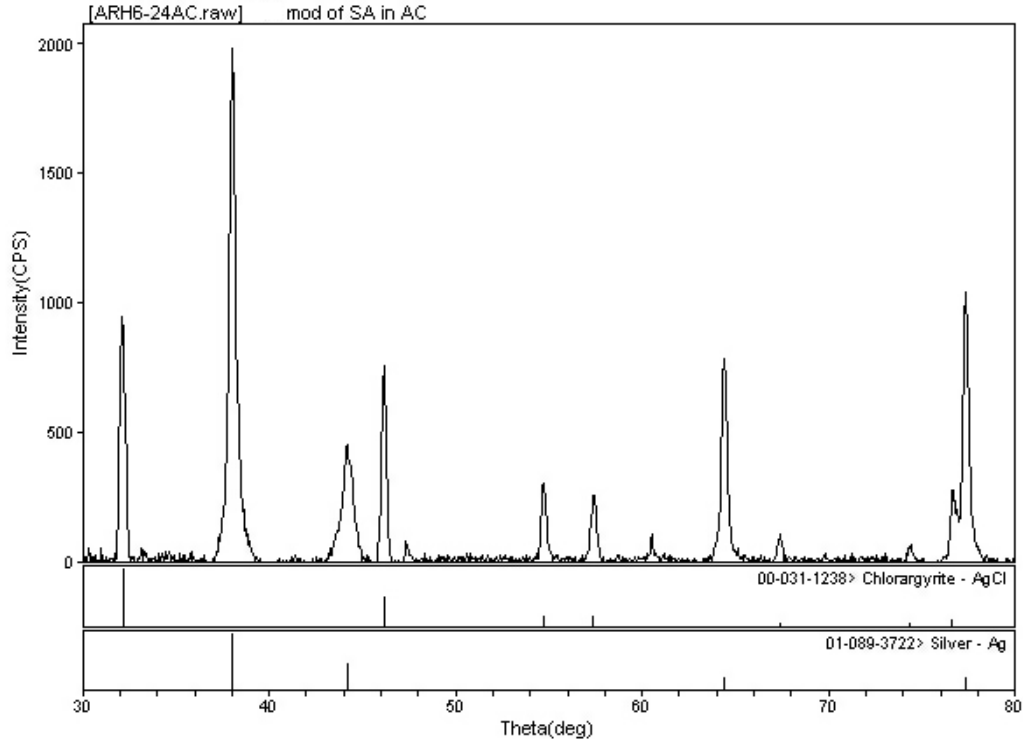


Figure 6. An X-ray diffractogram of silver nanomaterial synthesized using Fern's procedure 83B with a modification in the ratio of silver nitrate to Pluronic 123 surfactant. The primary peaks are indexed to silver (01-089-3722) with the primary peak at 38.053° . anomalous peaks are indexed to AgCl impurities. The average crystalline size according to Scherrer formula analysis is 21 nm.

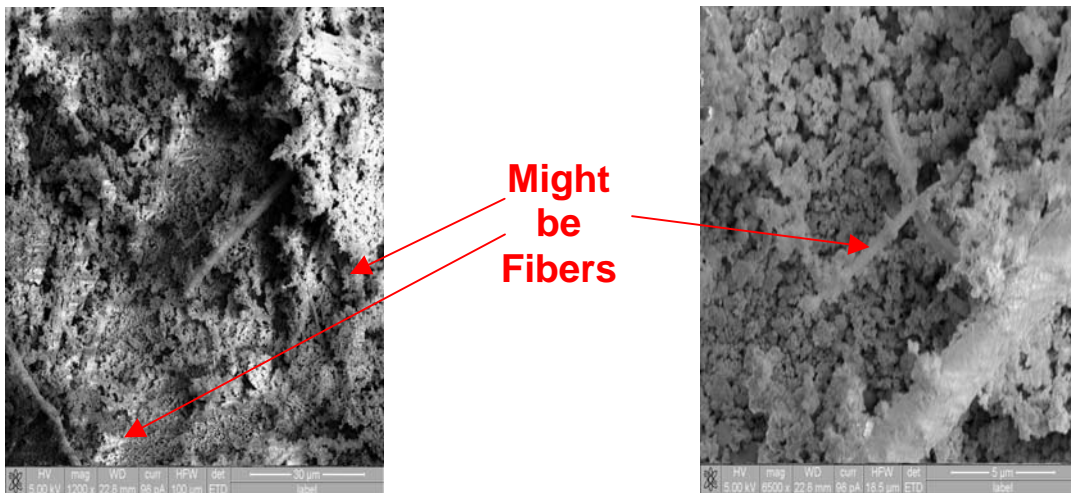


Figure 7. Focused Ion Beam (FIB) images of the procedure described in Figure 6.

Optimization of Conditions for the Synthesis of Silver Nanowire

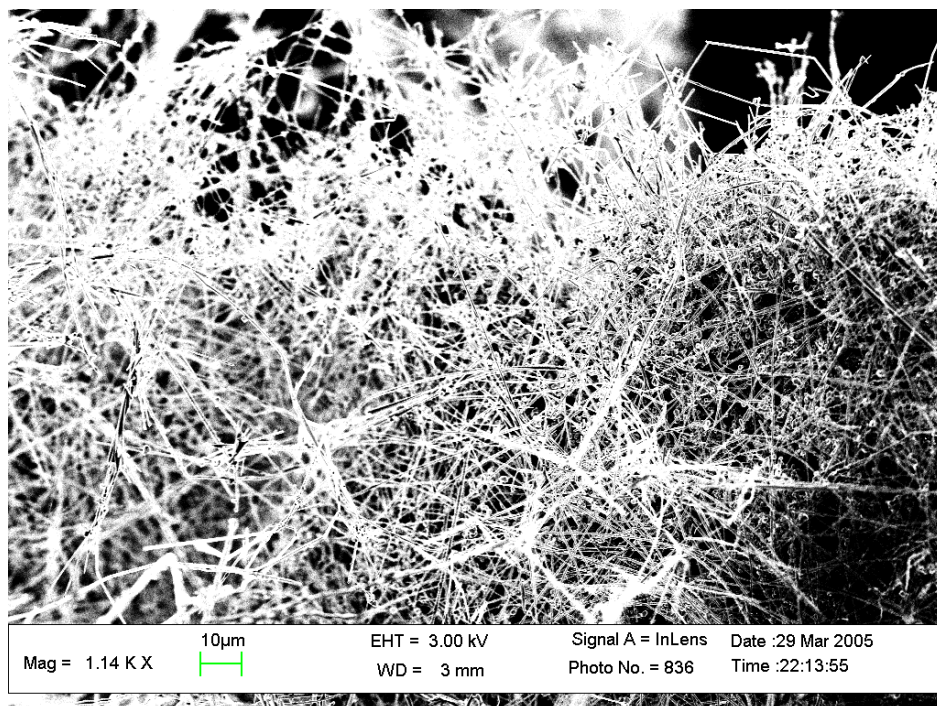
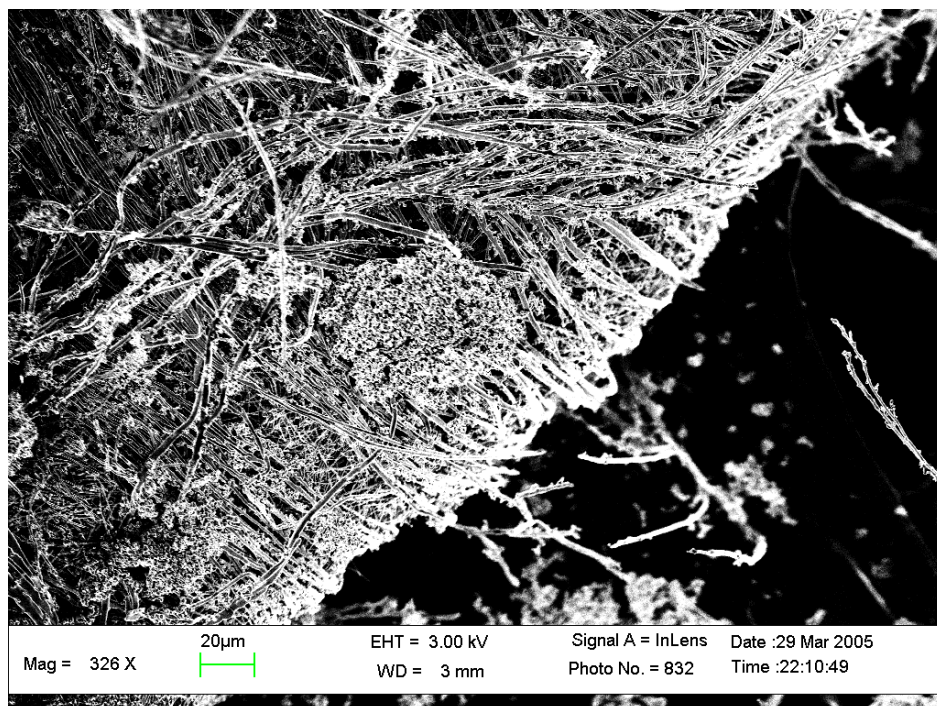


Figure 8A and 8B. Scanning Electron Microscope (SEM) images of silver nanowire synthesized by Chunrong Xiong using an autoclave at 135°C. (Procedure A)

Optimization of Conditions for the Synthesis of Silver Nanowire

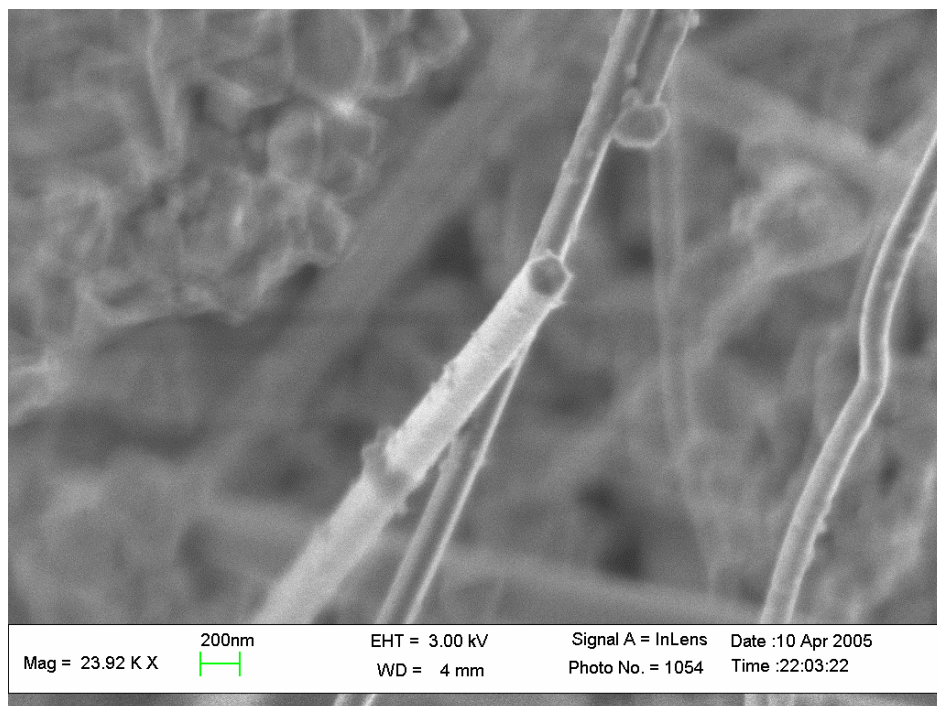
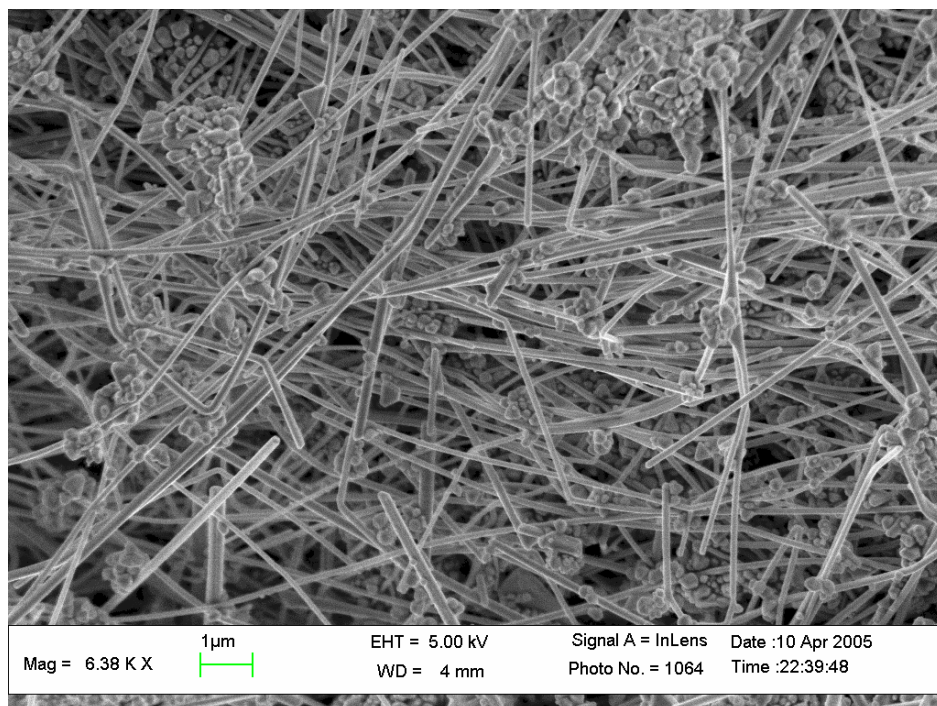


Figure 9A and 9B. SEM images of silver nanowire synthesized by Chunrong Xiong using an autoclave at 135°C. The bottom picture shows the pentagonal cross section of silver. (Procedure C)

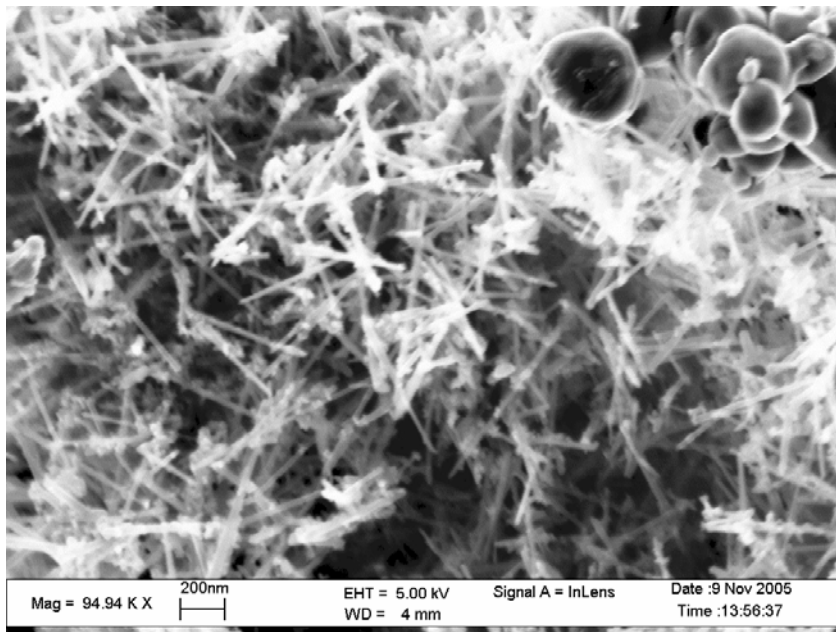


Figure 10. SEM images of silver nanoparticles and nanorods as synthesized by Fern Edwards. Nearly 75% of the product was nanorods with average length of 15 nm. (Procedure 78)

Optimization of Conditions for the Synthesis of Silver Nanowire

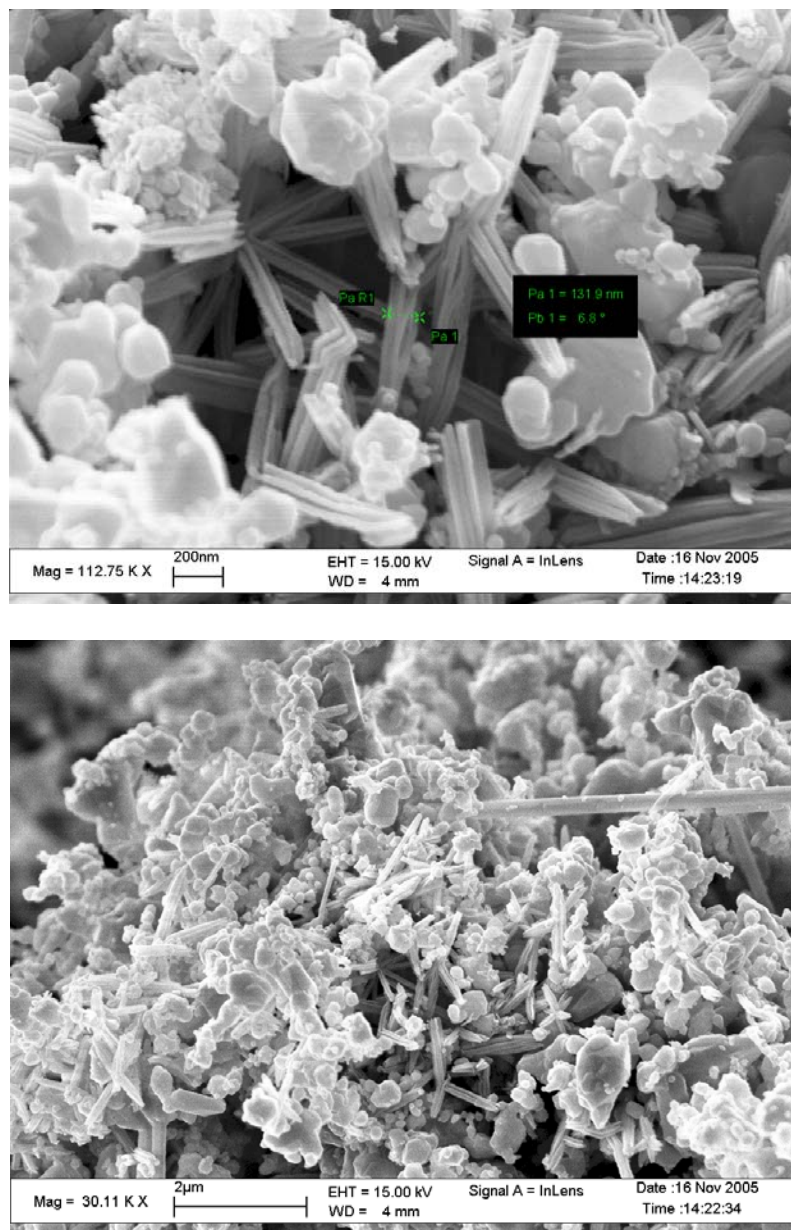


Figure 11A and 11B. SEM images of silver nanowires and nanoparticles synthesized by Fern Edwards. The fused rods are of ~130 nm length. (Procedure 83B)