

## Research Article

# High-Yield Synthesis of Long Silver Nanowires via Chromic Chloride and a Stable Reaction Environment

**Bosheng Zhang** , **Rui Dang**, **Qigao Cao** , **Panchao Zhao** , **Kunkun Chen**,  
and **Hanqi Meng**

*Northwest Institute for Nonferrous Metal Research, Xi'an 710016, China*

Correspondence should be addressed to Bosheng Zhang; 410302479@qq.com and Qigao Cao; caoqigao@c-nin.com

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The search for suitable synthesis methods and parameters capable of controlling the length, diameter, and yield of silver nanowires (AgNWs) is still an emerging strategy today. Therefore, a method for high-yield synthesis of long AgNWs via chromic chloride and a stable reaction environment was proposed. The results show that  $\text{Cr}^{3+}$  could restore the adsorbed atomic oxygen quickly and provide a high efficiency in the prevention of the oxidative etching, for the ion of  $\text{Cr}^{2+}$  oxidized to  $\text{Cr}^{3+}$  has a lower standard electrode potential, and a more stable reaction environment provided by the coupling method could avoid disturbing the growth of the {111} reactive sites of the wires; then, the yield and length of the AgNWs were improved. The length of the AgNWs was over  $75\ \mu\text{m}$  and even  $160\ \mu\text{m}$ ; the yield of the AgNWs was over 90%, which provides the referable basis for the synthesis of ultralong AgNWs.

## 1. Introduction

For the special optical, thermal, catalytic, and electronic properties [1–3], one-dimensional (1D) metallic nanostructures have garnered a significant amount of research attention and found application in various devices [4, 5]. For example, silver nanowires (AgNWs) have excellent electrical, transmittance, and flexible properties; also, the source of raw materials is wide and cheap [6, 7]. AgNWs are now being used as a substitute for indium tin oxide (ITO), which is the dominant transparent conductor material. In addition, AgNWs are now regarded as the next generation of flexible transparent materials and have been used in the fields of solar cells [8, 9], organic light-emitting diodes (OLEDs) [10], flexible liquid crystal displays (LCDs) [11], and even medical textiles.

Many methods have been developed on the synthesis of AgNWs, such as the template method, ultraviolet irradiation technique, soft solution method, hydrothermal method, and polyol process [12–18]; the polyol process has been the most promising method for its simplicity and high repeatability. The polyol process was developed by Sun et al. [19]; afterward,

many research groups have explored different approaches and factors such as reaction times, ratio of chemicals, reaction temperature, additive agents, and stirring speed in order to improve the morphology and yield of AgNWs [20–26]. Bergin et al. [27] claimed that low temperatures of  $130^\circ\text{C}$  and long reaction times of about 12 h can achieve  $25\ \mu\text{m}$  long wires, which contained large diameter and the length up to  $25\ \mu\text{m}$ . Coskun et al. [28] suggested that stirring speed could control the length and diameter, while the AgNW length was not exceeding  $30\ \mu\text{m}$ . Lee et al. [29] prepared the AgNWs in the range of 15–22 nm and lengths up to  $20\ \mu\text{m}$  under the pressure of 200 psi. Tang et al. [30] improved the yield of AgNWs in the presence of  $\text{N}_2$  and at a higher temperature of  $198^\circ\text{C}$ , while the lengths of AgNWs were not exceeding  $30\ \mu\text{m}$  and the yield for calculation contained large, but shorter, AgNWs, which do not exceed  $5\ \mu\text{m}$ . Above all, the length and yield of AgNWs are not perfect. Also, Im et al. [31] prepared a high-performance, flexible, and robust metal nanotrough-embedded transparent conducting hybrid film, which proved that the long metallic nanowires could promote the performance of transparent electrodes. Ji et al. [32] prepared flexible

and transparent cellulose hybrid films with high dielectric performance, which showed that the length of the nanofibers has a direct influence on the performance of films. A similar viewpoint was also reported by Jang [33]. Therefore, the search for suitable synthesis methods and parameters capable of controlling the length, diameter, and yield of AgNWs is still an emerging strategy today.

The key to the formation of uniform silver nanowires was believed to be the use of PVP as a polymer capping reagent and the formation of multiple twin particles; the PVP cap the multiple twin {100} side faces, leading {111} side faces to grow [12]. During the AgNW growth, the adsorbed atomic oxygen could block the {111} reactive sites from adding Ag atoms efficiently; then, oxidative etching would happen on the {111} side, which could lead to a lower yield and shorter AgNWs, so the variable valence metal ions are necessary to reduce the efficiency of oxidative etching by restoring the adsorbed atomic oxygen. Meanwhile, it is reasonable and necessary to avoid disturbing the growth of the {111} reactive sites for the growth of crystals.

Therefore,  $\text{Cr}^{3+}$  was considered a promising metal ion and first used in this paper; the standard electrode potential of  $\text{Cr}^{2+}$  oxidized to  $\text{Cr}^{3+}$  ( $E_{(\text{Cr}^{3+}/\text{Cr}^{2+})}$ ) is  $-0.42\text{ V}$ , more efficient to restore the adsorbed atomic oxygen and prevent the oxidative etching than other metal ions, such as  $\text{Fe}^{3+}/\text{Fe}^{2+}$  [34–36], which would lead to a positive promotion of the growth of the yield and length of AgNWs. On the other hand, a coupling method with the hydrothermal method and polyol process could provide a more stable reaction environment than the traditional polyol process, which would avoid disturbing the growth of the {111} reactive sites and lead to a positive promotion of the growth of the {111} reactive sites; then, the yield and length of AgNWs would be improved. In this paper, the efficiency of preventing oxide etching, the yield and morphology of AgNWs affected by different standard electrode potentials of metal ions, and the effect of reaction environment stability on the yield and morphology of AgNWs were first proposed to be studied using a coupling method together with the hydrothermal method and polyol synthesis.  $\text{Cr}^{3+}$  was considered a promising metal ion and first used in this paper. We think that the thesis discussed in this paper is novelty.

## 2. Experiment

**2.1. Materials.**  $\text{AgNO}_3$  (AR),  $\text{NaCl}$  (AR),  $\text{CrCl}_3$  (AR),  $\text{FeCl}_3$  (AR),  $\text{CoCl}_2$  (AR), and PVP (Mw = 360,000) were all purchased from a Kelong reagent factory of Chengdu, China. Ethylene glycol (AR) was purchased from a Macklin reagent factory of Shanghai, China. Ethanol (AR) was purchased from a Tianjin chemical reagent factory. All the materials were used as received without further purification. Deionized water purified from a Daojing (Beijing, China) ultrapure water system was used throughout the experiments.

**2.2. Methods.** For a typical synthesis, 110 mM  $\text{AgNO}_3$ , 150 mM PVP, and 5 mM  $\text{CrCl}_3$  solutions in EG were prepared. Then, 12 mL of the  $\text{AgNO}_3$  solution, 12 mL of the PVP solution, and 420  $\mu\text{L}$  of the  $\text{CrCl}_3$  solution were mixed

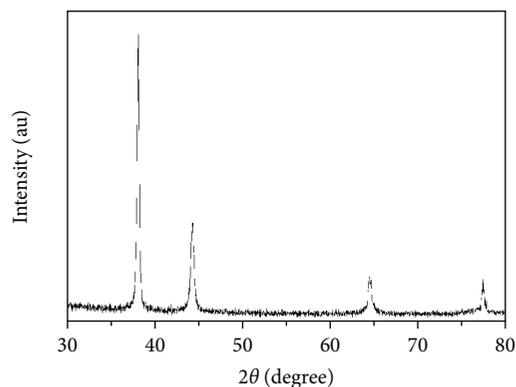


FIGURE 1: XRD pattern of AgNWs synthesized through the coupling method.

by magnetic stirring for 3–5 minutes. The mixture was immediately transferred into the hydrothermal synthesis reactor; then, the reactor was placed into a heated oven at  $160^\circ\text{C}$  for 3 h. After the reaction, acetone and ethanol were used to wash the precipitate with a centrifugation of 3500 rpm for 10 min; the washed AgNWs were redispersed in ethanol for future use. The crystal type of AgNWs was observed through X-ray diffraction (XRD); the morphology of AgNWs was observed through a scanning electron microscope (SEM).

## 3. Results and Discussion

The X-ray diffraction pattern of the AgNW powder samples synthesized through the coupling method by adding  $\text{Cr}^{3+}$  reveals four prominent diffraction peaks in Figure 1, which are from the {111}, {200}, {220}, and {311} faces (the same as JCPDS file 04-0783). The AgNWs were confirmed as a typical face-centered cubic structure.

A series of experiments were carried out to study the morphology of AgNWs affected by kinds of metal ions, which contained  $\text{NaCl}$ ,  $\text{CoCl}_2$ ,  $\text{FeCl}_3$ , and  $\text{CrCl}_3$ . The concentration of metal ions was all  $86\ \mu\text{M}$  in a mixed solution; other parameters are all described in Table 1. SEM images in Figure 2 show the effect of different metal ions on the length of AgNWs. It is clear that the length of AgNWs was affected by kinds of metal ions; the length of AgNWs gradually increases as shown from panel (a) to panel (d). The average length of AgNWs is summarized in Figure 2(e) by counting 250 wires to measure the linear distance between the two ends of AgNWs in SEM using the software ImageJ.

The length of wires with  $\text{NaCl}$  existing was about  $30\ \mu\text{m}$ , which was shorter than the others. It was because oxidative etching occurred on the {111} reactive sites in the reaction process. For  $\text{CoCl}_2$  addition, the standard electrode potential of  $E_{(\text{Co}^{3+}/\text{Co}^{2+})}$  is  $1.808\text{ V}$ , higher than the oxygen standard electrode potential of  $1.23\text{ V}$ , which leads to a small prevention of oxidative etching, so the length of AgNWs was similar to the length of AgNWs with  $\text{NaCl}$  existing.

For  $\text{FeCl}_3$  and  $\text{CrCl}_3$ , the standard electrode potentials of  $E_{(\text{Fe}^{3+}/\text{Fe}^{2+})}$  and  $E_{(\text{Cr}^{3+}/\text{Cr}^{2+})}$  are  $0.771\text{ V}$  and  $-0.42\text{ V}$ , respectively, lower than the oxygen standard electrode potential, which would restrain the oxidative etching, confirming the

TABLE 1: Experimental conditions with different metal ions.

Coupling method	Experimental conditions		
Metal ions	Co <sup>2+</sup>	Fe <sup>3+</sup>	Cr <sup>3+</sup>
Concentration of AgNO <sub>3</sub> :PVP:M <sup>n+</sup> :Cl <sup>-</sup> in a mixed solution	54 mM:73.7 mM:86 μM:258 μM		
Temperature	160°C		
Reaction time	3 h		

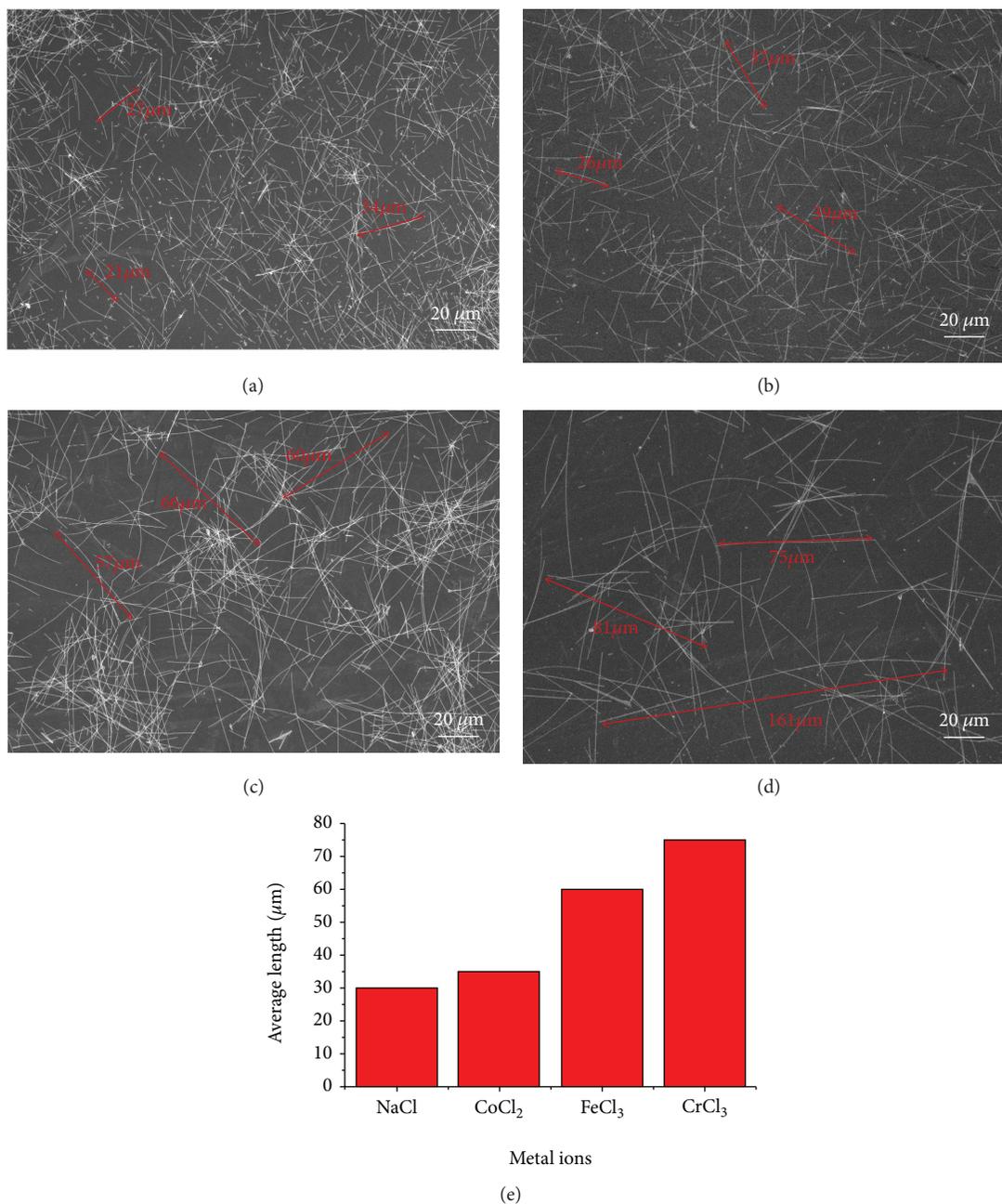


FIGURE 2: The SEM images of AgNWs synthesized with different metal ions (a) NaCl, (b) CoCl<sub>2</sub>, (c) FeCl<sub>3</sub>, and (d) CrCl<sub>3</sub> and (e) the tendency of the average length of AgNWs with different metal ions.

AgNW synthesization to be longer. We could also find that the length of AgNWs with FeCl<sub>3</sub> existing was 60 μm and the length of AgNWs with CrCl<sub>3</sub> existing is 75 μm, which is

the longest; the yield of AgNWs with CrCl<sub>3</sub> existing was over 90%, which is summarized by counting the weight of the raw material of Ag divided by nearly all the dry AgNWs. The

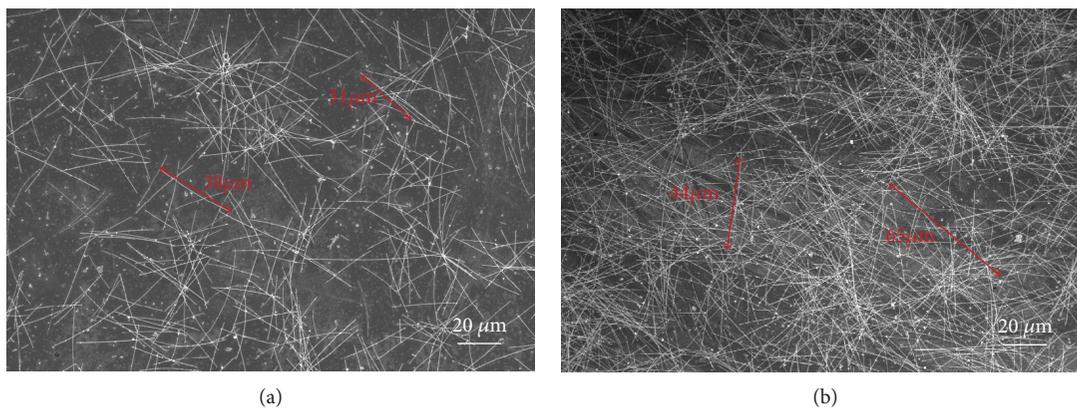


FIGURE 3: The SEM images of AgNWs synthesized with low concentration metal ions: (a) FeCl<sub>3</sub> and (b) CrCl<sub>3</sub>.

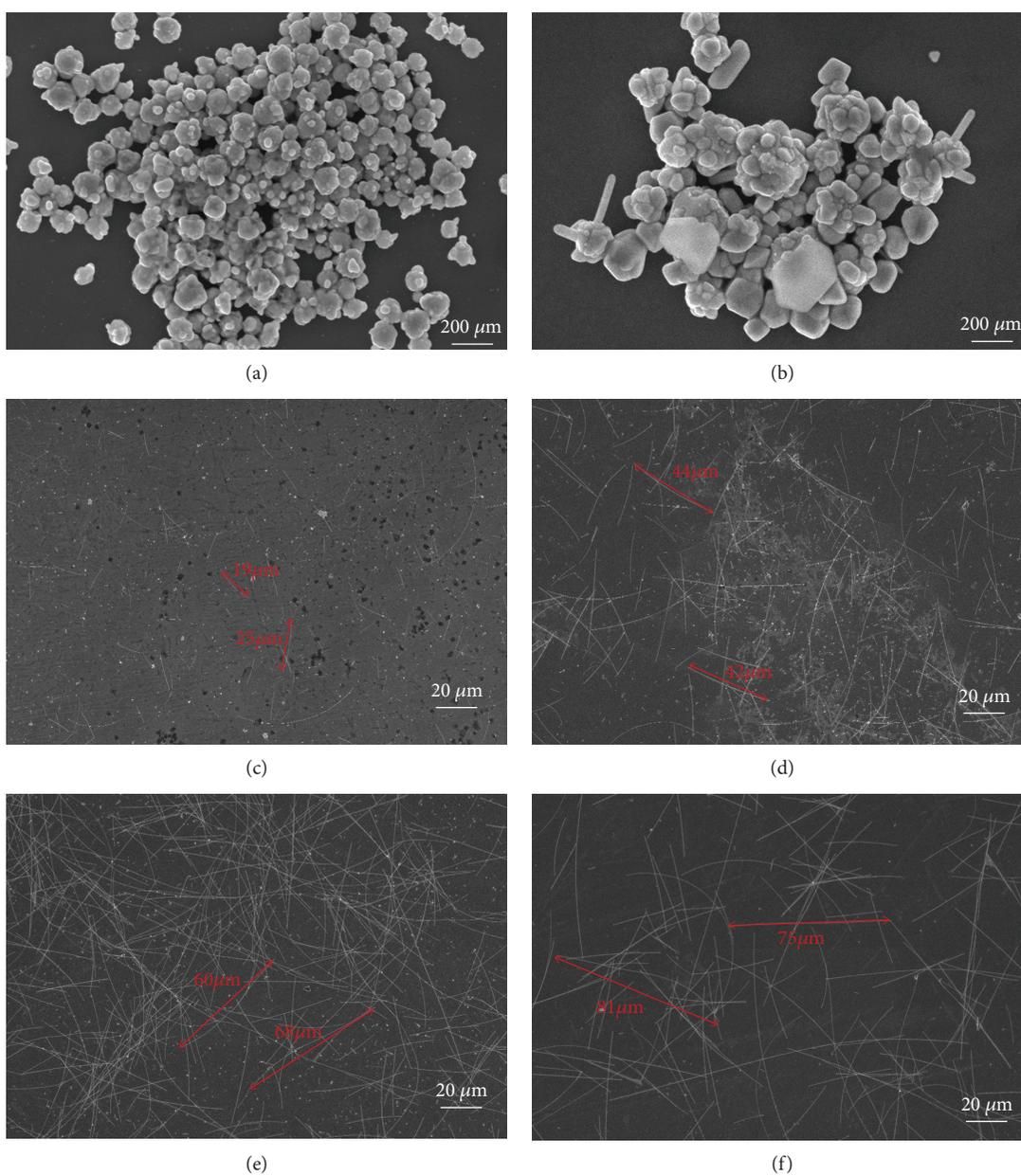


FIGURE 4: The SEM images of AgNWs synthesized by the coupling method for (a) 3 min, (b) 10 min, (c) 20 min, (d) 1 h, (e) 2 h, and (f) 3 h.

TABLE 2: Experimental methods and conditions.

	Polyol process	Polyol process	Coupling method
Concentration of $\text{AgNO}_3$ :PVP:CrCl <sub>3</sub> in a mixed solution		54 mM : 73.7 mM : 86 $\mu\text{M}$	
Temperature		160°C	
Reaction time		3 h	
Stirring speed	400 rpm	0 rpm	0 rpm

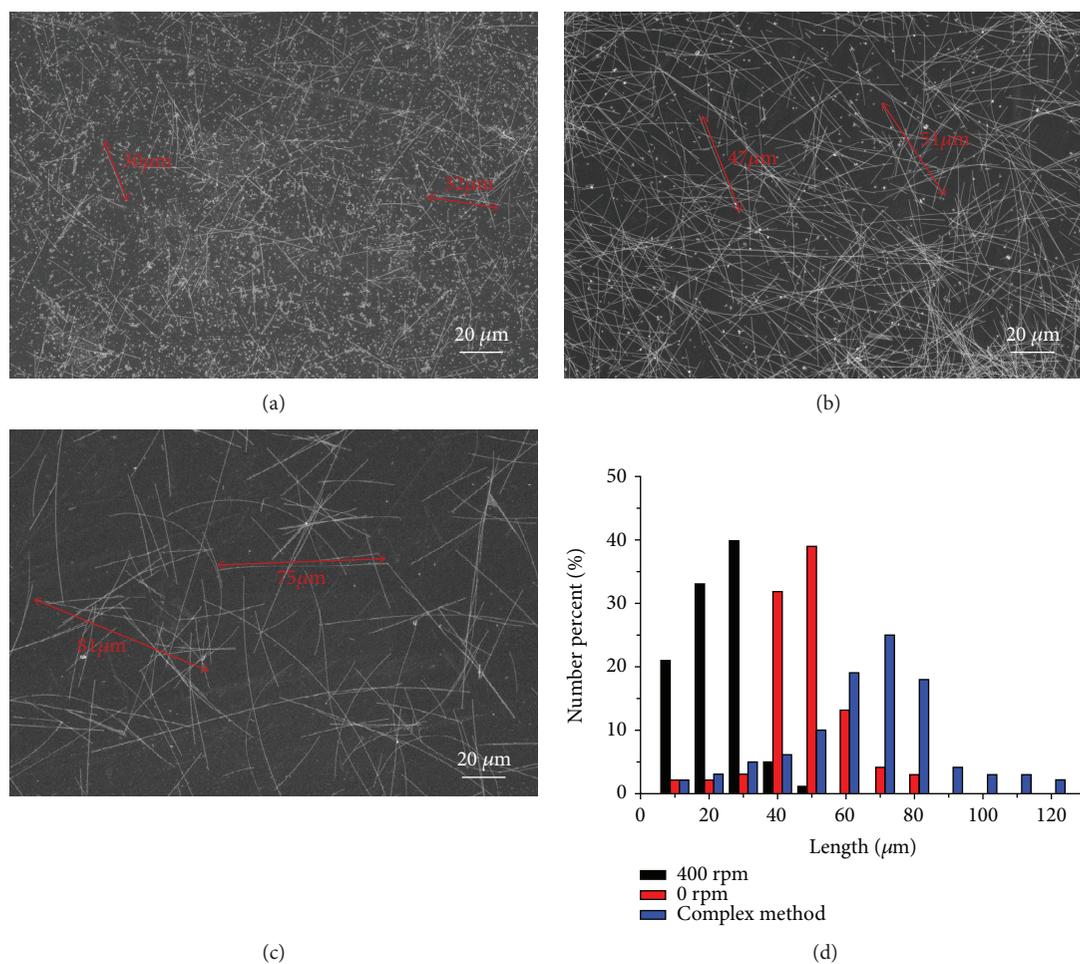


FIGURE 5: The SEM images of AgNWs synthesized by the polyol process with stirring speeds of (a) 400 rpm and (b) 0 rpm. (c) The coupling method with the described parameters in Methods and (d) the length distribution of AgNWs with different stirring speeds and methods.

length of the AgNWs with different ions existing showed regularity as their standard electrode potential. During the AgNW growth, the adsorbed atomic oxygen should be restored quickly to prevent oxidative etching, which would lead to a positive promotion of the growth of the yield and length of AgNWs. So, a metal ion with low standard electrode potential was necessary. Also, the length of AgNWs with CrCl<sub>3</sub> existing in this paper was longer than the 60  $\mu\text{m}$  length of AgNWs reported in References [34–36] with FeCl<sub>3</sub> existing.

Besides, the effect of a low concentration of Cr<sup>3+</sup> and Fe<sup>3+</sup> on the length of AgNWs was also researched, the concentration of metal ions was all 17  $\mu\text{M}$  in a mixed

solution, and other parameters were all described in Methods. The SEM images are shown in Figure 3; the length of wires with CrCl<sub>3</sub> existing was much longer than the length of wires with FeCl<sub>3</sub> existing. It is because Cr<sup>3+</sup> has more strong capacity of scavenging adsorbed oxygen on {111} reactive sites than Fe<sup>3+</sup> due to its lower standard electrode potential; fewer Cr<sup>3+</sup> could reduce the occurrence rate of oxidative etching, which would ensure that the AgNWs have a favourable growth. Meanwhile, the thought for the effects of the standard electrode potential on the morphology of AgNWs was proven.

SEM images of AgNWs synthesized by CrCl<sub>3</sub> and a stable reaction environment in different reaction times were shown

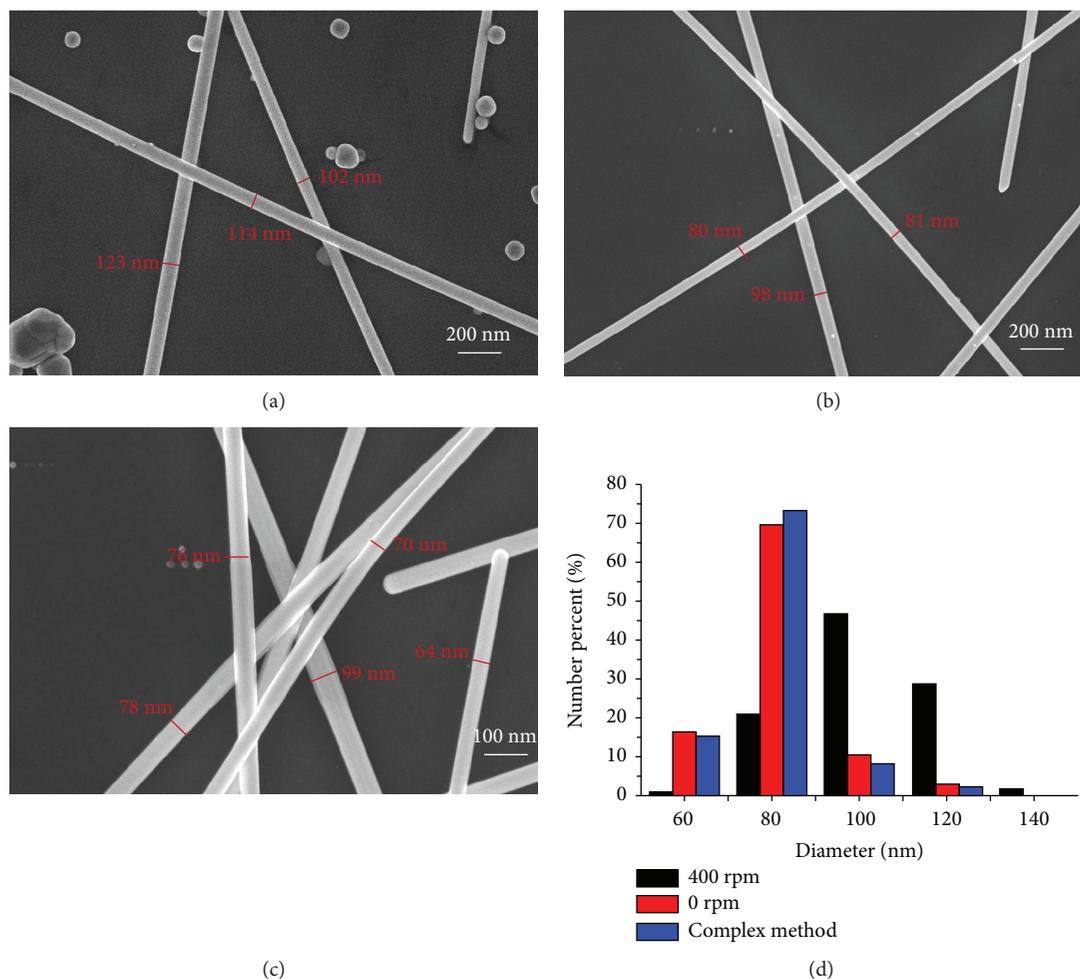


FIGURE 6: The SEM images of AgNWs synthesized by the polyol process with stirring speeds of (a) 400 rpm and (b) 0 rpm. (c) The coupling method with the described parameters in Methods and (d) the diameter distribution of AgNWs with different stirring speeds and methods.

in Figure 4. Multiple twin particles were observed after the reaction lasting 3 minutes. 10 min later, the particles grew larger as well as the wires produced from the large particles. Few and short AgNWs were formed after 20 min. 1 h later, more and more AgNWs were formed, and early AgNWs grew longer. The scale of the AgNWs became more and more large, and the numbers of particles dropped off after 2 h. 3 h later, the nanoparticles nearly completely disappeared, and the grow process of AgNWs is perfectly reflected. During the synthesis process, Ag nanoparticles start to form via homogeneous nucleation; as the process continues, some of the Ag nanoparticles can grow into multiple twin particles; then, the PVP cap the multiple twin {100} side faces, leading the {111} side faces to grow. The AgNW growth had benefited from the stable reaction environment.

SEM images of AgNWs synthesized in different reaction environments provided by the coupling method and polyol process with the same described parameters in Table 2 are shown in Figure 5(c). The length of AgNWs synthesized by the coupling method is found to be  $75 \mu\text{m}$ , which is longer than the length of  $50 \mu\text{m}$  synthesized by the polyol process without stirring. The diameter of AgNWs synthesized through two methods is nearly the same, about 80 nm, which

is shown in Figure 6. The length and diameter distribution are summarized in Figures 5(d) and 6(d) by counting 200 wires to measure the linear distance between the two ends of AgNWs in SEM using the software ImageJ. The yield synthesized by the coupling method was 90%.

In this paper, the method is more simple, and its yield is higher than that of the conventional polyol process because of the more stable reaction environment and higher restoring efficiency in adsorbing atomic oxygen on the {111} reactive sites. After the reaction, acetone and ethanol were used to wash the AgNWs with a centrifugation of 3500 rpm for 10 min, the washed AgNWs were dried in a vacuum environment at  $60^\circ\text{C}$  after taking the samples, and the yield is summarized by counting the weight of the raw material of Ag divided by nearly all the dry AgNWs.

The results also indicate that the growth of long wires requires a stable reaction environment, which could prevent disturbing the oriented growth of the wires. The speed of 400 rpm produced short wires as well as an amount of silver particles in the polyol process (Figure 5(a)). No speed solution produced long AgNWs with few silver particles in the polyol process (Figure 5(b)). However, the coupling method produced the longest wires without inseparable nanoparticles

(Figure 5(c)), for the reaction environment is the most stable without physical perturbation and air agitation. The growth of the AgNWs is maybe a process of spontaneous growth and free assembly after the seeds of AgNWs formed. On the other hand, the saturated vapor pressure of EG in 180°C is just 58.1 kPa, which is much less than the water, so the coupling method is safer than the traditional hydrothermal method.

Traditional polyol synthesis methods reported so far were mainly conducted in a mouth flask; the reaction environment may be disturbed by shaking, gas disturbance, and stirring, which will disturb the growth of the {111} reactive sites of the wires; then, the length and yield of the AgNWs could be affected to reduce. In this manuscript, the method could provide a more stable reaction environment than polyol synthesis; the more stable reaction environment promoted the growth of the {111} reactive sites of the wires; then, the length and yield of the AgNWs could increase.

On the other hand,  $\text{Cr}^{3+}$  as an etching agent was first used in this manuscript. Adsorbed atomic oxygen on the {111} reactive sites of the wires will etch the growth of the {111} reactive sites. The  $\text{Cr}^{2+}$  ion oxidized to  $\text{Cr}^{3+}$  has a lower standard electrode potential than  $E_{(\text{Fe}^{3+}/\text{Fe}^{2+})}$ ; the adsorbed atomic oxygen could be restored quickly; then, the length and yield of the AgNWs could increase.

#### 4. Conclusion

The AgNWs with the length over 75  $\mu\text{m}$  and the yield over 90% were synthesized through  $\text{CrCl}_3$  and a stable reaction environment. The standard electrode potential of  $E_{(\text{Cr}^{3+}/\text{Cr}^{2+})}$  is -0.42 V, much lower than the standard electrode potential of oxygen, which could restore the adsorbed atomic oxygen quickly and provide a high efficiency in the prevention of the oxidative etching with respect to  $\text{Fe}^{3+}$  and  $\text{Co}^{2+}$ ; then, the yield and the length of AgNWs would be promoted. In addition, a more stable reaction environment provided by the simple coupling method could avoid disturbing the growth of the {111} reactive sites of the wires; then, the AgNWs would be longer than those done by the traditional polyol process without stirring, and the diameter was nearly the same.

#### Data Availability

The data used to support the findings of this study are available from the corresponding author upon request.

#### Conflicts of Interest

The authors declare that they have no conflict of interest.

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