Research Article

High-Yield Synthesis of Uniform Ag Nanowires with High Aspect Ratios by Introducing the Long-Chain PVP in an Improved Polyol Process

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Polyvinyl pyrrolidone (PVP) with different molecular weights was used as capping agent to synthesize silver nanowires through a polyol process. The results indicated that the yields and aspect ratios of silver nanowires were controlled by the chain length of PVP and increased with increasing the molecular weight (MW) of PVP. When the long-chain PVP-K90 (MW = 800,000) was used, the product was uniform in size and was dominated by nanowires with high aspect ratios. The growth mechanism of the nanowires was studied. It is proposed that the chemical adsorption of Ag^+ on the PVP chains at the initial stage promotes the growth of Ag nanowires.

1. Introduction

Nanometer-sized noble metals are of great interest not only for their unique properties [1–3], but also for their potential applications in the fields of biomedical diagnostics [4], catalysis [5], optical devices [6], and data storage [7]. Among them, silver nanomaterials are particularly interesting due to their roles as substrates in studies of the surface-enhanced Raman scattering [8, 9], nonlinear optical properties [10], and catalysis [11]. For the optical properties, absorption associated with the collective oscillation of the conduction electrons or surface plasmon resonance (SPR) has been studied for many decades. Both experimental and theoretical studies show that the number and position of plasmon resonances as well as the spectral range for the surface-enhanced Raman scattering (SERS) of metal nanostructures strongly correlate with their exact morphology and aspect ratios of the nanostructures [12, 13]. In general, cylindrical Ag nanowires show one resonance, whereas several resonances are expected for Ag nanorods with triangle profiles [14]. For Ag nanoprisms, in-plane dipole SPR can be tuned across the entire visible spectrum from ∼400 nm to the near-infrared

regions [15]. More recently, Ag nanowires have attracted much attention due to their important applications for fabricating nanoscale fluorescence sensors [16, 17], plasmonic fibers [18–21], and so on.

For fundamental and applied sciences, various methods have been adopted to prepare Ag nanostructures [22–25]. In recent years, Ag nanostructures have yielded a great deal of literature featuring on the anisotropic growth of crystals into defined morphologies, ranging from nanowires, nanorods, nanoplates, nanocubes, and branched multipods to highly faceted particles [13, 26–29]. Among these methods, the common chemical reduction method based on the polyol process, which is convenient, versatile, and low costing, is usually adopted for the synthesis of noble metal nanoparticles. By changing the preparation parameters in the system, all kinds of nanostructures with different morphologies can be obtained, and the morphology could be further well controlled through introducing capping agent. One good example is the synthesis of Ag nanowires using polyvinyl pyrrolidone (PVP) as structure-directing reagent developed by Xia and coworkers [23, 30]. Based on the polyol method, large-scale uniform silver nanowires with high aspect ratios

TABLE 1: Products obtained by using PVP with different MWs. ($T = 150°C$ and $R = 1$). Note that D and L mean the average diameter and length of Ag nanowires, respectively.

No	PVP	Final product (yields) and size of nanowires
(1)	K17 (MW = 15000)	Nanoparticles (~60%) and nanowires (~40%); D: ~120 nm, L: ~3 μm
(2)	K25 (MW = 38000)	Nanoparticles (~50%) and nanowires (~50%); D: ~100 nm, L: ~3 μm
(3)	K30 (MW = 58000)	Nanoparticles (~10%) and nanowires (90%); D: ~180 nm, L: ~8 μm
(4)	K60 (MW = 200000)	Nanoparticles (~5%) and nanowires (~95%); D: ~120 nm, L: ~8 μm
(5)	K90 (MW = 800000)	Nanowires (~99%); D: ~100 nm, L: ~10 μm

were successfully achieved with [24] or without exotic seeds [23, 31].

Although the polyol process is a popular method of preparing metal nanostructures, so far most of the published works mainly focused on the synthesis process. In the case of the effect of capping agent PVP, although there are lots of documents studying the effect of the amount of PVP on determining the shape and size of Ag nanostructures, the investigations on the role of the molecular weight are still scarce and the effects on the synthesis of Ag nanostructures are not very clear until now [23, 24, 29].

In our previous work, we reported the preparation of Ag nanostructures with various shapes including nanoparticles, nanowires, nanorods, and nanocubes by the improved PVP-mediated polyol process (PVP with MW = 40000) [26, 27]. It was found that both the PVP/AgNO$_3$ ratio R and reaction temperature T played important roles and the favorable experimental parameters for synthesizing Ag nanowires were usually $R = 5$ and $T = 150°C$. If the R value decreased to 1, the perfect Ag nanocubes were the major products [27]. On the other hand, our recent inspiring investigation reveals that when the PVP with higher molecular weight was used, Ag nanowires with high yields and high aspect ratios were obtained at the condition of $R = 1$ with the same temperature, indicating that the molecular weight of PVP is also a critical parameter for the synthesis of Ag nanowires.

In this paper, we successfully synthesized large-scale uniform silver nanowires with high aspect ratios by introducing the long-chain PVP (MW = 800000) in an improved polyol process and investigated the effect of PVP molecular weight on the synthesis of Ag nanowires. The morphological evolution of the Ag nanostructures at various PVP molecular weights was studied in detail, and the growth mechanism of Ag nanowires is proposed linking both the structural characteristics of Ag nanostructures and the roles of PVP surfactants.

2. Experimental

AgNO$_3$ (≥99.8%, Sinopharm Chemical Reagent Co., Ltd.) and ethylene glycol (EG, Nanjing Chemical Reagent Co., Ltd.) were used in this work. PVP powders (Boai New Kaiyuan Pharmacy Co., Ltd.) with various average MW of 15000, 38000, 58000, 200000, and 800000 were chosen, which were marked as K17, K25, K30, K60, and K90, as shown in Table 1. All reagents were not purified further.

The improved PVP-mediated polyol process was used to synthesize the Ag nanowires. Firstly, 0.204 g of AgNO$_3$ and 0.1332 g PVP were, respectively, solved in a 5 mL EG solvent. Then both solutions were simultaneously injected dropwise into 20 mL of EG solvent which was magnetically stirred at 150°C in a round-bottom flask. The injection time lasted about 3 min. Magnetic stirring was applied throughout the entire synthesis. The molar ratio between the repeating unit of PVP and AgNO$_3$ was fixed at $R = 1$.

The reactant mixture changes quickly from colorless to yellow after the addition of a few drops of PVP and AgNO$_3$ solutions. The mixtures turned from yellow to slightly opaque after ~30 min and finally formed slightly green colloids for PVP-K17 and PVP-K25 after ~65 min or gray white colloids for PVP-K30, PVP-K60, and PVP-K90 after ~80 min. For characterization, the mixture was sampled at different times and was then centrifuged repeatedly to remove possible contamination (EG and excess PVP).

For field-emission scanning electron microscopic (FE-SEM; Sirion200, FEI, Holland) and X-ray diffraction (XRD; Ultima-III; Rigaku, Tokyo, Japan) measurements, the condensed products were dispersed on the copper sheets and glass substrates, respectively. For transmission electron microscopy (TEM; JEM-2000EX; JEOL) and high-resolution (HR) TEM (Tecnai G2 F20, FEI) observations, the products were diluted with deionized water (~×30), and a few drops of dispersions were placed onto the carbon-coated copper grids and dried at room temperature. Optical absorption spectra for the diluted samples were recorded on a UV-Vis-NIR spectrophotometer (U-3410, Hitachi). Fourier transformed infrared (FT-IR) spectra were obtained on a Bruker Vector 22 FT-IR spectrophotometer by using KBr pellets.

3. Results and Discussion

In our experiment, it was found that both yields and aspect ratios of the obtained Ag nanowires synthesized at $T = 150°C$ and $R = 1$ increased with increasing the MW of PVP. Table 1 presents the yields and sizes of Ag nanowires with different PVP types. Figure 1 shows some typical FE-SEM results of samples no. 1–no. 5 in Table 1. It can be seen that the Ag nanowires are not uniform in size and shape and a lot of irregular particles appear in the products when PVP-K17 and PVP-K25 were used (Figures 1(a) and 1(b)). For the case of sample-no. 3 (PVP-K30), the Ag nanowires (together with some cubic, tetrahedral, and other shaped nanoparticles) with high yields are not uniform with diameter ranging from
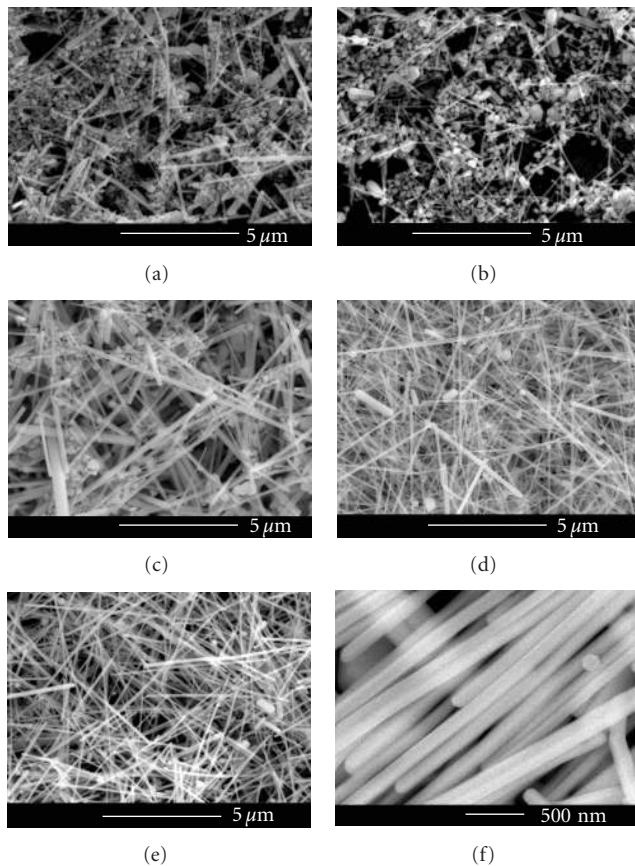
(a)   (b)

(c)   (d)

(e)   (f)

FIGURE 1: ((a)–(e)) FE-SEM images of the final products of sample no. 1–no. 5. (f) A magnified image of sample no. 5.
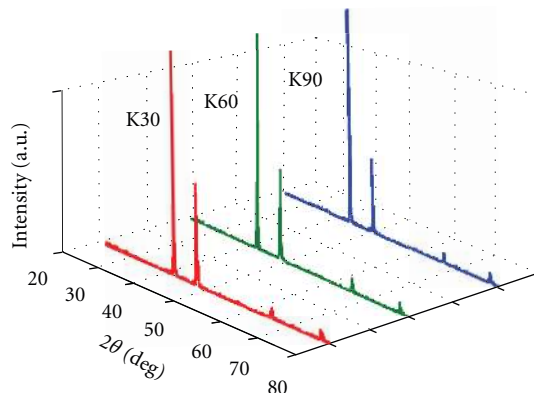

FIGURE 2: XRD patterns of the Ag nanowires synthesized using different MWs of PVP (K30, K60 and K90 with MW = 58000, 200000 and 800000). The ratio of intensity between the (111) and (200) peaks was 2.18, 2.4 and 2.9 respectively.

40 nm to 380 nm, as shown in Figure 1(c). With the MW of PVP increasing, the yields of the Ag nanowires increase further and almost no particles could be obsesrved in the product, as shown in Figures 1(d) and 1(e). Figure 1(f) is a magnified image of sample-no. 5 (PVP-K90); it is seen that the diameter of Ag nanowires is quite uniform and tends to self-assemble into two-dimensional arrays with solution evaporation during preparing sample.

Figure 2 presents the XRD pattern of sample no. 3–no. 5. The XRD pattern indicates that Ag nanowires synthesized using this solution-phase method are purely in the face-centred cube (fcc) phase. The lattice constant calculated from this XRD pattern was 4.092 Å, which is perfectly in agreement with the literature value of 4.086 Å. It is worth noting that the ratio of diffraction intensity between the (111) and (200) planes changed from 2.18 to 2.90 as PVP MW increased from 58000 to 800000, possibly because of the higher yields of the Ag nanowires in the final products.

The TEM images (Figures 3(a), 3(b), and 3(c)) also indicate that the Ag nanowires of sample-no. 5 possess uniform diameter and high yield, just as what we observed in the SEM measurements. In order to obtain the detailed structural information of the Ag nanowires, we further performed the selected-area electron diffraction (SAED) pattern characterization and HRTEM observation, as shown in Figures 3(d)

and 3(e). The SAED pattern consists of a superimposition of contributions from different subunits. The spots from superimposition of [112] and [001] zone axes are indicated. According to the HRTEM image, the fringe spacing is about 0.145 nm, which is in agreement with the (220) lattice spacing of Ag crystal, as shown in Figure 3(e). These results imply that the Ag nanowires should be fivefold symmetry rounding the long wire axis in the [110] direction, as widely reported about Ag nanowires [32–34].

Since Ag nanostructures with different shapes and sizes exhibit different SPR bands at different frequencies, we subsequently carried out UV-vis spectrum measurements for the samples. Figure 4 shows the optical extinction spectra of Ag colloid solutions synthesized using different MWs of PVP. One observes that all the products show the evident plasma peaks at ~355 nm and ~390 nm, which should be attributed to the out-of-plane quadrupole resonance and out-of-plane dipole resonance of the Ag nanowires, respectively [35–37]. This observation indicates that all the synthesized products contain Ag nanowires. For the curves (a) and (b), there is another obvious peak at ~445 nm ascribed to the SPR resonance of the Ag nanoparticles, and a shoulder peak around ~445 nm also appears in curves (c) and (d), which indicates that the final products should be a mixture of Ag nanowires and a small amount of nanoparticles.

It is interesting that, with the MW of PVP increasing, the peak intensities at ~355 nm and ~390 nm increased while the peak intensity at ~445 nm decreased. When the PVP molecular weight reaches 800000, only the peak at ~355 nm and ~390 nm could be obsserved, indicating that there are almost only Ag nanowires in the final product. (see curve (e) for sample-no. 5). This is consistent with the results of SEM and TEM observations that Ag nanowires were more favorable products synthesized using higher MW of PVP. For the first time, to the best of our knowledge, uniform silver nanowires with high yields were successfully synthesized by using so-long-chain PVP (MW = 800000) in a simple polyol process. Generally, PVP with MW = 40000 or 55000 is used to prepare Ag nanowires.
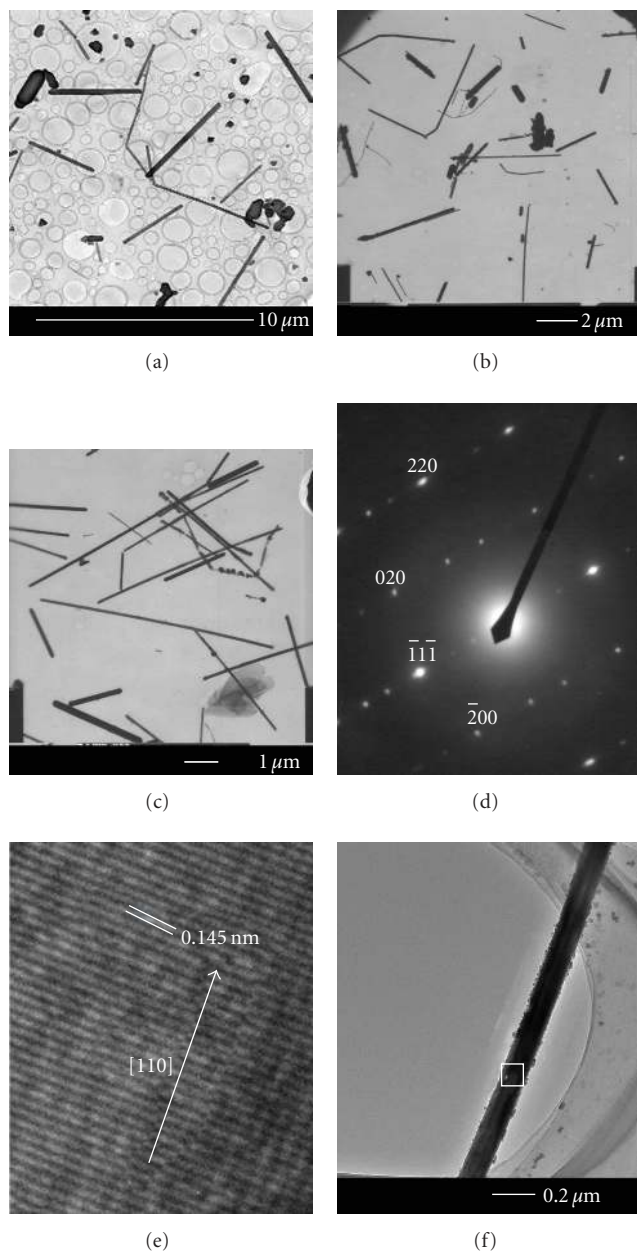
(a)

(b)

(c)

(d)

(e)

(f)

Figure 3: TEM images of the Ag nanoproducts using different MWs of PVP:(a) K30 (MW = 58000), (b) K60 (MW = 200000), and (c) K90 (MW = 800000). (d) A typical SAED of one Ag nanowire; the spots from superimposition of the [112] and [001] zone axis are indicated. (e) A typical HRTEM image of a selected area of an individual Ag nanowire. (f) The nanowire of sample no. 5 used for the SAED and HRTEM studies with the area marked with a white pane indicating where the HRTEM image was recorded.

According to all above results, we could conclude that the yields as well as the aspect ratios of the Ag nanowires increased with increasing the MW of PVP. In terms of the process of growth and reduction, EG is generally used as a reducing reagent while the PVP plays the role of surfactant during the preparation. This is true for the reduction of Ag$^+$

(a) PVP-K17 (d) PVP-K60
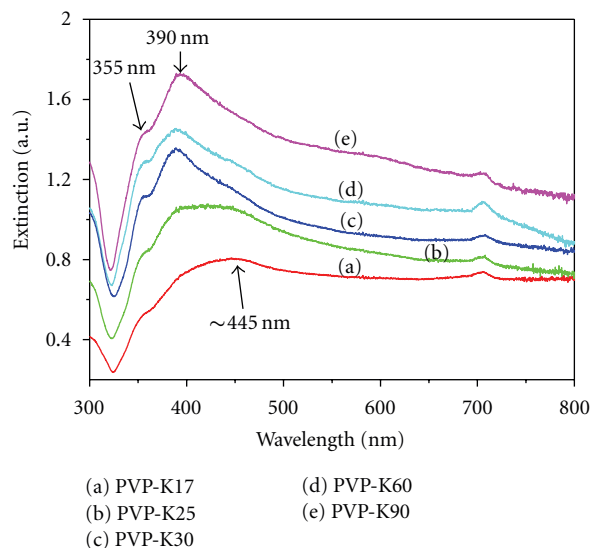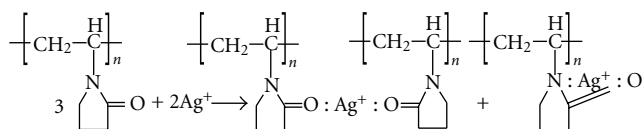(b) PVP-K25 (e) PVP-K90
(c) PVP-K30

Figure 4: UV-visible extinction spectra of the final products synthesized using various MWs of PVP (a) K17 (MW = 15000), (b) K25 (MW = 38000), (c) K30 (MW = 58000), (d) K60 (MW = 200000), and (e) K90 (MW = 800000). All solutions had been diluted by ×30 with deionized water.

ions in hot EG solution, which was proved in our previous work [38], and the chemical reaction is as follows:

$$2HOCH_2CH_2OH \longrightarrow 2CH_3CHO + 2H_2O$$

$$2Ag^+ + 2CH_3CHO \longrightarrow CH_3CO–OCCH_3 + 2Ag + 2H^+. \tag{1}$$
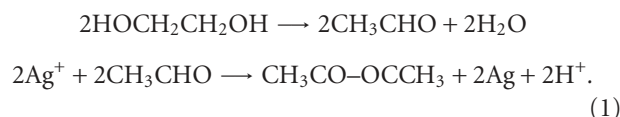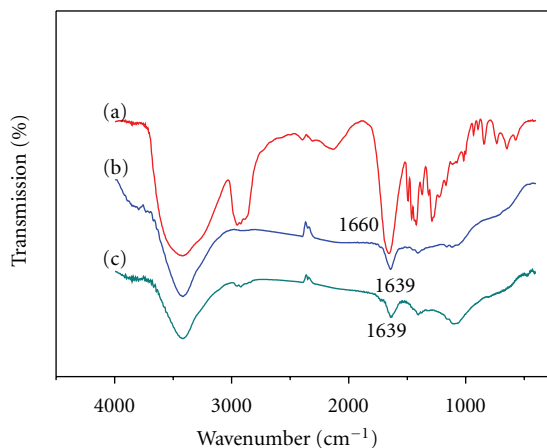
We now discuss the effect of PVP on the formation of Ag nanostructures. It is known that the growth morphology is often determined by the surface free energies under thermal equilibrium. On the nanometer scale, the transition fcc metals are favourable for the formation of fivefold twinned structure with their surfaces bounded by the lowest-energy {1 1 1} facets. However, crystal growth is usually far from the thermal equilibrium; thus, the shape is not characterized by minimizing the surface energy, but rather the growth rate of each face as determined by the kinetics [39]. In the study of the growth mechanism of anisotropic Ag nanostructures fabricated by the polyol process, a common attitude is that PVP interacts more strongly with Ag atoms on the {1 0 0} facets than those on the {1 1 1} facets, which act as a structure-directing agent as well as the capping agent preventing aggregation [30].

We know that PVP has the structure of polyvinyl skeleton with strong polar group (pyrrolidone ring), so it has an affinity toward many chemicals to form coordinative compounds. The polar groups, such as the >C=O groups of PVP chain, can interact with metal ions and form coordinating complex (as seen in Scheme 1) [40]. In our previous work [41], Fourier transform infrared spectra of PVP and PVP-AgNO3 film were measured, and a shift in frequency of the C=O in pyrrolidone ring indicates an interaction between the AgNO3 molecules and the PVP chains as in Scheme 1. These

Scheme 1: Complexation of Ag ions with PVP chains.

Scheme 2: Schematic diagram of 1D structure for PVP-Ag coordination compound. The red, blue, and white spheres represent the atoms of oxygen, nitrogen, and carbon, respectively.
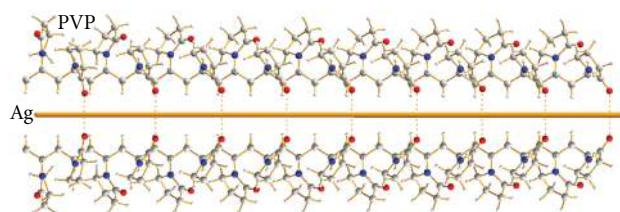

(a) PVP-K90
(b) PVP-Ag before washing
(c) PVP-Ag after washing

Figure 5: Infrared transmission spectra for PVP-K90 and the corresponding final products before and after washing.

reactions would create a state in which Ag^+ ions can be reduced to Ag^0 to form extremely fine Ag embryos. Thus the PVP-Ag coordination complex compound would promote the formation of crystals once the mixture is dissolved in hot EG solution.

In our experiment, the chain length of PVP plays an important role in determining the yields and sizes of the silver nanowires. As for the long-chain PVP, which has higher degree of polymerization (DP) compared with the short one, the average number of repeating units in one PVP macromolecule chain n is very high (from the PVP-K90 to PVP-K17, the average number n is about 7207, 1802, 522, 342, and 135, resp.). Thus, there are more carbonyl groups in one PVP macromolecule and more silver ions coordinated along the long chain of PVP. So it is suggested that the PVP with higher MW should more easily induce the PVP-Ag coordination compound to arrange in a one-dimensional (1D) manner and that linear Ag embryos with a longer size should be formed (as in Scheme 2). Accordingly, the embryos would continue to grow becoming silver nanowires, while the reduction reaction takes place in hot EG. As for the PVP with low MW, a relatively few silver ions coordinated along the short chain of PVP should result in the formation of silver nanorods with a shorter length, and those free silver ions which do not react with PVP would be attributed to the formation of Ag nanoparticles. So the previous adsorption of Ag^+ on the PVP chains in the initial stage and the chain length of PVP both are critical factors to synthesis of 1D silver nanostructures. We suggest that the coordination compound

of Ag^+ and the PVP with high MW should facilitate the formation of Ag nanowires with high aspect ratios.

Furthermore, to explore the surface chemical state of the final Ag nanowires, we also investigated the infrared spectra of pure PVP and the final product before and after washing. Figure 5 presents the infrared transmission spectrum for PVP-K90 and the corresponding final product which shows that the peak of the C=O at $1660 \, cm^{-1}$ was slightly widened and shifted to approximately $1639 \, cm^{-1}$ after coordination with silver. The results suggest that the bonding between PVP and the metal crystal remains even when the metal ions are reduced. The fact that there still exists the C=O peak at $1639 \, cm^{-1}$ in the spectra of the product after washing verified a certain chemical action between PVP and silver. This result is accordant to investigations of some other groups. For example, Xie et al. suggest that one monolayer of the PVP molecules might be adsorbed on the surface of the Ag nanowires through Ag-O coordination according to the results of thermogravimetric analysis (TGA), X-ray photoelectron spectroscopy (XPS), and Fourier transform Raman spectra (FT-Raman) [42]. The Ag nanowires modified by long-chain PVP would be helpful for self-assembly into ordered two-dimensional arrays for the PVP layer providing possible interaction among the neighboring Ag nanowires.

4. Conclusions

We have successfully synthesized uniform Ag nanowires with high yields by using a simple and improved PVP-mediated polyol process through only adjusting the MW of PVP at $150°C$ and $R = 1$. We found the chain length of PVP played a critical role in directing the 1D growth of Ag nanostructures. With the MW of PVP increasing from 15000 to 800000, the yields of Ag nanowires improved from 40% to 99% and the average length increased from $3 \, \mu m$ to $10 \, \mu m$; moreover, the uniformity of size became better. The growth mechanism of silver nanowires proposes that the chemical adsorption of Ag^+ on the PVP chains promotes a linear arrangement of the reduced Ag atoms and that more Ag atoms or small particles should be attributed to the growth of Ag nanowires naturally. High-yield synthesis of prefect silver nanowires with high aspect ratios requires only the long-chain PVP usage and need not introduce any exotic seeds and ions. The method is very simple and economical and promotes the potential applications of silver nanowires in lots of areas such as SERS, electronic, photonic, and optoelectronic devices.
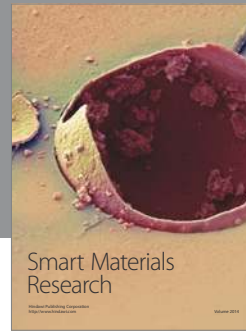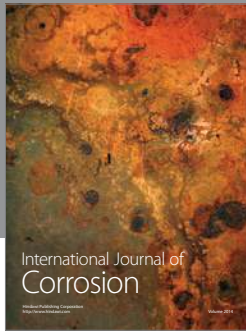
Acknowledgments

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