Research Article

A Facile Fabrication of Silver-Coated Copper Nanowires by Galvanic Replacement

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We demonstrated a general strategy to fabricate silver-coated copper nanowires by a galvanic replacement, which is guided by the chemical principle that metal ions (silver ions) with a relatively high reduction potential can galvanically etch nanostructure made from a less metal (copper). Well-dispersed and high-yielded copper nanowires were initially synthesized and then introduced into silver-ammonia solution for the growth of silver nanocrystals on the nanowire surfaces under vigorous oscillation. The results of X-ray diffraction, scanning electron microscope, and transmission electron microscope revealed that the silver nanocrystals were uniformly distributed on the copper nanowire surfaces to form Cu-Ag heterostructures. The concentration of silver-ammonia solution and the time of replacement reaction determine the size and density of the silver nanocrystals. Our investigation might pave the way to the synthesis of other bimetallic nanostructures via a facile, fast, and economical route.

1. Introduction

Bimetallic nanostructures have received great attention due to the compound performance, enhanced activity, and physicochemical stability compared to the individual components. Particularly, the new properties can be obtained owing to the electronic coupling between two different metals, providing more promising applications for the bimetallic nanostructures in catalysis, plasmonics, electronics, and sensing [1–21]. For instance, Stewart et al. fabricated Cu-Ag, Cu-Au, and Cu-Pt core-shell nanowires. Compared with pure copper nanowires (Cu NWs), the resistance to oxidation of the transparent electrode based on these bimetallic nanowires can be effectively improved [7]. Liu et al. have synthesized Cu-Pt nanotubes by a galvanic replacement. The nanotubes exhibit superior electrocatalytic activity and stability during oxygen reduction reaction compared with a commercial Pt/C catalyst [11]. Jiang et al. reported the synthesis of Cu-Ag hybrid nanowires with strong surface-enhanced Raman scattering sensitivity [20].

As well known, one-dimensional metal nanostructures have been attracting intense attention due to their excellent photoelectric properties [22–25]. Therein, Cu NWs have been widely investigated in the fields of catalysis and photoelectric devices ascribed to the excellent electronic transport properties and low cost in materials [16, 17, 26–31]. However, poor oxidation stability of the Cu NWs always hinders further practical application. To improve the stability of the Cu NWs, additional coatings are usually deposited on the NW surface, for example, Au, Ag, Pt, or Pd metallic coatings [16, 17, 30–32]. The outer metallic coatings with higher stability not only provide effective passivation against oxidation of the Cu NWs, but also maintain good conductivity. Among these coating materials, silver is one of competitive candidates since it has better electrical conductivity and stability than copper, and it has lower cost than other noble metals. According to the different combinations of two metals, bimetallic nanostructures can be classified into core-shell structure and heterostructure [1, 2, 5, 6, 8]. In order to obtain large surface area of the metallic nanowires to further improve the catalysis
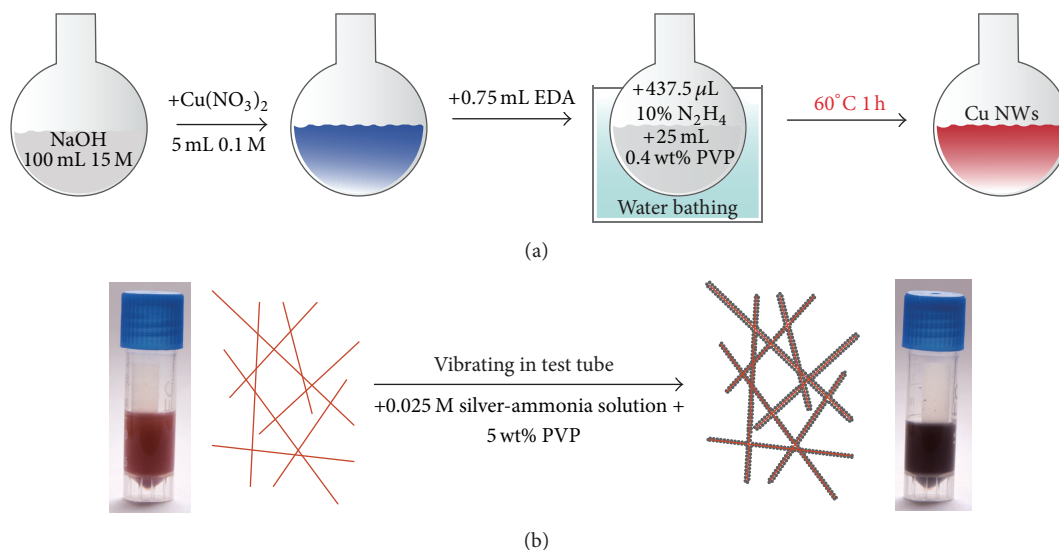
(a)

(b)

FIGURE 1: Schematic illustration of the fabrication procedure of pure Cu NWs (a) and silver-coated Cu NWs (b), respectively.

activity, silver nanocrystals are usually supposed to be coated on the surface of the Cu NWs to fabricate silver-copper heterostructure rather than core-shell nanostructure.

Based on this purpose, we developed a facile method to synthesize silver nanocrystals on the surface of high-yielded and well-dispersed Cu NWs by galvanic replacement. Although the coating method is usual in the fabrication of bimetals, the experimental parameters need to be accurately controlled in order to synthesize uniform coating. In our work, the replacement reaction took place under vigorous oscillation without any other additives. Silver nanocrystals uniformly grow on the Cu NW surface and formed Cu-Ag heterostructure with Cu NWs. The surface areas of the bimetallic nanowires are supposed to greatly increase; meanwhile the oxidation of the Cu NWs is hindered by the coating of the silver nanocrystals. Therefore, this work reveals that more metals with different reduction potentials could facilely synthesize the bimetallic heterostructures by galvanic replacement.

2. Experimental

2.1. Synthesis of Cu NWs. Cu NWs were synthesized using a modified chemical reduction [33]. The fabrication procedure is shown in Figure 1(a). Typically, 1.5 mol sodium hydroxide (NaOH) was dissolved using deionized water of 100 mL in a flask to obtain 1.5 M NaOH solution. Then, 5 mL aqueous solution of copper nitrate ($Cu(NO_3)_2 \cdot 3H_2O$, 0.242 g in 10 mL of deionized water) was slowly added to NaOH, yielding a blue transparent solution. The high concentration of NaOH was required to prevent copper ions from forming copper hydroxide precipitates. 0.75 mL (99 wt%) solution of ethylene diamine (EDA) was injected into the blue solution. Following this, 437.5 μL (10 wt%) hydrazine (N_2H_4) was dropwise added to the flask to reduce copper ions, and 25 mL (0.4 wt%) solution of poly(vinyl pyrrolidone) (PVP, K30) was added to improve the dispersibility of product. The white precipitates appeared and then the mixture became transparent again as

the added amount of N_2H_4 increased. The flask was capped and exposed to 60°C water bathing for 1 h. Finally, the red product containing Cu NWs was obtained. Cu NWs were washed with deionized water containing 1 wt% PVP and 3 wt% N_2H_4 several times.

2.2. Synthesis of Silver-Coated Cu NWs. Silver-coated Cu NWs were synthesized by a galvanic replacement, and the fabrication procedure is displayed in Figure 1(b). Before replacement, Cu NWs were washed utilizing ethanol and glacial acid acetic to remove the surfactant and oxide. Silver source derived from silver-ammonia solution, which was prepared according to the following steps. 0.170 g silver nitrate ($AgNO_3$) was dissolved in 25 mL H_2O, and then ammonia ($NH_3 \cdot H_2O$) was dropwise added to $AgNO_3$ solution until white precipitate disappeared, generating silver-ammonia solution. Cu NWs with a certain quality were introduced into silver-ammonia solution in a test tube along with fierce vibrating. After tens of minutes, the red Cu NWs turned crimson, demonstrating that the silver-coated Cu NWs were synthesized. The final products were repeatedly washed by ethanol to remove the redundant $Ag(NH_3)_2^+$ ions.

2.3. Characterization. The structures and compositions of the products were characterized using an X-ray diffractometer (XRD) (X'pert Pro MFD) with a Cu Kα radiation (λ = 1.54178 Å). The morphologies of the nanowires were observed using a field emission scanning electron microscope (FESEM) (Nova™ NanoSEM 430, FEI), and energy dispersive spectra (EDS) were also conducted on the SEM 430. A transmission electron microscope (TEM) measurement and the element mapping characterization of the nanowires were carried out with JEOL-2100F electron microscope.

3. Results and Discussion

Figures 1(a) and 1(b) show a schematic illustration of the fabrication procedure of pure and silver-coated Cu NWs,

respectively. During the synthesis process of pure Cu NWs, the aggregation of Cu NWs can be inhibited by the addition of surfactant PVP. Good dispersibility of the Cu NWs is essential to obtain uniform distribution of silver nanocrystals on the Cu NW surface.

Figures 2(a) and 2(b) display typical SEM images of the product before replacement reaction, indicating that high-yielded nanowires in a large scale were synthesized. The straight nanowires possess smooth surfaces and uniform diameters, and the length of the nanowires is up to dozens of microns. Figure 2(c) shows diameter distribution histogram of the obtained nanowires, revealing that the average diameter of the nanowires is approximately 107 nm. The corresponding EDS pattern of the nanowires is shown in Figure 2(d), which confirms that main element of the nanowires is Cu, whereas element Si peak originates from the substrate, and element C and O weak peaks derive from the capped surfactant or adsorbed air. Furthermore, XRD measurement was utilized to further explore whether copper had been oxidized in the nanowires. Figure 2(e) displays XRD pattern of the high-yielded nanowires and the standard card of cubic copper (JCPDF number 96-710-1265), respectively. Three obvious and sharp diffraction peaks are detected at 43.2, 50.3, and 74.0° for the nanowires, which can be assigned to (111), (002), and (022) reflections of face centered cubic structure of metallic copper, respectively. No obvious peaks from copper oxide can be observed, indicating high purity of the Cu NWs.

Before replacement, the Cu NWs were repeatedly washed by ethanol and glacial acid acetic and then dried in vacuum oven at 60°C. The dried Cu NWs with a certain quality were redispersed in ethanol and glacial acid acetic and then introduced into various concentrated silver-ammonia solution to keep molar ratio of Cu and Ag 2 : 1 and 1 : 2. The mixed dispersions were persistently vibrated in a test tube to synthesize the silver-coated Cu NWs. Two products were named as samples A and B, respectively. The silver ions possess a relatively high reduction potential compared to copper ions, which can lead to a galvanic replacement between silver ions and copper at ambient condition. Therefore, in our work, it is believed that metallic Cu NWs can reduce the $Ag(NH_3)_2^+$ ions adsorbed on their surfaces, leading to the formation of metallic silver.

Figures 3(a) and 3(b) present representative SEM images of samples A and B, respectively. The inset of Figure 3(b) is a magnified image. Several nanocrystals are observed on the smooth surface of Cu NWs after galvanic replacement, which can be ascribed to silver nanocrystals. Once $Ag(NH_3)_2^+$ ions were reduced to metallic silver on Cu NW surface, a large strain can be generated due to the lattice mismatch between silver and copper at the interface, providing driving force to form silver nanocrystals [5]. As the concentration of silver-ammonia solution increased, the surface roughness of the Cu NWs is greatly improved, and the numbers and diameters of the nanocrystals are increased. Compared to the pure Cu NWs, the surface areas of the bimetallic nanowires thus are effectively enlarged, potentially promoting the catalytic activities. Furthermore, the nanocrystals are uniformly distributed on the surface of the Cu NWs via galvanic replacement under

vigorous oscillation, and no individual nanocrystals can be observed in solution. It indicates that the replacement of $Ag(NH_3)_2^+$ by metallic Cu just takes place on the surface of the Cu NWs. The oscillation offers lots of opportunities for the collisions between $Ag(NH_3)_2^+$ ions and Cu NW surface, resulting in uniform distribution of silver nanocrystals. The individual nanocrystals cannot be synthesized in solution due to the absence of any other reducing agent. Moreover, if silver nitrate substitutes for silver-ammonia solution in this work, the replacement reaction would take place very quickly. The mixed solution turned black in several seconds, and uneven coatings were observed. It is believed that the $Ag(NH_3)_2^+$ chelate ions are beneficial to a mild replacement reaction, generating uniform coatings of the nanocrystals on the Cu NW surfaces.

Figures 3(c) and 3(d) display EDS patterns of the nanocrystals-coated Cu NWs in samples A and B, suggesting that the main elements of the nanowires are Cu and Ag, and the content of element Ag in sample B is higher than that in sample A. However, the contents of element O in two samples are not markedly increased compared to the pure Cu NWs, demonstrating that the nanocrystals on the nanowire surfaces can effectively hinder the oxidation of the Cu NWs. Moreover, the atomic ratios of element Cu to Ag in two samples are larger than the predefined ratios of 2 and 0.5. The surface of Cu NWs provides active sites for the replacement reaction. Once the silver nanocrystals occupy all active sites on the surface, the further galvanic replacement almost stops. The redundant $Ag(NH_3)_2^+$ ions in the solution are hard to be further reduced to metallic silver due to the lack of any reducing agent in the solution, leading to a high atomic ratio of element Cu to Ag in two samples. If we extended the reaction time for sample A, the sizes and numbers of the silver nanocrystals could further increase.

Figure 3(e) shows XRD patterns of samples A and B, and the standard cards of cubic copper and silver are provided for reference. In two patterns of samples A and B, the diffraction peaks located at 43.2 and 50.4° can be assigned to (111) and (002) reflections of face centered cubic structure of metallic copper (JCPDF number 96-710-1265). The diffraction peak originated from silver cannot be observed in the pattern of sample A due to low loading of silver nanocrystals on the Cu NW surface. However, as the content of silver increased, the peak at 38.0° appeared, which matches well with the (111) reflection of face centered cubic structure of metallic silver (JCPDF number 01-087-0597). It is worthy to note that the peak positions of metallic copper in sample B do not shift as silver nanocrystals formed, revealing that the nanostructure of the Cu NWs cannot be changed by silver nanocrystals. The obtained product is heterostructure of Cu NWs and silver nanocrystals, rather than bimetallic alloy.

Figure 4(a) presents a typical TEM image of several silver-coated Cu NWs. It can be clearly observed that many small black dots are uniformly attached on the surface of every single nanowire. Particularly, more dots are located at the junctions of the straight nanowires. Combined with the above discussion, the attached dots are silver nanocrystals. Figure 4(b) shows TEM image of single silver-coated Cu NW, indicating a uniform distribution of the silver nanocrystals
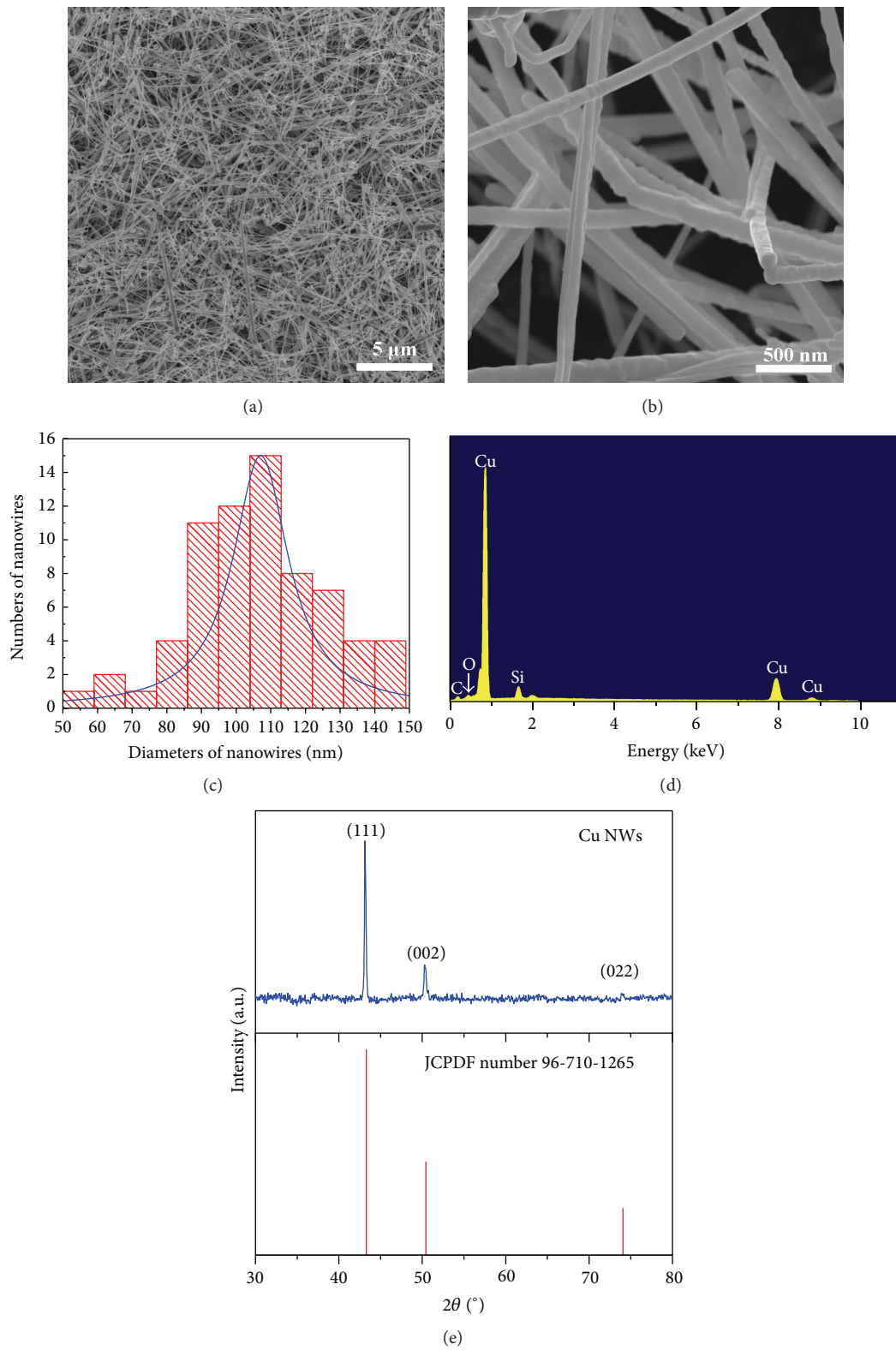
(a)

(b)

(c)

(d)

(e)

FIGURE 2: SEM images of high-yielded Cu NWs with low (a) and high (b) magnification; (c) diameter distribution histogram of the nanowires; (d) corresponding EDS pattern of the nanowires; (e) XRD pattern of the Cu NWs, and the PDF card of cubic copper (JCPDF number 96-710-1265) is provided for reference.
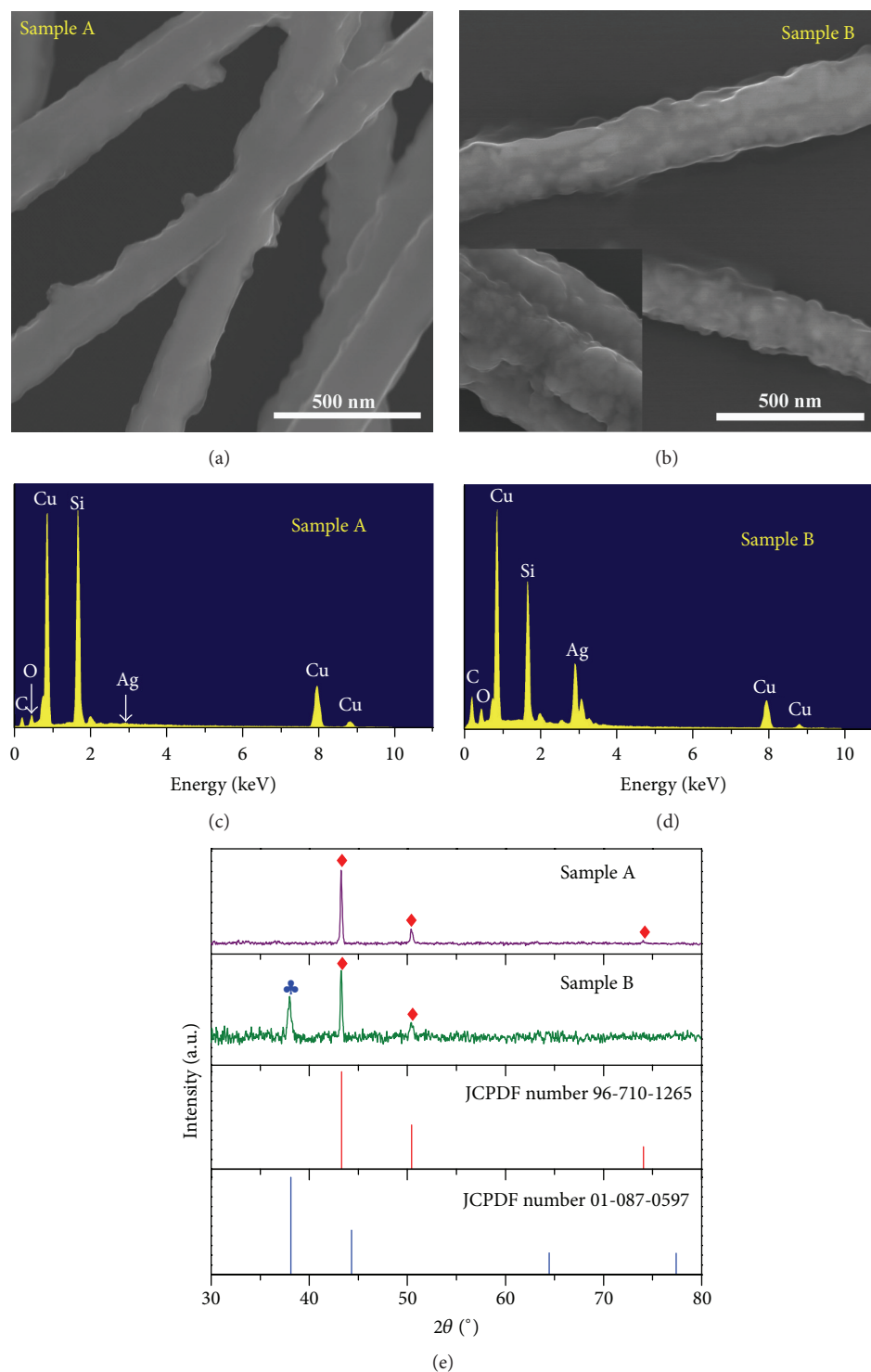
FIGURE 3: SEM images of the silver-coated Cu NWs for samples A (a) and B (b); the inset of (b) is a magnified image; EDS patterns of the silver-coated Cu nanowires for samples A (c) and B (d), respectively; (e) XRD patterns of the silver-coated Cu nanowires for samples A and B, and the PDF cards of cubic copper (red line, JCPDF number 96-710-1265) and silver (blue line, JCPDF number 01-087-0597) are provided for reference. The red rhombus represents the reflection of face centered cubic structure of metallic copper (JCPDF number 96-710-1265), while the blue star represents reflection of face centered cubic structure of metallic silver (JCPDF number 01-087-0597).

(a)   (b)   (c)
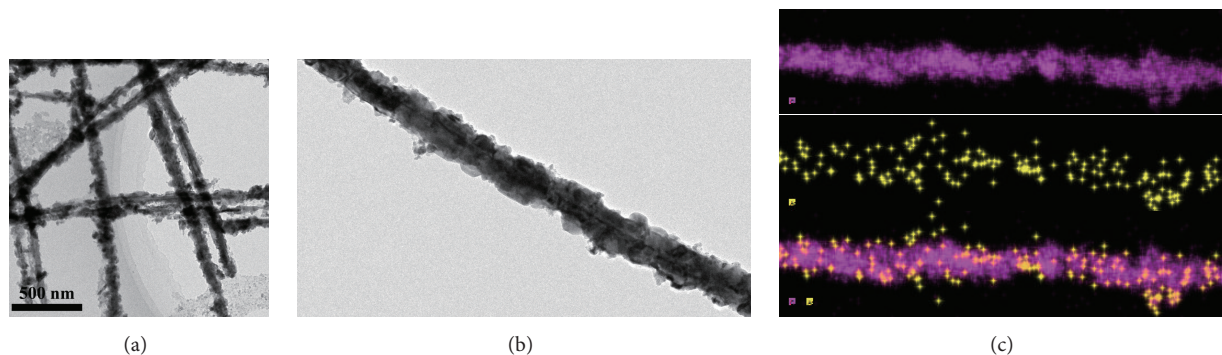
FIGURE 4: (a) TEM image of the silver-coated Cu NWs; (b) TEM image of single silver-coated Cu NW and its element mapping images (c); the purple and yellow dots represent elements copper and silver, respectively.

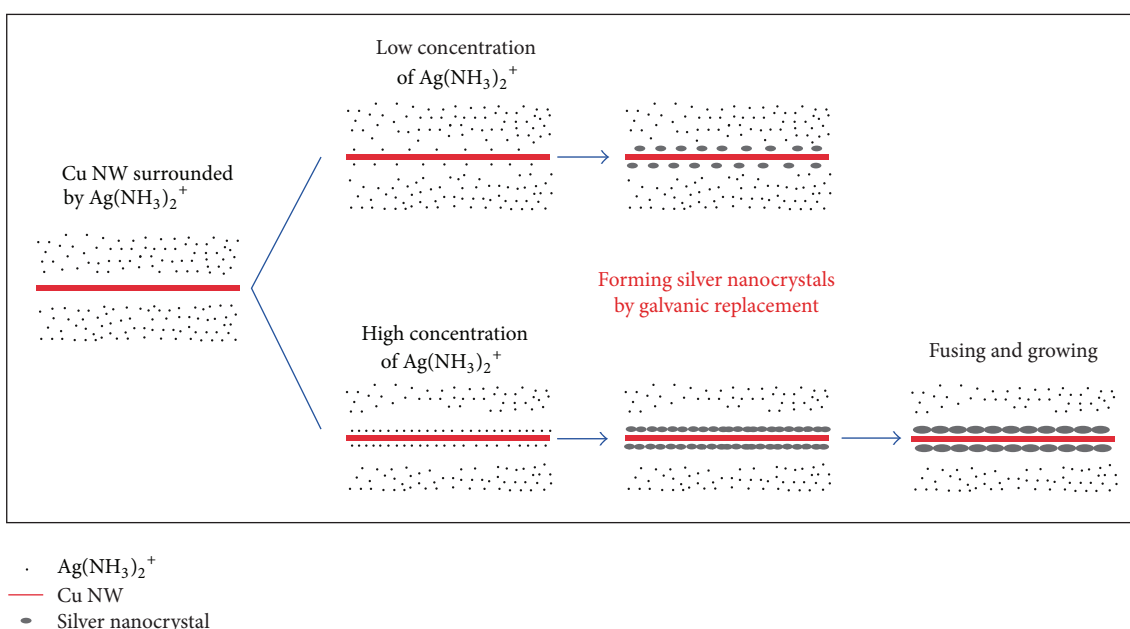
- · Ag$(NH_3)_2^+$
- — Cu NW
- • Silver nanocrystal

FIGURE 5: Schematic diagram of a possible growth process of silver-coated Cu NWs.

on the Cu NW surface, and initial smooth surface of Cu NW appears to be relatively rough. The element mapping images of one selected silver-coated Cu NW are displayed in Figure 4(c), in which the purple and yellow dots represent elements Cu and Ag, respectively. The images reveal that the straight nanowire consists of principally copper, and silver nanocrystals are decorated on the surface of the nanowire. The TEM results are consistent with the above SEM analysis.

Figure 5 illustrates a schematic diagram of the synthesis of silver-coated Cu NWs with low and high Ag$(NH_3)_2^+$ concentration, respectively. The Cu NWs were initially introduced into silver-ammonia solution, thus surrounded by many Ag$(NH_3)_2^+$ ions. The adsorbed Ag$(NH_3)_2^+$ ions on the surface of Cu NWs could be reduced to metallic silver via galvanic replacement. The large strain generated at the Cu-Ag interface can provide driving force to form silver nanocrystals. The numbers and sizes of the nanocrystals synthesized with a high concentration of Ag$(NH_3)_2^+$ ions are larger than those that are synthesized with a low concentration of Ag$(NH_3)_2^+$ ions. It can be considered that high concentration of silver source provides more possibility for adsorption or collision between the Ag$(NH_3)_2^+$ ions and Cu NW surface during the same reaction time, resulting in more silver nanocrystals formed. In this case, a lot of crystal boundaries are generated among the nanocrystals. The small nanocrystals can fuse and further grow to form one with a larger size. Certainly, if the reaction time is further extended, the numbers and sizes of the nanocrystals with a low concentration of Ag$(NH_3)_2^+$ ions can also be considerably increased.

4. Conclusion

In summary, uniform coating of the silver nanocrystals on the Cu NW surface could be synthesized by facile galvanic replacement under vigorous oscillation. Good dispersibility of the pure Cu NWs is important to improve the distribution of the silver nanocrystals. The silver ions are reduced to

metallic silver on the surface of the Cu NWs to generate large strain at the Cu-Ag interface. The strain can provide driving force to form silver nanocrystals. The resistance of oxidation to the Cu NWs can be effectively improved by depositing silver nanocrystals. Our work suggests a facile, fast, and low-cost way to synthesize desired heterostructures consisting of different metals.

Competing Interests

There are no competing interests related to this paper.

Acknowledgments

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