

Research Article

Synthesis of One Dimensional Gold Nanostructures

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Gold nanostructures with shapes of rod, dumbbells, and dog bone have been fabricated by an improved seed-mediated method. It is found that the pH change (the addition of HNO₃ or HCl) and the presence of Ag⁺ ions have a great influence on the growth process and aspect ratios of these Au nanocrystals. UV-Vis-NIR absorption spectra for the Au colloidal show that the transverse plasmon absorption band locates at ~520 nm, while the longitudinal plasmon absorption band shifts in a wide spectra region of 750–1100 nm. The obtained Au nanostructures have been investigated by transmission electron microscopy, high-resolution transmission electron microscopy, and X-ray diffractometer. Based on the characterizations and FDTD simulations, most of the obtained Au nanorods are single crystals, possessing an octagonal cross-section bounded by {110} and {100} faces. One model for the anisotropic growth has been proposed. It is found that slow kinetics favor the formation of single-crystalline Au nanorods.

1. Introduction

Noble metallic nanostructures are of great interest due to their unique properties and promising applications in the fields of optics, electronics, magnetism, and catalysis [1–6]. Pt nanoparticles, for instance, were applied as catalyst for the hydrogenation of organic and for selective reactions depending on the crystallographic planes exposed by their surfaces [5, 6]. For the case of optical properties, absorption associated with the collective oscillation of the conduction electrons or surface plasmon resonance (SPR) has been studied for many decades. In recent years, several special shapes and structures have been studied for Au nanoparticles, such as nanorings, nanoplates, dog bones, and nanoprisms. At present, the SPR absorption for arbitrary geometries can be theoretically calculated by discrete dipole approximation or finite difference time domain (FDTD) solutions (FDTD supplies a simple, convenient, and systematic approach to calculate the optical response of a nanostructure with arbitrary symmetry and geometry by solving Maxwell's equations on discrete grids). Both experimental and theoretical studies show that the number and position of the plasmon resonances as well as the spectral

range for surface-enhanced Raman scattering (SERS) of metal nanostructures have a strong correlation with their exact morphology and the aspect ratios [1, 7–12]. For example, cylindrical Ag nanowires show one resonance whereas several resonances are expected for Ag nanorods with triangle and other profiles [8, 9]. Two distinctive plasma resonances usually appear in the optical absorption spectra. In addition to a weak transverse surface plasmon resonance (SPR_T) roughly in the visible spectral region (500–530 nm with different solvents), a strong longitudinal surface plasmon resonance (SPR_L) shifts from the visible to the near-infrared (Vis-NIR) region with increasing aspect ratios of Au nanorods [10–12]. This strong polarization sensitivity of SPR absorption in the NIR region is an efficient converter of photon energy to thermal energy, which opens new possibilities for many attractive applied fields. Due to their facile synthesis, ease of functionalization, biocompatibility, and inherent nontoxicity, Au nanoparticles are being developed as ideal biological applications, such as gene delivery, cell imaging, photothermal therapy, and anticancer drug-delivery technology [1, 13–17]. More recently, it is reported five-dimensional (the wavelength, polarization, and 3 spatial dimensions) optical recording mediated by the SPR of Au

nanorods, which allow multiple patterns being stored in the same volume [18]. Under irradiating recording layers doped with Au nanorods of different aspect ratios, the selected nanorods with a certain SPR_L on resonance with the laser light wavelength and polarization would melt. When the readout laser light is implemented, the contrast exhibits.

For fundamental and applied interest, study on the well-controlled shapes and novel structures of Au nanostructures, therefore, has become a very important issue. For one dimensional Au nanorods, different approaches have been demonstrated for synthesizing Au nanorods, including hard templates (such as Al_2O_3 membrane and mesoporous silica) assistant deposition approach [12, 19], electrochemical or photochemical reduction with surfactants [20, 21], and seed-mediated methods [22]. Recently, some groups reported the synthesis of branched and multipod-shaped metal nanostructures with sharp edges and corners [23–27]. Calculation on the fields around nanoparticles show that surface charges are accumulated at the sharp corners that exhibit strong enhancement of an electromagnetic field [28], which makes the nanostructure an excellent candidate as SERS substrates. At the same time, contrast studies on the SPR of these quasi-one-dimensional Au nanostructures demonstrate that the shape and position of the SPR_L was not only sensitive to the aspect ratio but also influenced by additional details of the rod shape and in particular by the natural transition from cylindrical to flared, dog bone, bipyramidal, and dumbbell [29–32].

Here, we report the synthesis of Au nanorods, dumbbells, and dog bone through an improved seed-mediated at room temperature. It is found that the addition of acid, Ag^+ ions and a second surfactant is crucial in both improving the shape and controlling the aspect ratios of Au product. A growth model was proposed based on the results.

2. Experimental Section

2.1. Materials. Hydrogen tetrachloroaurate ($HAuCl_4 \cdot 4H_2O$) was purchased from Shanghai Chemical Reagents Company. L-ascorbic acid (AA, 99.7%) and sodium borohydride ($NaBH_4$, 96%) were obtained from Sinopharm Chemical Reagent Company. Cationic cetyltrimethylammonium bromide (CTAB, 99%) and benzyltrimethylhexadecyl ammonium chloride (BDAC) were obtained from Nanjing Robiot Company. All other reagents were used without further purification. Deionized water used throughout the experiments was purified by a MilliQ system (18.25 M Ω).

2.2. Synthesis. Seed solutions were generated firstly with a strong reducing agent ($NaBH_4$), followed by preparation of a growth solution with a weaker reducing agent (AA) to grow Au nanorods. In the improved process, chemicals of HNO_3 , $AgNO_3$, and BDAC were applied in the growth solution for the understanding of growth mechanism of final product. The details of the process are the following.

2.2.1. Synthesis of Au Seeds. 10 mL CTAB (with molar concentration $[CTAB] = 0.1$ M) was mixed with 0.05 mL $HAuCl_4$

solution ($[HAuCl_4] = 0.05$ M). Then, 0.6 mL $NaBH_4$ ($[NaBH_4] = 0.01$ M) was injected into the solution under vigorous stirring. The as-obtained seeds are very active indicated by the color change from brownish to red within one day.

2.2.2. Synthesis of Au Dog Bones, Dumbbells, and Nanorods. CTAB was dissolved into 10 mL deionized water ($[CTAB] = 0.1$ M) as growth solution. Then, solutions of 0.2 mL $AgNO_3$ ($[AgNO_3] = 0.004$ M) and 0.1 mL $HAuCl_4$ ($[HAuCl_4] = 0.05$ M) were added to the growth solution. When 0.06 mL AA ($[AA] = 0.1$ M) was added, the solution color changed from orange to colorless. Finally, 12 μ L of the as-prepared seed solution was injected into the growth solution. Under this condition, Au nanostructures with shapes of dog bones and dumbbells were synthesized. In order to fabricate Au nanorods, different amount (0.1 mL, 0.2 mL, 0.3 mL, and 0.4 mL) of acid (HNO_3 or HCl with concentration of 1 M) was employed in the growth solution. When BDAC was used as a cosurfactant, Au nanorods with large aspect ratios were obtained.

2.3. Characterization. The absorption spectra of the prepared samples were collected using a UV-Vis-NIR spectrometer (SP-752PC) in the wavelength range of 200–1100 nm. The products were purified by centrifugation at 14000 rpm for 20 min. Precipitates were centrifugated repeatedly with deionized water. Then, the samples were deposited on copper grids covered by an amorphous carbon film, HRTEM grids, and glass slides for further measurements. Microscopic observations were carried out using transmission electron microscope (TEM: JEOL-100CX) and high-resolution TEM (HRTEM: JEOL-2011). XRD measurement was performed on a diffractometer (Ultima-III, Rigaku).

3. Results and Discussion

3.1. Microstructure and Optical Absorption of Au Nanostructures. As described in the experimental section, Au dog bones and nanorods were prepared, respectively, in the absence and with the presence of HNO_3 with CTAB as surfactant. Figure 1(a) shows the UV-Vis-NIR absorption spectra for the Au colloids of dog bones and nanorods. In contrast to Au nanorods, the SPR_T of dog-bones is wide. Upon addition of HNO_3 , the SPR_L position changes from 767 nm to 860 nm, indicating the aspect ratios of Au nanorods increase. It is found that the reduction rate of Au^{3+} ions decreases when acid (HNO_3 or HCl) is added in the growth solution, which facilitates the formation of Au nanorods with large aspect ratios. The structures of the products were characterized by TEM measurements. The SAED patterns indicate the single-crystalline nature of the obtained Au nanocrystals. The dog bone is measured to be 50 nm in average length and 11 nm in diameter for the middle section, and some of the products take the shapes of dumbbell and cube, as exhibited in Figure 1(b). The aspect ratio for Au nanorods in Figure 1(c) is ~ 4.5 . Usually, Au nanorods were available over Au dog bones by

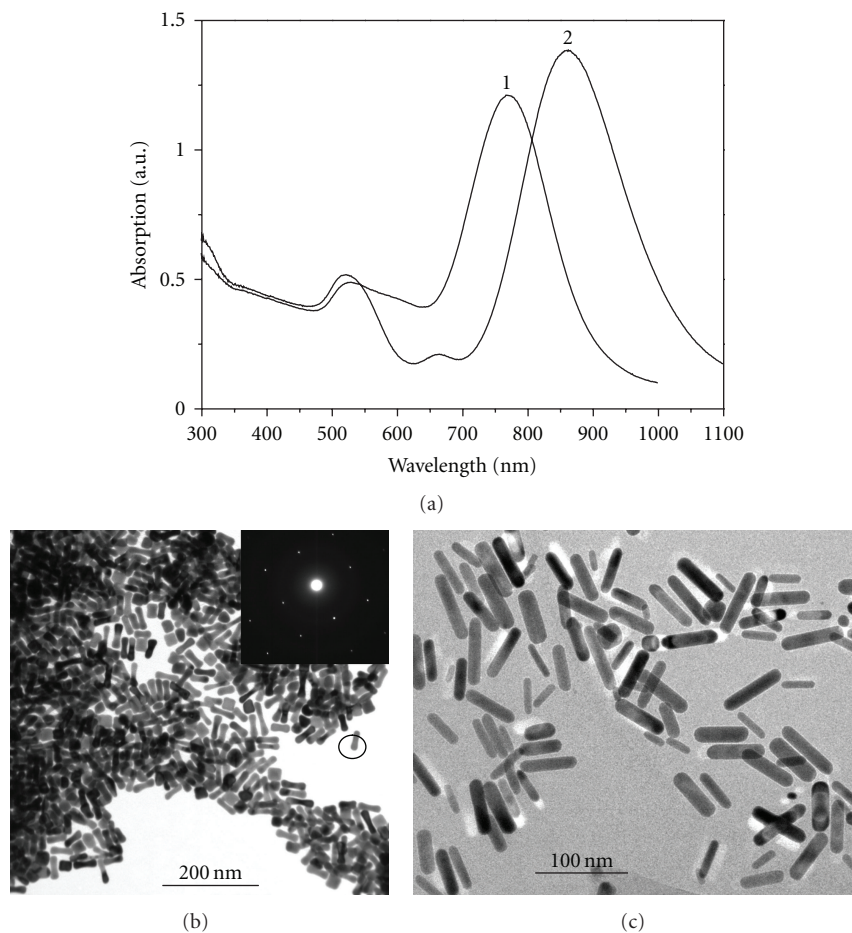


FIGURE 1: (a) Vis-NIR absorption spectra of Au dog bones (curve-1) and Au nanorods (curve-2) prepared in the absence and with the presence of HNO_3 , respectively. (b) and (c) TEM images and SAED pattern of Au dog bones and Au nanorods corresponding to spectra 1 and spectra 2, respectively.

decreasing the content of AA or increasing the content of “seed” solution even in the absence of acid [33, 34]. Instead of these two strategies, it confirms that the addition of HNO_3 is more efficient in modifying the dog bone in our current work. Additionally, the chemical CTAB purchased from different supplies would lead to quite different results; sometimes no Au nanorods were obtained [35]. This could be attributed to the difference of capping ability which significantly influences the morphology of the final products.

To produce Au nanorods with large aspect ratios, a surfactant mixture contained CTAB and BDAC ($[\text{BDAC}]/[\text{CTAB}] = 1.25$) was applied in the growth solution. Figure 2(a) shows the UV-Vis-NIR absorption spectra for the colloid of Au nanorods produced with the presence of CTAB/BDAC sampled at different stages. It is clear that the absorption intensity of Au nanostructure increases during aging, suggesting the increase of number and (or) volume of Au nanostructures. The red shift of the SPR_L position indicates that the aspect ratio of Au nanorods increases with time, which is opposite to the reported trend [36]. The recorded spectra also show that Au nanorods maintain their overall shapes during growth process, implying proper

rate of Au supply in the colloidal solution. Thus, proper reduction rate favors the formation of nanorods. For the growth of Au dog bones and dumbbells, the formation originates additional deposition of Au cluster at the ends of the as-formed nanorods which could act as seeds.

In our experiments, the presence of HNO_3 or HCl plays an important role in synthesizing Au nanorods. For fully understanding the role of acid, a series of syntheses were performed by altering the content of HNO_3 . As shown in Figure 2(b) for samples after addition of seed solution for ~ 24 h, the SPR_L band blue shifts with increase the content of HNO_3 from 0.1 to 0.4 mL, which offers a useful means to systematically control the shape and aspect ratios of Au nanostructures. Further results show that HNO_3 can slow down the growth rate effectively. For example, the solution became light red after 3 h when 0.2 mL HNO_3 was added, while 5 h was needed for this change with addition of 0.3 mL HNO_3 . When HCl was applied, similar results were observed. In the TEM image of Au nanorods, several Au nanocubes were also observed, together with some small nanoparticles as byproducts, as shown in Figure 2(c). The aspect ratio of these nanorods is estimated to be

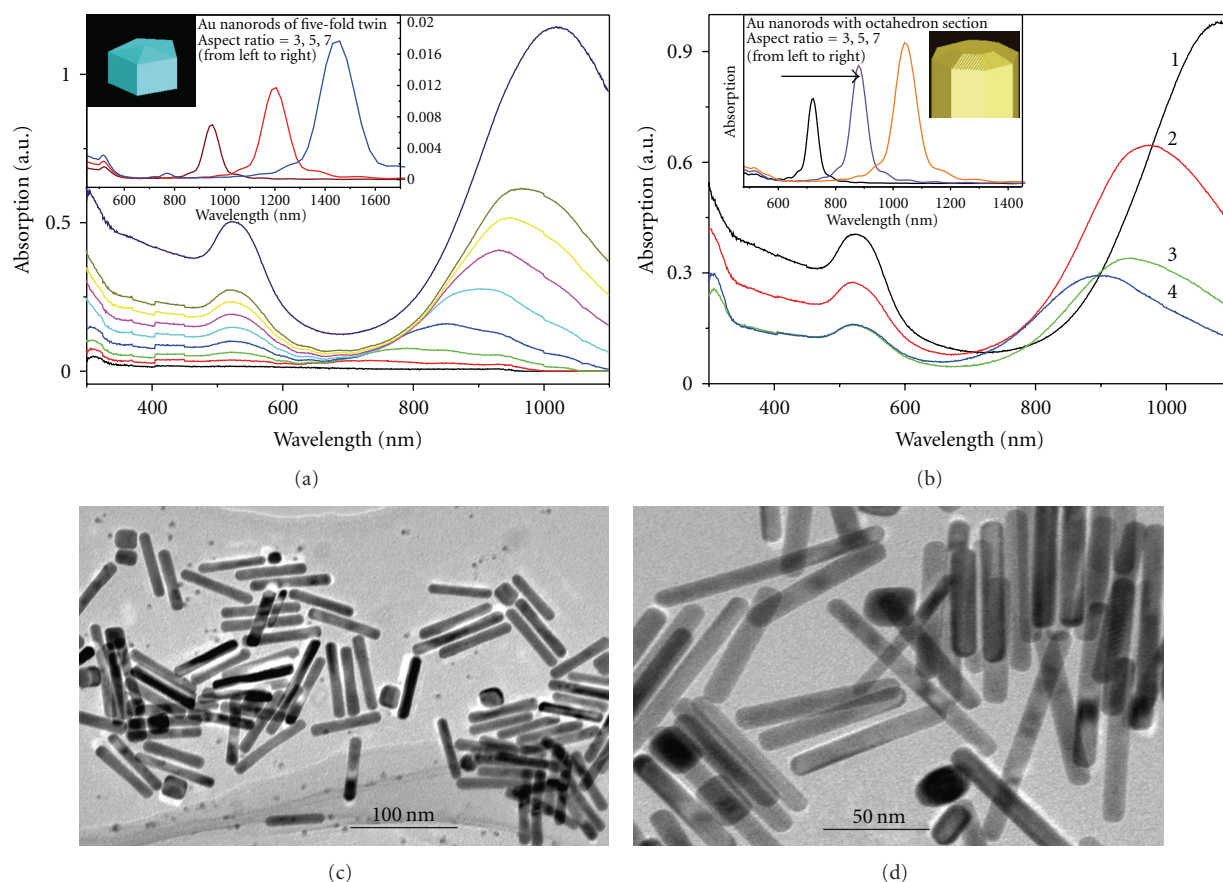


FIGURE 2: (a) Vis-NIR spectra of colloidal Au nanorods aged for various times. Spectra from bottom to top correspond to samples aged for 1–8 hour and 1 day. (b) Vis-NIR spectra of 4 identical growth solutions with increasing HNO_3 (curve-1: 0.1 mL, curve-2: 0.2 mL, curve-3: 0.3 mL, and curve-4: 0.4 mL). Insets of (a) and (b) are the calculated spectra using FDTD solution for Au nanorods with fivefold twin and octahedron cross section (water as medium). TEM images of Au nanorods prepared with HNO_3 (0.1 mL) (c) and HCl (0.2 mL) (d) in a binary surfactants of CTAB and BDAC.

~ 7 (the average lengths and diameters of nanorods are, resp., ~ 70 nm and ~ 10 nm for Figure 2(c) and ~ 84 nm and ~ 12 nm for Figure 2(d)), corresponding to the SPR_L band position at 1050 nm. Inserted in Figures 2(a) and 2(b) are the FDTD calculated optical absorption spectra for the fivefold twined Au nanorods and Au nanorods with octahedron cross section (with aspect ratios of 3, 5, and 7, using water as medium). The simulations indicate that the obtained Au nanorods are not five-fold twined crystal, but single-crystalline Au nanorods with octahedron cross section.

Figure 3 shows HRTEM images of representative Au nanorods and dumbbells. The SAED patterns indicate that these structures are single crystals. Au nanorods appear to grow along [001] direction with side faces bounded by {100} and {110} facets. Clear fringes parallel and perpendicular to the growth axis are shown in Figure 3(a). The fringe spacing is 0.200 nm, corresponding well with the lattice spacing of {200} planes for Au crystal (0.203 nm). Similar structures are found for Au dumbbells. Occasionally, the presence of {111} facets were observed at two ends of Au dumbbell, as detailed in Figure 3(b).

To further determine the structure characterization of the Au nanorods, an X-ray diffraction experiment was carried

out. Figure 4 shows the typical XRD result of one sample. In the XRD profile, four diffraction peaks can be indexed to fcc Au crystal though the background information is clear due to a very thin layer of Au nanorods covered on the quartz glass substrate. The XRD profile of the Au nanorods shows a strong (111) diffraction peak, and the intensity of the (200) diffraction peak is also strong. The enhanced (111) and (200) diffractions indicate that the stack and elongation of {111} and {100} facets mainly account for the crystals growth.

3.2. Growths of One Dimensional Au Nanocrystals

3.2.1. The Role of Halogen Ions. According to [37], ion additives, such as NaCl, KCl, and NaNO_3 , can serve as useful “tools” to tailor the shape, aspect ratio, and yield of Au nanorods. These additives caused the SPR_L band red shift within the critical concentrations. Ionic strength in colloidal solution has an effect on the size of soft template and suitable ionic concentration can improve micellar structure through reducing the repulsion between the neighboring head group of CTAB [38]. However, further increasing the ionic concentration may prompt a template transfiguration from rod to bone. A series of studies, concentrated on

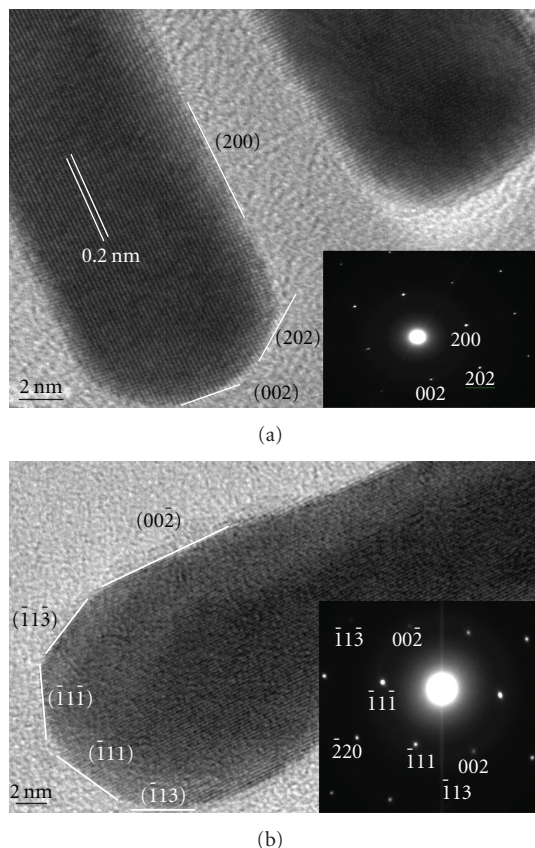


FIGURE 3: (a) HRTEM image of two Au nanorods recorded along [100] direction. The inset shows the corresponding SAED pattern. (b) HRTEM image of one Au dumbbell recorded along [110] direction. The inset shows the corresponding SAED pattern.

Br^- ions pointed out that Br^- ions aided the formation of structures covered with $\{110\}$ and $\{100\}$ facets [39, 40]. We suggest that the major function of halogen is chemisorption on Au crystal surface or chemical interaction between Au crystals and surfactant. Proper concentration of halogen ions avails the formation of single-crystalline Au nanorods with large aspect ratios.

3.2.2. The Role of Ag^+ Ions. Originally, the seed-mediated method without addition of AgNO_3 was reported by Jana et al. Au nanorods with high aspect ratio (~ 18) were obtained for citrate-capped seeds, but the yield was very low [41]. Then, this method was improved to increase the yield of Au nanorods through adding Ag^+ ions to the growth solution [42]. In addition to a high yield of Au nanorods, the SPR_L can be tuned in a wide wavelength range upon increasing the concentration of Ag^+ ions. Based on the soft template of CTAB, AgBr will be formed with addition of AgNO_3 , which would decrease the charge density of adsorbed CTAB molecules on Au facets and the consequent repulsion between the neighboring head groups, favoring the CTAB template elongation. Another model is that AgBr would adsorb preferentially on special facets and direct the rod-shape growth [33, 39]. Therefore, ideas can be expected

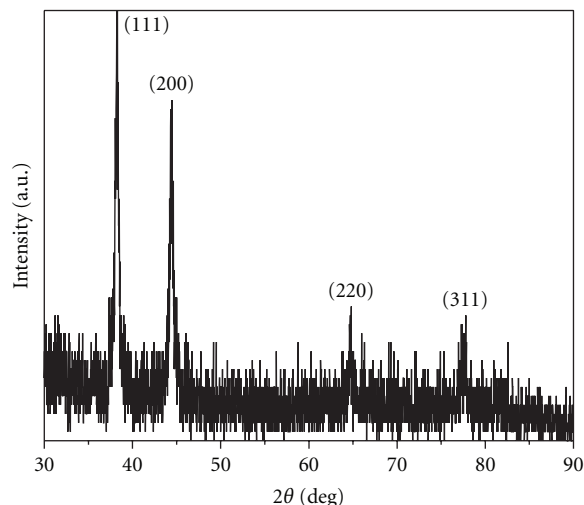


FIGURE 4: X-ray diffraction profile of the as-prepared Au nanorods.

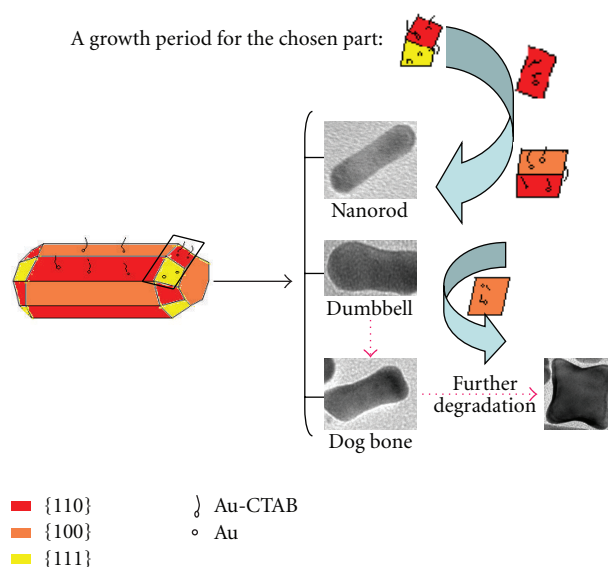


FIGURE 5: Schematic illustration of proposed growth mechanism responsible for the formation of nanorods, dumbbells, and dog bones.

for the application of AgNO_3 . (i) AgBr adsorbs on the single crystalline Au “seed”, promoting anisotropic growth into nanorods. (ii) AgNO_3 has an effect on making Au nanorods stable and protecting them from evolving into other shapes. Otherwise, when no AgNO_3 was introduced, the growth solution turned red within 1 min after the addition of “seed” solution, and the product is dominated by Au nanospheres. In the experiment results, no Ag nanostructures were obtained. This should ascribe to the fact that Ag^+ cannot be reduced in the slightly acidic growth conditions (due to the fact that any AuCl_4^- present is an oxidant with respect to $\text{Ag}(0)$).

3.2.3. The Role of CTAB and BDAC. Undoubtedly, the key factor for prompting the formation of Au nanorods is the

molecules of CTAB. CTAB possesses an ammonium head-group and a hydrocarbon tail which maybe a driven force for the combination between Au and CTA⁺. CTAB forms micelles above its critical micelle concentration. It is widely accepted that CTAB prefer to bind to the side surface of Au nanorods through Br⁻ ions chemisorbing on the side surfaces [43]. Thus, the side surface is well protected from further deposition. When all conditions remained the same except for the length of the surfactant tail, Au nanorods with high aspect ratios were obtained upon increasing the length of surfactant chain [39, 44]. Others proposed that CTAB micelles acted as a soft template for controlling the shape and size of particles. Au atoms reduced were trapped by the preformed templates, leading to the formation of Au nanorods [37, 38]. In our experiment, solutions of CTAB and CTAB/BDAC were prepared and kept at 10°C. CTAB precipitate was observed in the solution within 30 min, but it took much longer time for the appearance of CTAB precipitate in the mixture solution. This observation demonstrated that the solubility of CTAB is improved dramatically in the CTAB/BDAC mixture solution, and that the templates became more flexible, as evidenced by the elongation of Au nanorods upon aging. Moreover, it is found that the growth of Au nanorods is more time saving in the single-component surfactant than in the binary surfactant mixture.

3.2.4. Growth Model for Different Shapes. For the case of crystal growth in solution phase, the final structure of crystal is determined by both kinetic and thermodynamic effect [9, 45–47]. It is known that thermodynamic control always attempts to minimize the total interfacial free energy of a crystal. However, in the chemical synthesis with the presence of surfactant, the slow reduction rate of Au³⁺ ions provides the possibility for the highly anisotropic growth. The coverage of {110} facets on nanorods seems to deviate from thermodynamic control. This should ascribe to that the surfactant CTAB molecular usually binds to {110} facet with preference, and that the Au atom spacing on the side faces ({110} or {100}) is comparable to the size of the CTA⁺ headgroup [39, 48]. Therefore, the single-crystalline Au nanorods have an octagonal cross-section covered by flat {100} facets (the truncated corners are bounded by {111} and {110} facets) with sidewalls of {110} and {100} facets.

In our postulation, the free Au and Au-CTAB complex coexist in the growth solution with different affinity to crystal facets. The Au-CTAB complex are preferred to the {110} facets at the corner of Au rods, while the free Au to the {111} facets. The deposition rate of free Au on {111} facets is faster than that of the bonded Au on {110} facets, which results in the elongation of {110} facets. The growth mechanism of these Au nanostructures can be depicted as schematically illustrated in Figure 5. The {110} facets are not thermodynamically stable and easily evolve into {100} facets with the aid of surfactants, forming the octagonal cross-section covered by both {110} and {100} facets. With increasing the kinetic rate, the rods change from a cylindrical form to a dumbbell and further to dog-bone. A large kinetic rate facilitates the appearance of {100} facets and thus, dog

bones dominate the products and then degrade into the cubes.

4. Conclusions

In summary, we conducted the synthesis of single-crystalline Au nanorods, dumbbells, and dog bones in solution phase that contains surfactants with the addition of acid. Through comparing the results, we can see that changes of PH value (by adding HNO₃ and HCl) can modify the morphology and tune the aspect ratios of the final products. The SPR_L of Au nanorods can be tuned in a large spectra range of 750–1100 nm, which is important for the potential applications related to this property. A growth model is proposed, in which kinetics and thermodynamics are employed.

Acknowledgments


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