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Journal of Molecular Structure

journal homepage: www.elsevier.com/locate/molstruc

Self-assembled synthesis of Ag nanodendrites and their applications to SERS

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A R T I C L E I N F O

Article history: Received 23 January 2011 Received in revised form 23 April 2011 Accepted 25 April 2011 Available online 5 May 2011

Keywords: Self-assembly Ag nanodendrites SiO₂ micro reactor SERS

ABSTRACT

In this paper, SiO₂ nanoparticles were synthesized using self-assembly for their fabrication. The SiO₂ particles as a micro reactor were soaked into silver nitrate (AgNO₃) aqueous solution. The dendritic Ag nanoparticles were prepared by a reaction of AgNO₃ with template agent (CTAB) and AgBr decomposed into elemental Ag nanoparticles under visible light irradiation on the SiO₂ micro reactor. The morphology and structure of synthesized Ag dendrites were determined by employing following techniques: X-ray diffraction (XRD), scanning electron microscopy (SEM), transmission electron microscopy (TEM), ultraviolet visible spectroscopy (UV-vis). The correlated surface-enhanced Raman scattering (SERS) effects have been investigated when 2-Mercaptobenzothiazole (2-MBT) was used as a Raman probe. Especially, the possible growth process was proposed by arresting the growth at a series of intermediate morphology stages during the shape evolution of Ag dendrites based on TEM observations. The results clearly showed that the dendritic Ag nanostructures could be obtained through oriented attachment of nanoparticles along a crystallographically special direction and the morphology of the dendrites was dependent on the reaction duration. Moreover, the investigation of the SERS revealed that the Ag nanodendrites were an excellent substrate with significant enhancement effect.

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1. Introduction

During the past decades, fabrication of noble metal nanostructures with small uniform size and morphologies have become a central issue in modern material chemistry research because they have potential applications in electronics, catalyst, optical and sensing properties, etc. [1–5]. Generally, the noble metal nanoparticles were synthesized on some supporting materials, such as inorganic oxide (silica, titanium oxide) [6,7]. Recently, novel hierarchical selfassembly technique constructed by nanorods, nanowires, nanorings and nanodendrites have attracted considerable attention [8–11]. The preparations of nanostructures of the noble metals are mostly based on reduction reaction by using a reducing agent. Dendritic nanostructures have received scientists' interest owing to their attractive supramolecular structures [12-14], and the deneritic metal nanoparticles have been shown to have potential applications in catalysis, chemical, photonic crystals, plasmonics and surface-enhanced Raman scattering-based analytical devices and so forth [15–18]. There have been many discussions about the various factors affecting the formation mechanism of the dendritic nanostructure. Many scientists think that the dendrites are produced by the hierarchical assembly under non-equilibrium growth conditions [19]. As a result, much attention has been paid to their preparation and a large amount of preparative methods have been developed [20,21]. The synthesis of these nanodendrites has implication for the fundamental study of the growth process and shape control.

At present, most of the dendritic nanostructures were prepared by using wet-chemical routes in solutions [22] or polymers or by electro deposition methods [23]. In this paper, the mono-disperse, highly crystalline and nanotextured Ag dendritic nanoparticles, about several micrometers in size are synthesized from a selfassembly process by soaking in aqueous AgNO₃ at room temperature. The sample has been applied in the surface-enhanced Raman scattering enhancement. Compared to the reports referred above, the reaction does not demand any additional reagents. This work demonstrates that a facile method has been developed to prepare the Ag nanoparticles with interesting dendritic morphologies and the possible dendritic formation mechanism is discussed. The Raman scattering enhancement efficiency of the Ag nanodendrites is investigated by using 2-MBT as the model Raman probe and the results shown that these dendrites can effectively enhance the Raman signal of 2-MBT.

2. Experimental section

2.1. Chemicals

Tetraethyl orthosilicate (TEOS), sodium hydroxide (NaOH), cetyl trimethyl ammonium bromide (CTAB), gelatin, silver nitrate



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(AgNO₃), 2-Mercaptobenzothiazole (2-MBT) and other chemicals were used as received. All the chemicals were obtained from Nanjing Chemical Reagents. These were of analytical grade and the deionized water was used for all types of sample preparations.

2.2. Synthesize of Ag nanodendrites

The SiO₂ nanoparticles were first prepared by self-assembly techniques with precursors: TEOS, NaOH, CTAB and gelatin. 0.7 g of TEOS and 0.2 g NaOH were mixed with 12.5 mL deionized water in a 25 mL beaker by magnetic stirring at 25 °C for 25 min. Then this colorless and clear solution was transferred to the sample trough. Subsequently, a solution having 0.55 g of CTAB (0.55 g), 0.15 g of gelatin in 22 mL water was poured gently over that colorless solution. The container was sealed and left undisturbed at 21 °C for 48 h until a film was observed at the air–water interface. This as-prepared SiO₂ film was transferred to glass substrates and soaked into 0.05 mol·L⁻¹ silver nitrate (AgNO₃) aqueous solution. The formation of the dendritic nanoparticles occurred subsequently.

2.3. Characterization of the materials

X-ray diffraction (XRD) analysis was carried out on a Bruker D8 Advance diffractometer using Cu K α radiation (λ = 0.1542 nm). Scanning electron microscope (SEM) of JEOL-6380 LV and energy dispersive spectrometer (EDS) with EDAX Genesis 2000 were applied to detect the sample. Transmission electron microscopy (TEM) measurements were made on a JEOL-2100 microscope operated at an accelerating voltage of 200 kV and a Gatan 794 chargecoupled device (CCD) camera. Samples for TEM examination were made by placing a drop of the dispersion of the deionized water on a carbon-coated copper grid after ultrasonic dispersion for 10 min and air-dried at room temperature. UV-vis absorption and transmission spectra were obtained using a Beijing Eraic UV-1201 spectrometer in the range of 300-700 nm at room temperature. The Raman spectra were recorded on a Renishaw Invia Raman microscope excited by an argon ion laser beam (514.5 nm, 20 mW). The laser power at the sample position was 10 mW with a spot size of ca. 1–2 μ m. The data acquisition time used in the measurement was 25 s. Replicate measurements on different areas were made three times to verify that the spectra were a true representation of each experiment.

3. Results and discussion

3.1. The XRD patterns of these nanoparticles

The low-angle (2.1–7°) X-ray diffraction patterns of SiO₂ nanoparticles at room temperature before and after soaking in AgNO₃ solution are shown in Fig. 1. As seen from Fig. 1, a predominant peak can be observed for two samples. In addition, there is no obvious reflection in the range from 7° to 30° for these samples (data not shown), suggesting these as-prepared SiO₂ nanoparticles are amorphous components. According to some literature [24], the recorded *d* value corresponding to the diffraction peak located at $2\theta < 10^{\circ}$ suggested that a well-ordered structure i.e. spacing between lamellar nanostructures, was present in this synthesized oxide particles. The predominant peak of SiO₂ particles (a) is appeared at $2\theta = 2.6^{\circ}$ with a *d* spacing of 3.39 nm. In comparison with the XRD pattern of the two samples, the Bragg peak of SiO₂ nanoparticles after soaking (b), initially at $2\theta = 2.5^{\circ}$, corresponds to a d spacing of 3.46 nm. The diffraction peak of SiO₂ samples shifts to higher angles marginally with an increase in Ag contents after soaking, the *d* spacing is a bit larger for the SiO_2 particles.

Fig. 1. LAXRD patterns of SiO₂ nanoparticles before and after soaking.

A typical X-ray diffraction (XRD) pattern of the synthesized nanodendrite is shown in Fig. 2. It is found that a very broad peak around $2\theta = 22^{\circ}$ corresponds to the amorphous silicon, indicating the self assembly SiO₂ in existence. The result is consistent with the analysis of Fig. 1. It exhibits peaks at 2θ angles of 38.2° , 44.4° , 64.4° and 77.3° , which are assigned to the $(1\ 1\ 1)$, $(2\ 0\ 0)$, $(2\ 2\ 0)$ and $(3\ 1\ 1)$ reflections of the face-centered cubic (f.c.c) structure of metallic Ag, respectively. The lattice parameter calculated from the XRD pattern is 0.4078 nm, in agreement with the literature report (0.4078 nm, JCPDS File No. 04-0784). It is worth noting that the intensity ratio of the $(2\ 0\ 0)$ and $(2\ 2\ 0)$ diffraction peaks are much lower than the conventional value (0.2 and 0.12 versus 0.36 and 0.2), confirming that these Ag particles are primarily dominated by $(1\ 1\ 1)$ facets [25], indicating that the dendrites are crystalline Ag which can be obtained by soaking in solution.

3.2. The morphology of Ag nanodendrites

The morphology of the synthesized dendritic Ag nanostructure is characterized by SEM. The image of these particles is shown in Fig. 3a which indicates that they are mono-dispersed and about $1-2 \,\mu\text{m}$ in size having dendritic shape. The dendrites are composed of a main trunk, several plump branches and numerous leaves, which look like leaves with multilevel nanostructures. From



Fig. 2. HAXRD patterns of SiO₂ nanoparticles after soaking.





Fig. 3. SEM images and EDS spectra of Ag nanodendrites (soaked 10 min), the Pt signals come from the plated element.

Fig. 3b, it can be seen that the composition of the Ag nanodendrites are checked using EDS based on SEM images. EDS analysis reveals that the composition of the dendrites mostly consists of C, O, Si, Pt and Ag. The detailed element contents are shown in the table. The Pt signals come from the plated element. The peaks for Si and Ag can be discerned in the spectra of the composite particles (Fig. 3b), indicating that Ag nanodendrites have been successfully assembled by using the air–water interfacial SiO₂ particles as micro reactor.

TEM analysis provides more information about the microstructure of the Ag nanodendrites. In order to understand the growth mechanism of Ag dendrites, the typical TEM images of the products obtained by different reaction durations are shown in Fig. 4. Fig. 4a and b depict that the TEM images of the numerous small nanoparticles, clearly indicating that the sample possesses a small spherical nanostructure at the beginning of the reaction. At low magnification, the non uniform spherical nanoparticles with a diameter of 10-20 nm are recognized. When the reaction time is over 10 min, Fig. 4c and d are the TEM image of Ag nano cluster surface with SiO₂ as micro reactor, which have a leaf-like character. Fig. 4c is showing that the overall appearance of the sample is connected in networks. An amplified image (Fig. 4d) clearly shows the presence of leaf-like structure. With an increase in the reaction time from 10 to 30 min, the nano clusters began to transform into dendrites, as shown in Fig. 4e, which have a wheatear-like character and crystalline structure. When the reaction time increases to 1 h, the products become elegantly dendritic, indicating that the growth process of Ag nanodendrites is completed, which is showing a hierarchical structure composed of trunks, branches and leaves. It is clear that these nanodendrites are symmetric, and the angles between the stems and the branches are approximately 60°. The appearance of lattice orientation of Ag in the inset HRTEM image in Fig. 4f also indicates the formation of crystals and the dendritic products which grow in a random pattern with lattice spacing of approximately 0.40 nm, corresponding to the distance between two (1 1 1) crystal planes of the Ag structure. Therefore, the hierarchical structure of the Ag nanocrystals is consistent with the XRD results.

3.3. The UV-vis spectra of Ag nanodendrites

For the sake of comparison, the UV-vis absorption spectra of Ag nanodendrites at different periods obtained through soaked reaction are shown in Fig. 5. The spectrum measured for bare silica colloids is featureless (curve e), but the nanoparticles after soaking display sharp absorption peak due to the Mie plasmon resonance excitation from the Ag nanoparticles during the whole process [26]. The peak appearing at 398 nm depicts the existence of the Ag nanoparticles at soaked time for 10 min (Fig. 5a), it is related well to the surface plasmon absorption of these Ag⁰ particles [27]. The peak at around 435 nm is attributed to the AgBr, which is prepared by a reaction of Ag ions and Br ions from CTAB. As the reaction time increases, the plasmon resonance peak is redshifted gradually [28] and the peak of AgBr is disappearing gradually due to much larger nanoparticles. The AgBr decomposes into elemental Ag particle under visible light irradiation as indicated in Fig. 5 (curve b and c). The shift can be interpreted as the result of nano-size effect [29]. The absorption peak at 408 nm is ascribed to the presence of the dendritic Ag nanoparticles in 6 h (curve d). It is shown that the growth process of Ag nanodendrites completed in 1 h, which is agreed with the results of TEM.

3.4. Formation mechanism

Based on these experimental results, we believe that the electrostatic interactions and oriented attachment may play an important role in the formation of these nanodendrites. Therefore, a possible crystal growth mechanism for the formation of Ag nanodendrites can be proposed and illustrated in Fig. 6. First, two CTAB molecules is coupled through Vander Waal's forces to produce a dimeric bar, which served as a new template [30]. The SiO₂ particles from its precursor hydrolysis carry negative charges in the strong alkaline condition, which is interacting with the positively charged CTAB head groups through electrostatic interactions. As a micro reactor, this template of SiO₂ plays a key role in the following assembly processes [31]. Second, the SiO₂ air-water interfacial particles, which are soaked into the AgNO₃ aqueous solution, the AgBr–SiO₂ nanocomposite particles can be prepared by a reaction of Ag⁺ and Br⁻ ions from CTAB. Then a traditional photographic exposure process of black-white film is employed to generate elemental Ag. Namely, AgBr decomposes into elementary Ag nanoparticle under visible light irradiation and produces perfect single crystallites Ag nanostructures [32]. Third, the small Ag nanoparticles can move freely into the aqueous solution. There is a rotation between the particles driven by Brownian motion and the shortrange interactions by particles sharing a common crystallographic orientation and forming a coherent particle-particle interface during this process. The result leads to the reduction of total energy of the solid-liquid interface and an increase in entropy [19]. When surfaces are not atomically flat, coherence achieved by lattice distortion in the interface, which lead to the formation of dislocation between the neighboring particles, an imperfect oriented attachment can also occur [8]. At the same time, coarsening of the



Fig. 4. TEM images of dendritic Ag nanostructures obtained at different reaction durations: (a and b) 2 min; (c and d) 10 min; (e) 30 min; (f) 1 h. The inset is an HETEM image of Ag.

crystallites occur in this case. These Ag nanoparticles subsequently aggregate through the oriented attachment mechanism.

3.5. Surface-enhanced Raman scattering effect

The spherical Ag nanoparticles of self-assembly technique have been employed extensively for SERS studies because they exhibit excellent SERS enhancement [33]. To demonstrate the SERS enhancement ability of Raman spectral intensity for the dendritic Ag substrate, a SERS spectrum of the dendritic Ag is compared by using 2-MBT as a probe. The spherical Ag nanoparticles are prepared by the same method without adding the precursor (TEOS). Both the samples are prepared by immersing into 10^{-5} mol·L⁻¹ aqueous solution of 2-MBT. After 24 h, the samples are removed and measured. Fig. 7 shows the Raman spectrum of 10^{-5} mol·L⁻¹ 2-MBT solution (curve a), the SERS spectra of 10^{-5} mol·L⁻¹ 2-MBT adsorbed on the spherical Ag nanoparticles (curve b) and 10^{-5} mol·L⁻¹ 2-MBT adsorbed on the Ag dendrites (curve c). It is found that 10^{-5} mol·L⁻¹ 2-MBT solution alone cannot give any Raman signals. Fig. 7b and c are showing comparison of SERS spectra of 2-MBT on the sphere Ag with the dendritic Ag. The SERS spectrum of the 10^{-5} mol·L⁻¹ 2-MBT molecules adsorbed on the Ag dendrites is shown in Fig. 7c. In curve c, four relative strong Raman peaks and several weak peaks are observed. As expected, the band at 1321 cm⁻¹ is attributed to in-plane C–C stretching vibrations, the characteristic strong bands at 1447, 970 and 1583 cm⁻¹ are



Fig. 5. UV-vis spectra of Ag nanodendrites: (a) 10 min; (b) 30 min; (c) 1 h; (d) 6 h; (e) bare silica colloids.

assigned to the frame vibration and the stretching vibration with aromatic ring modes [34], respectively. The peak at 745 cm^{-1} is corresponding to the C-H bond out of plane bending mode. The band at 1024 cm^{-1} is probably a succinimidyl N-C-S stretch overlapping with aromatic ring modes [35]. It is shown that the dendritic Ag is higher than that of the spherical Ag nanoparticles in SERS enhancement ability. The significant difference in the enhancement ability between these different kinds of nanoparticles may be explained by the electromagnetic mechanism (EM). It can be seen from Fig. 4 that the roughness of the dendritic Ag nanoparticles show stronger absorption than the spherical at the excitation wavelength (514.5 nm). The size of dendritic Ag is in the range of 10-100 nm (Fig. 4a), which is the suitable size for the EM enhancement. Therefore, the dendritic Ag nanoparticle gives rise to a stronger resonance enhancement effect. Thus, the difference of the structure leads to the difference in the enhancement ability of the two kinds of nanoparticles. It is clear that the Raman signals of 2-MBT adsorbed on the Ag dendrites are enhanced remarkably relative to 2-MBT solution. In general, the structure and morphology of Ag nanoparticles are the key factors for strong SERS effect [36].

Fig. 8 shows that SERS spectra of 2-MBT in the range of 10^{-6} – 10^{-10} mol·L⁻¹ are measured on the Ag dendrites in the same scan time. The figure presents the SERS spectra recorded for various



Fig. 7. Raman SERS spectra of (a) 10^{-5} mol·L⁻¹ 2-MBT solution, (b) 10^{-5} mol·L⁻¹ 2-MBT adsorbed on the spherical Ag nanoparticles and (c) 10^{-5} mol·L⁻¹ 2-MBT adsorbed on the Ag nanodendrites.



Fig. 8. SERS spectra of 2-MBT at different concentration adsorbed on the Ag nanodendrites (a: $10^{-6} \text{ mol} \cdot L^{-1}$; b: $10^{-7} \text{ mol} \cdot L^{-1}$; c: $10^{-8} \text{ mol} \cdot L^{-1}$; d: $10^{-9} \text{ mol} \cdot L^{-1}$; e: $10^{-10} \text{ mol} \cdot L^{-1}$).

concentrations of 2-MBT solution in the region from 250 to 2000 cm^{-1} . The SERS spectrum of $10^{-10} \text{ mol}\cdot\text{L}^{-1}$ 2-MBT solution



Fig. 6. Formation mechanism of dendritic Ag nanostructures.

on the dendrites displays no sensitivity (curve e in Fig. 8), which means that the 2-MBT molecule cannot be detected on Ag dendrites. So, it is confirmed that 10^{-10} mol·L⁻¹ is determined to be the lowest detection limit. The figure reveals a steady increase in SERS intensity or peak height of the 2-MBT Raman band is observed with increasing 2-MBT concentration. These indicate that the morphology of the nanocrystals play a critical role in the SERS enhancement. SERS is a very local phenomenon occurring at rifts of the rough metal surface [37]. The dendritic nanostructure with many branches leads to formation of many rifts. Therefore, it can be concluded that the Ag dendrites can be used as a surface-enhanced Raman scattering (SERS) substrate, which is very valuable in trace detection for environment evaluation, and so on [38].

4. Conclusion

In this research work, a simple method, based on a selfassembled fabrication approach, to produce SERS substrates in a reliable way is investigated. In short, we fabricate a SERS-active substrate by the simple reaction without employing reducing agent, which can reflect SERS effects of financially dendrites by other methods. The sample initially grows quickly with the reaction time in the SiO₂ micro reactor and tends to adopt a dendritic shape in 1 h. SEM and TEM images shown that these samples have the morphology of dendrites. The probable formation mechanism for dendritic structures has been explained by the strong electrostatic interactions in strong alkaline conditions and the oriented aggregation. It is also found that the self-assembled process occurs between relatively large single-crystalline Ag particles, which may provide new possibilities for designing novel metal nanostructures of large nanoparticles at room temperature. The synthesized material has excellent SERS enhancement ability due to the dendritic supramolecular nanostructures. It is found that this method is relatively simple, because no complicated equipment or special reagents are required. The prepared dendritic nanostructured surface can also be applied in other fields, such as surface catalysis and biological sensors.

Acknowledgments

This project was financially supported by the Natural Science Foundation of China (Grant No. 20974045) and the Natural Science Foundation of Jiangsu Province (No. BK2009385). We also would like to thank Dr. Tariq Mahmud of Institute of Chemistry, University of the Punjab in Pakistan for his helpful discussion.

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