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Hongru Zhang

Xi'an Jiaotong University

Xianjun Shao

State Grid Zhejiang Electric Power Company (China)

Jiangyang Zhan

State Grid Zhejiang Electric Power Company (China)

Yizhou Jiao

Xi'an Jiaotong University

Run Yang

Xi'an Jiaotong University

Panli Rao

Xi'an Jiaotong University

Yu Zheng

State Grid Zhejiang Electric Power Company (China)

Zhulu Wang

State Grid Zhejiang Electric Power Company (China)

Junping Zhao

`junping_zhao@mail.xjtu.edu.cn`

Xi'an Jiaotong University

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Ultrathin AAO-Confined Pulsed Electrodeposition of Ordered Ag Nanocolumn Arrays as Sensitive and Uniform SERS Substrates

Hongru Zhang[1] Xianjun Shao [2] Jiangyang Zhan[2] Yizhou Jiao[1] Run Yang[1] Panli Rao[1] Yu Zheng[3] Zhulu Wang[3] Junping Zhao*[1]

[1] **High Voltage Division, School of Electrical Engineering, Xi'an Jiaotong University, Xi'an 710049, China**

[2] **State Grid Zhejiang Electric Power Co., Ltd. Research Institute, Hangzhou, China**

[3] **State Grid Zhejiang Electric Power Co., Ltd. Quzhou Power Supply Company, Quzhou, China**

Ultrathin anodic aluminum oxide (AAO) templates provide a promising route for constructing ordered plasmonic nanoarrays, but their application in SERS substrates is often limited by insufficient template–substrate contact and nonuniform pore filling during electrodeposition. Herein, we apply ultrathin AAO template-assisted confined electrodeposition to fabricate ordered Ag nanocolumn arrays for SERS substrates. Surface modification was introduced to improve the wettability and interfacial contact between the AAO template and the Au-coated substrate, thereby maintaining template stability during electrodeposition and suppressing uncontrolled Ag growth outside the confined nanochannels. By further regulating the pulsed electrodeposition potential, uniform Ag nanocolumn growth within the AAO nanochannels was achieved. The optimized Ag nanocolumn array substrate exhibited a limit of detection of 10^{-10} M for Rhodamine 6G, an enhancement factor up to 10^8 , and a point-to-point relative standard deviation of 7.03%. This work provides a practical strategy for constructing structurally uniform ordered nanocolumn arrays with stable SERS responses.

Keywords: plasmonic nanostructures; template-assisted electrodeposition; ultrathin AAO; Ag nanocolumn arrays; SERS substrates

1. Introduction

Surface-enhanced Raman spectroscopy (SERS) combines high sensitivity with molecular fingerprint recognition, making it a powerful technique for trace detection in environmental monitoring, food safety, and biomedical diagnostics [1]. For SERS to move toward practical analytical applications, the design of substrates capable of providing both strong Raman enhancement and spatially uniform signals is of central importance[2]. Randomly roughened metal surfaces[3] have played an important role in revealing the contribution of nanoscale gaps, surface roughness, and high surface area to SERS enhancement[4]. Building on these insights, increasing attention has been devoted to ordered plasmonic nanoarrays[5], in which key structural parameters such as feature size, spacing, and spatial distribution can be more precisely controlled[6].

Among various ordered plasmonic architectures, nanocolumn arrays are particularly attractive for SERS because they combine large surface area, controllable intercolumn gaps, and strong localized electromagnetic coupling[7]. Their well-defined geometry enables more systematic regulation of nanocolumn diameter, height, and spacing, facilitating the formation of relatively regular hot-spot distributions, which is beneficial for obtaining uniform SERS responses across the substrate [8]. In this context, anodic aluminum oxide (AAO) template-assisted synthesis provides a promising route for constructing ordered nanocolumn arrays, owing to the long-range periodic arrangement of nanopores, independently tunable pore diameter and interpore spacing, and compatibility with large-area fabrication[9]. In particular, template-assisted electrodeposition enables the confined growth of metallic nanocolumns or nanorods within AAO nanochannels, allowing the structural parameters of one-dimensional nanoarrays to be regulated by both the template geometry and the deposition conditions [10].

AAO-template-assisted electrodeposition has been extensively investigated for the fabrication of ordered metallic nanowire and nanorod arrays. In particular, transferable ultrathin AAO membranes enable ordered nanoarrays to be directly integrated onto conductive substrates and have been explored in the fabrication of functional

nanostructures for optical and magnetic applications [11]. For example, Galindo et al. fabricated highly ordered nickel nanowire arrays using ultrathin AAO templates and investigated their magnetic properties[12]. These studies demonstrate the feasibility of combining transferable ultrathin AAO templates with electrodeposition to construct well-defined metallic nanoarrays.

In the field of SERS, AAO-template-assisted methods have been used to fabricate ordered Ag and Au nanorod arrays with controllable diameter, spacing[13], and aspect ratio[14]. Huang et al. prepared large-area Ag nanorod array substrates using AAO-template-assisted fabrication and investigated the influence of nanorod geometry on the SERS response[15]. Ultrathin AAO membranes have attracted increasing attention as transferable nanostructured templates because they enable direct integration of ordered plasmonic architectures onto various substrates[16]. However, the use of transferred ultrathin AAO membranes as confined electrochemical templates for the fabrication of uniform Ag nanocolumn SERS substrates remains insufficiently explored. Unlike conventional self-supported AAO templates, transferred ultrathin membranes require close and stable contact with the conductive substrate to maintain effective confinement during electrodeposition[11]. Moreover, the nucleation, axial growth, and overgrowth of Ag nanocolumns are strongly affected by the pulsed electrodeposition conditions[17]. Therefore, both the template contact state and the electrodeposition parameters must be regulated to obtain Ag nanocolumn arrays with uniform morphology and spatially consistent SERS responses.

In this work, surface modification was combined with voltage-regulated pulsed electrodeposition to fabricate ordered Ag nanocolumn arrays within transferred ultrathin AAO nanochannels. The surface treatment was used to improve the wettability and contact between the ultrathin AAO membrane and the Au-coated substrate, while the pulsed electrodeposition conditions were optimized to regulate Ag nucleation, pore filling, and overgrowth. Under the optimized conditions, Ag nanocolumn arrays with improved structural uniformity were obtained. The resulting substrate exhibited a limit

of detection of 10^{-10} M for Rhodamine 6G, an enhancement factor of up to 10^8 , and a point-to-point relative standard deviation of 7.03%, demonstrating high SERS sensitivity and good spatial signal uniformity. This study provides a practical strategy for controlling Ag nanocolumn growth in transferred ultrathin AAO templates and offers guidance for the fabrication of structurally uniform ordered SERS substrates.

2 Experimental Section

2.1 Equipment and Materials

Table 1. Composition of the Electrochemical Deposition Solution

Component	Concentration (g·L ⁻¹)	Main Function	Mechanism Description
AgNO ₃	10	Silver Ion Source	Provides Ag ⁺ ions for deposition.
EDTA	5	Complexing Agent	Forms stable Ag ⁺ complexes, preventing precipitation and ensuring uniform deposition.
Na ₂ SO ₃	50	Complexing stabilizer	regulates free Ag ⁺ concentration and suppresses uncontrolled precipitation/aggregation.
K ₂ HPO ₄	20	Buffering and supporting electrolyte	Maintains pH stability and enhances conductivity.

The glass substrates used in this study are flat quartz glass with a diameter of 20 mm. The magnetron sputtering was performed using the MSP-300B magnetron sputtering system from Chuangshiweina Technology Co., LTD, with a Au target material purity of 99.99%. The chemical reagents used were of analytical grade and were purchased from Macklin Co., LTD. The electrochemical deposition was conducted using an electrochemical workstation Ivium company CompactStat.h. Electrochemical deposition was carried out using a three-electrode configuration, with a mercury sulfate electrode serving as the reference electrode. The compositions of the solutions used are shown in the Table 1. The ultrathin AAO template was purchased from Shenzhen Topmembranes Technology Co., Ltd.

2.2 Substrate Preparation Process

The preparation process includes the following steps:

Step 1: Fabrication of the Gold Conductive Layer. First, the quartz glass substrates are thoroughly cleaned, soaked in ethanol, and subjected to ultrasonic cleaning. Afterward, the substrates are completely dried using nitrogen. The cleaned quartz glass surfaces are coated with a 5 nm titanium layer as a transition layer by magnetron sputtering, followed by a 50 nm gold film deposition.

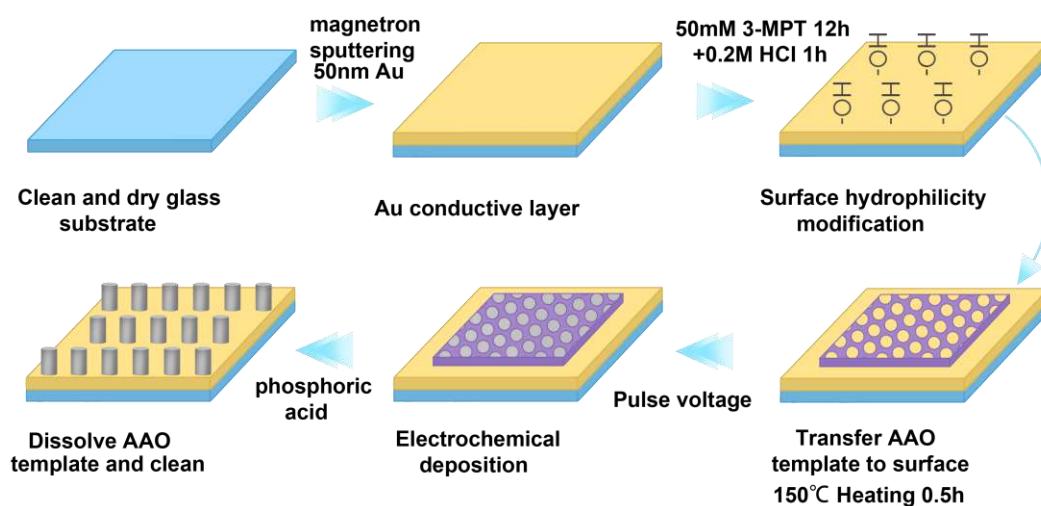


Figure 1. SERS substrate preparation flow chart

Step 2: Hydrophilic Modification of the Gold Surface[18]. The substrates are immersed in a 50 mM 3-mercaptopropyltrimethoxysilane (3-MPT) solution and reacted for 12 hours to form a 3-MPT self-assembled monolayer[19]. The substrates are then transferred to a 0.2 M hydrochloric acid solution and soaked at 30°C for 1 hour to induce hydroxylation on the surface, creating a hydrophilic surface, and finally cleaned. The AAO template was soaked in 30% hydrogen peroxide at room temperature for 2 hours to generate a large amount of hydroxyl groups to enhance hydrophilicity.

Step 3: Transfer of the Ultrathin AAO Template to the Substrate Surface. A commercial nano ultrathin double-channel AAO template with a pore size of 80 nm, pore spacing of 100 nm, and template thickness of 250 nm is selected. The AAO template is placed on the gold film surface, held with tweezers, and immersed in acetone to dissolve the PMMA support layer. Meanwhile, the surface tension of acetone is used to spread the AAO template in the liquid.

Step 4: Thermal Treatment for Interfacial Stabilization. The samples are heated at 150°C for 30 minutes. During thermal treatment, residual solvent and interfacial water were removed, which helped improve the contact between the AAO template and the modified Au film. The surface treatment promoted intimate template–substrate contact and helped maintain the integrity of the confined nanochannels during electrodeposition.

Step 5: Electrochemical Growth of Nanocolumns. Pulsed electrodeposition was carried out with a cathodic pulse potential of 0.25 V relative to the initial open-circuit potential (E_{OCP}) which was measured using a mercury sulfate electrode as the reference electrode. In our experimental system, $E_{\text{OCP}} \approx -0.35$ V vs. Hg/Hg₂SO₄. The pulse program consisted of a 0.2 s deposition step at $E_{\text{OCP}} - 0.25$ V and a 0.5 s rest step at E_{OCP} . The electrochemical deposition conditions were optimized in preliminary experiments by varying the deposition potential (0.1–0.6 V) and pulse cycle, with the goal of achieving uniform nanocolumn filling within the AAO nanopores. To concentrate the current in the AAO template area, other areas are insulated with transparent nail polish.

Step 6: Dissolution of the AAO Template Using 5% Phosphoric Acid Solution to Expose the Nanocolumns. Afterward, the samples are immersed in distilled water for 12 hours to remove any residual impurities on the surface.

2.3 Characterization Methods

The surface morphology of the substrates was observed using a scanning electron microscope (SEM), with measurements taken on a Hitachi SU8230. For Raman spectroscopy performance testing, a Horiba LabRAM ARAMIS confocal Raman spectrometer was used. The laser wavelength was set to 532 nm, with a laser power of 10 mW and an integration time of 1 second, using a 10× objective lens. The acquired Raman spectra were processed using Origin Pro software, applying baseline correction (ALS method) and spectral smoothing (S-G method). In the experiments, Rhodamine 6G (R6G) was used as the analyte, and different concentrations of R6G aqueous solutions were prepared as test samples. Each sample was soaked in the corresponding concentration of Rhodamine solution for 1 hour, and after air drying, Raman spectroscopy measurements were performed.

3 Results and Discussion

3.1 SEM Testing Results

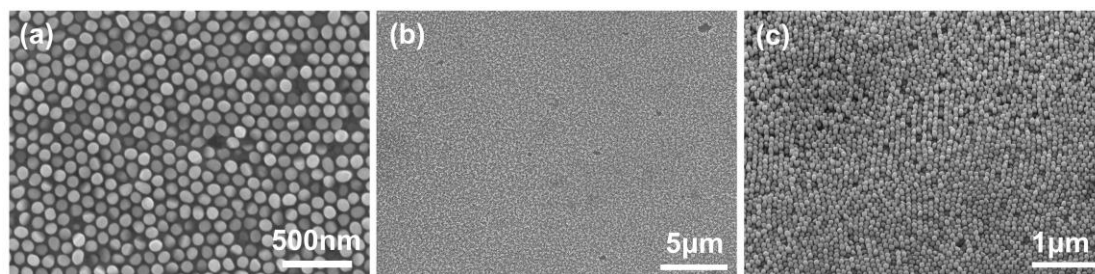


Figure 2 SEM image of SERS substrate

Figure 2 shows the morphology at a deposition potential of $E_{OCP} - 0.25V$. From the SEM images, it can be observed that densely packed nanocolumn arrays were successfully obtained on the surface. Under the constraint of the AAO template, the nanocolumns are arranged in an array along the template pores, with a diameter of approximately 80 nm, narrow size distribution and a good hexagonal arrangement. In the low-magnification SEM images, the nanocolumns are evenly distributed over a large area.

To quantitatively evaluate the structural uniformity of Ag nanocolumn arrays, a self-developed Python program was used for statistical analysis of SEM images. The program first performs grayscale and threshold segmentation on the SEM image, extracts the contour of a single nanocolumn through connected domain recognition, and calculates its equivalent circle diameter as the column diameter. Subsequently, the center distance between adjacent nanocolumns is calculated based on the centroid coordinates of each nanocolumn, and the intercolumn gap distance is calculated by combining the diameters of adjacent nanocolumns. Finally, the statistical distribution of the diameter, center distance, and inter column gap of the nanocolumns were obtained, and their mean and standard deviation were calculated to evaluate the size uniformity of the array structure. Image analysis showed that the diameter of the nanocolumns is 75.39 ± 13.35 nm, the gap distance is 21.48 ± 8.03 nm, the center

distance of the nanocolumns is 98.38 ± 18.26 nm, and the porosity is about 4.8%. The quantitative results show that the uniformity of the nanocolumn array is good.

3.2 Optical Testing Results

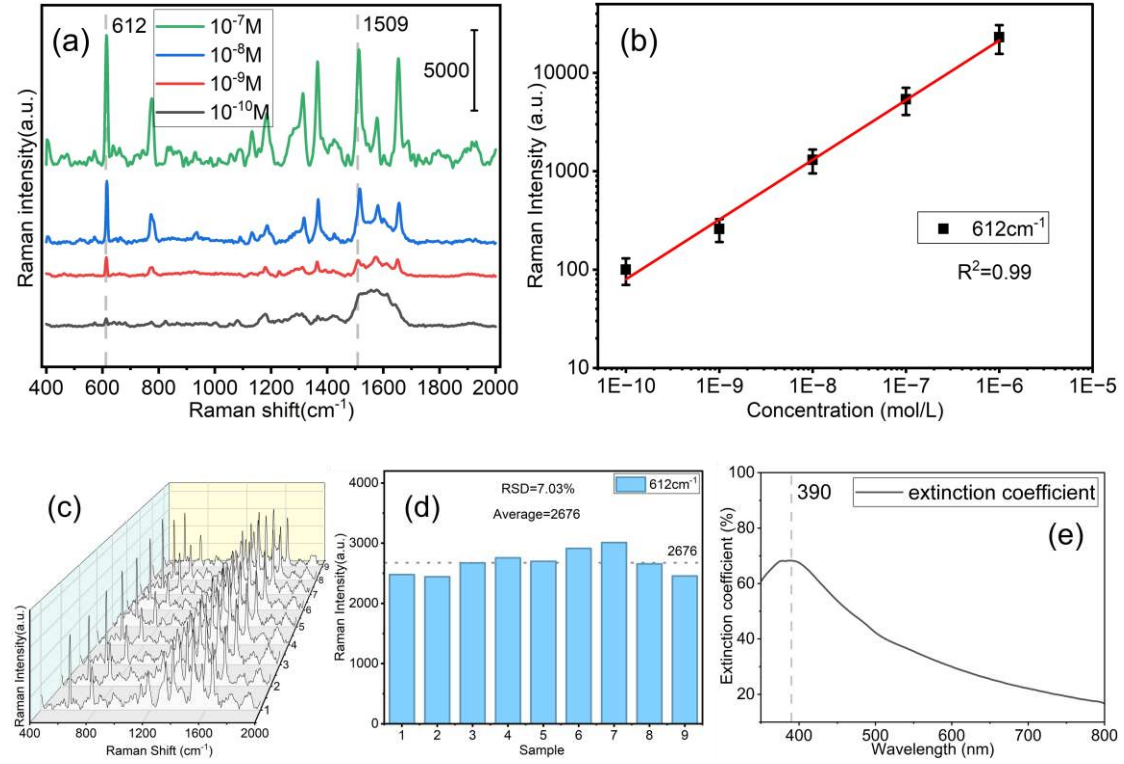


Figure 3 Raman spectroscopy and extinction spectroscopy (a) Raman spectrum test results at different concentrations (b) Relationship between concentration and Raman intensity (c) Multi-point repeated test results (d) Results of RSD (e) Extinction spectrum

The enhancement performance of the SERS substrates was evaluated in terms of the enhancement factor (EF) and the limit of detection (LOD). The LOD was defined as the analyte concentration corresponding to a signal-to-noise ratio (S/N) of 3. The EF was calculated using

$$EF = \frac{I_{SERS} / N_{SERS}}{I_{RS} / N_{RS}} \quad (1)$$

where I_{SERS} and I_{RS} are the Raman intensities of the selected characteristic band obtained from SERS and normal Raman measurements, respectively, and N_{SERS} and N_{RS} represent the corresponding numbers of molecules contributing to the signals.

Raman spectroscopic measurements demonstrated that the fabricated SERS substrate exhibits excellent enhancement performance. Using the characteristic Raman band of R6G at 612 cm^{-1} as the reference, the calculated LOD reached as low as 10^{-10} M , compared with the ordinary Raman spectra obtained by directly dropping R6G onto silicon wafers, the EF was as high as 10^8 . After multiple measurements at different points, the RSD was 7.03%, indicating good uniformity of the enhanced substrate. Additionally, by measuring the extinction spectrum of the SERS substrate, it was found that the LSPR resonance peak is located around 390 nm, and in the visible range, the extinction coefficient decreases with increasing wavelength.

3.3 FDTD Simulation of Electromagnetic Field Distribution

Finite-difference time-domain (FDTD) simulations were performed to investigate the local electromagnetic field distribution of the Ag nanocolumn structure. The simulation model was constructed according to the experimentally observed morphology. The Ag nanocolumn array had a period of 100 nm, a nanocolumn diameter of 80 nm, and a height of 200 nm, corresponding to an intercolumn gap of approximately 20 nm. The incident light wavelength was set to 532 nm, consistent with the Raman excitation wavelength used in the SERS measurements. The light was incident vertically from the top of the nanocolumn array, and the polarization direction was set parallel to the lateral direction of the array. Periodic boundary conditions were applied in the lateral directions to represent the ordered periodic nanocolumn array, while perfectly matched layer (PML) boundary conditions were used along the light propagation direction. The optical constants of Ag were taken from the material database of FDTD Solutions, and a refined mesh size of 0.5 nm was used around the Ag nanocolumns and intercolumn gaps to ensure accurate calculation of the localized near-field distribution.

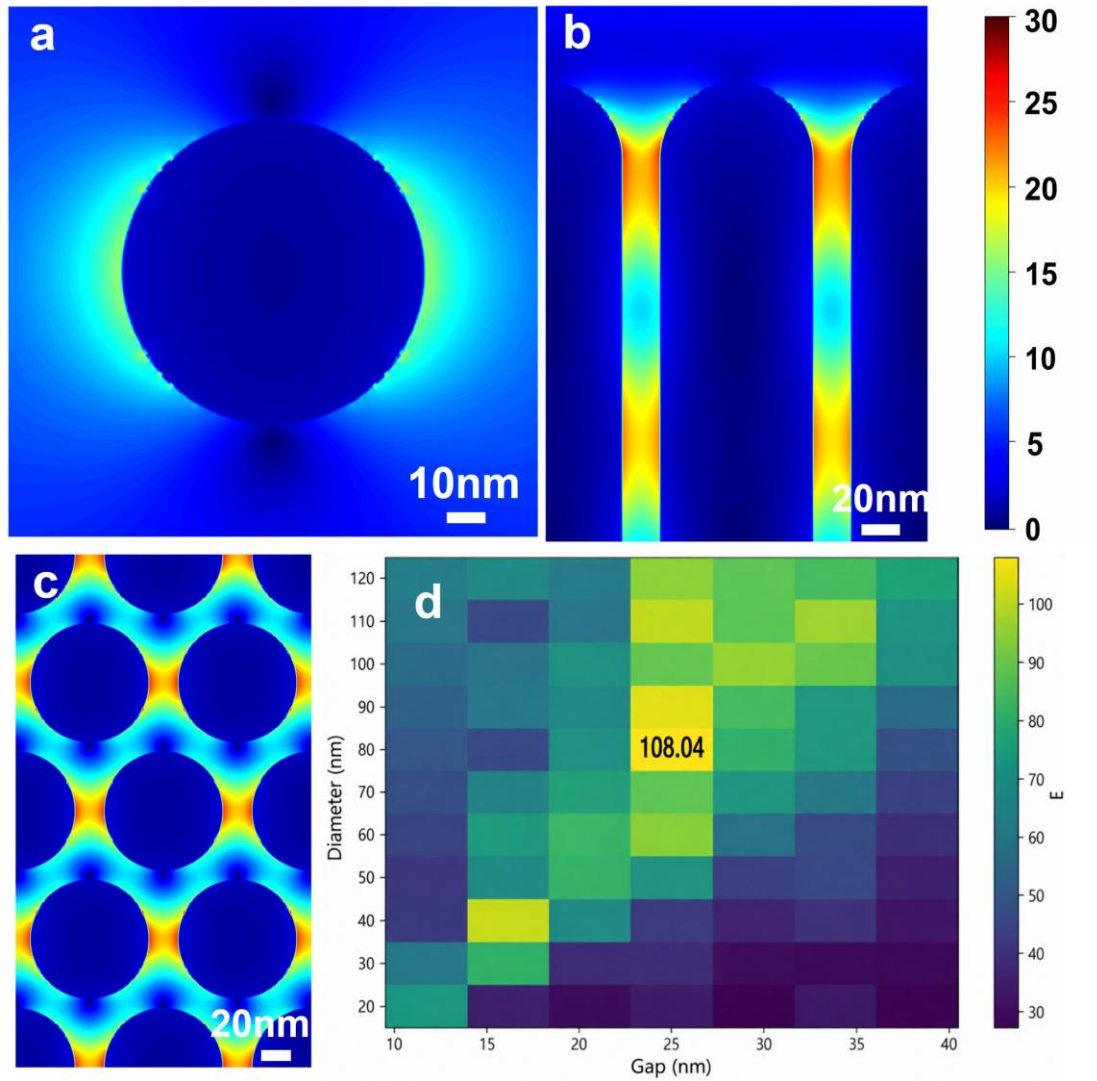


Figure 4 FDTD simulation results. (a) Single nanocolumn simulation results (b) Side view of nanocolumn array (c) Top view of nanocolumn array (d) Heat map of maximum electric field generated by nanocolumns arrays with different diameters and gap distances

As shown in Figure 4, the single Ag nanocolumn exhibited limited electromagnetic field enhancement. In contrast, the Ag nanocolumn array generated stronger and periodically distributed localized electric fields, especially in the intercolumn gaps and at the edges of the nanocolumns. Since the electromagnetic enhancement contribution in SERS is generally proportional to $|E/E_0|^4$, the stronger local electric field in the ordered nanocolumn array indicates its higher potential for SERS enhancement. These results confirm that the ordered Ag nanocolumn array is more favorable for producing strong and spatially regular electromagnetic enhancement than isolated nanocolumn structures. Figure 4 (d) calculates the electric field generated by different diameters of nanocolumns and gaps. It is found that the maximum electric field value is generated when the diameter of the nanocolumns is 80 nm and the gap between the nanocolumns

is 25 nm, these dimensions are close to the experimentally measured diameter of 75.39 ± 13.35 nm and gap of 21.48 ± 8.03 nm.

3.4 Influence of the Cathodic Potential Step on Nanocolumn Growth

Deposition potential is a key parameter in the electrochemical deposition process because it directly determines the cathodic overpotential and thereby strongly influences the reaction kinetics at the electrode/electrolyte interface. The overpotential can be expressed as[20]:

$$\eta = E_{\text{applied}} - E_{\text{eq}} \quad (2)$$

where η represents the overpotential, E_{applied} is the applied potential, and E_{eq} is the electrochemical equilibrium potential. According to classical electrode kinetics, an increase in overpotential accelerates the charge-transfer reaction and thus increases the cathodic current density at the electrode surface. Under kinetically controlled conditions, the metal deposition rate is positively correlated with the current density and can be described by Faraday's law[21]:

$$\frac{dm}{dt} = \frac{MiA}{nF} \quad (3)$$

where m is the mass of deposited Ag, M is the molar mass of Ag, i is the current density, A is the effective electrode area, n is the number of electrons transferred per Ag ion, and F is the Faraday constant. Classical electrode kinetics are used here only as a qualitative framework for interpreting the effect of the cathodic potential step[21]. In the confined nanopores of the ultrathin AAO template, the actual growth behavior is additionally affected by restricted ion transport and geometric confinement[22].

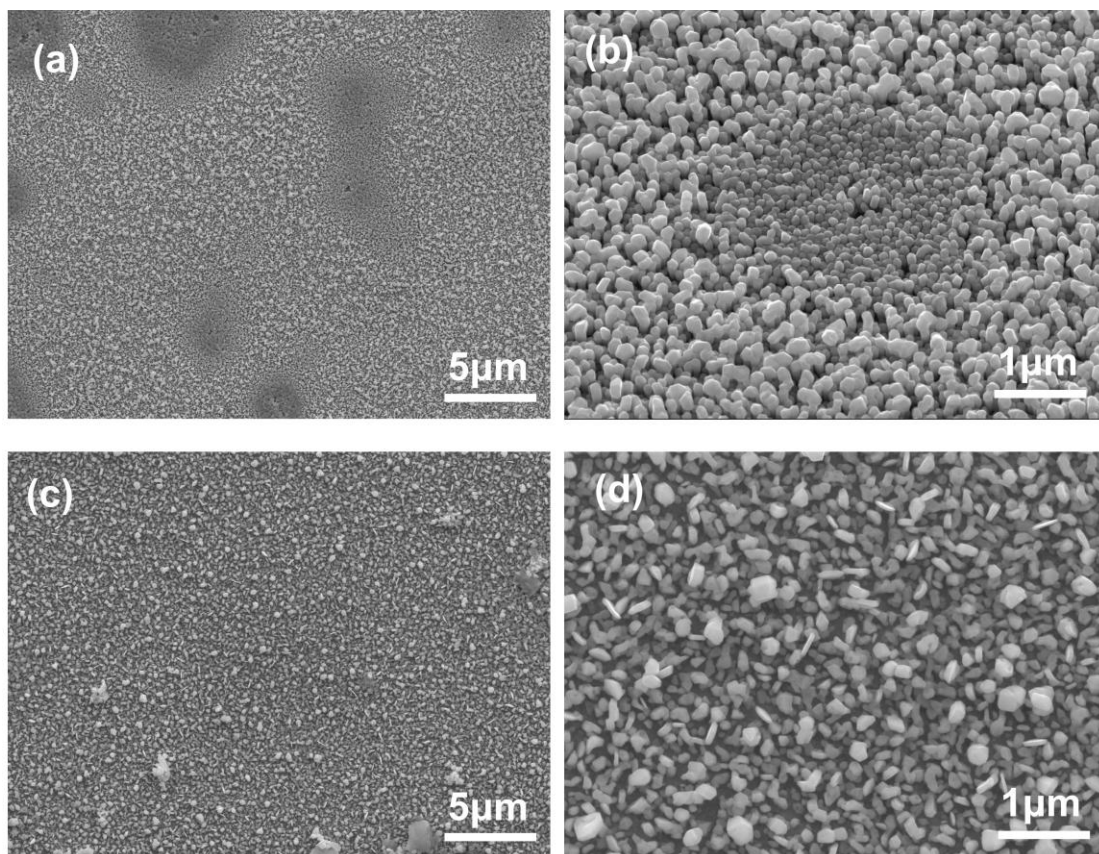


Figure 5 (a-b) SEM images at deposition potential of $E_{OCP}-0.5V$ (c-d) SEM images at deposition potential of $E_{OCP}-0.15V$

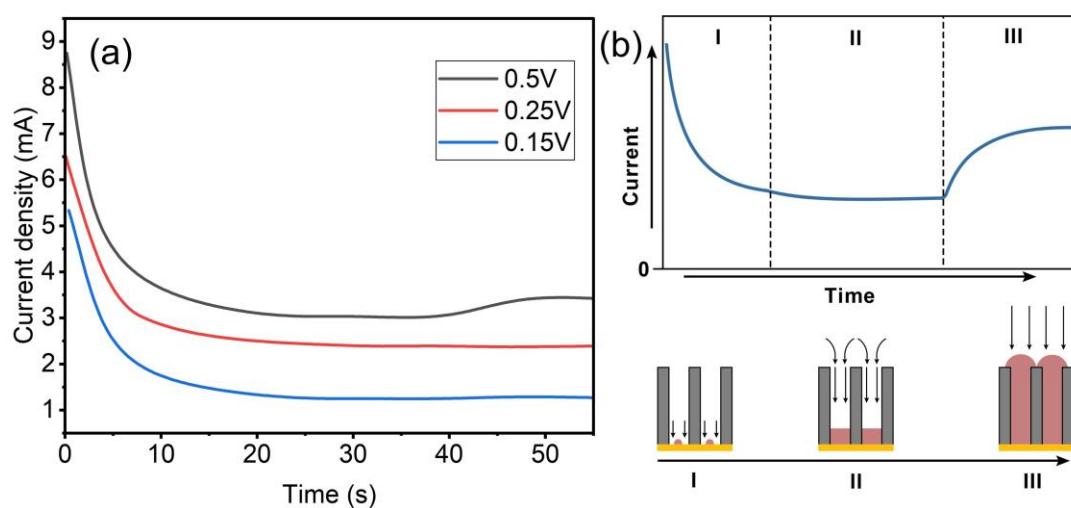


Figure 6. On-pulse current-density envelopes and proposed growth mechanism during pulsed electrodeposition (a) On-pulse current-density envelopes recorded during pulsed electrodeposition at different working potentials. The pulse program consisted of a 0.2 s

working pulse and a 0.5 s rest interval; only the current density during the working-potential stage is plotted, while the rest-period current is omitted for clarity. (b) Schematic illustration of the three-stage confined electrodeposition process, including nucleation and concentration-gradient establishment, confined growth within AAO nanochannels, and free growth after the deposits emerge from the template.

As shown in Figure 5, the deposition potential strongly affects the morphology of the Ag nanostructures. At $E_{\text{OCP}} - 0.50$ V, the high electrochemical driving force accelerates Ag^+ reduction and nanocolumn growth. However, rapid Ag^+ consumption may cause local ion depletion and uneven growth among different nanochannels. Once the deposits emerge from the AAO template, the loss of geometric confinement further promotes free growth and aggregation above the template surface[23], resulting in the overgrown structures observed in Figure 6a and b.

At $E_{\text{OCP}} - 0.15$ V, the electrochemical driving force is insufficient to induce efficient nucleation and continuous growth in all nanochannels. Consequently, some pores remain unfilled or incompletely filled, while Ag deposition preferentially occurs at a limited number of active sites, producing discontinuous nanocolumns and localized aggregates, as shown in Figure 5c and d. These results indicate that both excessively high and excessively low working potentials are unfavorable for the formation of uniform Ag nanocolumn arrays.

The on-pulse current-density envelopes in Figure 6a provide further insight into the growth process. Based on the current evolution and the confined geometry of the AAO template, the deposition process can be divided into three stages[24], as illustrated in Figure 6b.

In stage I, the rapid current decrease may arise from the combined effects of double-layer charging, Ag nucleation at the conductive pore bottoms, and the establishment of a local Ag^+ concentration gradient.

In stage II, Ag grows vertically within the AAO nanochannels under geometric confinement. Ag^+ consumption during the working pulse and ion replenishment during

the rest interval gradually approach a dynamic balance, resulting in a relatively stable current density. This confined-growth stage is critical for the formation of ordered nanocolumn arrays.

In stage III, the Ag nanocolumns emerge from the AAO template and enter a free-growth regime. The exposed Ag tips experience less restricted ion transport, increasing the deposition rate and current density. This behavior is particularly evident at $E_{\text{OCP}} = -0.50 \text{ V}$, where the current density increases after approximately 40 s, consistent with the overgrown morphology observed in Figure 5a and b.

Although a relatively low working potential can suppress premature overgrowth, it may also result in insufficient nucleation within some nanochannels. To improve the initial nucleation uniformity without continuously increasing the deposition rate, a transient high-voltage pulse was introduced at the beginning of the electrodeposition process.

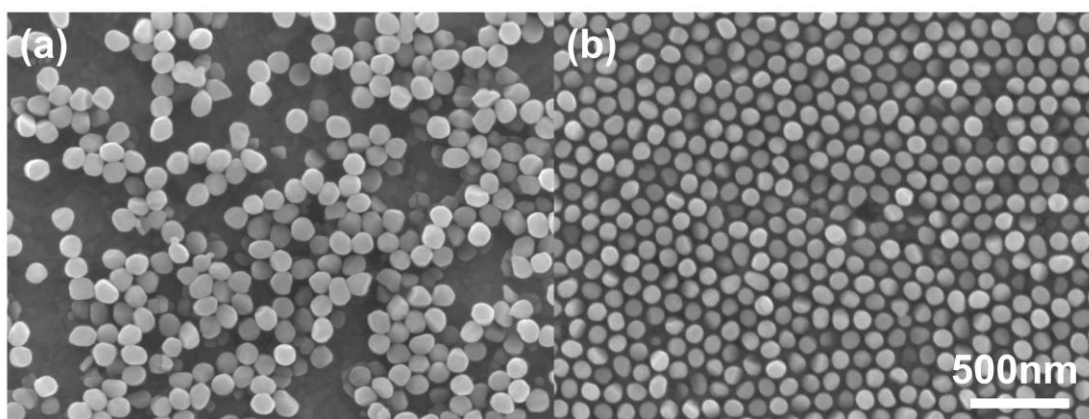


Figure 7 Comparison of confined electrodeposition with and without an additional high-voltage pulse during the initial stage. (a) No additional high voltage pulse applied (b) An additional $E_{\text{OCP}} -0.9 \text{ V}$ high-voltage pulse was applied for 1 s.

During electrochemical deposition, the initial high-voltage pulse accelerated nucleation and increased the density of nucleation sites within the confined pores, thereby reducing the occurrence of unfilled pores. Compared with the control group, the experimental group subjected to the $E_{\text{OCP}} -0.9 \text{ V}$ pulse for 1 s exhibited a markedly lower empty-pore fraction, indicating that transient high-voltage activation promotes more uniform and complete confined electrochemical growth. After conducting image statistics, it was

found that the porosity decreased from over 40% to 4.8%. This fully demonstrates the role of high-voltage pulses in reducing porosity.

Overall, the formation of uniform Ag nanocolumn arrays requires coordinated control of the nucleation and growth stages. An appropriate working potential balances Ag^+ reduction, axial growth, and ion transport, whereas a short initial high-voltage pulse promotes homogeneous nucleation. The combination of transient nucleation activation and subsequent confined growth improves the filling efficiency and structural uniformity of the Ag nanocolumn arrays.

4 Conclusion

In this work, an ultrathin AAO template-assisted pulsed electrodeposition strategy was developed to fabricate ordered Ag nanocolumn array SERS substrates. A 3-MPT-mediated surface treatment promoted intimate contact between the transferred AAO membrane and the Au-coated substrate, helping maintain stable confinement during electrodeposition. By regulating the deposition potential and introducing a transient initial high-voltage pulse, Ag nucleation, pore filling, and subsequent nanocolumn growth were effectively controlled, resulting in arrays with improved structural uniformity.

The optimized substrate exhibited a limit of detection of 10^{-10} M for Rhodamine 6G, an enhancement factor of up to 10^8 , and a point-to-point relative standard deviation of 7.03%. FDTD simulations indicated that strong localized electromagnetic fields were concentrated mainly within the intercolumn gaps and near the nanocolumn edges. These results demonstrate that coordinated regulation of template–substrate contact and pulsed electrodeposition conditions provides a practical route for constructing structurally uniform Ag nanocolumn array SERS substrates.

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