



# Porous GaN electrode for anodic stripping voltammetry of silver(I)



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## ABSTRACT

Here we demonstrate porous GaN electrode can be applied for trace Ag(I) detection. Compared to traditional planar electrodes, porous GaN electrode can detect lower concentration of Ag(I) as it possesses more deposition sites (crystal defects) and larger surface area. Under the optimum conditions, porous GaN electrode shows a linear voltammetric response in the Ag(I) concentration range from 1 to 100 ppb with the detection limit of 0.5 ppb. Such an unmodified, high-porosity and chemically stable electrode is promising to operate in real samples.

## 1. Introduction

With the widespread use of silver compounds and silver-containing processes in industrial activities, the ever-increasing silver content in ecological environment and biological organisms has drawn much attention due to its potential threat to human health [1–3]. In order to determine the concentration level of Ag(I), different analytical techniques such as fluorimetry [4], atomic absorption spectroscopy (AAS) [5], inductively couple plasma mass spectrometry (ICP-MS) [6] have been developed and applied. However, these methods are not suitable for in-situ monitoring applications. In this respect, electrochemical technique has advantages over the above approaches owing to its easy operation, low cost, high sensitivity and fast response. Among them, stripping voltammetric analysis has been widely used for measuring trace heavy metals [7–9].

The mercury-film and hanging mercury drop electrodes had played a great role in the development of stripping voltammetry [10]. Nevertheless, because of the toxicity of mercury, environment-friendly coating materials such as bismuth [11,12] and antimony [13] replaced mercury and became new generation electrodes. Whereas the addition of coating materials such as mercury and bismuth into samples makes the determination process more complicated. Ideally, non-film electrode materials are favored over above film electrodes due to their simplified measuring operation [14–17].

Gallium nitride (GaN) as a direct wide band gap semiconductor has been long prized for its superior optical properties [18,19]. Nevertheless, GaN, with its adjustable carrier concentration and conductivity type, has the potential to be a good electrode substrate. Furthermore, GaN possesses excellent chemical stability under harsh

conditions, which is vital for some practical applications. Last but not least, the characteristic of high electron mobility means GaN can generate less noise and thus spot smaller signals. Above facts endow GaN with capacities to be a highly accurate biosensor or chemical sensor [20]. In this work, we demonstrate porous GaN can be a good electrode material for Ag(I) detection using anodic stripping voltammetric technique. This porous GaN electrode without any surface modification is more favorable industrially due to its easy electrode fabrication and measurement process. The low limit of detection (0.5 ppb) and good anti-interference ability prove porous GaN electrode has the potential to be applied in real water samples.

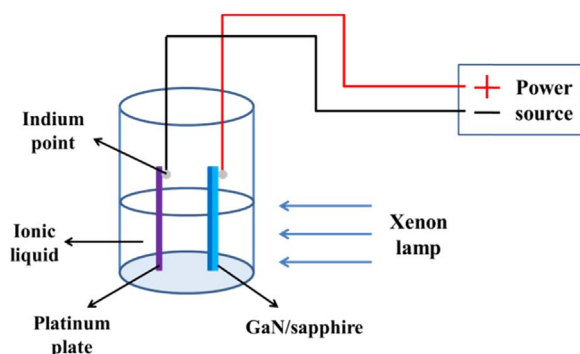
## 2. Experimental

### 2.1. Fabrication of porous GaN electrode

Single-crystal n-type GaN(0001) film was grown on sapphire(0001) substrate by hydride vapor phase epitaxy (HVPE). The Si-doped GaN layer is 5 μm thick with the carrier concentration of  $4.8 \times 10^{18} \text{ cm}^{-3}$ . The size of planar GaN electrode is 1.3 × 0.3 cm. Porous GaN electrode was fabricated by photo-assisted electrochemical etching technique. Planar GaN with front-side ohmic contact made by indium point and platinum plate were used as the anode and cathode, respectively. The front-side of planar GaN electrode was illuminated by a 300 W Xenon lamp; the applied etching voltage and time were 5 V and 5 min, respectively. Ionic liquid 1-ethyl-3-methylimidazolium trifluoromethanesulfonate ( $\text{C}_7\text{H}_{11}\text{F}_3\text{N}_2\text{O}_3\text{S}$ ) was used as the etchant. The schematic diagram of etching device is shown in Fig. 1. After etching porous GaN electrode was obtained by completely washing. The surface morphology

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**Fig. 1.** Schematic diagram of fabricating porous GaN electrode by photo-assisted electrochemical etching using ionic liquid as the etchant.

of porous GaN electrodes was characterized by scanning electron microscopy (SEM, Hitachi S-4800). CHI 660D (CH Instruments) was used in all electrochemical measurements.

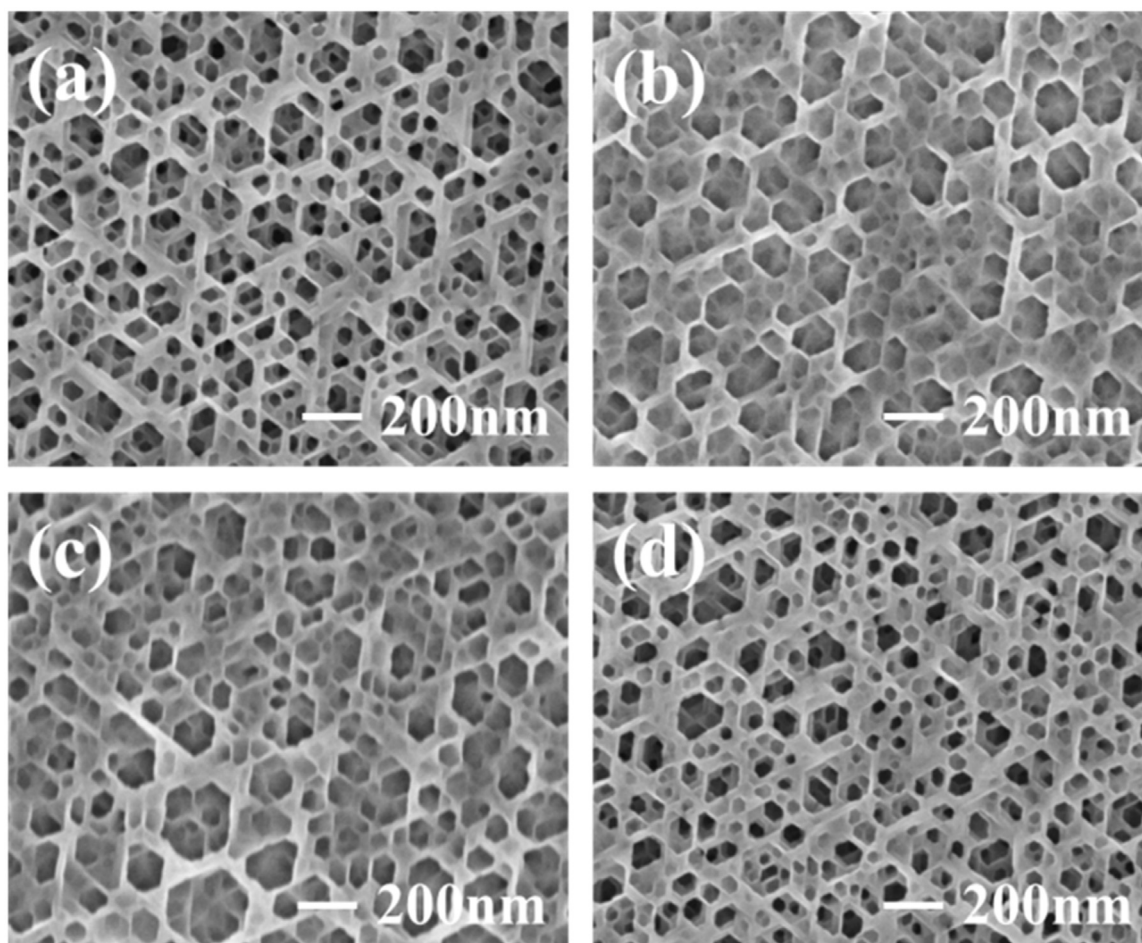
## 2.2. Analytical procedure

The analysis of Ag(I) was carried out using porous GaN, platinum wire and Ag/AgCl electrodes as working, counter and reference electrodes, respectively. The three electrodes were immersed into a 50 mL electrochemical cell, containing 0.1 M acetate buffer (pH 4.5) and different concentrations of Ag(I). All measurements were implemented at room temperature in the presence of dissolved oxygen. The detailed square-wave anodic stripping voltammetric (SWASV) mea-

surements were performed as follows. (a) The preconcentration process was carried out at the applied potential of  $-0.5$  V for 2 min under magnetic stirring (800 rpm). (b) The stripping voltammogram was recorded by applying a positive-going square-wave voltammetric potential scan from  $-0.1$  to  $0.4$  V with a frequency of 20 Hz, amplitude of 25 mV, and potential step of 5 mV. (c) The regeneration step was executed at the potential of  $+0.6$  V for 5 min under magnetic stirring (800 rpm), prior to the next measurement.

## 3. Results and discussion

**Fig. 2** shows SEM images of porous GaN under different stages of SWASV process. The surface morphology of freshly prepared GaN is shown in **Fig. 2a**. The pore density is estimated up to  $6.5 \times 10^9$  per square centimeter. The pore diameter is between 20 and 160 nm, and the average pore diameter is 58 nm. Moreover, the pore shape is hexagonal, which is consistent with the wurtzite structure (hexagonal system) of GaN. The etching mechanism of ionic liquid has been speculated in our previous report [21]. The pore morphology of porous GaN after preconcentration procedure demonstrates Ag(I) can enter into the inner pores and the deposited Ag fills most of the pores of GaN, which is shown in **Fig. 2b**. This result also proves porous GaN electrode can be fully wetted by the electrolyte. **Fig. 2c** exhibits there is still a significant amount of Ag in the pores of GaN, this result suggests the stripping process can not wipe off all Ag deposit. The previous report had demonstrated the Ag deposit could not all be stripped voltammetrically [22]. The main reason may be ascribed to the unique interface between Ag and GaN, which needs a more positive potential and longer time to oxidize Ag to Ag(I). **Fig. 2d** shows porous GaN recovers to its



**Fig. 2.** SEM images of porous GaN (a) cleanly, (b) after preconcentration step, (c) after stripping step, and (d) after regeneration step.

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