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A Simple Method for Fabrication of Gold Nanoelectrodes

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Abstract: A facile method has been developed for the fabrication of Au nanoelecrodes (Au NEs). The tip of Au NEs can be controlled within the range from dozens to hundreds of nanometer.

Keywords: Gold nanoelectrode, electrochemical etching.

Recently, the study of nanoelectrodes (NEs) has grown rapidly. Pt[1], carbon fiber[2], W[3] and Pt-Ir[4] NEs have been reported. In this work, we developed a method to make Au NEs. A Au wire in 1 cm length with a 20 μm diameter was adhered to a 100 μm copper wire by FHS203 silver epoxy (Fenghua Ltd, Guangdong, China). It was inserted into a 150 μm ID capillary and protruded *ca.* 0.5 cm. The Au wire was sealed at one end of the capillary with HC704 high temperature resistant adhesive (HTRA, Xiguang Organic Silicon Material Factory, Wuxi, China). Then, the 100 μm copper wire was adhered to a 500 μm copper wire. Finally, they were inserted into a *ca.* 1 mm ID glass tube. Both ends of the glass tube were sealed with HTRA. After 24 h, the Au wire was electrochemically etched in a solution containing 60% saturated $CaCl_2$, 4% HCl, and 36% H_2O (by volume) reported in Ref. 4 with an alternating current voltage of 18 V against a Pt coil until the current was dropped to zero. The electrode was cleaned with ethanol, 0.01 mol/L NaOH, 1 mol/L HNO_3 and double-distilled water, respectively. Then it was dipped into a plating shield adhesive (PSA). After being drawing out from the adhesive, the electrode was heated at 190℃ for 20 min. PSA on the surface of the Au wire was shrunk and the end of the PSA-coated Au wire was exposed. The procedure was repeated. The size of the Au NE depends on the layers of the coated PSA and can be controlled in the range from dozens to hundreds of nanometer. The scheme of the Au NE and its scanning electronic microscopy (SEM) image are showed in **Figure 1 and 2A**, respectively. The linear sweep voltammogram of the Au NE in a solution consisted of 5 mmol/L $K_3Fe(CN)_6$ and 1 mol/L KCl is shown in **Figure 2B.** Assuming that the exposed area of the Au NE is a disk, its radius can be calculated by equation (1).

$$i_{lim} = 4nFDc^{o}r \qquad (1)$$

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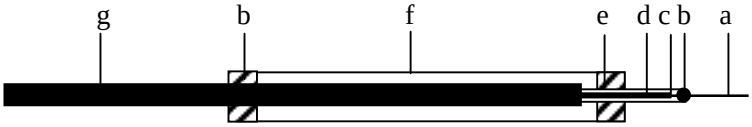
Where i_{lim} is steady-state diffusion-limited current, c^o and D are the buck concentration and the diffusion coefficient of $K_3Fe(CN)_6$, respectively, F is the Faraday constant, and n is the number of electrons transferred. From the detected i_{lim} of 31 pA, the effective radius can be calculated to be 211 nm.

Figure 1 Schematic drawing of a Au NE.



a. PSA-coated Au wire, b. HTRA, c. silver epoxy, d. 100 μm copper wire, e. capillary, f. glass tube, g. 500 μm copper wire.

Figure 2A SEM image of a Au NE.

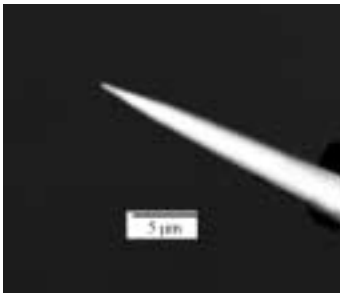
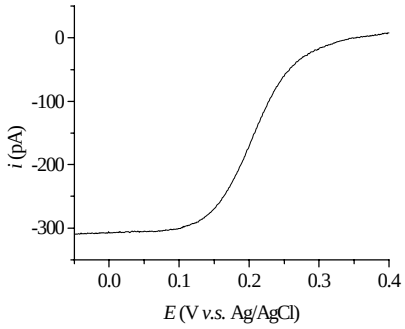


Figure 2B Linear sweep voltammogram of a Au NE.



5 mmol/L $K_3Fe(CN)_6$ and 1 mol/L KCl with a scan rate of 10 mV/s.

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