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Evaluation of using xurography as a new technique for the fabrication of disposable gold electrodes with highly reproducible areas

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ABSTRACT

This communication describes the use of xurography as a new technique for the rapid fabrication of gold disk electrodes with highly reproducible areas. Adhesive vinyl films and a cutting plotter were used to produce masks containing the electrode layouts, and recordable compact disks (CDs) were used as gold source. Electrode area was defined by transferring the vinyl adhesive mask to the gold surface. The electrochemical behavior of the "vinyl-CDtrodes" was studied by cyclic and square wave voltammetry experiments on model systems such as potassium ferrocyanide, sulfuric acid, and dopamine. The electrodes constructed using this technique meets the most important characteristics of a disposable device: low cost, reproducibility between different devices, and possibility of large-scale production.

1. Introduction

Gold is one of the most used metallic solid electrodes in electrochemical and electroanalytical techniques. Besides its high stability over a wide range of potential, high purity, and inertness in the presence of almost all reagents, gold electrodes can be fabricated in different formats such as disks, bands, wires, and arrays. Moreover, the fabrication of self-assembled monolayers (SAMs) through strong Au – S bonds is recently driving the attention to this material due to the vast assortment of possibilities in biomolecule immobilization and biosensing applications [1–3].

Photolithography is a well-known technique used for manufacturing gold-based electrodes and sensors arrays with highly reproducible features [4,5]. However, photolithography is an expensive fabrication technique that requires sophisticated instrumentation and clean rooms that sometimes are not readily available. Consequently, low-cost technologies to construct such devices are being developed [6,7]. Aiming to address this issue, a technique based on heat-transference of toner masks onto gold surface of peeled CDs was described by Daniel and Gutz [6] for the fabrication of single or multiple coplanar gold electrodes of any shape and with sizes down to 100 μm. Lowinsohn and co-workers [8] extended the application of the technique to produce disposable gold disk electrodes and Richter et al. [7] reported the fabrication of microelectrode arrays by using the same methodology. The combination of screen printing and gold electrodeposition techniques was also reported as a good alternative to fabricate low-cost gold electrodes and sensor arrays [9].

In this scenario, a simple and inexpensive method based on xurography is presented in this communication for rapid fabricating disposable gold disk electrodes with highly reproducible areas. Xurography was firstly proposed by Bartholomeusz et al. in 2005 [10] as an alternative technique for rapid prototyping microstructures for microfluidic applications. This technique uses a cutting plotter commonly used in the sign industry for cutting graphics in adhesive vinyl films. When compared to other rapid prototyping techniques, xurography has some advantages due its short fabrication time, independence of clean room facilities, and low equipment and material cost [10,11]. Potential applications explored in the first work include shadow masking, micromolds for PDMS, multilayered three-dimensional channels, and electroplating [10]. In electroplating, the authors have produced patterned masks to define the exposed electrode area of gold coated glass slides, but have not extended its use to electroanalysis. More recent applications of xurography on microfluidics include the fabrication of microchannels and reactors [12], PCR devices [13], and DNA sequencing devices [14]. In the following sections, it will be demonstrated that xurography is also suitable to produce disposable gold electrodes, since reproducible analytical parameters were obtained with a wide set of electrodes in different electrochemical characterization experiments.

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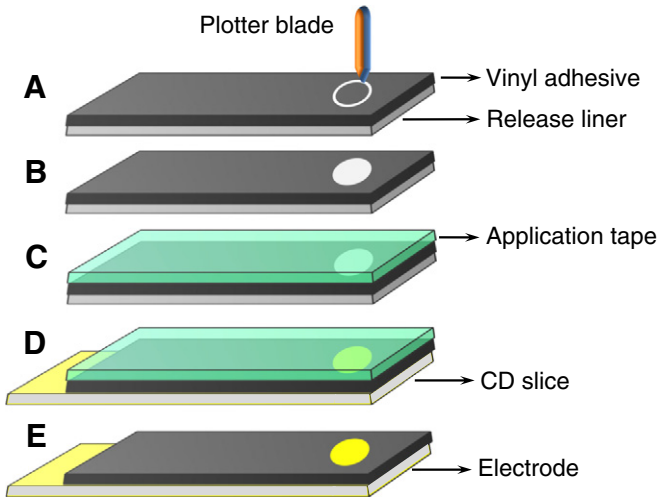

Fig. 1. Schematic diagram showing the vinyl-CDtrodes fabrication procedure. A, the layout of the electrodes is transferred to the vinyl adhesives using a cutting plotter. B, the undesired portions is peeled off the release liner. C, an application tape is placed on the vinyl surface. D, the vinyl adhesive is transferred to the gold surface. E, the application tape is removed, leaving the vinyl adhesive attached to the gold surface.

2. Experimental

2.1. Materials and instrumentation

Dopamine hydrochloride, sodium chloride, sodium ferricyanide, sodium nitrate, and acid were purchased from Sigma-Aldrich (St. Louis, MO). Cyclic and square wave voltammograms were recorded with an Autolab PGSTAT20 Electrochemical Analyzer (Eco Chemie BV, Utrecht, Netherlands). The working electrode, saturated calomel reference electrode (Model ZE11311, Aldrich, St. Louis, MO), and platinum wire counter electrode were inserted into a 20 ml cell through holes in a Teflon cover. The diameters of the circular structures cut on the vinyl adhesives were measured from images digitalized using a Sony CCD camera (DXC-390P) using a graphics editing program.

2.2. Electrode fabrication

Fig. 1 schematically illustrates the electrode fabrication procedure. The layout of the electrodes was drawn using the CorelDraw 11.0 software package (Corel, Ottawa, Canada). The patterns were transferred to the vinyl adhesives (3M Scotchcal^{MR} D3000, 0.09-mm-thick) using a cutting plotter (Model GX-24, Roland DG Corporation, Hamamatsu, Japan) with nominal resolution of 100 μm (Fig. 1A). The undesired portions were peeled off the release liner using a needle, leaving only the portion that contains the electrode layout as a negative mask (Fig. 1B). Next, an application tape was placed on the vinyl surface to hold the structure in place while transferring to the substrate surface (Fig. 1C).

The gold electrodes were constructed using recordable compact disks (CDs) as the gold source. For this purpose, the CDs (Mitsui MAM-M Standard Gold CD-R, Mitsui & CO., Tokyo, Japan) were treated with concentrated nitric acid to remove the polymeric film that protects its metallic layer [1]. The CDs were then cut in small pieces with approximately 0.8 cm in width and 5 cm in length, using a paper guillotine. The vinyl adhesive containing the electrode layout was transferred to the gold surface using the application tape (Fig. 1D). The application tape was removed, leaving the vinyl adhesive attached to the gold surface, thus defining the disk format of the electrode and leaving the opposite edge to clamp a crocodile clip (Fig. 1E). An insulator ink was used for covering parts of the gold layer around the electrode edge avoiding the contact with the solutions.

3. Results and discussion

The cutting plotter utilized in this work was able to make well-defined circular structures down to 447 ± 12 μm (n = 3) in diameter and square shape structures (microchannels) down to 107 ± 13 μm (n = 3) in width. Depending on the quality of the cutting plotter, better resolutions can be obtained. Bartholomeusz et al. [10], for example, utilized a cutting plotter that was able to produce square shape structures down to 18 μm in width. Fig. 2 shows scanning electron micrographs of a vinil-CDtrode with a nominal diameter of 1.0 mm. The top view (Fig. 2A) shows the well defined circular structure that can be obtained by xurography. The roughness on the side wall of the vinyl adhesive (Fig. 2B) is inherent in the cutting method. As will be demonstrated below, this roughness is not a limitant factor of reproduibility in the electroanalytical measurements. As can be observed in the 1.5-mm-diameter electrode presented in the Graphical abstract, the side wall of larger circular structures tends to be smoother. Fig. 2C shows the CD tracking trails on the electrode gold surface [8]. The very low roughness of the CD gold surface combined with the ability of the cutting plotter to make highly reproducible structures yielded vinyl-CDtrodes with highly reproducible electrochemical responses.

Fig. 3 displays square wave voltammograms for 40 μmol L^{-1} $K_4Fe(CN)_6$ obtained at vinyl-CDtrodes with increasing nominal diameters from 0.50 to 3.0 mm (a–f) in steps of 0.50 mm. In agreement with previous reports [8,15], $Fe(CN)_6^{-4}$ yields a well-defined peak located at about +0.218 V (vs SCE) on gold surface. The high linearity (correlation coefficient of 0.9985) observed in the resulting plot of peak current versus electrode area (Fig. 3A) indicates the ability of the cutting plotter in making features with high precision, affording analytical responses proportional to the electrode area.

Fig. 3B shows the dependence between nominal and measured diameters of circular structures obtained with the cutting plotter. All nominal diameters studied were larger than the measured diameters

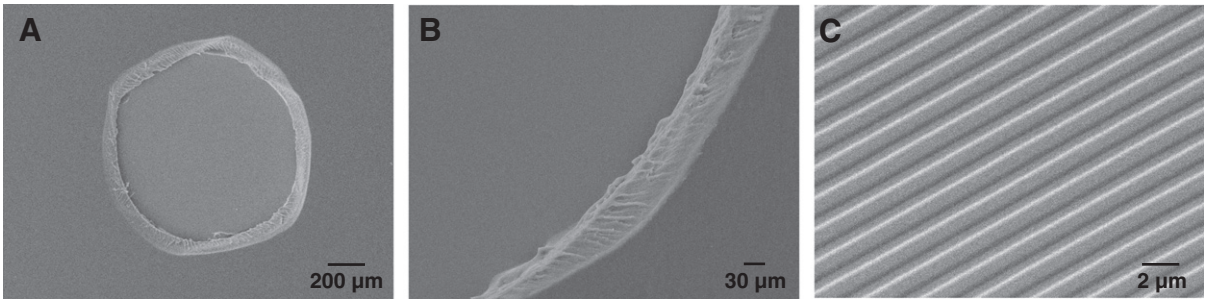

Fig. 2. Scanning electron micrographs of a vinil-CDtrode with a nominal diameter of 1.0 mm. (A) top view; (B) enlarged view of the adhesive side wall, and (C) CD tracking trails on the electrode gold surface.