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Use of Recordable Compact Discs to Fabricate Electrodes for Microchip-based Analysis Systems

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Abstract

This work demonstrates that recordable compact discs (CDs) that contain gold as a reflective layer can be used as an electrode substrate for microchip-based analysis systems. A fabrication procedure that enables the reproducible patterning of multiple electrodes has been developed. It is shown that the microelectrodes can be integrated within a PDMS-based fluidic network and used for amperometric detection of electroactive analytes at both single and dual microelectrodes. A detailed comparison is made between the CD-based patterned electrodes and electrodes made by the traditional method of sputtering gold and titanium adhesion layers onto a glass substrate. It is also shown that mercury can be electrodeposited onto a CD-based microelectrode and the amalgam electrode used to selectively detect thiols. Finally, it is demonstrated that a decoupler for microchip-based electrophoresis can be made by electrodepositing palladium onto a gold electrode and a separate downstream gold working electrode can be used for amperometric detection. These CD-based patterned electrodes are attractive alternatives for situations where device cost is of a concern or sputtering facilities are unavailable.

Introduction

The use of electrochemical detection in microchip-based analysis systems has become very popular. This is due to the inherent advantages of miniaturizing electrodes,¹ the fact that, as opposed to fluorescence detection, many analytes (including catecholamine neurotransmitters) can be detected directly without derivatization, and the selectivity that can be achieved with either judicious control over the detection potential or the use of multiple electrodes.^{2–5} The utilization of metal electrodes (usually either platinum or gold) in these devices has been widely documented. While there have been examples of placing metal wires into the microchannels,^{6–8} the most popular way of fabricating devices containing metal electrodes is to sputter metal layers onto a glass substrate (usually an adhesion layer such as titanium followed by the metal layer of interest), pattern the electrode design through traditional lithographic steps, and bond these thin-layer (~0.1–0.5 μm) electrodes with a separate layer that contains fluidic channels.^{3,9–16} These types of electrodes have been used to detect a wide variety of analytes; however, there is a significant cost (from the sputtering system and metal targets) and time (from pump-down and controlled deposition rates) associated with fabricating planar electrodes in this manner. While we^{4,10,11,13,17,18} and others^{3,9,12,14–16} have used these plates for many interesting studies, the overall cost is somewhat prohibitive for the often stated goal of inexpensive, disposable microchip devices.

One possible way to produce more economical thin-layer planar electrodes for microchip-based analysis systems is through the use of recordable compact discs (CDs). These long-term storage devices have been reported to contain a 50–100 nm reflective gold layer on a polycarbonate

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substrate and are relatively inexpensive (~\$1 per CD). A gold layer is more resistant to oxidation, as compared to other common reflective materials such as aluminum, and is preferred for long-term archival storage. Fig. 1A demonstrates the different layers that are found in such CDs. By removing the protective coating and lacquer layers, the gold layer is exposed and can be used for electroanalytical purposes.¹⁹⁻²⁴ Previous studies have defined the working electrode area either by dicing the CD^{19,22} or adding an insulating toner layer on top of the gold layer.^{20,21,23,24} The resulting electrodes have been used for static studies including cyclic voltammetry,^{19,20} potentiometric stripping,^{19,21} and self-assembly of monolayers.²² In addition, electrodes made with a toner-based method have been used with capillary²³ and microchip electrophoresis²⁴ in an end-column configuration, where the electrodes are aligned at the column/channel exit.

The purpose of this paper is to investigate whether archival CDs can be used as an electrode substrate for microchip-based analysis systems. A fabrication procedure that enables the reproducible patterning of multiple electrodes has been developed and it is shown that the microelectrodes can be integrated within a PDMS-based fluidic network and used for amperometric detection of electroactive analytes at both single and dual microelectrodes. A detailed comparison is made between the CD-based patterned electrodes and electrodes made by the traditional method of sputtering gold/titanium onto glass substrates. It is also shown that mercury can be electrodeposited on the microelectrodes and the resulting Hg/Au amalgam electrodes used to detect thiols at low potentials. Finally, it is demonstrated that a decoupler for microchip-based electrophoresis can be made by electrodepositing palladium onto a gold electrode and a separate downstream gold working electrode can be used for amperometric detection. These CD-based patterned electrodes are attractive alternatives for situations where device cost is of a concern or sputtering facilities are unavailable.

Experimental

Microchip Fabrication

Fig. 1 outlines the fabrication procedure that was used to pattern electrodes on the recordable CDs (Archival Gold[®] CD-R, Delkin Devices Inc., Poway, CA). According to the manufacturer, the CD consists of the 5 layers shown in Fig. 1A. To pattern the electrodes (see Fig. 1B), the first step involves removing the lacquer and protective coating layers by immersing the CD in 6 M HNO₃ at 80 °C for ~20 mins. Next, a positive photoresist (AZ[®] 1518, AZ Electronic Materials) was dynamically dispensed onto the CD at a spin rate of 500 rpm for 20 s, after which time the spin rate was ramped to 3000 rpm for 40 s. The CD was heated at 100 °C for 1 min prior to UV exposure (9 s @ 5.75 mW/cm², UV flood source, Optical Associates Inc., Milpitas, CA) through a positive photomask. For most studies, a high resolution positive transparency was used for the mask (3600 dpi, The Negative Image, Saint Louis, MO); however, for the 10 µm electrodes shown in Fig. 2, a positive chrome mask was utilized (Advance Reproductions Corp., North Andover, MA). The CD was then heated at 100 °C for 1 min followed by development in AZ[®] 300 MIF. Unexposed and thus still polymerized positive resist remained after the development step to protect the underlying gold layer. Any unprotected gold was wet etched with aqua regia (42 mL of HCl, 6 mL of HNO₃, and 48 mL of H₂O) at room temperature. The CD was then over-exposed with the UV flood source (30 s @ 5.75 mW/cm²) to breakdown the remaining photoresist, which was then developed in AZ[®] 300 MIF. Finally, the exposed dye layer was removed with isopropanol. Copper wire was connected to the electrode plate using J-B Weld (J-B Weld Co., Sulfur Springs, TX). Colloidal silver (Ted Pella Inc., Redding, CA) was used to make electrical contact between the copper wire and gold microelectrode.

A glass electrode plate with sputtered adhesion (titanium) and gold layers was used for comparison studies and fabricated as previously described.¹⁰ The Nanofabrication Facility at

Stanford University was responsible for sputtering a layer of titanium (200 Å) followed by gold (2000 Å) on high quality borosilicate glass. Upon receipt of the plates, electrodes were patterned in a similar fashion as described above by using a positive photoresist and UV exposure through a positive transparency. Following photoresist development, the unprotected gold was etched with aqua regia (at room temperature) followed by etching of the exposed titanium layer with Titanium Etchant (Transene Company Inc., Danvers, MA) at 85 °C. The remaining resist was then removed by an acetone rinse.

Masters used in the production of poly(dimethylsiloxane) (PDMS) microchannels were fabricated based on previously published methods.^{4,10,17} Raised microstructures of the desired dimensions were formed on a 4-inch silicon wafer using either SU-8 50 (for microchip-based electrophoresis channels) or SU-8 10 (for microchip-based electrophoresis) negative photoresist (MicroChem Corp., Newton, MA). Structure heights were measured using a surface profiler (Dektak³ ST, Veeco Instruments, Woddbury, NY), which corresponded to the channel depth of the desired PDMS structures. For experiments involving microchip-based flow analysis, the 2.6 cm (in length) PDMS flow channel had a cross-section of 100 (width) × 108 (height) μm and was made with a 20:1 mixture of Sylgard 184 elastomer base and curing agent. A hole punch was used to create a outlet reservoir, an inlet hole for fluid introduction was made with a 20 gauge luer stub adapter (Becton Dickinson and Co., Sparks, MD), and the microchip was reversibly sealed to the CD in a manner where the flow channel intersected the desired microelectrode (Fig. 2B). The effective electrode width is defined by the microchannel width (100 μm) and the electrode length was varied in this study. For experiments involving microchip-based electrophoresis, the PDMS flow channel had a cross-section 50 (width) × 27 (height) μm and was made with a 25:1 mixture of Sylgard 184 elastomer base and curing agent. The separation channel length (from intersection to the decoupler) was 1.4 cm and the side channel lengths were 0.7 cm. A hole punch was used to create buffer reservoirs. The effective electrode width is defined by the microchannel width (50 μm) and the electrode length was 500 μm.

Microchip Operation

The setup for microchip-based flow injection analysis is similar to previous work from our labs.^{10,17} The buffer flow stream was continuously pumped (Harvard 11 Plus Syringe Pump, Harvard Apparatus) through an off-chip 4-port rotary injection valve (Valco Instruments) and to the microchip. A 360 μm o.d., 50 μm i.d. fused silica capillary, fitted on one end with a 794 μm o.d. tubing sleeve (Upchurch Scientific) was used to transition from the injection valve to the microchip (see Fig. 2B). The injection valve enabled reproducible, discrete 200 nL injections of sample into the PDMS-based flow channel. For single electrode studies involving catechol as the analyte, an LC Epsilon Potentiostat (Bioanalytical Systems) was used in a 2-electrode format with a platinum counter electrode inserted into the outlet reservoir. Dual electrode studies were done in a similar manner except with a CH Instruments Bipotentiostat (812B). Studies involving the use of Hg/Au amalgam electrodes used the Epsilon potentiostat except in a 3-electrode format using Ag/AgCl reference and platinum auxiliary.

A LabSmith HVS448 3000V High Voltage Sequencer (LabSmith, Livermore, CA) was used as the voltage source for studies involving microchip-based electrophoresis. A gated injection sequence was utilized. The separation was performed by applying +400V to the buffer reservoir and +300 V to the sample reservoir, with the sample waste and decoupler set at ground. An injection was accomplished by applying 0 V to the buffer reservoir for 0.5 sec. Injection plug sizes were obtained using fluorescein and a fluorescence microscope (as previously described¹¹) and were found to be 0.32 nL. Detection was achieved at +450 mV with a CH Instruments potentiostat (810B) in a 2-electrode format using a platinum counter electrode inserted into the buffer waste reservoir.

Several studies involved the use of electrodes modified with different metals *via* electrodeposition. All depositions were carried out with a CH Instruments potentiostat (810B) in a 3-electrode format with a Ag/AgCl reference and a platinum auxiliary electrode. The gold electrode of interest was amalgamated with Hg by suspending a 5.8 mM $\text{Hg}_2(\text{NO}_3)_2 \cdot 2 \text{H}_2\text{O}$ salt solution containing 1.0 M KNO_3 and 0.5% HNO_3 over the electrode and applying a potential of -230 mV for 45 sec. For the microchip electrophoresis studies, a palladium decoupler was created by electrodepositing Pd onto a gold electrode. This was done with a 1011 mg/L palladium (II) chloride solution that was 5% in HCl (Sigma Aldrich, St. Louis, MO) and applying a deposition potential of -100 mV for 120 sec.

Results and Discussion

Fabrication and Characterization

Previous studies involving the use of recordable CDs for electroanalytical purposes have involved first removing the protective coating and lacquer layers (exposing the underlying gold layer) and either dicing the CD or adding an insulating layer on top of the gold layer to define the working electrode area.¹⁹⁻²² In order to determine whether these CD-based gold electrodes can be used as an electrode substrate for microchip-based analysis systems, a fabrication procedure had to be developed that leads to well-defined electrodes that are individually addressable, can be integrated with a fluidic network, and can be used to analyze discrete bands. The procedure outlined in Fig. 1B was developed to produce this type of electrode. After removing the protective coating and lacquer layers, a positive photoresist was spin-coated onto the gold layer and UV exposure through a positive mask was used to define the eventual electrode dimensions. A wet etching step with aqua regia was used to etch the gold layer in regions as defined by the photoresist. Previous studies involving the patterning of metal layers on glass substrates have used an acetone rinse to remove photoresist after the wet etching step.^{4,10,11,13,17,18} The use of acetone had to be avoided here, as the gold layer is on a polycarbonate substrate and acetone dissolves this polymer. Other organic solvents such as isopropanol did not adequately remove the resist; therefore, the remaining resist was removed by over-exposing the CD with UV light (using no mask) and rinsing with an aqueous developer solution.

As shown in Fig. 2, multiple electrodes of varying dimensions and spacings could be patterned with this procedure, with the size and spacings being defined by the lithographic mask. This ranged from dual 3 mm electrodes with a 1 mm spacing (Fig. 2A), a 3 mm electrode and 500 μm electrode with a 1 mm spacing (Fig 2B), and a set (12) of 10 μm electrodes with a 100 μm spacing (Fig. 2C). Previous reports of CD-based electrodes have stated that the gold layer is between 50 and 100 nm in thickness. With the archival CDs tested here, the resulting patterned electrodes were measured (*via* a surface profiler) to also be in this range, with an average step height of $70 \pm 23 \text{ nm}$ ($n=8$). PDMS-based fluidic channels could be reversibly sealed over these electrodes as demonstrated in Fig. 2B. A syringe pump was used to hydrodynamically introduce buffer into a 2.6 cm (in length) flow channel ($108 \times 100 \mu\text{m}$ cross-section) at flow rates varying from 1.0 – 5.0 $\mu\text{L}/\text{min}$ (0.15 – 0.77 cm/s) and no leakage was seen around the electrode/channel interface. Studies involving the deposition of metal layers onto glass substrates usually involve an adhesion layer (such as titanium) to improve the stability of the electrode upon exposure to a buffer solution.^{3,9-16} As shown in Fig 1A, there is no such layer in these CDs. The sealed devices could be used without any signs of delamination under most of the experimental conditions described below; however, the electrodes did occasionally delaminate when the reversibly-sealed PDMS layer was removed from the CD, with the gold layer coming up with the detached PDMS layer. The electrodes were stable for long periods of time as long as the device was not disassembled. Therefore, similar to irreversibly-bonded glass devices, the final sealed device could not be routinely disassembled and re-used.

Integration with Microchip-based Flow Analysis

Microchip-based flow injection analysis, with an off-chip injection scheme,²⁵ was used to characterize the CD-based patterned gold electrodes and determine the efficacy of using the electrodes in microchip-based analysis systems. Microchip-based flow injection analysis has been used to measure enzyme kinetics,²⁶ catecholamine release from adherent cells,²⁷ and intracellular glutathione from erythrocytes.¹⁰ In the previously described experimental setup,^{17,25} an off-chip rotary injection valve is used to inject 200 nL of sample into a capillary and to the microchip as shown in Fig. 2B. Since the most established method of using metal electrodes in microchip devices involves metal layers that are sputtered onto a glass substrate, the first characterization study involved a comparison between the CD-based electrodes and electrodes made by patterning a glass plate containing 200 Å Ti (adhesion layer) and 2000 Å Au (electrode layer). The electrode size was constant, with the width being defined by channel width (100 µm) and the length being 3 mm. A hydrodynamic voltammogram (HDV), the results of which are shown in Fig. 3A, was determined for each electrode by injecting a sample of catechol and varying the electrode potential relative to a platinum counter electrode. As shown in the figure, the HDVs are similar, with both reaching a current limited plateau at +450 mV. A calibration curve comparison was also made, with a calibration curve (1-200 µM) from the use of 2 different patterned CD electrodes being compared to a curve generated with Ti/Au electrodes patterned on a glass plate. As can be seen in Fig. 3B, the calibration curves for each are superimposable and each has similar correlation coefficients. These results show the CD-based patterned electrodes, when integrated with microchip-based flow analysis, exhibit a similar analytical performance as electrodes made by the more traditional method of sputtering well-defined layers of Ti and Au onto a glass substrate.

The CD-patterned electrodes were further characterized by repeated injections of a catechol solution into a device where the effective electrode area was 100 µm (defined by the channel width) × 500 µm (length). As shown in Fig. 4A, 5 injections of this catechol solution led to an average response of 6.80 ± 0.45 nA. The same device was subsequently used to perform a limit of detection (LOD) study for catechol. The LOD ($S/N = 3$) was estimated from the injection of a 500 nM catechol solution and found to be 240 nM. It was also seen that multiple electrodes can be patterned and integrated with microchip-based flow analysis. The set of dual electrodes shown in Fig. 2A were used to monitor the chemically reversible redox couple of catechol by operating the upstream generator electrode at +450 mV and the downstream collector electrode at 0 mV. Repeated injections of a 200 µM catechol solution led to a collection efficiency (ratio of ratio of the cathodic and anodic currents) of 35.6%.

Modification of the Patterned Electrodes

While gold electrodes can be used for the direct detection of a variety of analytes, they can be modified to increase their selectivity. One possibility is to form a Hg/Au amalgam electrode that one can use to indirectly measure thiols *via* mercury oxidation at low potentials.^{10,28,29} As we previously described, thin-layer Au electrodes can be amalgamated with Hg *via* a deposition procedure where Hg⁰ is produced *in situ* by electrochemically reducing a Hg(I) solution with the electrode of interest.¹⁰ A similar procedure was used here to create a CD-based Hg/Au microelectrode that was subsequently used to analyze a cysteine solution. The A HDV was determined by injecting a cysteine solution and changing the potential relative to a Ag/AgCl reference electrode (Fig. 5). As has been previously shown,^{10,28,29} the maximum current is achieved at a low potential (+100 mV) and at potentials higher than +200 mV the current began to decrease due to oxidation of mercury from the electrode surface. The inset of Fig. 5 shows the results from 2 consecutive injections of the cysteine solution at the optimal potential.

One popular method of integrating electrochemical detection with microchip electrophoresis involves the use of a palladium decoupler. This electrode is integrated within the fluidic network to provide an electrophoretic ground (cathode) and absorb hydrogen produced from the reduction of water.^{8,13,18,30} The decoupler provides a field-free region for downstream detection electrodes that can also be integrated within the fluidic network so that band broadening is minimized.¹³ In this work, two electrodes were patterned, a 3 mm Au electrode onto which palladium was electrodeposited¹¹ as well as a 500 μm detection electrode (1 mm spacing between the two electrodes). A PDMS microchip with a 1.4 cm separation channel (Fig 6A) was reversibly sealed over the electrodes, with the Pd/Au electrode being used as a decoupler. A gated injection scheme and 140 V/cm field strength were used to analyze a dopamine and catechol solution. As can be seen in Fig 6B, a baseline resolution was achieved with an average of 74,740 plates/m for dopamine. The response for each was also reproducible, with average peak heights being 0.51 nA for dopamine (1.4% RSD) and 0.28 nA for catechol (0.7% RSD). The Pd/Au decoupler effectively dissipated the hydrogen produced and was stable over the course of a day at this field strength; however, the use of a higher field strength (350 V/cm) led to delamination of the decoupler from the CD surface after ~ 30 mins of operation. Since this field strength can be used with sputtered palladium electrodes that include an adhesion layer,¹³ the delamination is probably due to the absence of the adhesion layer. While a baseline resolution separation was achieved for this 2 component mixture, lengthened serpentine electrophoresis channels could be used to improve the resolution¹¹ while maintaining a field strength where the CD-based decoupler is stable.

Conclusion

In this paper we have developed a fabrication procedure to reproducibly pattern single or multiple electrodes from archival CDs. It is shown that these electrodes can be integrated with PDMS-based fluidic channels and used for amperometric detection of electroactive analytes at both single and dual microelectrodes. In addition, the electrodes can be modified with mercury (for selective thiol detection) or palladium (for use as a decoupler with microchip electrophoresis). In addition to the applications shown here, these types of electrodes could be useful for researchers who are trying to develop inexpensive/disposable microchip devices, those who are interested in using electrochemical detection in microchip systems but do not have access to a fabrication facility, or for teaching purposes.³¹ While we have used photolithography to pattern the electrodes, other non-lithography methods can be used to mask metal layers for etching steps.^{24,32}

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Fig 1.

A) Depiction of the layers in a recordable CD; B) Outline of the fabrication steps used to pattern CD-based gold electrodes for use in microchip-based analysis.

Fig 2.

A) Picture of a typical patterned CD electrode plate; B) Picture of a PDMS-based flow channel reversibly sealed over a patterned CD electrode plate with the capillary being connected to an off-chip 4-port injection valve (not shown); C) Micrograph of patterned electrodes with the electrodes being 10 μm in width and separated by 100 μm.

Fig. 3.

A) Comparison of HDVs (200 μM catechol) obtained with a CD electrode plate and a traditional sputtered glass plate using microchip-based flow injection analysis; B) Comparison of calibration curves (analyte = catechol) obtained with 2 different CD electrode plates and a sputtered glass plate using microchip-based flow injection analysis (+450 mV vs. Pt counter electrode). For each comparison, electrode size = 3 mm $\times$ 100 μm , flow rate = 3.0 $\mu\text{L}/\text{min}$, and the buffer = 10 mM phosphate (pH 7.0).

Fig. 4.

A) 5 consecutive injections of a 200 μM catechol solution with detection at a 500 $\mu\text{m} \times 100 \mu\text{m}$ CD-based gold microelectrode (+450 mV vs. Pt counter electrode). B) 3 consecutive injections of a 200 μM catechol solution followed by dual electrode detection at 3 mm $\times$ 100 μm electrodes (1 mm spacing). For each the flow rate = 3.0 $\mu\text{L}/\text{min}$ and the buffer = 10 mM phosphate (pH 7.0).

Fig. 5. Hydrodynamic voltammogram (200 μ M cysteine) obtained with a CD-based Hg/Au amalgam electrode ($250 \times 100 \mu$ m in size) using microchip-based flow injection analysis. Inset shows 2 consecutive injections of the cysteine solution at the optimal potential of +100 mV (vs. Ag/AgCl reference). For these experiments, the flow rate = 3.0 μ L/min and the buffer = 10 mM MES (pH 5.5).

Fig. 6.

A) Picture showing PDMS-based electrophoresis microchip sealed over the CD-electrode plate. Inset shows micrograph of electrophoresis channel and the detection electrode. B) Electropherogram showing 3 consecutive injection, separation and detection events. Analytes = 200 μM dopamine (DA) and catechol (CAT). Decoupler = 3 mm, electrode size = 500 × 100 μm. Buffer = 25 mM boric acid with 1 mM SDS (pH = 9.2). Reservoir abbreviations: B = buffer; BW = buffer waste; S = sample; SW = sample waste.