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Characterization and fabrication of disposable screen printed microelectrodes

Rashid O. Kadara, Norman Jenkinson, Craig E. Banks *

Faculty of Science and Engineering, School of Biology, Chemistry and Health Science, Division of Chemistry and Materials, Manchester Metropolitan University, Chester Street, Manchester, M1 5GD Lancs, UK

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ABSTRACT

We report the fabrication of disposable and flexible screen printed microelectrodes which are characterised with microscopy and cyclic voltammetry. These new type of screen printed electrochemical platforms consist of micro-sized graphite typically with radii of 60 to 100 microns are defined by an inert dielectric. The advantage of this type of electrochemical sensing platform is that each microelectrode is disposable and cost effective and thus does not require extensive cleaning or electrode pre-treatment between measurements. Prior to measurements the screen printed microelectrode needs only to be calibrated with a suitable redox probe, as is typically the case with microelectrodes. We show proof of concept that the screen printed microelectrodes are advantageous for electro-analytical measurements with the example of determination of lead via cathodic stripping voltammetry. The use of graphite screen printed microelectrodes allows comparable detection limits to that obtained in the literature at insonated boron doped diamond electrodes, without the need for power ultrasound – which otherwise limits the widespread applicability and ease of measurement.

1. Introduction

In the toolkit of electrochemists, microelectrodes are versatile tools in the pursuit of mechanistic information from electrochemical processes and in addition are of high analytical interest. Microelectrodes due to the virtue of their small size experience high mass transport rates in comparison to macroelectrodes and exhibit high temporal resolution, high spatial precision and resolution and reduced solution resistance [1–4].

A variety of methods exist for the fabrication of microelectrodes and one of the most favoured approaches involves taking the desired microelectrode wire and sealing into an insulator tube such as borosilicate glass [5]. Careful choice of the borosilicate glass relative to the microelectrode wire is essential which is then heated to the glass transition temperature sealing the microelectrode wire upon cooling. This is ground down and polished to reveal the microelectrode, which should, but not always, reveal a well-sealed microelectrode. Others methods use micropipette pullers which use laser heating allowing quartz to be used in the production of microelectrodes. Whatever the chosen method, the microelectrode needs to be carefully polished to ensure a smooth and clean working electrode surface. The physio-chemical state of an electrode surface defines its performance and surface passivating layers, contaminants, chemisorbed or physically adsorbed particles which are encountered during electrochemical measurements need to be removed [5]. Additionally the microelectrode is calibrated using a known redox system to determine the electrochemically active area before measurements [5]. A major limitation is that if a careful and diligent polishing technique is not followed, voltammetric responses different from those anticipated are encountered since the electrodes can become recessed below the surface of the insulating surround [6].

One approach to avoid laborious electrode pre-treatment/polishing is through the use of disposable electrodes which can be fabricated in a variety of ways such as direct-pen, air-brushing, pad printing and screen printing [7–10] have been evaluated as novel electrode materials for electrochemical applications. Notably Fletcher and Horne have reported random assemblies of microdisks (RAM™ electrodes) where hundreds or thousands of disk-shaped microelectrodes are sealed within epoxy resin [11]. Each approach has its pros and cons depending on the applications. An established technique which has been demonstrated to be adaptable to industrial mass-production of low-cost disposable devices such as those used by diabetics to monitor their blood glucose levels, is the use of screen printing [12]. Advantageous disposable electrode designs have been reported. For example, Chang and Zen [13] have fabricated screen printed edge band ultramicroelectrodes in the range of 0.18–1.35 mm length with a width of 20 μm. This approach has been adopted by Hart and co-workers for microband glucose biosensors which was applied in the analysis of serum [14] and also in the fabrication of a lactate biosensor [15]. Other novel approaches to the use and fabrication of disposable electrochemical sensors have been microtube electrode configurations with a

* Corresponding author.
E-mail address: c.banks@mmu.ac.uk (C.E. Banks).

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"detection well" where electrodes formed via pad printing have been laser etched, effectively drilling a hole through the cross-section of the electrode [16].

To the best of our knowledge, there are no reports of disposable microelectrodes produced solely by screen printing. Note that microelectrodes have been reported but these are constructed where a dielectric layer is printed over a carbon base with holes patterned onto the dielectric via a laser to reveal the underlying carbon base. This approach can produce recessed microelectrodes with a dielectric which is not efficiently sealed to the carbon based with changes to the microstructure of the underlying carbon surface which will affect electrochemical performance [17].

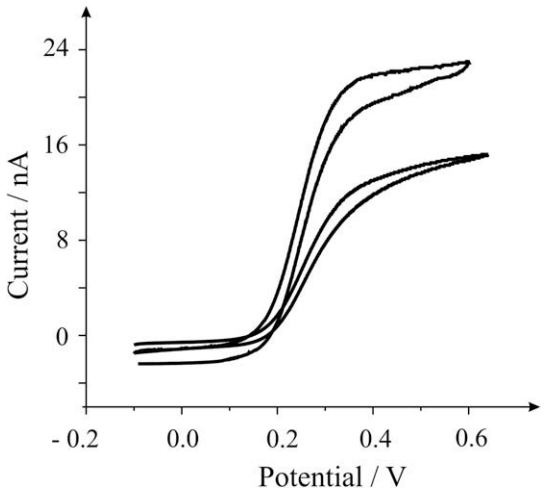

Fig. 1. Typical cyclic voltammetric profiles using a screen printed microelectrode recorded in 1 mM potassium ferrocyanide 1 M KCl at a scan rate of 5 mV s^{-1} vs. SCE.

In this paper we report the fabrication of flexible and disposable screen printed microelectrodes. The fabricated microelectrodes are characterised with microscopy and cyclic voltammetry and have radii typically in the range 60 to 100 microns. Cyclic voltammetric profiles of potassium ferrocyanide in aqueous media display low-noise, low-background, sigmoidal responses. The screen printed microelectrodes display analytical utility as demonstrated with the example of the cathodic stripping of lead which allows low micro-molar detection which allows comparable detection limits to that obtained in the literature at insonated boron doped diamond electrodes, without the need for power ultrasound – which otherwise limits the widespread applicability and ease of measurement.

2. Experimental section

All chemicals used were of analytical grade and were used as received without any further purification from Sigma–Aldrich. All solutions were prepared with deionised water of resistivity not less than 18.2 MΩ cm^{-1}.

Voltammetric measurements were carried out using a μ-Autolab III (ECO-Chemie) potentiostat using a three-electrode configuration. A saturated calomel electrode (reference electrode) and a platinum wire (auxiliary electrode) were used to complete the electrochemical cell setup. Square-wave voltammetry was employed with a step potential of 5 mV, amplitude of 25 mV and frequency of 25 Hz.

Screen printed carbon electrodes were fabricated in-house with appropriate stencil designs using a microDEK 1760RS screen printing machine (DEK, Weymouth, UK). Successive layers of two different inks, carbon–graphite ink (C2000802P2) and dielectric ink (C2070423D5) obtained from Gwent Electronic Materials Ltd. (Pontypool, UK) were printed onto a polyester flexible film. The carbon–graphite ink was first printed to obtain the conductive tracks. Onto this, the dielectric ink was used and manipulated to define the working area. The electrodes were cured in an oven at a suitable temperature before use. The detailed information of

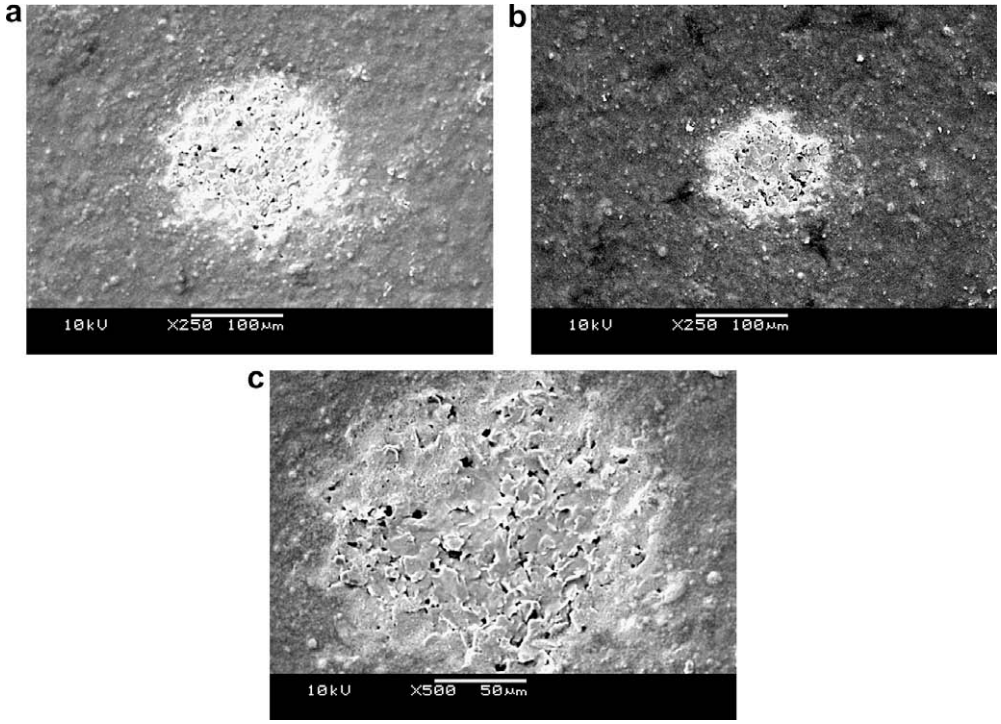

Fig. 2. Scanning electron micrographs of two screen printed microelectrodes (A, B) taken from a typical batch and a closer inspection of electrode B (C).