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Self-anchoring nickel microelectrodes for rapid fabrication of functional thermoplastic microfluidic prototypes

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ABSTRACT

Microfluidic devices play an increasingly important role in healthcare-related fields, but integration of electrodes and electronic components has been restricted at the prototyping stage of product development by a limited range of fabrication methods. In this work a new fabrication methodology is presented for embedding metallic microelectrodes in thermoplastic microfluidic devices. Microelectrodes are fabricated on steel wafers by means of photolithographic patterning and electrodeposition and then transferred to a thermoplastic sheet using hot embossing, resulting in embedded metal electrodes flush with the polymer surface. The unique shape of the microelectrodes provides an anchoring mechanism that ensures structural stability and reliability of the devices. A wide variety of thermoplastics can be used in this process including polycarbonate, polymethylmethacrylate (PMMA), and cyclic olefin copolymer (COC). Devices are assembled by a solvent-assisted bonding process, after drilling the inlets and outlets. This method allows for rapid fabrication of robust embedded electrodes and wiring connections from a broad range of metals for thermoplastic microfluidic devices. Finally, embedded interdigitated microelectrodes are used to measure conductivity within a microchannel via impedance spectroscopy analysis. The use of this technology is relevant to a wide range of analytical applications.

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1. Introduction

Microfluidic technologies have demonstrated great potential in a wide variety of fields, providing accurate and reliable handling, manipulation, and analysis of small sample and reagent volumes. Health care is particularly well-positioned to benefit from this technology, with an ongoing rise in demand for point-of-care health technologies based on microfluidics and MEMs. The wide use of this technology has allowed the development of a number of interesting applications in recent years, including concepts such as point-of-care diagnostics, organs-on-a-chip, drug screening, etc. [1–8].

Despite their promise, these lab-on-a-chip (LOC) devices have not been widely commercialized nor adopted. Several reviews have analyzed the challenges involved in LOC commercialization process, explaining some of the reasons for the delayed spread of devices into mainstream healthcare and highlighting aspects that are necessary for successful commercialization [9–11]. First, it is important to address LOC designs as a total analysis system rather than focusing on discrete modules, while still maintaining a clear and specific final analytical goal. A complete device should provide sample acquisition, preparation, reaction and analysis with complete autonomy to fully leverage the scaling benefits of microfluidic systems. Second, differences in the fabrication methods between the research process and in final industrial production should be minimized or eliminated. To this end, there has been a recent movement towards the use of industrial fabrication tools at the development level, including transitioning to commercially-viable materials like thermoplastics for the primary microfluidic device materials [12,13]. Equipment such as hot embossing machines are becoming more widely available in the research setting, opening up commercial fabrication methods for early stage prototyping in academic labs [14]. This change allows the development of low cost devices with a wide variety of material properties, increasing the potential scalability of the fabrication for both commercial applications and lab prototyping. This standardization of

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fabrication methods streamlines the translation of research outcomes into real commercial devices. Moreover, the integration of those methods at the research level will reduce the fabrication time and offer a wider selection of potential materials.

Integration of multiple materials in a single device expands the range of device capabilities. Electrodes in particular are an essential component of many devices, functioning as actuators, sensors and detectors. A common approach for integrating electrodes within microfluidic devices is to pattern and deposit a metallic layer onto a silicon or glass substrate, and then bond that to the microfluidic network (typically made of PDMS). Although this is a viable methodology, there are still some associated problems when trying to use plastics. Structural integrity and the adhesion of the metallic structures to plastic components are particularly challenging for some material combinations [15].

There are many applications in which the integration of microelectrodes within microfluidic devices is essential. The most popular current methods use Physical Vapor Deposition (PVD) or evaporation to create electrodes on silicon, glass or most recently, high temperature resistant polymers. Integration of microfluidics can be accomplished using different photoresists, like SU-8 series as a substrate for metallization or the creation of microchannels [16]. However, the material options and electrode thicknesses can be limiting. To address these limitations, alternative technologies have pursued embedding microelectrodes. Schrott et al. [15] use UV-curable PMMA as the backside to embed the microelectrodes in microfluidic devices. Jung et al. [17] or Chien et al. [18] proposed a similar approach using hot embossing to finish the embedding process. Again these works are limited to photocurable materials. Other approaches propose fabricating guides inside the PDMS device for inserting electrode wires [19] or even optical fibers [20]. And another interesting approach consists of fabricating electrodes as part of the microfluidic devices by mixing conductive particles with the polymer in defined areas [21,22]. This allows the possibility to build 3D microelectrodes within the device but requires a more complex fabrication process.

This research presents a novel methodology for the fabrication of whole plastic microfluidic devices with embedded metallic electrodes using a robust self-anchoring approach. Both the microfluidic network and the electrodes are fabricated using electrodeposition and hot embossing techniques with thermoplastic materials, enabling compatibility with scalable manufacturing processes and materials. The fast turnaround time enables this method to be incorporated from early stage prototyping through production steps of product development. In-channel electrolyte detection is characterized through impedance spectroscopy measurements using interdigitated microelectrodes (IDE) fabricated using this method.

2. Material and methods

2.1. Microfluidic device fabrication process

The microfluidic device was fabricated by means of hot embossing using a nickel insert prepared using photolithographic techniques and electrodeposition. The complete fabrication process has been depicted on Fig. 1, left column, by a 6-step workflow.

Steel wafers (90 mm diameter, 304 stainless steel 0.0293″ thick, #8 mirror finish, Stainless Supply, NC) were used as a substrate for electroplating. After a cleaning process using acetone, 2-propanol and distilled water, wafers were dried with a stream of nitrogen.

A photolithographic process was employed to transfer the microfluidic or electrode geometry to a photoresist. A 75 μm thick dry photoresist (Riston® GoldMaster Series, Dupont) was laminated on the wafers (Fig. 1A) and patterned via UV exposure with a positive mylar mask. After photoresist development in 1% w/v potassium carbonate, the back of the wafer was covered with a vinyl sticker to shield it from the electroplating process. Next, a nickel strike pretreatment was performed to promote strong adhesion of the deposited nickel layers to the steel substrate. Specifically, the patterned wafer was connected to the negative lead in a 55 °C bath of Wood's Nickel Strike (12.5% HCl 240 g/L Nickel chloride) solution, and a 1.0 A current was applied for 2 min with vigorous stirring in the bath. Next, nickel was deposited onto the open areas of the wafer (Fig. 1B) via electrodeposition in a 55° nickel sulfamate solution (#42027, Alfa Aesar, Inc). The remaining photoresist was removed in a 3% w/v potassium hydroxide solution. A variation on this method using vinyl adhesive rather photoresist was previously described by Novak et al. [14] for fabricating nickel masters for microfluidic devices.

Table 1
Glass transition temperatures (T_g), recommended embossing temperature (T_e) and demolding temperature (T_d) and bonding temperature (T_b) for thermoplastics tested in this work Biographies.

Thermoplastic	T_g (°C)	T_e (°C)	T_d (°C)	T_b (°C)
Cyclic olefin copolymer (COC)	136	155	115	110
Polycarbonate	147	160	120	120
Poly(methyl methacrylate) (PMMA)	105	120	85	80
Polytetrafluoroethylene (Teflon)	250	275	225	–
Polystyrene	95	110	75	–

The deposition rate is proportional to the applied current-controlled electrical field density. Low current was used to maintain low current density in the small features. This also helps achieve more homogeneous deposition and avoid the lipping of feature edges. Currents between 0.15 and 0.5 A were used for electrodeposition depending on the sizes of the structures. Features were electroplated to a height of 50 μm.

Hot embossing was then used to produce the microfluidic device in 1.5 mm thick polycarbonate (McMaster-Carr, Inc.). The nickel-patterned wafer and polycarbonate were placed in contact between two heated platens on a hydraulic lab press. After the system was heated to 10–15 °C above the glass transition temperature (T_g), a pressure of 5–40 MPa (Fig. 1D) was applied. The system was then cooled to 20 °C below the T_g, and the polycarbonate was separated from the wafer, leaving the embossed microchannels (Fig. 1E). Table 1 shows the temperatures used in this work for different materials in the fabrication of both the microfluidic devices and the embedded microelectrodes.

2.2. Microelectrodes fabrication process

Microelectrodes were fabricated with a modified version of the microfluidic channel fabrication process (Fig. 1, right column). A spin-coated positive photoresist (2 to 5 μm thick, S1818, Shipley Microposit) was used to define the microelectrode geometry. During the electroplating step, the nickel strike pretreatment was omitted to allow a weak bond between the nickel and the steel wafer. The nickel was over-electroplated by allowing the nickel height to exceed the thickness of the photoresist, with typical heights from 5 to 15 μm. Electrodeposition rates of 0.5 to 1 μm/min were used. As the plated nickel exceeds the photoresist height, it begins to accumulate horizontally as well as vertically, generating "hook" features. These features create a self-anchoring mechanism allowing the electrodes to separate from the steel wafer and remain securely embedded in the thermoplastic during the hot embossing process. The cross section of the electrodes resembles a mushroom shape after removing the photoresist with acetone (Fig. 1C).

Hot embossing was used to embed the microelectrodes using the same parameters as previously described. During this process, the plastic flows to accommodate the electrodes and fill the space

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