A FABRICATION METHOD FOR INTEGRATING ELECTRODES IN NANOFLUIDIC CHANNELS

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ABSTRACT

Nanofluidic channels are unique bioanalytical tools that can separate, detect, analyze and concentrate biomolecules. Embedding electrodes in such systems increases the functionality by allowing for unique flow control, label-free sensing, and concentration enhancement. In this work, we describe the fabrication process of nanofluidic channels with integrated electrodes, as well as the characterization of such channels. The key aspects of our surface-micromachined process is the use of a side-release etch to overcome diffusion limited etching of long thin channels, and a low-temperature process (<300 C), to allow for for the integration of addressable electrodes for a variety of bioanalytical applications.

KEYWORDS: Nanofluidic, Fabrication, Integrated Electrodes

INTRODUCTION

Compared to more conventional nanoporous systems, such as electrophoresis gels, microfabricated nanochannels provide better dimensional precision and control, resulting in new functionality and enhancing device performance [1-11] for biomolecule separation [1-4], sieving [5-6], and pre-concentration [7]. Furthermore, embedded electrodes can be fabricated within nanofluidic channels to manipulate biomolecules both spatially and temporally, impart a surface charge on the channel [12], change the polarity for concentration enhancement [13], and/or carry radio-frequency signals for sensing applications [14]. Additionally, such channels can be used to study fundamental and unique coupled physics at the surfaces of substrates by both sensing molecules in-situ and controlling the surface potential [12, 14, 22]. Despite these promises, the nanofluidic fabrication process is a key limitation.

The fabrication methods for such channels can be classified into two main categories, wafer bonding and surface micromachining with sacrificial etching. The bonding method offers process simplicity and has no limit on channel length since the channel is etched into one wafer and bonded to the other. However, this approach is limited to a single level and the process often demands either high temperatures (>1000°C for bonding for fused silica) [3-4] or the inclusion of adhesives and precise wafer alignment for bonding [15]. The sacrificial micromachining method is based on surface MEMS techniques where the channels are formed by removing a thin sacrificial layer by wet, plasma or vapor phase chemical etching. While wet chemical release provides good selectivity, the presence of liquids poses severe challenges on structural integrity and surface contamination [16]. Plasma release avoids those problems but is ineffective in removing materials without line of sight. Vapor release provides the advantages of both approaches. In particular, XeF₂ has been demonstrated to have superior selectivity between silicon and silicon dioxide [17]. However, removing sacrificial layers from long nanochannels remains a challenge due to the diffusion-limited long etching times described by the Deal-Grove model [17].

In this paper, we describe a fabrication method for centimeter-long nanochannels with metal electrodes using a novel side-release based sacrificial micromachining method with XeF₂. We demonstrate that all steps in this process are kept under 300°C, so that this method can safely integrate metal layers into centimeter-long nanochannels. The resulting devices are compatible with CMOS processes and therefore a major step toward truly handheld devices.

FABRICATION

The general fabrication process is illustrated in Fig. 1. The key to our fabrication method is the formation of intermediate sacrificial structures that are released on along the length of the channel features and the sealing step of the nanoscale gap with a CVD layer. This allows for us to overcome the difficulty of diffusion-limited etching [18] in these high aspect ratio channels. The minimum lateral dimension of the resulting channels is determined by the lithographic step defining the lateral edges of the channels and the amount of sacrificial material deposition in the thin channel gap.

For our devices, the substrate is a 525 µm thick, 100 mm diameter, double side polished fused silica wafer (supplier: Hoya Corp.; model# 4W55-325-15C-STD), on which we deposit a 500 nm thick silicon nitride (SiNx) layer with parameters shown in Table 1 by plasma-enhanced chemical vapor deposition (PECVD). After annealing the wafers at 300°C to stabilize the nitride film and reduce defects in subsequent depositions, a 50 nm thick layer of SiO₂ is deposited by PECVD which will act as the floor of the nanofluidic channel. The metal electrodes are patterned using a 2-layer photolithography lift-off process with a sputtered Cr-Au-Cr stack. Next, 200 nm of sacrificial amorphous silicon (a-Si) and 20 nm of SiO₂ are deposited by PECVD. The thickness of the sacrificial layer determines the height of the nanochannel and the 20 nm SiO₂ layer forms the top wall of the nanochannel. Patterning of the sacrificial layer sandwich is done using CF₄/O₂ chemistries which etch SiO₂ anisotropically at ~150 nm min⁻¹ and a-Si at ~180 nm min⁻¹. A 50 nm thick layer of PECVD SiO₂ is deposited followed by a
1 μm thick PECVD SiN_x, both at 100°C. The SiO_2 and SiN_x layers form the channel wall materials and are subsequently patterned to expose the a-Si sacrificial layer by using a CHF_3-based etch. We then use pulsed XeF_2 vapor to release the channel through an isotropic etch. For XeF_2, the pulsing removes the gaseous waste of the etching reaction and a 60 s pumping time is implemented at the end of each etch cycle. The etch process has a >1000:1 reported selectivity over SiO_2 [19-21] and is highly sensitive to moisture. For a 200 nm thick sacrificial layer and channel width of 5 μm, a single pulse of 90s each at 4 Torr was sufficient to complete the etching process. Next, we apply a high density oxygen plasma at 250°C to remove autofluorescence and change the surface of the channel from hydrophobic to hydrophilic for better compatibility with bioanalytical systems. These properties are a result surface fluorination caused by XeF_2.

At this point, a gap is formed between the substrate and channel wall. The size of the gap is determined primarily by the thickness of the sacrificial layer and the stress gradient in the released films. A 500 nm thick PECVD SiO_2 layer (process parameters described in Table 1) is conformally deposited and seals this gap, thus completing the nanochannel. Finally, a CHF_3-based etch step is used to create access to the channel and the photoresist used to pattern the access holes is removed by O_2 plasma. Note that the channel is completely sealed during the last lithographic step, thus minimizing channel contamination.

Table 1. Typical deposition parameters for nanochannel process

<table>
<thead>
<tr>
<th>Process name</th>
<th>Parameters</th>
</tr>
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<tbody>
<tr>
<td>PECVD SiO_2</td>
<td>SiH_4/O_2/Ar flow rates: 7.5/15/20 sccm; Pressure: 5 mTorr; Power: Source=800W, Bias= 20W; Temperature: 100°C</td>
</tr>
<tr>
<td>PECVD SiN_x</td>
<td>SiH_4/N_2/Ar flow rates: 15.5/8.5/20 sccm; Pressure: 10 mTorr; Power: Source=800W, Bias=120W; Temperature: 100°C</td>
</tr>
<tr>
<td>PECVD a-Si</td>
<td>SiH_4/Ar flow rates: 15/20 sccm; Pressure: 1.5 mTorr; Power: Source=400W, Bias=35W; Temperature: 100°C</td>
</tr>
<tr>
<td>Post deposition annealing</td>
<td>250°C with 5°C/min ramping speed</td>
</tr>
</tbody>
</table>
RESULTS AND DISCUSSION

We have completed and characterized, surface micromachined centimeter long nanofluidic channels without integrated metal electrodes [18], as shown in Figure 2. Fabricated devices were characterized using capillary filling and current monitoring techniques [22]. The channels behaved as electokinetic flow theory predicts [18] and therefore are viable channels for useful nanofluidic based bioanalytical systems.

Figure 2: Device characterization for surface micromachined nanochannels (adapted from [18]). The wafer consisting of 8 different channel geometries is placed on top of a microscope for inspection during both capillary filling and current monitoring experiments. For capillary filling, a camera (Andor, IXON +) records image data of parallel channels filling. For current monitoring experiments, electrodes are placed in the channel well and the current is monitored for various applied voltages and ionic concentrations, following that of [22].

Furthermore, we have completed fabrication of channels with electrodes, however, channels have not been completely released due to errors in the tolerances used to define the fluidic mask. Figure 3 shows a fully released gate electrode fabricated into an unsealed 7 µm wide nanogap with a 10 µm wide addressable gate electrode spanning the width of the channel. Errors in the tolerance of the features on the mask resulted in release lengths greater than 10 µm on the “cross geometry” features and therefore resulted in nanochannels that were not complete, as shown in Figure 4. Attempts at more cycles of XeF₂ failed due to the inefficiency of the diffusion limited process for long release lengths. Given the success of our previous work [18], future work to complete these channels should be straightforward with better designed masks using the same conformal CVD sealing and CHF₃ fluidic access etch.

CONCLUSION

Nanofluidic systems are in need of novel fabrication methods in order to incorporate metal electrodes within the channels for sensing, flow control, and concentration enhancement applications. Here, we developed a low-temperature fabrication process for nanofluidic channels with integrated addressable electrodes, characterized homogenous channels fabricated using this process, and showed results of more complicated heterogeneous channels. Compared to conventional methods, our process eliminates the maximum length limitation imposed by the Deal-Grove diffusion by using a side release and CVD seal.
The low temperature process and dry release method have enabled integration of metals which can be used in a variety of bioanalytical applications.

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