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Procedia Engineering 25 (2011) 827 – 830

**Procedia
Engineering**www.elsevier.com/locate/procedia

Proc. Eurosensors XXV, September 4-7, 2011, Athens, Greece

TMMF dry film resist as masking layer in deep etching of Pyrex-glass for microfluidic chip fabrication

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Abstract

We present the application of dry film resist (TMMF) as new masking technology to process glass wafers using standard wet etching. The considered microfluidic chip features patterned metal electrodes for impedance sensing and hydraulic access holes fabricated on a Pyrex wafer. Therefore, deep etching of through holes, while protecting the deposited metal structures on the Pyrex glass wafer, is required. To achieve this, amorphous silicon (a-Si) was used as masking layer on one side and unexposed TMMF dry film resist was used as mask on the other side of the wafer. The a-Si and TMMF are excellent masking layers in deep-wet etching for Pyrex wafers with etch duration more than 1 hour in 49% hydrofluoric (HF) acid.

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Introduction

In many microfluidic device fabrications Pyrex glass is among the materials of choice due to its excellent optical accessibility, biocompatibility, bondable to silicon substrates and established processing technology. Microfabrication on Pyrex glass substrates is normally performed through a wet etching process which produces isotropic wall profiles. Alternatively, micromachining of Pyrex glass using deep reactive ion etching (DRIE) yields vertical wall profiles, but the etch rate is relatively slow [1]. In some applications Pyrex glass substrates are used as enclosures to form micro channels. In order to introduce the liquid into the micro channel, suitable liquid access holes are fabricated and frequently situated on the

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Pyrex glass substrates. Depending on the complexity of the design, machining through holes on Pyrex glass substrates can be performed using mechanical drilling, sand blasting and ultrasonic drilling [2]. An alternative to these techniques is the deep wet etching process that is cost efficient as well as suitable for batch fabrication. The primary requirement for the wet etching process is the masking layer to selectively protect the Pyrex glass from the concentrated hydrofluoric (HF) acid etching solution. There are several masking candidates that are capable to withstand at certain duration during the etching process in a HF bath. A list of such masking candidates has been reported for example in [2, 3]. However, to achieve deeper etch depth, the masking material must withstand the HF for an extended period of time. In general the etch rate depends on the oxide composition in the glass material, the concentration of hydrofluoric acid and the temperature.

An interesting material that can be considered as mask for deep etching of Pyrex glass is epoxy based dry film resist. Since most epoxy based materials are known to have good electrical, thermal and chemical resistance, also a dry film epoxy resist should have similar properties and should protect well against HF acid. In this work we present the fabrication process for a microfluidic dispenser chip used for impedance spectroscopic study of biomaterials using two different materials as etch masks. The Pyrex glass substrate features the patterns of metal electrodes and of the hydraulic access holes. The fabrication of Pyrex substrate was performed using standard deep wet etching process with two different masking materials. A dry-film resists (TMMF) was used as protection to metal electrodes at one side of the Pyrex glass wafer and amorphous silicon was used as masking layer on the other side.

Chip design

The microfluidic dispenser chip as shown in fig. 1 is designed to perform single cell printing by confining single cells into picoliter sized droplets[4]. This chip features a micro channel to transport cells and embedded electrodes for electrical detection of single cells. The fluid channel was fabricated on the silicon substrate while the electrodes, electrical pad interfacing and hydraulic access holes were fabricated on the Pyrex glass substrate. Both substrates were bonded together by anodic bonding and subsequently diced to form the microchip.

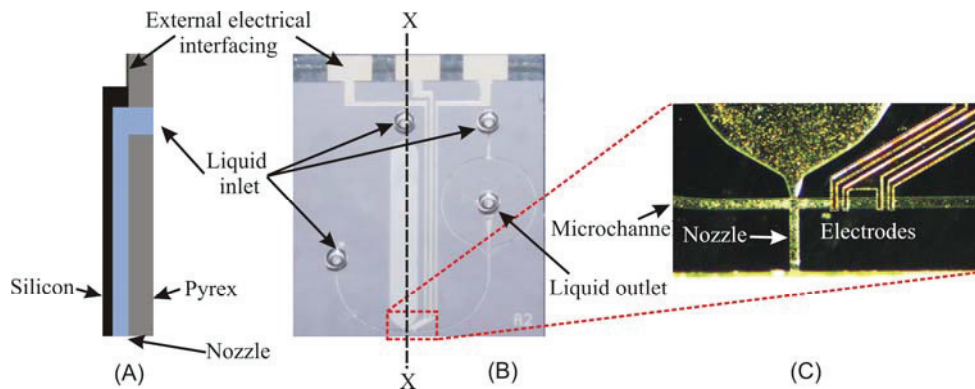


Fig. 1. The microfluidic dispenser chip fabricated from silicon and Pyrex. The schematic drawing shows (a) a cross sectional view X-X, (b) a photograph of a finished microfluidic chip with 8mm x 10mm size and (c) enlarged view at the nozzle and co-planar pair electrodes.

Fabrication technology and discussion

Chip fabrication was divided into two sections: processing the silicon wafer, and processing the Pyrex wafer. The fabrication process for the silicon wafer is summarized in fig. 1(a). The micro channel was etched 40 μm depth via the Deep-RIE (DRIE) process. To fabricate the window opening for external electrical interfacing, the DRIE process was performed on the other side of the wafer as well. Finally the wafer underwent a thermal oxidation process to grow 500 nm thick SiO_2 as passivation layer to prevent Ohmic contact between the electrodes and the silicon.

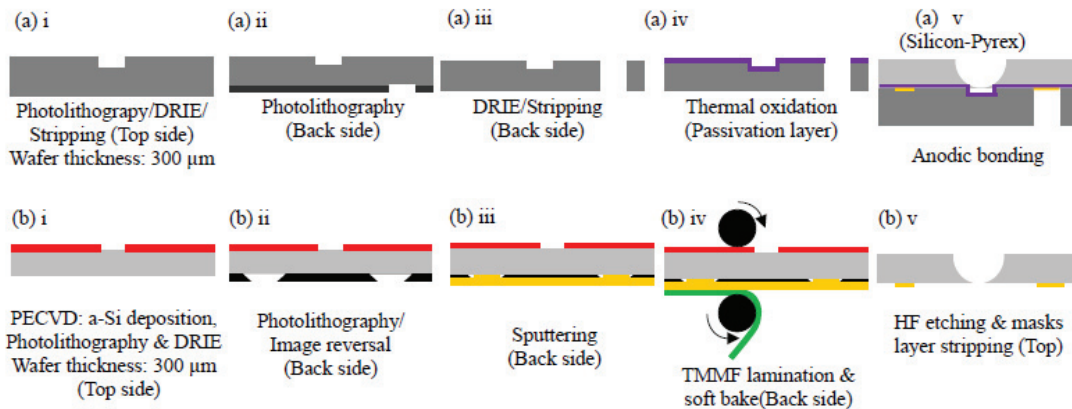


Fig. 2. Fabrication process steps for the microfluidic-chip. (a) i to (a) v: The fluid channels and electrical access holes are developed on the silicon wafer. (b) i to (b) v: The electrodes of the sensor and hydraulic access holes are developed on the Pyrex wafer (bottom). (a) v: Both wafers are bonded via anodic bonding

Processing the Pyrex wafer via deep wet etching requires masking layers on both sides to protect the electrodes and glass from HF attack. Fig. 2b shows the fabrication steps to process the 300 μm thick Pyrex wafer. Firstly, 500 nm thick a-Si was deposited on one side of the Pyrex wafer via PECVD. Then, fluidic access holes were defined on the wafer via photolithography (AZ 4533 – MicroChemicals GmbH, Germany) followed by DRIE opening of the a-Si layer. Subsequently, the 100/200 nm thick Ti-Wt / platinum electrodes were patterned on the other side of the Pyrex wafer via image reversal (photoresist : AZ 5214E – MicroChemicals GmbH, Germany) followed by a sputtering process. The lift-off of the sacrificial metal layer was not yet performed at this stage (see below). In order to protect this surface from HF attack, a layer of TMMF dry film resist (30 μm) was laminated, followed by soft bake at 80°C for 3 minutes.

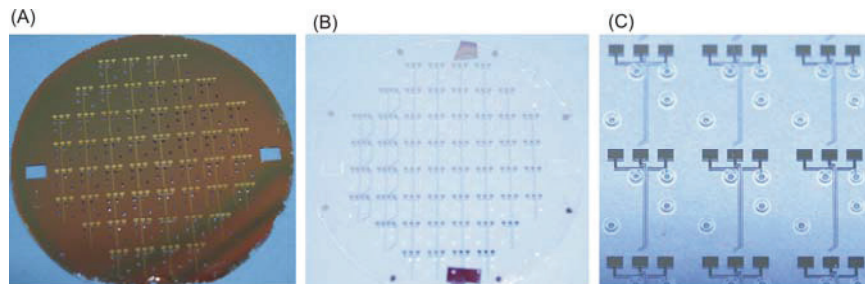


Fig. 3. Post-etching process (mask stripping). (a) After stripping the TMMF layer in SU8 developer solution and lift-off. (b) After DRIE process to remove the thin a-Si layer. (c) A closer view of the Pyrex wafer with patterned platinum electrodes and fluid access holes shows the good definition of metal structures as well as through holes.

The TMMF layer was not developed after the soft bake. It remained unexposed throughout the whole wet etching process. Therefore, the entire wet etching process was done in a yellow room by using concentrated HF acid (49% v/v) until the through holes were completely etched through the wafer.

Stripping-off the masks after the etch requires three process steps: First the TMMF layer was delaminated. Since the TMMF was not cross-linked (unexposed), it can be easily striped-off or dissolved into Propylene glycol monomethyl ether acetate solution (SU8 developer) within 10 minutes. The subsequent stripping step of the sacrificial photo resist layer (see above) to create the metal structure was performed by a standard lift-off process. Final, stripping-off the a-Si layer on the other side of the Pyrex wafer was achieved either by a DRIE process (3 minutes) or wet etching in Tetramethylammonium hydroxide (TMAH) solution.

Another important step to improve the duration of the a-Si layer to remain intact throughout the etching process was to hard bake and to maintain the photoresist layer that has been used for patterning the a-Si. Using this approach, a-Si layers can withstand for more than one hour in HF solution [5]. The etch rate for Pyrex glass in 49% HF solution was approximately 5 $\mu\text{m}/\text{min}$. Etching the through holes on the 300 μm Pyrex wafer lasted approximately 60 minutes. It turned out that unexposed TMMF resist layers as well as a-Si can withstand etching duration for one hour or even longer. Fig. 1c shows the final dispenser chip after anodic bonding of the silicon and Pyrex wafers. The metal structure was well defined and preserved. The Pyrex wafer surface was free from residuals and bonded hermetically with the silicon wafer.

Conclusion

We have presented epoxy based dry resists as alternative masking material for deep wet etching of Pyrex glass wafers. The TMMF layer applied in this work demonstrated excellent masking properties which remained intact in concentrated HF acid for more than one hour.

Acknowledgements

We acknowledge support from clean room staff (RSC-IMTEK) for helping in device fabrication. A. Y. acknowledges the Ministry of Higher Education Malaysia for granting the graduate scholarship.

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