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ScienceDirect
Nuclear Instruments and Methods in Physics Research B 260 (2007) 362–365
NIM B
Beam Interactions with Materials & Atoms
www.elsevier.com/locate/nimb

Rapid prototyping of micro/nano poly (methyl methacrylate) fluidic systems using proton beam writing

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Available online 14 February 2007

Abstract

A technique has been developed for the rapid prototyping of enclosed micro/nano polymethyl methacrylate (PMMA) fluidic systems using proton beam writing (PBW) and thermal bonding. Micro/nano structures consisting of channels and reservoirs were fabricated in a PMMA resist layer coated on to a Kapton substrate using a focused MeV proton beam. By thermal bonding these structures are fixed to a top bulk housing of PMMA, peeling off the Kapton substrate, and bonding the remaining exposed side to PMMA, enclosed high-aspect-ratio nano/microchannels can be fabricated. The key to the process is bonding the PMMA housing to the patterned resist under suitable conditions, to ensure that the bond strength is higher than the adhesion between the resist to the Kapton substrate, while ensuring that the deformation of the patterned structures caused by bonding temperature and pressure is minimised. Experiments showed that the optimum bonding condition is at 105 °C with a pressure of 1.2 Bar for 2 h.

PACS: 07.78.+s; 85.85.+j

Keywords: Proton beam writing; PMMA nano enclosed channels; Fluidic chips

1. Introduction

In the past decade, micro fluidic systems have been successfully designed and mass-produced for both basic research into chemical and biological phenomena, and used in chemical and medical applications [1,2]. This is a high potential area, and is rapidly growing to meet the demand for fast analytical processes for applications of high-throughput screening, gene expression analysis and pharmacogenomics [3].

Micro fluidic systems typically consist of three dimensional channels and are conventionally fabricated from silicon or glass using optical lithography. This technology has been adopted mainly as a consequence of the expertise gained during decades of research and development in the IC industry [4]. However, the high cost of a glass or silicon micro fluidic system limits applications, and more recently polymer micro fluidic systems have been suggested as good alternatives. Polymer fluidic devices can be fabricated from a master by thermal embossing, injection molding and thermal forming etc., which are cheap to produce and therefore disposable [3,5].

In an ever increasing trend for miniaturisation, there is also an increasing interest in developing sub-micron and nano fluidic devices for the future. Nano fluidic systems however are still at the research stage, since channel dimensions can be of the order of the size of the molecules under investigation, and therefore it is necessary to study molecular behavior at the single-molecule level [6–11,12]. Currently a major research interest of nano fluidic devices is the study of single molecules, especially bio-molecules e.g. DNA detection and handling [6,12–14]. Though this is a young and challenging field, it promises important applications in biological science such as quick mapping of restriction cut genomic DNA segments, and localizing transcription factors for protein synthesis to a specific gene or even a specific binding site [6].

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doi:10.1016/j.nimb.2007.02.047

However, fabricating fluidic devices with sub micro feature dimension is currently expensive and challenging which is the main barrier for research to explore the tremendous potential of nanofluidics [14]. Since there are technical difficulties in pushing feature sizes below the optical diffraction limit of photolithography, other fabrication technologies such as UV and electron beam lithographies are coming into play. In addition, polymer materials are considered as promising materials due to their various mechanical, optical, electrical properties, giving more choices for application and fabrication [1,3,5].

Among the new nano fabrication technologies, proton beam writing (PBW) has been demonstrated as a successful one-step process for direct writing high aspect ratio structure with smooth vertical walls in a relative thick PMMA and SU8 resist at sub-100 nm resolutions [15]. Because PBW can fabricate high aspect ratio deep channels with sub micron feature dimensions (for 2.0 MeV protons, the lateral straggling and proximity effects are confined within 100 nm down to a depth of 13 μm below the surface in PMMA resist [20]), and PMMA is a biologically compatible material with good transparency, PBW is very suitable for fabricating nano fluidic systems for biological applications.

Since PBW is direct write and therefore a serial process, PBW is not economic for mass production: However PBW is a rapid direct writing process, which does not require a mask, and hence it can be used as an efficient prototyping tool. In a comparison with focused ion beam (FIB) milling, which uses a low energy heavy ion beam, PBW is approximately one million times more efficient in removal rate per incident ion [18]. We have also shown that PBW when coupled with 248 nm UV lithography to fabricate the bulk part of a pattern with feature sizes greater than a few microns [21], considerably increases the efficiency of PBW for fluidic chip fabrication. For constructing a nanochannel system, a pattern up to $500 \times 500\ \mu m^2$, can be written with a electromagnetically scanned proton beam in Center of Ion Beam Applications, National University of Singapore, and scanned patterns can be combined up to $25 \times 25\ mm^2$ by coupling with precise stage movement. Depending on the spatial resolution and complexity, a pattern of $25 \times 25\ mm^2$ can be realized in a few hours [18].

Although it is possible to fabricate a fluidic device with open channels [14], it is normal to construct such a device with enclosed channels to limit evaporation. A typical fluidic system is therefore constructed with enclosed channels and chambers. For microfluidic systems, there are commercially available processes to seal silicon or glass channels, while for polymer micro fluidic systems, adhesive bonding is normal practice [16]. However for nano fluidic systems, damage or blockages may occur with traditional bonding processes, e.g. in adhesive bonding the adhesive may be sucked into the nano fluidic channels by capillary force. Therefore new bonding technologies have been developed such as the shadowing technique which involves sputtering silicon dioxide over the nanochannels at a wide distribution of angles to seal the channel off at the upper surface [17]. Here angled sputtering particles deposit more on the side walls of the nanochannel and the bottom part of the channel is hidden by the shadow of the side walls and is left as a void. Another sealing process is to imprint a template of SiO_2 with nanochannels into a thin polymer film coated on a glass substrate [13]. Since the polymer film is too thin to bury the channel completely, the bottom part of the channel is left as void. However, such sealing technologies are expensive and the efficiency is not high.

To seal channel and chamber structures fabricated by PBW, we developed a technique by which patterned PMMA resist can be transferred to a bulk PMMA housing from a substrate and then sealed with a thin PMMA sheet by thermal bonding.

By using this method, enclosed fluidic channel systems can be constructed which can work as prototypes not only for commercial device development but also for research investigations which need small numbers of devices. Because the PBW pattern can fabricated into a nickel master by electroplating [19], the process can also be used for for mass production using hot embossing. This type of prototyping technology has high potential, since it can be transferred to mass production after initial optimization.

2. Experimental

A schematic representation of the process of rapid prototyping of a micro/nano fluidic system using PBW is

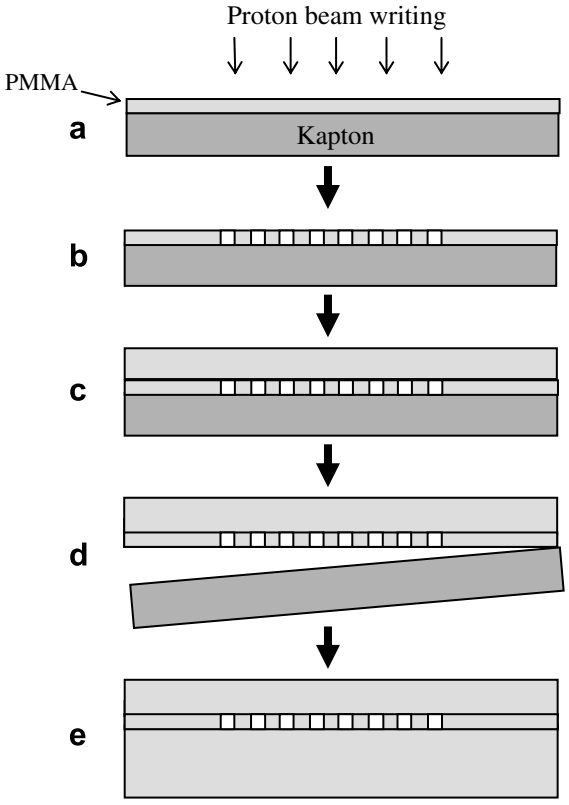

Fig. 1. Schematic representation of the process of the enclosed channel fabrication using proton beam writing.

shown in Fig. 1. This process involves: (a) Coating a PMMA resist layer on a Kapton substrate and then patterning the resist layer by PBW. (b) Developing the exposed resist, and then aligning a top housing of bulk PMMA on the patterned PMMA resist layer. (c) Thermal bonding the top housing to the patterned resist layer. (d) Cooling and pealing the Kapton substrate from the resist. (e) Bonding the top housing with the transferred patterned resist layer to a thin PMMA sheet to get a total PMMA fluidic device with enclosed channels.

The Kapton substrate is a 50 μm thick commercial film (Kapton® HN from DuPont), which was cleaned by acetone. On to the Kapton substrate, a layer of 10 μm PMMA resist (950 k molecular weight, 11 wt% in anisole, from Microchem) was spin coated in multiple coating and baking steps, the baking steps were at 170 °C for 10 min. In addition a 2 μm thick PMMA resist layer was coated on to an other Kapton substrate in a single coating step process. The lithographic patterning was carried out using the PBW facility at the National University of Singapore [15,19]. The 10 μm thick PMMA resist was machined with a focused 2 MeV proton beam while the 2 μm thick PMMA resist layer was machined with a focused 2 MeV H_2^+ beam.

After PBW exposure, the PMMA resist layers were developed in IPA-water (7:3) developer for 10 min at room temperature without agitation.

We then investigated the bonding process by bonding the top housing to the patterned PMMA resist on the Kapton substrate at a variety of different temperatures and pressures. For each set of parameters we peeled off the Kapton substrate and observed the exposed structure under SEM for signs of distortion and damage. To decrease deformation of the patterned resist layer, we placed a PDMS (Sylgard 184, Dow Corning Corp.) cushion under the Kapton substrate during the bonding process to equalize pressure.

The most critical part of the process is to bond the bulk PMMA top housing to the patterned resist layer with a strength higher than the resist to the Kapton substrate while minimising any deformation. Once the structure is sealed (as in step (e) of Fig. 1) it is then difficult to assess any distortion. However we also observed a chip with nanochannels after it was completely sealed (as in step (e) in Fig. 1) by machining an enclosed nanochannel structure with a 2 MeV H_2^+ beam to expose cross-sections of the enclosed channels (see Results section).

3. Results

Following the process steps in Fig. 1, a pattern with two orthogonal rows of 4×4 μm^2 pillars with a spacing of 1 μm was fabricated on a 10 μm thick PMMA layer by PBW. The patterned resist was then bonded to a bulk PMMA top housing and the Kapton substrate was pealed off gently (see Fig. 1 step (a)–(d)). We did not proceed with the step (e) in order that the transferred structures can be observed under SEM. Experiments showed that when the bonding temperature and pressure are low, the bonding strength between PMMA bulk housing to the patterned resist layer is low and the patterned PMMA resist will stay on the substrate rather than be transferred to the bulk PMMA housing. However when bonding temperature and pressure is high the deformation of the patterned resist layer is large and the channels will not be precise and may even detach after bonding. Our results indicated that the process is optimized at a pressure of 1.2 Bar and with a bonding interface temperature of 105 °C. As shown in Fig. 2(a) and (b), with the optimized process, the pattern did not show obvious signs of deformation after it was successfully transferred to the bulk PMMA top housing. This top bulk PMMA housing with the transferred micro channel structure can be easily sealed by bonding a PMMA film (see Fig. 1 step (e)). Because the in this process step, the pressure and temperature is the same as step (d), so the structure will have the same precision as after process step (d). This enclosed channel system is a fluidic chip to sort biological cells using a micro-sieve and electrophoresis.

A chip with nanochannels was also fabricated for DNA separation. A 2 MeV H_2^+ beam was focused into a 200×300 nm^2 spot size and scanned as a series of linear channels with cross micro channels over a 2 μm thick PMMA resist layer spin coated onto a 50 μm thick Kapton substrate. The full procedures (a)–(e) were carried out as

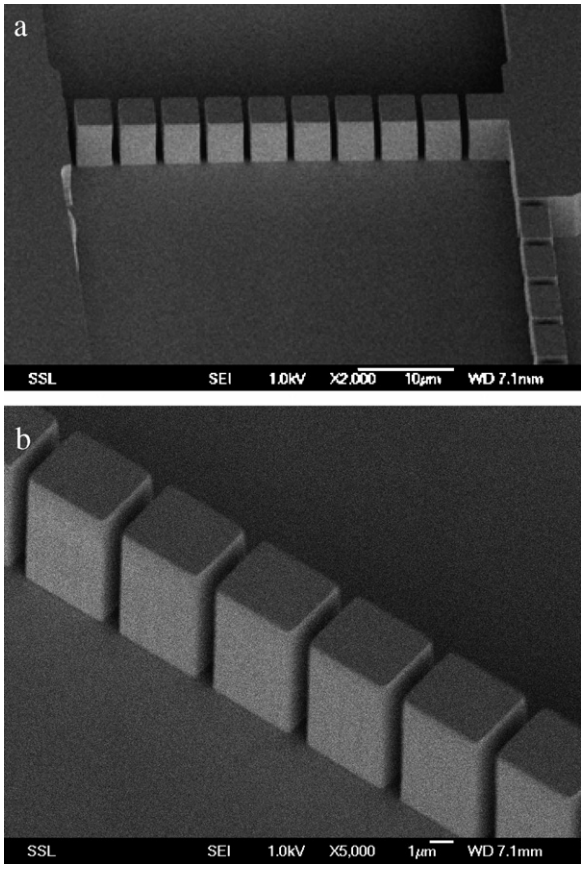

Fig. 2. (a) PMMA micropillars transferred to bulk PMMA from a Kapton substrate, (b) high resolution image of (a). The grooves are 800 nm wide.

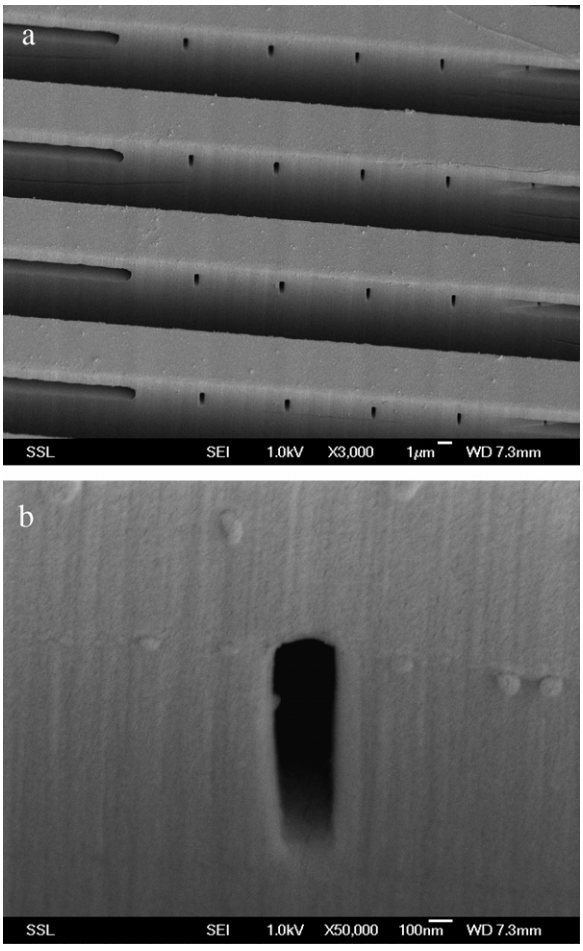

Fig. 3. (a) PMMA enclosed nanochannels fabricated using proton beam writing coupled with thermal bonding, and (b) close up of one of the buried channels. The channels are 200 nm wide and 2 μm deep.

described in Fig. 1. In order to visualize the enclosed channels, the final structure was micromachined laterally across the nanochannels at a depth of around 20 μm using proton beam writing in order to expose the cross-sections of the enclosed channels. As shown in Fig. 3(a) and (b), the channels are well formed with minimal deformation. Because a 3 mm thick PDMS cushion was placed under the 50 μm thick Kapton substrate or the thin PMMA sheet in the step (d) and (e) during bonding process, the pressure is rather uniform due to the flexibility of the PMDS, thin Kapton substrate and the PMMA sheet. The temperature is also uniform at the bonding interface when heating system approaching a thermal balance during the bonding process. Therefore the channel structure has uniform deformation throughout the device. Although we only test this bonding process in a single device level, it should be able to be applied to a wafer level bonding with a uniform pressure and a temperature control.

4. Conclusion

We have demonstrated a new way to make sealed fluidic micro/nano chips using proton beam writing and thermal bonding in PMMA. Micro fluidic chips with micro sized features, for cell and DNA separation have been realized with this process. This process is efficient for fast prototyping and for fabricating small numbers of identical chips. Chips with 200 nm width enclosed channels have also been fabricated by this process, and we have shown that the enclosed nanochannels have good precision.

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