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Sub Micron Poly-Dimethyl Siloxane (PDMS) Replication Using Proton Beam Fabricated Nickel Moulds

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Abstract. We present a process to fabricate Ni moulds on proton beam written (PBW) PMMA structures. These Ni mould are use to replicate PDMS lab on a chip devices featuring 300 nm details of high verticality, smooth sidewalls and high aspect ratios. The lifespan of the Ni mould can be extended and the functionality improved by means of introducing a 5 nm thick Teflon AF release layer. Following this method, PDMS chips have been fabricated for microfluidic experiments.

Introduction

In recent years, microfluidic systems have become an important platform for biological and chemical research, and associated applications [1-5]. The complexity of microfluidic systems is increasing rapidly [5-7] and new ways of detecting and investigating biological cells and molecules with higher sensitivity are being developed [8-10].

Silicone elastomer PDMS, which is 50 times cheaper than silicon on per volume basis, is used for making microfluidic lab on chip devices. Key characteristics are easily replicated though moulding, good optical transparency, biological compatibility and ease of bonding and sealing of patterned structures [1]. However, most of these PDMS chips have critical dimensions larger than 1-10μm. Such structures can be realized with photo lithography using a low cost membrane mask and replication through casting. Studies on PDMS fluidic structures at sub micrometer dimensions are limited because of the difficulty in fabricating high smoothness masters at these dimensions.

To fabricate structures with feature dimensions of several microns or larger on glass or silicon, optical lithography is an ideal choice [3, 4]. To pattern nanostructures in resist, electron beam lithography (EBL) is often the choice. The patterned resist layer, which in general is very shallow, acts as a mask for further etching into the substrate to produce nano structure in silicon or glass. Recently imprinting, embossing and other contact replication technologies have been used for replicating nano structures, where the imprinting or embossing moulds are often fabricated using EBL.

PBW, which can be classed as a deep writing analogue of EBL, has been recently shown to be a powerful tool for fabricating high aspect ratio nanostructures. PBW is characterized by high aspect ratio structuring, exhibiting vertical and smooth walls, in resists such as PMMA, SU8 and HSQ [11, 12]. PBW can realize much deeper structures than EBL because the proton is much more massive than the electron and can therefore penetrate much deeper in materials whilst maintaining a straight path. PBW can therefore realize 3 dimensional structuring directly without further etch steps.

To allow higher volume production of lab on chip devises, research has been carried out to transfer PBW nanostructures to a Ni mould which can be used in hot embossing [13]. Because PDMS is a soft material with low surface energy, it promises low stress replication and easy release from the mold after replication. In this paper we report the replication of PDMS nanostructures directly from Ni moulds electroplated on PBW structures.

Compared with polymer resist patterns on a Si substrate, the Ni mould is mechanically stronger, better able to withstand chemical solvents, allowing washing with organic solvent using ultrasonic agitation and promises a long lifespan.

The effective separation of micron-sized particles, such as DNA larger than 100kb, is still a challenging work and the separation based on the rectification of Brownian motion caused by the ratchet effect is reported with promising result [14, 15], however all these works are based on silicon or quartz based microchips, which is expensive and have to be fabricated piece by piece. In this paper we used the PBW patterned Ni mould to fabricate cheap and disposable Brownian chips for micro particle separation.

Materials and Method

A Si wafer was pre-coated with 20nm Cr and 60 nm Au as a seed layer for electroplating followed by spincoating of a 2μm thick PMMA resist layer (950k molecular weight, 11 wt% in anisole, from Microchem). PBW was carried out at the Centre for Ion Beam Applications (CIBA) in the Physics Department of the National University of Singapore with a standard process [21-23] (Fig.1a). After exposure, the resist was developed in an IPA based developer (IPA/DI water at 7:3) for 2 minutes and a second seed layer of Ti (2.5 nm) was coated (Fig. 1b) by using a FCVA machine (CS-2121 from Nanofilm Technologies International). The sample then was plated with Ni (Fig. 1c) in a commercial plating machine (RD.50 from Technotrans AG) with an alternating current regime. In one cycle, the forward current lasts for 1000 ms and is half of the reverse current value which lasts for 100 ms. We set the plating speed at 0.015μm per min. for the initial 30μm after which the plating speed is fixed at 0.3μm per min. The total thickness is aimed at 300μm. Next the Ni mould plus Si wafer are soaked in acetone to remove the resist and separate the stamp from the Si wafer, see Fig 1c.

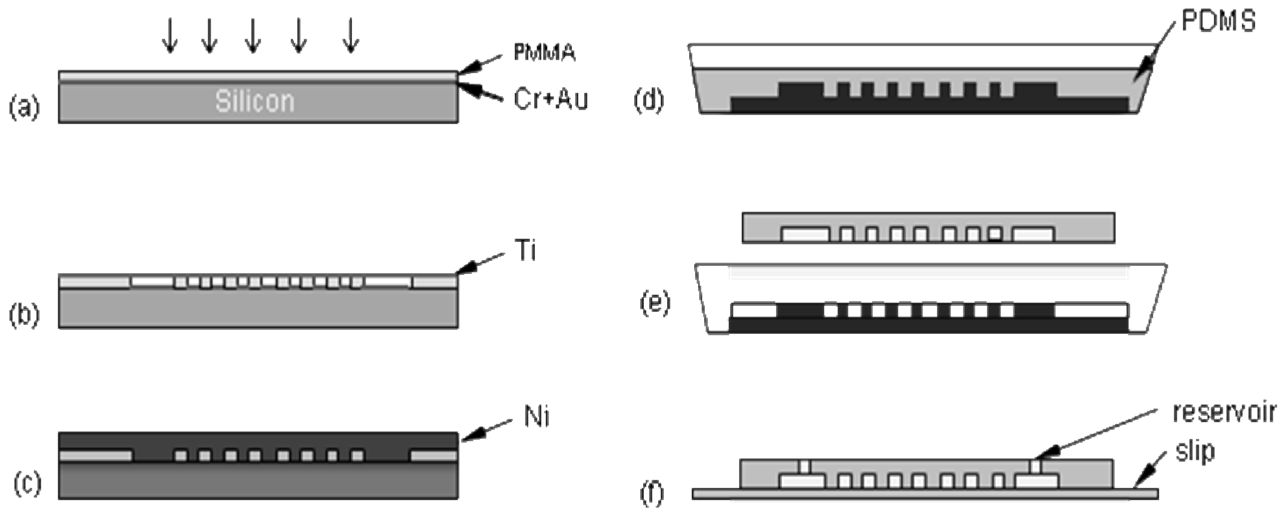

Figure 1 Schematic of the PDMS) replication using proton beam fabricated nickel moulds

A 5nm thick Teflon AF 1600 layer was coated on the Ni mould to improve the release properties of PDMS (Sylgard 184 from Dow Corning). PDMS was cast on the Ni stamp (Fig. 1d) with a standard process [13], and cured at 60°C for 4 hours. After releasing from the stamp, holes were punched in the PDMS replica which serves as fluidic reservoirs. Next a cover slip and the PDMS mould are treated with air plasma in a Harrick Plasma cleaner (250 mTorr for 15s). Finally the 2 pieces are brought in contact and bonded together (Fig. 1e and 1f). Scanning electron microscope photos (SEM) were taken with JSM-6700F SEM (from JEOL Ltd).

Results and discussion

Fig. 2a shows a precise 2μm thick Ni pattern of a ratcheted fluidic chip with details down to 300 nm. This Ni mould was obtained after PBW and electroplating following the procedure as shown in Figs. 1a-1c. Without the coating of an Teflon release layer this Ni mould can't be properly replicated into PDMS, since it features aspect ratios up to 4 and sharp corners. The obstacles in the ratchet structure have sharp corners which weaken and easily initiate cracks in the PDMS during demoulding. To reduce the demoulding stress, Teflon AF 1600 is coated onto the Ni mould. This release layer guarantees that the PDMS patterns are a faithful replica of the Ni mould. After plasma treatment the PDMS chip is bonded onto a microscope cover slip and forms an enclosed fluidic lab on chip (Figs. 1f and 2c).

The chip is now ready for fluidic experiments with buffer solution containing fluorescent spheres (Fig. 2d). When applying an electric field across the chip through platinum cathode and anode at the inlet and outlet of the fluid chip (Fig.2c), spheres of 500 nm flow through this chip driven by electrophoresis. Spheres diffusing down are blocked and deflected back to the original gap, whereas those diffusing up are deflected to the next gap on top. The movement of the spheres is recorded and tracked with a MATLAB code (Fig. 3). Initial work shows that the spheres are deflected over an angle of 14°±3°. Investigation of particles of different diameters will be carried out in future.

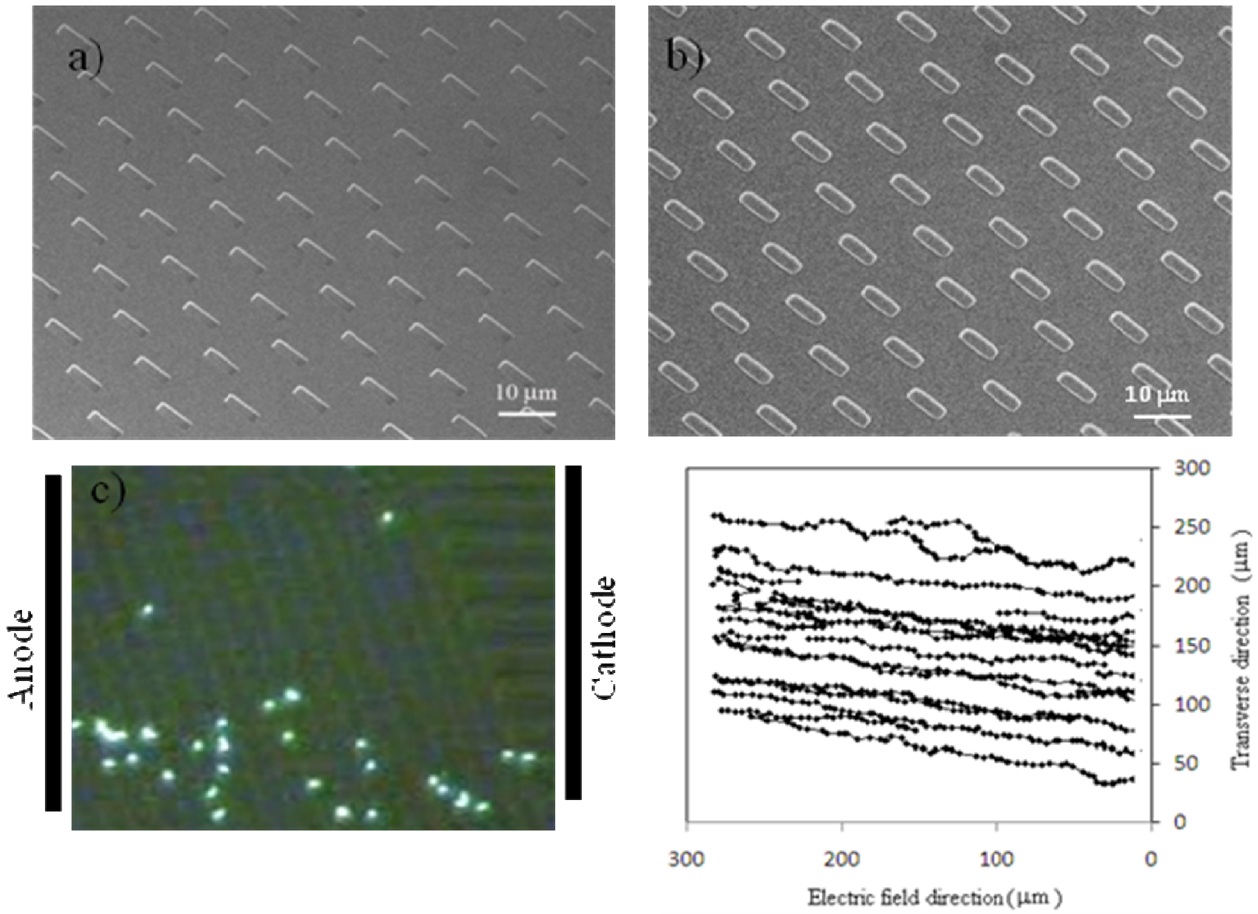

Figure 2 a) Nickel stamp fabricated using PBW b) PDMS Brownian structure replicated from a) c) the chip test on a inverted microscope d) the chip test under microscope with inset of schematic of the chip. d) Paths of 0.5µm spheres in the chip driven by field strength of 10V/mm.

Conclusion

We have shown here that PBW is a promising tool to fabricate high quality Ni moulds for PDMS replication featuring details down to 300 nm. A thin release layer of Teflon AF 1600 has been uniformly coated on the high aspect ratio Ni structures. This release layer improves the quality of the PDMS copies significantly enabling the replication of 300 nm details in PDMS featuring aspect ratios as high as 6. This has resulted in the fabrication of a PDMS Brownian ratchet array lab on a chip device with sub micrometer features. This PBW PDMS lab on a chip device can alter the path of nano particles away from the driving force along the electric field direction.

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