
Proton beam writing: a progress review

Jeroen A. Van Kan*, Andrew A. Bettiol,
Kambiz Ansari, Ee Jin Teo, Tze Chien Sum
and Frank Watt

Centre for Ion Beam Applications,
Physics Department, National University of Singapore,
Lower Kent Ridge Road, Singapore 117542

Fax: +65 6777 6126 E-mail: phyjavk@nus.edu.sg

E-mail: phybaa@nus.edu.sg E-mail: phyamk@nus.edu.sg

E-mail: phytej@nus.edu.sg E-mail: phywattf@nus.edu.sg

E-mail: phystc@nus.edu.sg <http://www.ciba.nus.edu.sg>

*Corresponding author

Abstract: A new direct write 3D nano lithographic technique has been developed at the Centre for Ion Beam Applications (CIBA) in the Physics Department of the National University of Singapore. This technique employs a focused MeV proton beam which is scanned in a predetermined pattern over a resist (e.g. PMMA or SU-8), which is subsequently chemically developed. The secondary electrons induced by the primary proton beam have low energy and therefore limited range, resulting in minimal proximity effects. Low proximity effects coupled with the straight trajectory and high penetration of the proton beam enables the production of 3D micro and nano structures with well-defined smooth side walls to be directly written into resist materials. In this review the current status of proton beam writing will be discussed; recent tests have shown this technique capable of writing high aspect ratio walls up to 160 and details down to 30 nm in width with sub-3 nm edge smoothness.

Keywords: proton beam writing; high aspect ratio; direct write; nano imprinting; SU-8; PMMA.

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Biographical notes: Jeroen Van Kan graduated from the Universiteit Van Amsterdam in 1992 with an MSc in Physics. He completed his PhD at the Vrije Universiteit Amsterdam in 1996. He joined the Centre for Ion Beam Applications (CIBA) as a research fellow in 1997. His area of specialisation is in proton beam writing.

Andrew Bettiol graduated from the University of Melbourne, Australia with a PhD in 1999. He then joined the Centre for Ion Beam Applications (CIBA) as a research fellow. His area of specialisation is in the application of p-beam writing to photonics.

Kambiz Ansari was a research fellow between 1994–2001 at the Van-de-Graaff laboratory of the Atomic Energy Organization of Iran. He graduated from Shahid Beheshti University of Tehran Iran in 1997 with an MSc in Solid State Physics. In 2001, he joined CIBA as an IAEA research fellow and later as a PhD student. He is focusing on the fabrication of sub-100 nm metallic stamps for nanoimprint lithography utilising proton beam writing.

Ee Jin Teo obtained her PhD degree in Physics from the National University of Singapore in 2002. She has been awarded a Singapore Millennium Foundation Fellowship to pursue her postdoctoral research on porous silicon and silicon micromachining at the Centre for Ion Beam Applications, National University of Singapore.

Tze Chien Sum graduated with a BSc and an MSc from the National University of Singapore in 1999 and 2001, respectively. He is currently pursuing his PhD at the Centre for Ion Beam Applications, Department of Physics, National University of Singapore.

Frank Watt graduated from the University of Newcastle upon Tyne (UK) with a BSc in Physics, and completed his PhD three years later in 1971. He was head of the Scanning Proton Microprobe group in the Department of Nuclear and Particle Physics, University of Oxford (UK), and subsequently moved to the National University of Singapore in 1992, where he is currently Professor of Physics and Director of the Centre for Ion Beam Applications.

1 Introduction

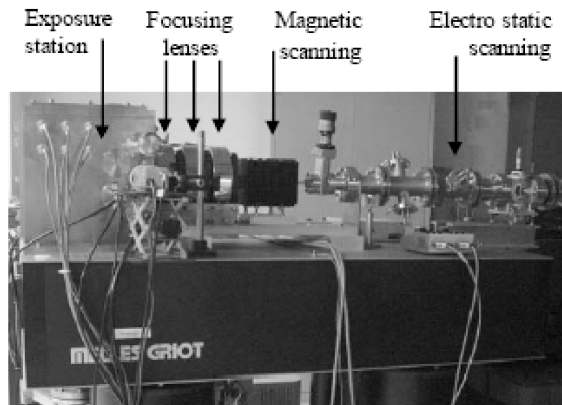
Current microelectronics production technologies are essentially two-dimensional (2D), and are well suited for the 2D topologies prevalent in microelectronics. As semiconductor devices are scaled down in size, and coupled with the integration of moving parts on a chip, there is expected to be a rising demand for smaller microelectromechanical systems (MEMS) and nanoelectromechanical (NEMS) devices. High aspect ratio three-dimensional (3D) microstructures with nanometre details are also of growing interest for optoelectronic devices. Therefore it is essential to develop new lithographic techniques suitable for the production of high aspect ratio 3D micro- and nano-components. Proton-beam (P-beam) writing is being developed at the Centre for Ion Beam Applications (CIBA), National University of Singapore and has been shown to be a promising new 3D lithographic technique [1,2]. P-beam writing is a new technique that utilises a focused beam of fast (MeV) protons written directly into a resist to produce a 3D latent image in a resist material. The relatively high energy of the incident protons produces high penetration into the resist (eg a 2 MeV proton will penetrate 60 μm into PMMA). P-beam writing is the only technique that offers the capability of direct-write high aspect ratio nano- and microstructures. P-beam writing is a fast direct-write lithographic technique; in a few seconds a complicated pattern in an area of $400 \times 400 \mu\text{m}^2$ can be exposed down to a depth of 150 μm . These features make p-beam writing a direct write technique of high potential for the production of high-aspect-ratio structures with sub-100 nm detail in the lateral directions for rapid prototyping. In combination with electroplating, p-beam writing can fabricate precise 3D metallic moulds and stamps that can be used for batch and high-volume production, using either nano-imprinting or flash and step imprint lithography.

A drawback of p-beam writing is that it is a new technology with no commercial instruments available as yet. Technical and commercial development of proton beam writing have been hampered by the difficulties encountered in focusing MeV ions to sub-100 nm dimensions: These difficulties have recently been overcome [3] and the first prototype p-beam writer has recently been constructed at CIBA [4].

2 Three dimensional nano writing facility

The p-beam writing work at the CIBA, has been carried out using two different accelerators. Details about the setup used in combination with the HVEC AN2500 Van de Graaff accelerator can be found in [5–8]. In 2000 a new 3.5 MV HVEC Singletron™ accelerator was installed in CIBA. P-beam writing with the new accelerator can routinely achieve spot sizes below the micron level because of its high brightness and increased beam stability. In addition a new nuclear nanoprobe facility has been developed at the CIBA [4], see Figure 1. This facility is the first of its kind dedicated to p-beam writing on a micron as well as on a nano scale. The p-beam writer utilises the Oxford Microbeams high demagnification lenses (OM52) in a high excitation triplet configuration. This lens system operates at an object distance of 7 m and a reduced image distance of 70 mm resulting in enhanced system demagnifications (228×60 in the X and Y directions, respectively).

Figure 1 P-beam writing end station set-up. In the exposure station wafers up to 6" can be exposed



The resist sample is mounted on a computer controlled Burleigh Inchworm XYZ stage which has a travel of 25 mm for all axes with a 20 nm closed loop resolution. The system has been designed to be compatible with Si wafers up to 6". This new focusing system is able to produce proton beams down to a $35 \times 75 \text{ nm}^2$ spot size, which can be used for mask-less direct write lithography.

During exposure the beam is scanned over the resist in a digitised pattern using a set of electromagnetic scan coils, located directly in front of the quadrupole lens system. In this way scan fields up to $0.5 \times 0.5 \text{ mm}^2$ can be achieved. To prevent deposition in the sample of any unwanted dose we use a beam blanking system where the beam is deflected out of the normal beam path using the field generated between a set of

electrostatic plates. The switching time for blanking is typically less than 1 μs . The scan system utilises a National Instruments NI 6731 Multi-I/O card which has four 16-bit digital-to-analog converters (DACs) and has a minimum update time of 1.0 μs . Two channels control the scan coils and a third DAC controls the beam deflection. The scan software supports AUTOCAD and Bitmap file format; more details about the scanning system can be found elsewhere [9].

The magnetic scan coils in the scanning system limit the writing speed due to their relatively long settling time. To overcome this problem, we have recently introduced a faster electrostatic scanning system to allow us to reduce exposure times by a factor of 15 [10]. Due to beam optical considerations, the p-beam writing system is limited to one pre-lens scanning system capable of large area scanning (currently magnetic scanning up to $0.5 \times 0.5 \text{ mm}^2$), which is located directly in front of the lenses. The new electrostatic scanning system, which is located upstream from the magnetic scanning assembly and is in a less effective position, is therefore currently limited to a maximum scan area of $50 \times 50 \mu\text{m}^2$.

To guarantee a constant proton dose per pixel as the beam is digitally scanned across the resist, we have developed two main methods for dose normalisation. Currently both methods rely on the detection of Rutherford backscattering (RBS) signals. In the first method the beam is moved to a new pixel in a scan after a fixed number of backscattered protons has been detected (pixel normalisation). In the second method the beam is scanned rapidly over a figure many times until a sufficient average dose has been reached (figure scanning). To facilitate the production of nano-structures with smooth sidewalls, it is advantageous to use more sensitive exposure dose feedback than can be achieved using RBS. For nano-writing, where beam currents are invariably smaller, provision is made to utilise feedback signals from either secondary electron emission or ion induced photon emission: These feedback signals typically have a much higher yield per proton compared to the number of nuclear backscattered events per proton. Recent studies have also shown that direct proton counting (using a photodiode) can be utilised to good effect for writing below 50 nm, and when used in combination with figure scanning produces the best quality nano-structures. This is in part due to the high beam current stability from the Singletron accelerator, leading to uniform exposure doses. Nanomachining at high accuracy requires the development of a focusing protocol, which ideally should include computer software for beam spot optimisation and high quality resolution standards to measure the beam spot size. Both of these are currently being developed at CIBA.

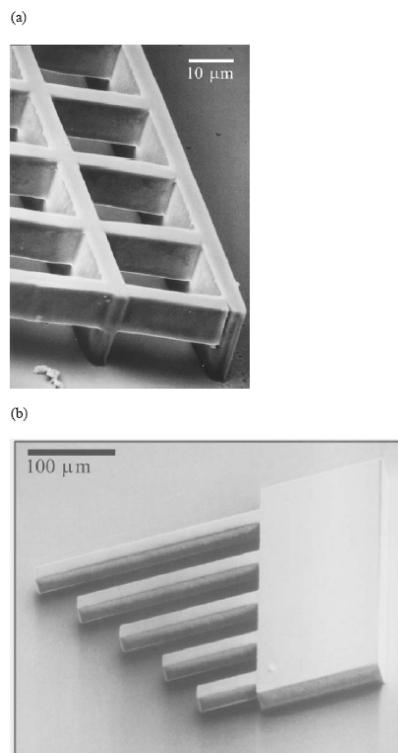
3 Proton interactions with resist

In p-beam writing the path of a high energy (MeV) proton in material is dependent on the interaction with the electrons and nuclei in the material. The probability that a proton interacts with an electron is a few orders of magnitude larger than for nuclear scattering in the first 50% of its trajectory. Therefore only proton-electron interactions will be discussed. Proton-electron interactions hardly change the trajectory of a proton because of the mass ratio ($m_p/m_e \sim 1800$). This implies that the path of a proton hardly deviates from a straight line. Since the energy transfer in these collisions is rather small, peaked around 100 eV, many collisions will occur before a proton comes to rest. Proton

trajectories can be accurately simulated by means of Monte Carlo calculations for example using the computer code SRIM [11]. These features make p-beam writing a predictable and an extremely powerful lithographic technique with the following key features:

Protons have a relatively long and well defined range in resist materials. The penetration depth depends on the proton energy; e.g. the penetration of a proton beam of energy 1.0 MeV in PMMA is 20 μm , whereas a 3.5 MeV proton will penetrate 160 μm [11]. This feature allows the production of slots and holes of well defined depth, and the creation of multi-level structures in one resist layer [8,12]. By exposing the negative resist SU-8 to protons at different energies, novel structures can be produced in one layer of resist. These structures include buried micro-channels, cantilevers, etc [8,13]. An example of a cantilever structure written with a 1.0 MeV and 2.0 MeV proton beam is shown in Figure 2. The cantilever shallow structures have been written with a 1.0 MeV beam, and the deeper anchors with a 2.0 MeV beam.

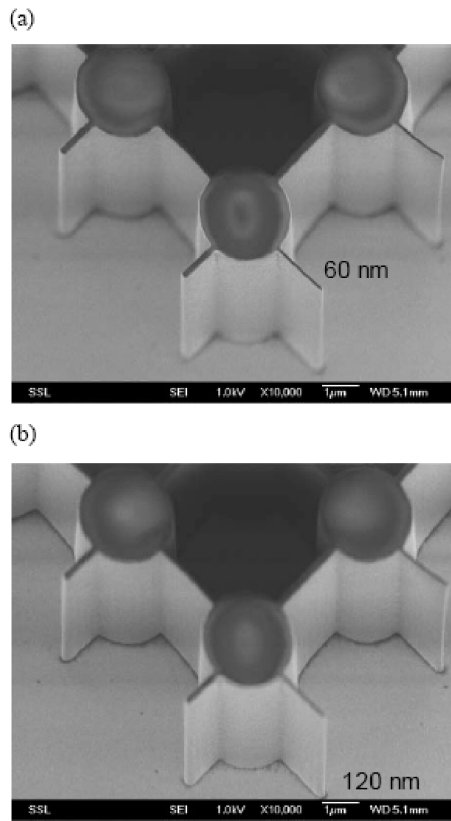
Figure 2 SEM image of suspended beams (a) and cantilevers (b) in SU-8, produced by two proton beam exposures at 1 and 2 MeV



The proton beam travels in a straight line, with very little small angle scattering except at the end of range. This allows the production of structurally accurate high aspect ratio structures. SRIM calculations [11] show that a point like 0.5 MeV proton beam penetrating a 200 nm thick PMMA layer applied on a silicon substrate will spread and have a radius of less than 1.0 nm when it enters the silicon substrate. The generated secondary electrons (delta rays) will have a typical range of 1.4 μm , in which 90% of the

electron energy is deposited [14]. This confinement coupled with an even energy deposition along the path of the proton beam generates smooth side walls. Our calculations show that lithography with MeV protons is potentially capable of producing high quality nano- and micro-structures with 1 nm smoothness and high aspect ratio. In Figure 3 we see an array of micro pillars connected via sub 100 nm walls in a 10 μm thick SU-8 layer. In Figure 3(a) the SEM is focused at the top and in Figure 3(b) the SEM photo is focused at the bottom indicating near vertical side walls. This corresponds to an extremely high aspect ratio in SU-8 of 160. We believe this to be state-of-the-art performances in SU-8; only Bogdanov and Peredkov [15] have reported similar aspect ratios for SU-8 structures, but with structures of 4 μm width or more.

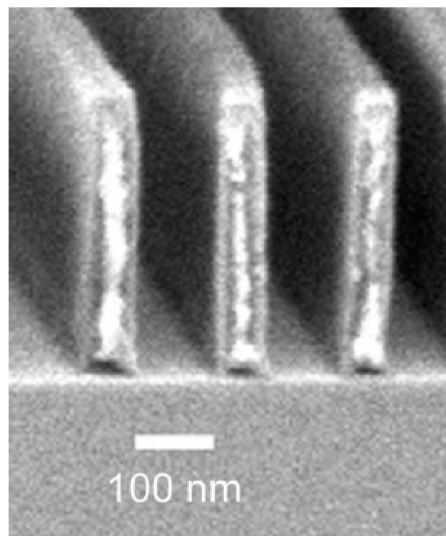
Figure 3 SEM image of 2 μm^2 diameter pillars written in a 10 μm -thick SU-8 layer. Linking the pillars are high aspect ratio walls of width of 60 nm (a) and of 120 nm (b). The structures were written with a 2 MeV proton beam



The proton beam has a relatively even dose distribution with penetration depth. Monte-Carlo calculations using SRIM [11] show that the energy deposition increases slowly with depth, and increases rapidly only at the end of range where energy loss due to proton/nuclear collisions increase. This feature ensures a relatively even exposure distribution with depth. This contrasts with 3D lithography using penetrating electromagnetic radiation (e.g. X-rays) which exhibits an exponential dose distribution with depth.

Reduced proximity effects. One big potential advantage of proton lithography is the virtual absence of high energy secondary electrons which can give rise to unwanted exposure of resist in e-beam lithography. In e-beam writing, much of the energy is dissipated in the form of secondary electrons with an energy of 2 to 50 eV, with a small but significant fraction of the secondary electrons having significant energies which can contribute to the proximity effect in the range of a few tenths of microns [16]. In Figure 4 we see 50 nm wide lines written with a 2 MeV proton beam in 350 nm thick PMMA. For the fabrication of these precise structures there was no special exposure strategy, i.e. the lines were exposed using uniform proton beam dose distribution.

Figure 4 SEM image of parallel lines written in a 350 nm-thick PMMA layer. The structure was written with a focused 2 MeV proton beam. The photo indicates a wall width of 50 nm



Although several resists have been tested for compatibility with p-beam writing [17], only polymethylmethacrylate (PMMA) and SU-8 (a chemically amplified, epoxy based resist) resist were found to be suitable for writing at the sub 100 nm level. Earlier tests have shown that PMGI can also be machined with a slightly reduced resolution compared to PMMA. Tests using Novolac resists have been disappointing, since these resists show positive as well as negative resist behaviour under p-beam writing [17]. In the exposure of PMMA, the protons cause chain-scissioning of the polymer chains. The resulting damaged resist, consisting of molecular chains with lower molecular weight, are then selectively removed using either GG developer at 30°C [18] or a less viscous developer for nano structures comprising of iso-propyl alcohol (IPA) and water (7:3). PMMA therefore is a positive resist under proton irradiation. On the other hand SU-8 cross-links under proton beam exposure. A suitable chemical developer can then be employed at room temperature to selectively remove the unexposed areas [17], making SU-8 a negative resist under proton beam exposure. The most suitable fluence to expose PMMA with 2 MeV protons is 80 nC/mm². In the case of SU-8 only 30 nC/mm² is needed for full cross-linking. Note that there is no post exposure bake required, tests have shown that a post exposure bake will limit the resolution of p-beam writing, reducing the effects of cracking and internal stress in the resist.

4 Applications

At CIBA, we are working on several application areas which we believe have high potential: e.g. nanoimprinting, silicon micro machining, biomedical applications, microfluidics and microphotonics. In this section we will discuss the latest progress in several of these application areas.

4.1 Nano-imprinting

P-beam writing is a flexible direct write technique, that is extremely suitable for rapid prototyping down to the nanometer level and therefore in combination with electroplating also extremely suitable for the production of high quality masters for nano-imprinting. Direct-write processes for a long time have been considered intrinsically slow and inefficient compared with masked processes for the mass production of large-area high-density low-dimensional structures. However, the increased technical complexities and predicted increased cost of producing feature sizes smaller than 100 nm has called into question the traditional role of masked processes as the universal method of mass production. Direct write processes therefore may have some distinct advantages when used in combination with nano-imprinting [19]. The p-beam written stamps have potential applications in all the developed technologies based on moulds and stamps [19–23], such as template fabrication for single molecule electronics, nano-photonics, nano-fluidics, biosensors, etc. Recently we have demonstrated [24] the production of 3D Ni stamps using p-beam writing in combination with Ni electroplating. In Figure 5(a) we show a SEM image of a 2 μm high and 100 nm wide Ni wall, corresponding to an aspect ratio of 20. The side wall roughness of similarly plated Ni structures was measured to be about 7 nm. This stamp has been successfully used to replicate a negative of this structure in PMMA using an Obducat Technologies AB, NIL-2-PL 2.5 inch nanoimprinter [25]. A SEM photo of the imprinted pattern, which has vertical sidewalls and flat surfaces identical to the stamp, is shown in Figure 5(b).

Figure 5 (a) SEM picture of a nickel stamp fabricated using proton beam writing and nickel electroplating, exhibiting vertical sidewalls, and a smooth surface (7nm)

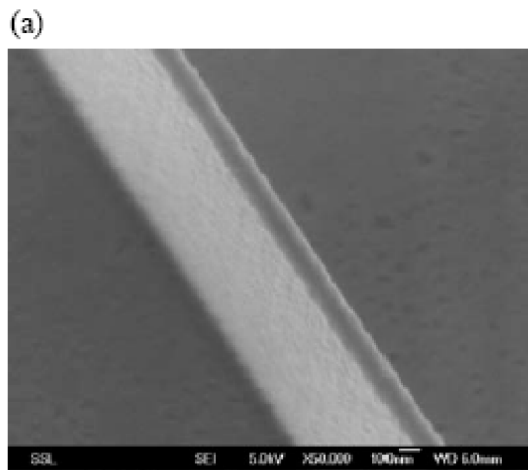
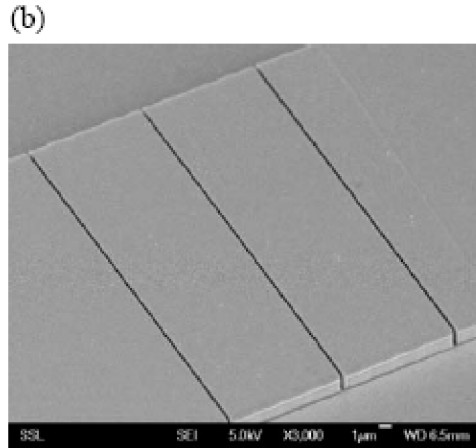
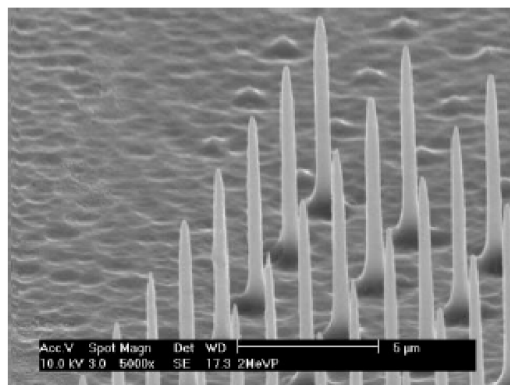


Figure 5 (b) imprinted negative copy in PMMA (continued)

4.2 Silicon machining

Proton irradiation prior to electrochemical etching allows the fabrication of 3D structures in bulk *p*-type silicon. The proton-induced damage increases the resistivity of the irradiated regions and acts as an etch stop for porous silicon formation. A raised structure of the scanned area is left behind after removal of the unirradiated regions with potassium hydroxide. Recent work in CIBA has shown the ability of p-beam writing to fabricate high aspect ratio silicon needles [26], with nano-metre size tips, see Figure 6. This has extensive applications in fabrication of multiple tip assemblies for dip-pen lithography, multiple head scanning tunnel microscopes, and integrated MEMS devices. One major limitation of conventional lithography and silicon etching technologies is the multiple processing steps involved in fabricating free-standing multilevel structures [27,28]. By using different proton energies to expose the silicon to different depths, p-beam writing can overcome these limitations and produce multilevel structures in Si.

Figure 6 Array of high aspect ratio silicon needles obtained by single spot irradiations

4.3 Biomedical applications

Assaying biomolecules by measurement of the electrical impedance between interdigitated electrodes in an electrochemical cell is of great topical interest. The change in electrical admittance between interdigitated electrodes with e.g. specific receptor molecules immobilised on the surface is determined by the presence of the specific target molecule [29,30]. These sensors are applicable for an extremely broad range of immunoassay applications from detecting simple toxins such as formaldehyde [31] to measuring bacterial metabolism [32] and detection of specific large molecules such as specific DNA sequences and hormones [29,33]. In more sophisticated indirect measurements the detection of HIV antibodies has been reported [34]. Much current interest is focused on sensors based on nanoscale electrode arrays with a high electrode spatial density and electrode gap d of 10–300 nm [35–37]. In Figure 7, a prototype biosensor structure produced using p-beam writing is shown. Prototype biosensor structures with gaps between metal electrode fingers of ~85 nm have been successfully written using a focused MeV p-beam [38]. P-beam writing is a potential candidate as a complementary technique for cases where the restrictions imposed by proximity exposure and limited aspect ratio are troublesome in conventional electron beam lithography [38].

We have also shown that p-beam written substrates can be used as scaffolds to grow cells. Although it is well known that the behaviour and function of cells are changed by geometric constraints and substrate surface properties, little work has been carried out on the effects of 3D microsubstrate geometry on cell behaviour. This knowledge is important for the success of tissue engineering so that cells can organise in suitable 3D environments and function properly as an organ *in vivo*. The major reason for this lack of information stems from the general unavailability of precisely patterned 3D microsubstrates. P-beam writing can produce 3D high-aspect ratio micromachined surfaces of different shapes and patterns [39]. A 3D structure was designed to test longitudinal movement along ridges: a circular ring structure containing a single 30 μm -wide, 20 μm -deep groove was interrupted with four radial 400 μm -long microridge outlets with widths of 5, 20, 25, and 30 μm , respectively (Figure 8(a)). In this structure, fibroblast cells migrated and spread outward until they reached the circular barrier. Cells were then observed to move along the three largest ridge channels (Figure 8(c–e)), but not along the narrowest ridge of 5 μm (Figure 8(b)). This result indicated that fibroblasts have restricted movement along a 5 μm -wide ridge, but can move easily along ridges of 20 μm or more. Interestingly, when cells pass along the ridges and move into the region outside the barrier, they then move back toward the main cell cluster, possibly sensing some chemical cues from the main body of cells still trapped within the circular barrier. Our results indicate that 3D microstructures can potentially deter fibroblasts from spreading and growing into a particular tissue environment. These 3D microstructures may be useful in tissue engineering when multiple cell types are needed to form a proper tissue and fibroblast overgrowth can be detrimental in the proper formation and function of the tissue. Our results also indicate that proton beam writing can be used to generate 3D microstructured substrates for studying cell behaviour. As a unique direct write micromachining 3D technique, random and complex microstructures can be generated that may reflect more closely the *in vivo* tissue environment, facilitating the study of cell–cell and cell–matrix interactions for tissue engineering.

Figure 7 SEM image of the prototype nano-biosensor structure after metal lift-off

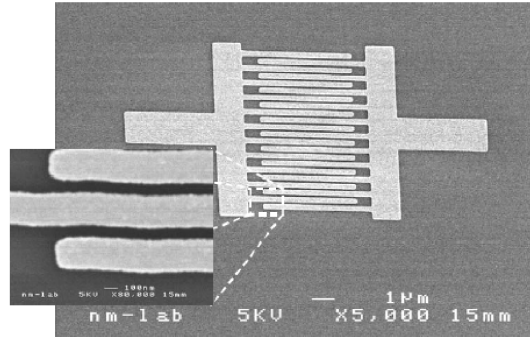
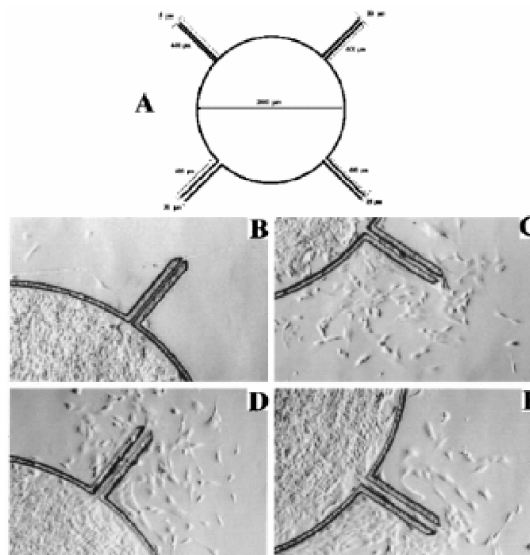


Figure 8 Fibroblasts selectively migrate and spread through smooth surface channels. A modified 3D microstructure corral containing four smooth channels with widths of 5, 20, 25, and 30 μm at the four corners is shown in (A). (B–E) Cell growth and spreading through the four channels on day 7. Channel width: (B) 5 μm ; (C) 20 μm ; (D) 25 μm ; (E) 30 μm



4.4 Photonics

There are two ways in which p-beam writing can be used for applications in micro-optics and microphotonics. The first involves the direct patterning of polymers to form micron sized components such as waveguides, gratings and microlens arrays. These micron sized structures are usually fabricated in resist layers spin coated on a suitable substrate such as glass or a thermal oxide layer on a silicon wafer. For applications in light guiding, the substrate and cladding material needs to be of a lower refractive index than the core material. SU-8 is a good material for making waveguides due to its high transparency, its low loss, smooth sidewalls, see Figure 9 [40]. Furthermore its refractive index (1.575 at 1550 nm) is slightly above that of common glass substrate materials and thermal oxide.

Micro lens arrays can be fabricated by first spin coating a resist layer on a transparent substrate such as a glass microscope cover slip, a patterning step is then used to fabricate the microlenses of the desired diameter. After development the polymer is thermally reflowed by heating the sample above its glass transition temperature. Under the influence of surface tension, the hemispherical microlenses are formed, see Figure 10(a). The focal length can be determined by choosing an appropriate combination of lens diameter and resist thickness. Other structures that can be fabricated by direct patterning include gratings and Fresnel zone plates, see Figure 10(b) [41].

Figure 9 SEM image of a prototype ring resonator fabricated in a 1 μm thick layer of SU-8 on Si. The inset shows a well defined space of 200 nm

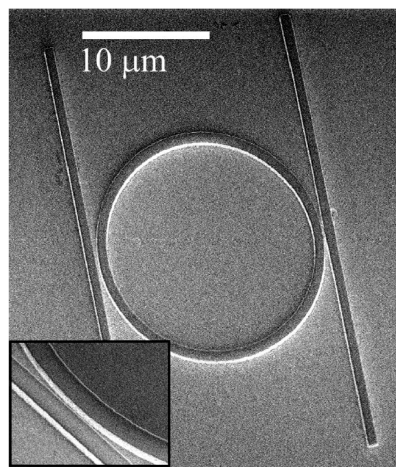


Figure 10 (a) Optical image of micro lenses fabricated in 15 μm thick PMMA

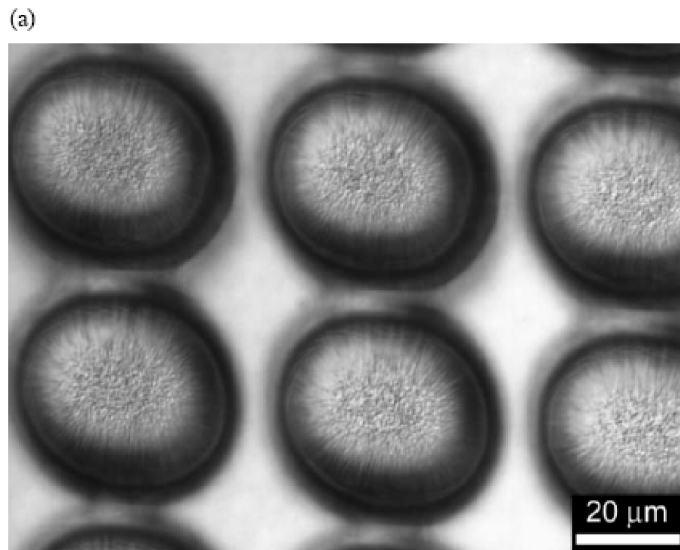
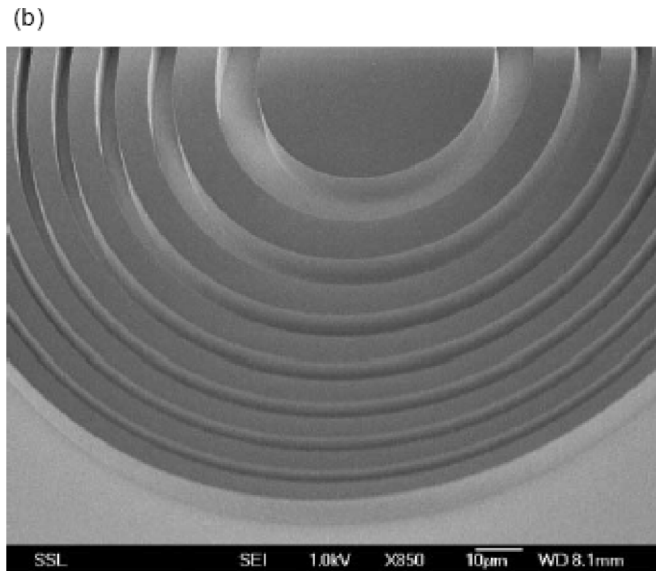


Figure 10 (b) SEM images of some micro-Fresnel lens elements micromachined in a 10 μm layer of SU-8 on Si (continued)

A second method of forming waveguides in bulk polymer or fused silica using p-beam writing involves direct write ion beam modification without a development step [42]. This is achieved by utilising the end of range of an energetic ion to create a buried channel waveguide in a substrate. Ions have the unique feature that the amount of energy they deposit into a substrate rapidly increases as the velocity decreases. Towards the end of range, the probability of an ion creating a vacancy also rapidly increases. The net effect of this is to create a buried region of damage resulting in a local increase in material density, and therefore a local increase in refractive index. This damaged region then can act as the core of a waveguide with the surrounding unirradiated region acting as the cladding.

5 Discussion and conclusion

Here we discussed the potential of p-beam writing and its wide range of application areas. In our p-beam writing facility, the proton beam can be focused down to $35 \times 75 \text{ nm}^2$ and directly scanned across a resist, thereby eliminating the need for a mask. Our resolution is currently the best performance in the world for MeV protons [3]. Although the technique of p-beam writing is still in its infancy, the technique shows great potential for 3D direct writing, particularly in the sub-100 nm range. The performance of p-beam writing is dependent on how well we can focus MeV protons. There is no scientific reason why this performance should not be improved, and due to the reduced proximity effects compared with the highly successful e-beam writing, p-beam writing may offer a new and novel way of producing 3D nano-structures.

Since the proton beam has a well defined range in resist (unlike x-rays), the depth of structures can be easily controlled by using different proton energies enabling the construction of slots, channels, holes, etc. with a well defined depth. The depth can be different for these slots, channels or holes in one single resist layer. In addition, by changing the angle of the resist with respect to the beam, complex shapes can be machined with very well defined sharp edges. Arbitrary shapes can be fabricated, high-aspect-ratios (more than 100) can be achieved in PMMA and SU-8 resist, and the smallest single line achieved so far in a PMMA layer is 30 nm [1].

In crossing the sub-100 nm resolution barrier, p-beam writing must now be taken seriously as a contender in the next generation lithographies. The advent of MEMS, nano-photonics, nano-magnetics [43], molecular nano-technology devices, tissue engineering and lab-on-a-chip systems, may benefit from p-beam writing technology either through rapid prototyping or nano imprinting. The direct write processes, which for a long time have been considered too slow for mass production, may have some distinct advantages when used in combination with nano-imprinting, since p-beam writing is ideal for producing high aspect ratio metallic stamps of precise geometry. The transfer of largescale patterns using nano-imprinting represents a technique of high potential for the mass production of a new generation of high area, high density, low dimensional structures.

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