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Abstract. Deep proton writing (DPW) is a fabrication technology developed for the rapid prototyping of polymer microstructures. We use polymethylmethacrylate (PMMA) substrates, which act as a positive resist, for irradiation with a collimated 12-MeV energy proton beam. Using 12 MeV enables the irradiation of increasingly thick PMMA substrates with less conicity of the sidewalls compared to the lower energies used in previous work. A microhole of 47.7 μm diameter over a depth of 1 mm is achieved, leading to a maximum aspect ratio of 21 : 1. The sidewalls of the irradiated structures show a slightly conical shape and their root-mean-square surface roughness is lower than 50 nm averaged over 72 measured areas of 56 $\mu\text{m} \times 44 \mu\text{m}$. This means that DPW components have optical surface quality sidewalls for wavelengths larger than 400 nm. Based on the trade-off among the sidewall roughness, conicity, and the development time, we determine that the optimal proton fluence for 12-MeV DPW in PMMA is $7.75 \times 10^6 \mu\text{m}^{-2}$. Finally, we discuss some high aspect ratio microstructures with optical surface quality that were created with DPW to be used for a myriad of applications, such as micromirrors, microlenses, optofluidic devices, and high-precision alignment structures for single-mode optical fiber connectors. © 2016 Society of Photo-Optical Instrumentation Engineers (SPIE) [DOI: 10.1117/1.JMM.15.4.044501]

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1 Introduction

Microfabrication technologies play an important role in today's life, enabling a myriad of applications, such as microelectromechanical systems, optical sensors, display and projection systems, and optical interconnections.^{1–4} Today, a plethora of these technologies exist and we can divide them into two groups: mask-based lithographic and direct writing technologies. Standard photolithography, stencil lithography,⁵ and LIGA^{6,7} (a German acronym for: Lithographie, Galvanoformung, Abformung) e.g., belong to the former group and can transfer a pattern from a mask to a substrate either by an additive or subtractive process. In case rapid prototyping is the goal, one of the main disadvantages of this group of technologies is the need for an often costly and high-qualitative mask, which needs to be fabricated before the pattern transfer can take place. Within direct writing technologies, we can further distinguish two groups: true three-dimensional (3-D) and so-called “2.5D” micromachining technologies, which do not allow the fabrication of overhanging parts. True 3-D technologies include aerosol jet writing,¹ laser-based 3-D printing,^{8–10} and two-photon polymerization.¹¹ The largest drawback of these technologies is that the desired structure is written voxel per voxel, which can be time consuming, especially if a true 3-D structure is not required. 2.5D micro-machining technologies allow for the creation of structures in a plane with a certain depth and profile, but no overhanging structures. These technologies include laser ablation,¹² electron-beam writing,¹³

and proton beam writing.^{14–16} For many applications, such as microneedles,¹² lab-on-a-chip devices,¹⁷ masks for lithography,¹³ and micromechanical optical fiber connectors,⁴ true 3-D structures are often not needed, and thus, 2.5D technologies are ideal for their rapid prototyping.

Over the last 15 years, we have developed a microfabrication technology, ideal for rapid prototyping, called deep proton writing (DPW), which can be used to achieve high aspect ratio structures with optical quality surfaces. In DPW, a cyclotron facility is used to generate a highly energetic (8.3 to 16 MeV) proton beam, which is subsequently used for irradiating a substrate material. The difference between DPW and what is commonly referred to as proton beam writing is that DPW uses a collimated proton beam at much higher energy for irradiation, rather than a focused beam at lower energy (typically <3 MeV^{14–16}). A higher energy allows the proton beam to penetrate deeper into the substrate material and, thus, allows for making higher structures (up to 2 mm for 16 MeV protons¹⁸). In this paper, we discuss the use of a 12-MeV collimated proton beam to structure polymethylmethacrylate (PMMA) substrates with a high thickness of up to 1 mm and a high molecular weight of $M_n \approx 10^5$ g/mol (Notz Hesaglas¹⁹). During irradiation, the highly energetic protons break the long polymer chains in the material, changing their chemical properties, in turn allowing us to etch the irradiated zones away, in a postirradiation development process. PMMA thus acts as a positive resist for DPW. Since we can irradiate with circular, elliptical, square, rectangular, and hexagonal proton beams with

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sizes ranging from ~ 33.5 to $300\ \mu\text{m}$ in size, DPW is a versatile rapid prototyping technology. To pattern the substrate, we move it perpendicularly to the proton beam and as such, arbitrary 2.5D patterns can be written. With a minimum aperture size of $33.5\ \mu\text{m}$, DPW does not allow for making very fine structures in the (sub-)micrometer-range. The strength of DPW rather lies in its versatility and in the possibility of rapid prototyping of high aspect ratio microstructures in thick substrates with optical surface quality.^{18,20}

In this paper, we first elaborate on the DPW setup and discuss the irradiation process at 12 MeV and the subsequent chemical development. Next, we investigate the effect of irradiating with different proton fluences, indicating the amount of protons that have penetrated the substrate in a certain area (expressed in μm^{-2}). Note that we focus on proton beams with an energy of 12 MeV in this paper, as opposed to the other proton energies, which were used in earlier work.^{18,20–22} In Sec. 3, we use a $50\text{-}\mu\text{m}$ -diameter (circular) proton beam to irradiate lines with various proton fluences and measure the resulting sidewall profile and root-mean-square surface roughness. From these measurements, we determine the optimal proton fluence for irradiation. Then, microholes and closely spaced lines are irradiated to determine the highest aspect ratio that can be achieved with our DPW setup, using a proton energy of 12 MeV. Finally, we discuss various application examples for which DPW-fabricated components have been used.

2 Technology Description and Experimental Methods

2.1 Deep Proton Writing

The proton beam used in our DPW setup is generated by a CGR-MeV model 560 cyclotron, which is capable of generating quasimonoenergetic ($\Delta E/E = 1\%$) proton beams in the energy range between 3 and 45 MeV.²³ In our case, the 12-MeV proton beam enters the irradiation setup, depicted schematically in Fig. 1, on the left-hand side. The setup is mounted in a vacuum chamber ($<10^{-4}$ mbar) to avoid scattering of the protons on air molecules. The proton beam first passes through a collimator, which reduces the beam diameter to 2 mm. Then, before finally reaching the substrate in the sample holder, the beam has to pass through a nickel

Fig. 1 Schematic representation (not to scale) of the DPW setup with the proton beam represented by the beam along the z-axis. If the proton beam is not blocked by the mechanical shutter, it passes through respectively the collimator, the stopping mask, and the substrate before finally reaching the current probe. The arrows represent movement capabilities of the components.

stopping mask. The goal of this mask is to limit the proton beam to a certain size and shape and stopping the rest of the protons. Stopping range of ions in matter (SRIM) data²⁴ show that a mask thickness of at least ~ 350 and $\sim 550\ \mu\text{m}$ of nickel is needed to fully stop protons with an energy of 12 and 16 MeV, respectively (we include 16 MeV as a requirement for the stopping mask to allow for a broader energy range with DPW). To increase the resolution of the irradiation, these masks should contain apertures with as small a diameter as possible. Since holes smaller than $50\ \mu\text{m}$ over a depth of $\sim 600\ \mu\text{m}$ are very hard to achieve in nickel, due to the very high aspect ratio, we opted for a stacking approach of thinner nickel plates to achieve the required thickness for the stopping mask. The mask-plates are fabricated according to the LIGA mould insert fabrication technology^{25,26} by a process combination of x-ray lithography, nickel electroforming, and mechanical postprocessing at the Karlsruhe Institute of Technology. Therefore, a $\sim 240\text{-}\mu\text{m}$ -thick PMMA sheet is glued on an 8-mm-thick copper plate as resist and is subsequently irradiated by hard x-ray radiation through a working mask with the desired structure layout. After wet chemical development of the structures, nickel electroforming is directly started from the bottom of the structure cavities (the upper surface of the copper substrate) without further metallization. When the nickel layer has reached the top of the PMMA structures and a slight overplating is realized, the galvanic nickel plating is stopped. To adjust the final thickness of the nickel plates and to generate a high-quality surface, mechanical grinding and polishing are performed. Finally, the polished mask-plates are removed from the thick copper plates by a combination of wire-electron discharge machining, wet-chemical copper etching, and resist removal from the through-holes using ethyl acetate. The resulting $\sim 210\text{-}\mu\text{m}$ -thick nickel mask-plates contain apertures in different shapes (circular, square, hexagonal, rectangular, and elliptical) with sizes ranging from 33.5 to $300\ \mu\text{m}$. By measuring the size and position of the apertures in the fabricated mask-plates with a Werth UA400 coordinate measuring machine (CMM), we selected the three mask-plates for which a maximum aperture overlap was measured. These three selected plates were then stacked by aligning them with mechanical transfer (MT-) pins, leading to a stopping mask thickness of $\sim 630\ \mu\text{m}$. MT-pins are very accurately machined (with a tolerance of only $0.25\ \mu\text{m}$ on their diameter of $698.75\ \mu\text{m}$) alignment pins, which are also used for aligning commercial optical fiber connectors, such as the mechanical push-on²⁷ and mechanical transfer registered jack²⁸ connectors. The stopping mask allows us to easily select the desired beam shape and size for irradiation by moving it perpendicularly to the proton beam. By moving high-accuracy ($0.3\ \mu\text{m}$) piezostages with a travel range of $50\ \text{mm}$, on which the sample holder is mounted, perpendicularly to the proton beam, we can irradiate arbitrary patterns on the substrate, resulting in a 2.5D shape where the depth is determined by the substrate thickness. To control the deposited proton fluence, we measure the amount of protons that have fully penetrated through the substrate using a coulomb meter with a resolution of $<250\ \text{fC}$ and integrator, which are positioned behind the sample holder.²³ Therefore, the maximum thickness of the substrates useable in our setup should be smaller than the penetration depth determined by the ion-matter interactions

Fig. 2 The energy (black solid line) and mass stopping power (red dashed line) of 12 MeV protons when penetrating in a PMMA substrate.

causing an energy transfer from the impinging ions to the substrate material. In Fig. 2, we show the result of a simulation of the penetration of H^+ ions with an energy of 12 MeV in a PMMA substrate. We can see that the so-called Bragg-peak of the stopping power is located at a penetration depth of about 1400 μm . This means that the protons will penetrate to a depth of about 1400 μm before finally losing all their energy and being stopped in the material. In addition, due to the divergence of the proton beam and the scattering of the protons within the substrate material, a slightly conical shape is irradiated as depicted in Fig. 3. This figure shows the deposited dose in the PMMA substrate for a 12-MeV proton irradiation with a fluence of $7.75 \times 10^6 \mu\text{m}^{-2}$ through a 50- μm hole in the stopping mask, calculated based on SRIM data. The calculations predict that the irradiated zone extends to about 300 μm in diameter after 1400 μm of penetration in PMMA. To limit the more pronounced scattering effect at a larger penetration depth in the PMMA and to be able to measure the proton current (requiring full traversal of the protons through the sample as explained earlier), we choose to limit the substrate thickness to 1 mm. In this case, the calculated dose deposition predicts a broadening from 50 to 102 μm in diameter, if

Fig. 3 The deposited dose of a 50- μm , 12-MeV proton beam throughout a 1400- μm -thick PMMA substrate with a fluence of $7.75 \times 10^6 \mu\text{m}^{-2}$ calculated based on SRIM data. The solid white line shows the conicity of the profile for the critical dose of 4.8 kJ/g for which the PMMA develops after 80 min in GG-developer at 38°C with ultrasonic stirring.

we consider a critical dose of 4.8 kJ/g for which the PMMA fully develops with the standard developing process explained below. We will measure the obtained sidewall profile and surface roughness after irradiation and development in Sec. 3.1.

The chemical development process after irradiation starts by submerging the irradiated substrate in the development agent for 80 min with ultrasonic stirring at 38°C. This so-called GG developer consists of 60% diethylene glycol monobutyl ether, 20% morpholine, 5% 2-aminoethanol, and 15% deionized water.²⁹ Afterward, the sample is submerged for 5 min in a stopping bath at room temperature, consisting of 80% diethylene glycol monobutyl ether and 20% deionized water. Finally the sample is rinsed with deionized water and dried. This etching process was optimized for 8.3-MeV DPW in previous work and remains unchanged except for a 20-min longer etching time for DPW at 12 MeV.^{23,30}

2.2 Molecular Weight of Irradiated Polymethylmethacrylate

Irradiating a PMMA substrate with 12-MeV protons causes the long polymer chains to break into pieces, hence lowering the substrate's molecular weight. This allows us to selectively etch away the irradiated zones in a subsequent step. To determine the effect of proton fluence during the irradiation, we measure the change in the PMMA's molecular weight due to proton irradiation with gel permeation chromatography (GPC). We use an Agilent PLgel mixed C column of 30 cm long at 30°C and should dissolve at least 6 mg of irradiated PMMA in tetrahydrofuran (THF) for analysis with an Agilent Chemstation. The resulting molar mass is calculated against polystyrene standards. To limit the mixing of irradiated and pristine PMMA material, we prepared a dedicated substrate using conventional micro-milling (using a VHF-CAM 100 with a resolution of 10 μm), as shown in Fig. 4(a). First, we glue a 1-mm-thick aluminum plate on top of a pristine 1-mm-thick PMMA substrate. An "L-shape" is then milled into this aluminum plate, and, in the same step, 2.3 mm $\times$ 2.3 mm squares are milled into the PMMA substrate, enabling an alignment accuracy of <10 μm between the L-shape and the squares. The size of the squares was chosen so that they contain ~ 6.3 mg of PMMA. TRIM simulations show that 12-MeV protons penetrate up to a depth of ~ 0.9 mm in aluminum. Hence, the 1-mm-thick aluminum plate will completely stop the protons and the milled L-shape thus allowing us to align the substrate with respect to the proton beam. This is done by determining the location of the corner of the L-shape by monitoring the proton current while moving the sample and locating the transition between minimum and maximum current. Knowing the relative location of the L-shape in the aluminum to the premilled squares, we can thus control the sample position with respect to the proton beam. To allow for small misalignments due to various positional tolerances and to make sure the entire premilled square is irradiated, we irradiate a zone extending at least ~ 50 μm outside the premilled square on all sides as depicted in Fig. 4(c). The complete premilled square is irradiated in steps of 0.5 μm with a square proton beam with a 300 μm side and a proton fluence ranging from 4.17×10^4 to $8.33 \times 10^6 \mu\text{m}^{-2}$. The irradiated sample is shown in Fig. 4(b) for

Fig. 4 Dedicated prestructured PMMA substrate for molecular weight analysis by GPC (a) before and (b) after irradiation. (c) Schematic representation of the 300 μm square proton beam and its irradiation path in steps of 0.5 μm to fully cover the 2.3 mm $\times$ 2.3 mm area of a premilled square, marked in (a).

Fig. 5 Number average molecular weight (M_n) as a function of proton fluence (F_p) during irradiation measured with GPC.

proton fluences between 4.17×10^4 and $2.08 \times 10^6 \mu\text{m}^{-2}$. Notice that due to the large surface to be irradiated, two samples were irradiated in two different sessions. After irradiation, the two small ledges connecting the premilled irradiated squares to the pristine PMMA are cut with a scalpel along the cut lines in Fig. 4(c). The irradiated PMMA is subsequently dissolved in THF and analyzed with GPC as discussed above. The results are shown in Fig. 5 where we can see

that a clear correlation exists between the proton fluence and the number average molecular weight (M_n). The higher the used proton fluence, the lower the molecular weight and thus the faster and more selective the chemical etching will be.³¹ Our pristine PMMA substrate has a number average molecular weight of $1.5 \times 10^5 \text{ g/mol}$. We can see that starting from a proton fluence of $8.33 \times 10^5 \mu\text{m}^{-2}$, a significant decrease (of more than an order of magnitude) in molecular weight is realized by the irradiation, and thus, a selective etching or development can start to take place. Note that the GG-developer attacks the irradiated PMMA starting at a fluence of $1.4 \times 10^5 \mu\text{m}^{-2}$, which corresponds to a molecular weight of $\sim 8.2 \times 10^4 \text{ g/mol}$.²⁹

3 Results

3.1 Geometrical Characterization

In any microfabrication technology, controlling the dimensions and the obtained geometry is important. Therefore, we investigate the effect of the proton fluence on the dimensions of irradiated structures. This is done by writing 500 μm long lines (cavities after development) in a 1-mm-thick PMMA substrate using a 50- μm -diameter proton beam and a proton fluence ranging from 7.25×10^6 to $8.75 \times 10^6 \mu\text{m}^{-2}$. This fluence range was selected based on prior knowledge on

Fig. 6 (a) Stereoscopic microscope image of lines irradiated and developed in PMMA with a width of 50 μm , a length of 500 μm , and a depth of 1 mm. (b) Sideview of the same lines after having been milled open. The 72 squares (6×12 array) indicate the location of the evenly spaced surface roughness measurements (not to scale) performed for each line. The five vertical dotted lines represent the approximate location of the stylus profilometer scan lines. (c) Schematic topview depicting the irradiated lines ($50 \mu\text{m} \times 500 \mu\text{m}$) and the dashed line along which they will be micromilled to make their sidewalls accessible for further metrology.

DPW at lower proton energy and with the aim of achieving a high selectivity of the etching process. After irradiation, the sample is developed in GG developer for 80 min at 38°C. The resulting lines can be seen in Fig. 6. Milling along the dashed line in the schematic top view in Fig. 6(c), allows us to measure the sidewall profile and surface roughness. For determining the profile, we use a Dektak8 (Veeco Inc.) stylus profiler with a horizontal and vertical resolution of 67 and 0.1 nm, respectively. The averaged sidewall profile, obtained from five line-scans per irradiated line, is shown in Fig. 7. Note that these are relative measurements and that all “upper left corners” of the measured data have been shifted to the same point to facilitate comparison between profiles of lines written with different proton fluences. We can see that for the lower proton fluence range the irradiated zone has not been fully developed, since there is still a development-artifact visible in the profile [which is also visible in Fig. 6(a)]. In Sec. 2.2, we have mentioned that with higher proton fluences, the resulting molecular weight of the irradiated zones is lower and, thus, the development rate is expected to be higher.³¹ Since the same development time was used for all different proton fluences, the sidewall profile

Fig. 7 Stylus profiler measurements of sidewall profile of developed lines (Fig. 6) which are irradiated with different proton fluences.

measurements confirm this. Note that in case the sample would be developed (much) longer, this development artifact would be developed away as well and a smooth profile would be obtained, at the cost of a more pronounced conical profile. Since the development rate is faster for higher proton fluences (and thus for lower molecular weight), the conicity of the irradiated line (i.e., the deviation from a perfectly vertical sidewall) becomes more pronounced for higher fluences. Considering only smoothness, longer developing times and/or higher proton fluences should thus be used. However, since higher fluences lead to increasingly long irradiation times, and since longer developing times lead to more conicity, we aim to use the lowest proton fluence possible. Thus, the optimal trade-off between the level of conicity, the irradiation time, and the obtained surface roughness, corresponds to a proton fluence of around $7.75 \times 10^6 \mu\text{m}^{-2}$. Irradiating with this proton fluence leads to a broadening of the line of about 22.5 μm over a depth of 1 mm, meaning that the line written with a 50- μm proton beam will have a width of 50 μm on the frontside (i.e., the side at which the protons are entering the PMMA) and about 95 μm on the back side of the PMMA sample. If we compare the measured broadening with what our calculations predict (102 μm at 1 mm depth, depicted in Fig. 3), we can see that the simulation overestimates the broadening slightly. This is because our model is not capable of accurately taking straggling effects caused by the energy spread of the originally accelerated proton beam nor the stochastically character of ion collisions into account.

To achieve the maximum aspect ratio possible with 12-MeV DPW in PMMA, we irradiate a 1-mm-thick PMMA substrate with a 33.5- μm -diameter proton beam (the smallest hole available in our nickel stopping mask stack) and a proton fluence between 8.77×10^6 and $1.17 \times 10^7 \mu\text{m}^{-2}$. The resulting holes after 80 min of development can be seen in Fig. 8(a). Only the holes with a fluence larger than $9.50 \times 10^6 \mu\text{m}^{-2}$ are fully developed after 80 min. We measure the diameter of the front and backside with the CMM, which are 33.6 and 47.7 μm , respectively, for the smallest fully developed microhole with a proton fluence of $9.50 \times 10^6 \mu\text{m}^{-2}$. We can thus fabricate microstructures with an aspect ratio of $\sim 21:1$. The maximum achievable aspect ratio could not be further increased by irradiating lines in close proximity. Our approach to test this consisted of measuring the thickness and height of the thin walls between irradiated lines after development, as illustrated in Fig. 8(b). For writing these lines, a 300- μm -diameter proton beam was used to reduce the required irradiation time. After measurement with the CMM, we achieved a width of 80 μm for a depth of 860 μm leading to an aspect ratio of 11:1. The smallest

Fig. 8 Stereoscopic microscope image of (a) microholes irradiated with a $\sim 33.5\text{-}\mu\text{m}$ -diameter proton beam and a proton fluence between 8.77×10^6 and $1.17 \times 10^7 \mu\text{m}^{-2}$ and (b) closely spaced lines irradiated with a 300- μm -diameter proton beam and decreasing spacing, leaving thin walls between the irradiated zones after development.

and highest aspect ratio wall achieved was measured to be $10\text{ }\mu\text{m}$ in width and $200\text{ }\mu\text{m}$ in height with an aspect ratio of 20:1. Due to the conicity of the irradiated zones, as depicted in Fig. 3, decreasingly small spacings between two irradiated lines lead to decreasingly small heights of the unexposed, undeveloped zones in between.

3.2 Sidewall Roughness

For optical microcomponents, being able to produce optical quality surfaces is key. The surface roughness of an optical component needs to be at least smaller than $\lambda/8$,³² where λ is the smallest wavelength at which the part will be used. Taking into account that PMMA has a high transmittance (>94%) for wavelengths higher than 400 nm ,¹⁹ the $\lambda/8$ optical surface quality requirement implies a surface roughness of maximally 50 nm . Using a Bruker contour GT-I noncontact optical profiler, the surface roughness of the sidewalls depicted in Fig. 6(b) is measured. We measure the root-mean-square surface roughness defined as $R_q = \sqrt{(1/n) \sum_{i=1}^n y_i^2}$ (where y_i is the vertical distance from the mean height line to the i 'th data point) over an area of $56\text{ }\mu\text{m} \times 44\text{ }\mu\text{m}$. The measurements are performed in a structured way over the entire surface for each irradiated section (and thus for each proton fluence used) with 72 evenly spaced measurements, represented schematically by the black squares in Fig. 6(b). This means that more than 38% of the surface of each line is measured and taken into consideration [notice that the rectangles of Fig. 6(b) are not drawn to scale]. The results of the surface roughness measurements are shown in Fig. 9 and one can see that by increasing the proton fluence, the average surface roughness decreases slightly. Considering the large variance however, there is no significant impact of the proton fluence on the surface roughness. We achieve optical surface quality sidewalls ($R_q < 46\text{ nm}$) for all considered proton fluences (between 7.25×10^6 and $8.75 \times 10^6\text{ }\mu\text{m}^{-2}$), satisfying the requirement of $\lambda/8$ for wavelengths larger than $\sim 370\text{ nm}$. This means that the conicity and development time are the more important factors to take into account when choosing a proton fluence for irradiation, as long as fluences higher than $7.25 \times 10^6\text{ }\mu\text{m}^{-2}$ are considered. These results are in good agreement with previous work, performed with 8.3-MeV DPW .³³

Fig. 9 Root-mean-square surface roughness obtained from 72 evenly spaced measurements with an area of $56\text{ }\mu\text{m} \times 44\text{ }\mu\text{m}$ using optical noncontact profilometry per irradiated line, irradiated with a different proton fluence.

3.3 Applications

Using DPW, we can prototype high aspect ratio, micro-sized structures with optical quality surfaces. Hence there are a myriad of possible applications. Originally we performed DPW using protons with an energy of 8.3 MeV . This allowed us to irradiate PMMA substrates with a thickness up to $500\text{ }\mu\text{m}$. From 2002 to 2005, spherical microlenses were the focus of our DPW research. Fabrication of these lenses is possible by using the in-diffusion of a monomer (MMA in this case) into the irradiated zones of the PMMA substrate, which causes the PMMA to swell.^{21,34,35} Starting in 2006, microholes were fabricated in which single-mode fiber arrays could be snap-fitted and could be aligned to each other, resulting in highly efficient optical interconnects.²² In addition, the coupling of light in and out of printed circuit board-integrated waveguides for short-reach optical interconnections was investigated. To this end, pluggable micro-optical out-of-plane-couplers²⁰ [depicted in Fig. 10(a)] and embedded micromirror inserts³⁶ were fabricated. For these applications, it would be beneficial to use thicker samples (since that would enable better angular alignment of inserted fibers in the fiber connectors, or the capability to address more than one board-integrated waveguides with the out-of-plane couplers) or reduce the conicity of the sidewalls to increase the mirror flatness. To be able to irradiate increasingly thick PMMA substrates and to reduce the conicity in $500\text{-}\mu\text{m}$ -thick substrates, we increased the energy of the protons used for DPW to 12 MeV . This was made possible by the stacking approach for making the stopping mask thicker, as explained in Sec. 2.1. The most recent application for DPW was the fabrication of high-precision micromechanical self-centering alignment structures for single-mode fibers. Such a DPW-fabricated prototype is shown in Fig. 10(b). These prototypes have been demonstrated to achieve insertion losses down to 0.1 dB at 1550 nm when assembled into a prototype fiber connector.⁴ The record-thickness for a PMMA component was achieved with DPW at 16 MeV . An optofluidic device for combined absorption and fluorescence measurements was successfully fabricated in 2-mm -thick PMMA.¹⁸ So far, DPW components have thus been used for micromechanical, micro-optical, and optofluidic applications. Currently, we are investigating the use of DPW to fabricate a high-precision mould for hydrogel casting, with the aim of creating structures that emulate the natural environment of tissue for the *in vitro* biological study of cells. To this end, we are currently optimizing DPW at 12 MeV in negative photoresist material (SU-8).

4 From Prototyping to Large-Scale Fabrication

It is clear that DPW is not a mass fabrication technology as such, but rather a flexible rapid prototyping technology. To be able to scale up the production of microcomponents based on DPW-fabricated prototypes, it is very important to show that DPW prototype components are compatible with replication technologies, such as hot embossing. To investigate this compatibility, the micro-optical out-of-plane-coupler²⁰ [depicted in Fig. 10(a)] was used as sacrificial master component to generate a mould insert by electroforming. The prototypes were bonded to a copper substrate after which nickel could be grown over the PMMA prototype. The prototype was subsequently dissolved with acetone, after which hot embossing replication with that mould insert

Fig. 10 (a) Prototype out-of-plane coupler and (b) prototype self-centering alignment structure for single-mode fiber alignment. Both prototypes are fabricated with DPW in 500- μm -thick PMMA.

was successfully achieved.³⁷ More recently, a mould insert of the high-precision micromechanical self-centering alignment structures for single-mode fibers was created. The difficulty for electroforming nickel over these structures lies in the fact that they contain deep trenches (i.e., with high aspect ratio) and that one has to ensure that the nickel starts to grow at the bottom of these trenches instead of on top of the PMMA prototypes. To overcome this difficulty, a seed layer was deposited on the component prior to electroforming.³⁸ Different bonding strategies were also investigated to minimize the unwanted filling of the trenches, due to capillary effects, with adhesive, used for bonding the prototype to a copper substrate.³⁸ The mould was subsequently used for the successful replication of self-centering alignment structures by hot embossing.³⁹ Hence, we have successfully proven the compatibility of DPW with replication through hot embossing. Notice that the mould inserts created from the DPW prototypes could also be used for other replication technologies, such as microinjection moulding.⁴⁰

5 Conclusions

We presented DPW, as a versatile microfabrication technology for the rapid prototyping of components with high aspect ratio and optical surface quality. In this work, we used a collimated proton beam with an energy of 12 MeV to irradiate PMMA substrates with high molecular weight ($\sim 10^5$ g/mol), which act as a positive resist for DPW. First, the molecular weight change in the PMMA due to irradiation was investigated for different proton fluences, using GPC. A proton fluence of $8.33 \times 10^5 \mu\text{m}^{-2}$ or higher causes a significant decrease (of more than one order of magnitude) in molecular weight and thus enables selective developing of the irradiated zones. Then a 50- μm -diameter proton beam was used to irradiate lines of which the quality of the resulting inner sidewall profiles was measured with stylus and optical noncontact profilometry. The results show that the sidewall profile has a conical shape causing an increase in line thickness on average from 50 to 95 μm over 1 mm of PMMA. The root-mean-square surface roughness was measured to be smaller than 50 nm (averaged over multiple areas of $56 \mu\text{m} \times 44 \mu\text{m}$) for a fluence above $7.25 \times 10^6 \mu\text{m}^{-2}$, meaning that optical surface quality was achieved. The optimal trade-off between conicity, surface roughness, and irradiation time was determined to be

obtained for a proton fluence of $7.75 \times 10^6 \mu\text{m}^{-2}$. Because DPW can achieve high aspect ratio structures with optical surface quality sidewalls, a myriad of applications, such as alignment structures for single-mode fiber, micromirrors, out-of-plane couplers, microlenses, (micro)fluidic devices, and even structures for biological research, are possible. We are currently investigating the use of SU-8 as a negative resist for 12 MeV DPW, to expand the possibilities even further.

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