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Direct E-beam Lithography of PDMS

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Introduction
Poly(dimethylsiloxane) (PDMS) is a versatile material frequently used in the fabrication of micro and nano scale devices. It has a unique combination of properties including excellent thermal and chemical stability and non-toxicity making it an attractive material for use in many fields of science, especially in biomedical research. Its sensitivity to electron radiation [2] has lead to its use as a resist for subsequent substrate patterning [3] albeit generally in a modified form [4-6]. Here we analyze the effects of exposing liquid PDMS to electron radiation over a large range of doses on the resulting elastic modulus and topography. The data shows that PDMS processed using e-beam lithographic techniques is a viable structural material capable of being utilized in the next generation of microfluidic and other micro devices.

Fabrication by E-Beam Lithography
PDMS with a zero shear viscosity of 1 Pa.s was decanted onto a clean Si wafers (IOB Technologies, UK): 2 mm native SiSOI layer and spun at a frequency of 33.3 Hz for 100 minutes, using a microprocessing (KEP-100API, Kurt Lesker Limited, UK). The resultant PDMS thin film thickness were in the range 1.5 µm to 50 µm and measured using an interferometer (Dektak 6M, Veeco). Ultraviolet (UV) exposure was performed over the wavelength range 250 - 800 nm. Exposure was carried out using an FEI XL30 SFG field emission scanning electron microscope (SEM) equipped with a pattern generator for lithography (Raith Ellipsiss). Arrays of 25 squares of 50 x 50 µm was exposed on the sample at a beam energy of 30 keV and beam current 1.02 nA, using the Raith pattern generator. After exposure the sample was dipped in toluene for 10 seconds to remove the unexposed areas of the PDMS from the substrate. Visualisation of topography (see Figs. 1 and 2) was performed using a MicroXAM interferometer (Omniscan, UK).

Experimental Analysis of Topography and Elastic Modulus
Acquisition of topographical and mechanical data were performed simultaneously using a Nanowizard II AFM (JPK, UK) operating in force scan mapping mode, at a temperature of 18 °C and a relative humidity in the range 25-35 %. This involved the use of a scanner with a maximum lateral range of 100 x 100 µm and a maximum vertical range of 30 µm in conjunction with a CellModem module (JPK, UK). Data acquisition was performed using rectangular 130 µm length Si cantilevers (type NSC30/RMN AL Mikro Masch, Estonia) having pyramidal tips with 10 nm nominal radii of curvature. Cantilever spring constants were on the order 0.2 N/m and were calibrated according to the method reported by Bowen et al. [7]. Data were acquired at 400 surface locations within the 100 x 100 µm scan area by driving the fixed end of the cantilever at a velocity of 20 µm/s towards the sample surface, whilst monitoring the deflection of the free end of the cantilever using a laser beam. Upon making contact with a surface feature, the height of the contact point was recorded, which was converted into a map of surface topology. A Hertzian model was fitted to data from four selected areas to assess the mechanical response of each exposed region. A maximum compressive load of 5 nN was applied to the surface during data acquisition, which corresponded to a small indentation strain.

The data in Fig. 3 shows how the Young’s modulus generally increases with increasing dose. At doses below ca. 150 µC/cm² the PDMS appears to be still mostly liquid-like with the apparent increase in elastic modulus corresponding to a change in the viscoelastic properties of the material. The noise at very low doses suggests that there is local agglomeration of partially cross-linked polymer chains through which the indentation is affected by the proximity of the substrate. The discontinuity at ca. 150 µC/cm² implies a critical cross-linking density at which solidification occurs. Topology data measured with the AFM, as exemplified in Fig. 4, was used to measure the thickness of the PDMS remaining after development. Fig. 5 shows that at doses above the critical value of ca. 150 µC/cm², the resultant thickness plateaus to a value close to that of the liquid film. The noise at doses above ca. 3000 µC/cm² in Fig. 3 is due to swelling effects due to excessive backscattering contributed to the increase in thickness in Fig. 5. In Fig. 4 and reduction in resolution as seen in Figs. 1 and 2. This suggests that between the limits of 150 µC/cm² and 3000 µC/cm², the PDMS is solid with consistent thickness and resolution and that the Young’s modulus is adjustable by three orders of magnitude from 1 MPa to 1 GPa.

Raman Spectroscopy
Large areas of approximately 1 x 1.3 mm were irradiated with electron doses of between 10 and 45,000 µC/cm² at a beam energy of 30 keV and beam current of between 21 and 780 nA. The XL30 final aperture was opened to increase beam current allowing faster exposures of the extremely large areas required for Raman measurements. The unexposed material was not removed prior to spectroscopy. Raman spectra of specimens were obtained using a Witec Alpha 3000R (LOT Orisit, UK) operating a 0.3 W single frequency 785 nm diode laser (Topica Photonics, Germany) and an Acton SP2300 triple grating monochromator/spectrograph (Pricinopt Instruments, USA) over the wavelength range 200 – 3000 cm⁻¹ at a spectral resolution of 1 cm⁻¹. Two 600 g/mm gratings were fitted in parallel on the prisms, covering the 200 – 500 cm⁻¹ and 500 – 3000 cm⁻¹ regions of interest. 

The chemical composition of PDMS is shown in Fig. 6. During irradiation, methyl groups are removed allowing the backbone of the polymer chains to become cross-linked. However, at large doses, the electrons also cause chain scission and cause other chemical species to form. This is what is observed in Figs. 3 and 5 where we see liquid like PDMS below ca. 150 µC/cm², solidified PDMS between 150 and 3000 µC/cm² and swelling effects above 3000 µC/cm². These chain formations and scissions corresponding to changes in phase may be perceptible in the Raman data. The results of the Raman analysis on samples with doses ranging from unexposed to 27517.7 µC/cm² are shown in Fig. 6. There are small peaks forming at higher doses in the highlighted areas in Fig. 7, and while they are weak, they are persistent, suggesting possible new bonds. This will form the basis of future analysis.

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