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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 led to its use as a resist for subsequent substrate patterning \(^3\) albeit generally in a modified form \(^4\). 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 SiO\(_2\) layer) and spun at a frequency of 33.3 Hz for 100 minutes, using a spin coater (WSL, Prom.Span, Ltd, Laurel, Laboratories, USA). The resultant PDMS film thickness were in the range 1.5 μm ± 50 nm and measured using an atomic force microscope (AFM, NanoS不少人(Toptica, UK) working in contact mode, in air). The data was corrected according to the method reported by Bowen et al. \(^5\). The PDMS was left to stand for 40 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 across 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 Horizontally model was fitted to data from the four selected positions to assess the mechanical response of each exposed region. A maximum compressive load of 5 mN was held on the sample for 10 seconds to remove the unexposed areas of the PDMS from the substrate. 

Experimental Analysis of Topography and Elastic Modulus
Acquisition of topographical and mechanical data were performed simultaneously using a Nanowizard II AFM (PJK, 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 × 100 μm and a maximum vertical range of 30 μm in conjunction with a CellMension module (PJK, UK). Data acquisition was performed using rectangular 130 μm length Si cantilevers (Type NSC30/n Al Mikro Masch, Estonia) having pyramidal tips with 10 nm nominal radius of curvature. Cantilever spring constants were on the order of 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 across 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 Horizontally model was fitted to data from the four selected positions to assess the mechanical response of each exposed region. A maximum compressive load of 5 mN was held on the surface during data acquisition, which corresponded to a small indentation strain.

Raman Spectroscopy
Large areas of approximately 1 × 1.3 mm were irradiated with electron doses of between 10 and 45,000 μC/cm\(^2\) at a beam energy of 30 keV and beam currents of between 21 and 780 nA. The XSL30 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 WETec Alpha 300R (LOT Oriel, UK) operating a 0.3 W single frequency 785 nm diode laser (Topica Photonics, Germany) and an Acto SP2300 triple grating monochromator/spectrometer (Princeton Instruments, USA) over the wavelength range 200 - 3000 cm\(^{-1}\). Mean spectra were composed of 100 accumulations, acquiring individual spectra using an integration time of 0.2 s.

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References

Fig. 1: Topography of PDMS irradiated with dose range 10-10,000 μC/cm\(^2\)
Fig. 2: Topography of PDMS irradiated with dose range 50-50,000 μC/cm\(^2\)
Fig. 3: Effect of dose on Young’s modulus
Fig. 4: 3D topology of developed sample
Fig. 5: Effect of dose on resulting film thickness
Fig. 6: Schematic of section of PDMS polymer chain
Fig. 7: Raman spectra – highlighted areas denote regions of interest