Supplemental material for

Accurate dosimetry in scanning transmission X-ray microscopes via the cross-linking threshold dose of poly(methyl methacrylate)

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1. Create *.xyt files for the PatternGen routine within STXM_Control

- Using a graphics program such as Microsoft Paint, create a black and white *.gif file. With the current version of STXM_Control, an uncontrolled exposure is made at the bottom left and top right of patterns. Intentionally adding an extra single pixel at the bottom left and top right of the *.gif image keeps these unintended exposures away from the desired pattern.

- Read the *.gif file into the program aXis2000 (Read → Images → Graphics → GIF → Data).
- Resize the image (Images → Set XY scale).
- Set the pixel size by remeshing (Utilities → Change mesh).
- Threshold mask the image so the pixel values fall between 0 and 1 (Images → Generate mask → Threshold, or Images → Multiply/divide).
- Write out the *.xyt file (Write → ALS-xyt).

Note that there is a limit to number of records in *.xyt files (i.e. points in the pattern). STXM_Control will crash if an *.xyt file with greater than 20000 records is executed. Individual pixel exposure times and positions can also be manually adjusted by opening the *.xyt file in a program such as Microsoft Notepad.

2. Execution of *.xyt files with the PatternGen routine within STXM_Control

- With the shutter set to AUTO, initiate a PatternGen scan in STXM_Control.

- Select the desired *.xyt file.

- Manually select the pattern origin point. The sample will be patterned beginning at this point from left to right and bottom to top.

- Choose a time multiplier value. For example, if all pixel values in the *.xyt file are between 0 or 1, and a time multiplier value of 300 is entered, the pixel exposure times will be between 0 and 300 ms. 10000 ms is the maximum multiplier value. In addition, the minimum time the shutter can go from closed to open to closed is about 1 ms.

- Estimate the time it will take to complete the pattern before executing by multiplying the number of pixels by the dwell time per pixel.

3. Derivation of a convenient form of the photon absorption rate

The photon absorption rate F is,

$$F = I_0 - I \qquad \text{(Eqn. 2)}$$

Transmission T is,

$$T = \frac{I}{I_0} \qquad \text{(Eqn. 6)}$$

Rearranging,

$$I = T(I_0) \qquad \text{(Eqn. 7)}$$

Subbing Eqn. 7 into Eqn. 2,

$$F = I_0 - T(I_0) \qquad \text{(Eqn. 8)}$$

Rearranging,

$$F = I_0(1 - T) \qquad \text{(Eqn. 9)}$$

Optical density *OD* is,

$$OD = -\ln\frac{I}{I_0} \qquad \text{(Eqn. 10)}$$

It follows that,

$$e^{-OD} = \frac{I}{I_0} = T \qquad \text{(Eqn. 11)}$$

Eqn. 3 is the result of subbing Eqn. 11 into eqn. 9.

$$F = I_0\left(1 - e^{-OD}\right) \qquad \text{(Eqn. 3)}$$

Supplemental figures

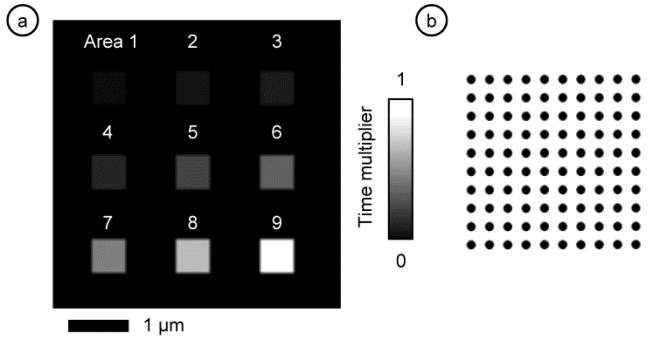

Figure S-1: a) The input file used for all patterning experiments. The white numbering serves to identify the areas and is not a part of the file. The dimensions of each square are 600 nm x 600 nm. b) Each square in a) consists of 10 x 10 individual exposures with a pitch of 60 nm.

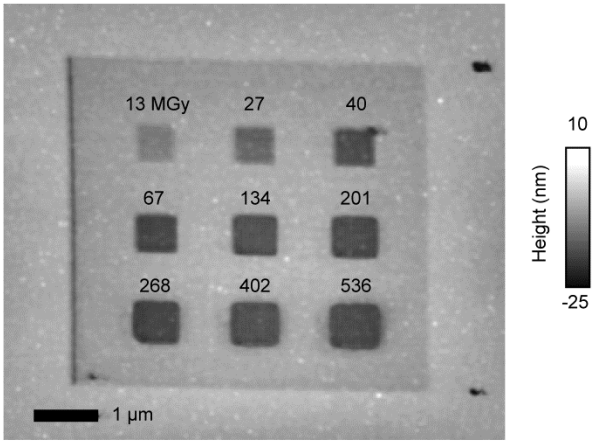

Figure S-2: Atomic force micrograph of a patterned area that had been previously imaged by STXM. The image parameters were 50/25/25 slits, 288.4 eV, 2 ms dwell time, 60 nm pixel size; the dose associated with collecting the image was 1 MGy. The nine area pattern was executed within the area that had been STXM imaged. The outline of the single STXM image is visible, and the dose associated with it caused a 2 nm reduction in height.

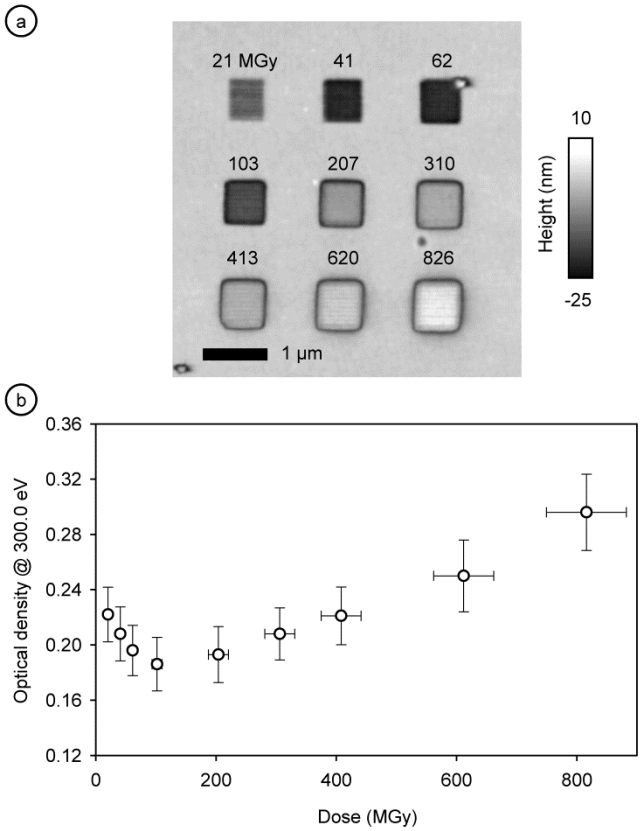

Figure S-3: a) Atomic force micrograph of as-spun PMMA, patterned with 10ID-1. The patterned areas initially decrease and then increase with increasing dose due to carbon contamination. b) STXM OD at 300 eV measurements of each area in a) plotted versus dose. The carbon signal rises with dose, even above the virgin PMMA film.