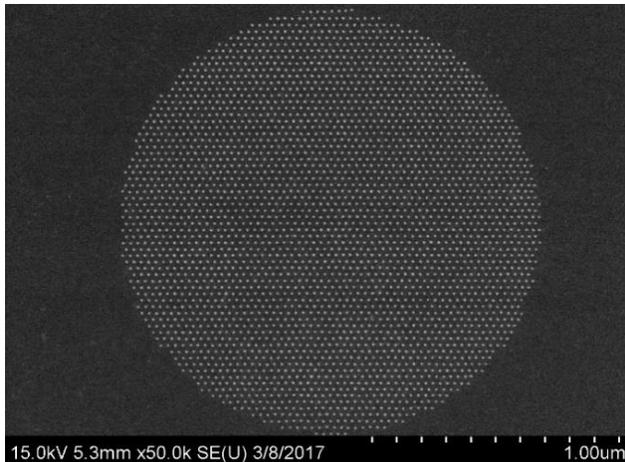


# AQM HSQ / H-SiO<sub>x</sub>

**H-SiO<sub>x</sub>** is a high purity, silsesquioxane-based solid with a long shelf life (>1 year when stored correctly). It is soluble in most organic solvents (e.g., anisole, *n*-butyl acetate, hexanes, methyl isobutyl ketone [MIBK], 2-propanol, toluene, xylenes). H-SiO<sub>x</sub> is commonly dissolved in MIBK, but carrier solvent investigations by AQM support an increased shelf-life in *n*-butyl acetate (HDPE container). The solutions are dispensed onto substrates/wafers and dispersed via spinning. After spinning, a film of H-SiO<sub>x</sub> remains that can be used as a resist or spin-on dielectric. H-SiO<sub>x</sub> can be used in electron beam (EB), extreme ultraviolet (EUV), and nanoimprint lithography systems.

Sufficiently dosed H-SiO<sub>x</sub> forms a low dielectric constant (low- $\kappa$ ) silicon-rich oxide that is resistant to removal by hydroxide-based developers, such as tetramethylammonium hydroxide (TMAH) (e.g., MF-319, MF-CD-26) and Salty Developer (NaOH/NaCl).

AQM is constantly synthesizing, testing, and improving its H-SiO<sub>x</sub> to meet and exceed customers' expectations. AQM has sold H-SiO<sub>x</sub> since 2017 and has distributors located in the United States (DisChem Inc.), Europe (EM Resist Ltd), China (GT Nano), Japan (OptoSirius), Korea (HunetPlus).

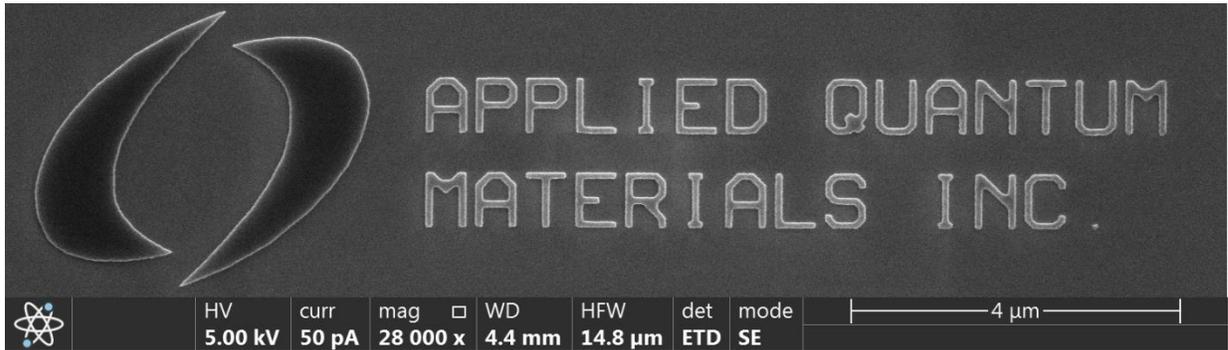


### Lithography features:

- Thin uniform films (5 nm – 2  $\mu$ m thickness)
- High resolution (capable of  $\leq 10$  nm patterning)
- Excellent line edge roughness
- Good dry-etch resistance

### Other Applications:

- Photoresist for mask making.
- Mask for etching, e.g. Si, SiO<sub>2</sub>, Si<sub>3</sub>N<sub>4</sub> or metals.
- Silicon-based photonics – waveguide components, grating couplers, and photonic crystals.
- Generation of stamps with nanopatterns.



**Figure 1.** SEM image of an AQM logo (~13.5 μm wide, ~80 nm thick layer) exposed at 400 μC/cm<sup>2</sup> (RAITH150 Two at 30 kV, 15 μm aperture), developed in MF-319 for 90 seconds, and then rinsed with deionized H<sub>2</sub>O for 60 seconds. Taken at the [UofA nanoFAB](http://uofa.nanoFAB).

### Purchasing:

AQM H-SiO<sub>x</sub> solid can be purchased separately or in a resist kit containing solid, HPLC grade *n*-butyl acetate (≥99.7%) or MIBK (≥99.5%), syringes with PTFE filters.

H-SiO<sub>x</sub> solutions can be made to standard 1, 2, 4, 6 or custom weight percentage. For the best results, it is recommended that freshly opened *n*-butyl acetate or MIBK is used. AQM is currently developing our own line of developer solutions: Salty Developer (NaOH/NaCl); TMAH (various concentrations). Contact us for details and pricing.



Examples H-SiO<sub>x</sub> solutions available:

Solution w/w	<i>n</i> -Butyl Acetate	H-SiO <sub>x</sub>
1 %	25.0 mL	0.223 g
6 %	17.8 mL	1.00 g
6 %	25.0 mL	1.41 g
20 %	4.5 mL	1.00 g
20 %	5.0 mL	1.10 g

Solution w/w	MIBK	H-SiO <sub>x</sub>
1 %	25.0 mL	0.202 g
6 %	19.6 mL	1.00 g
6 %	25.0 mL	1.28 g
20 %	5.0 mL	1.00 g

Solutions should be made with high accuracy measuring instruments (do not use glass) for the most consistent coating thicknesses.

HDPE containers are filled to 80% capacity due to shipping regulations. Common sizes available include 8, 30, 60, 125, 250, 500, and 1000 mL (Max volumes: 6, 24, 48, 100, 200, 400, and 800 mL, respectively)

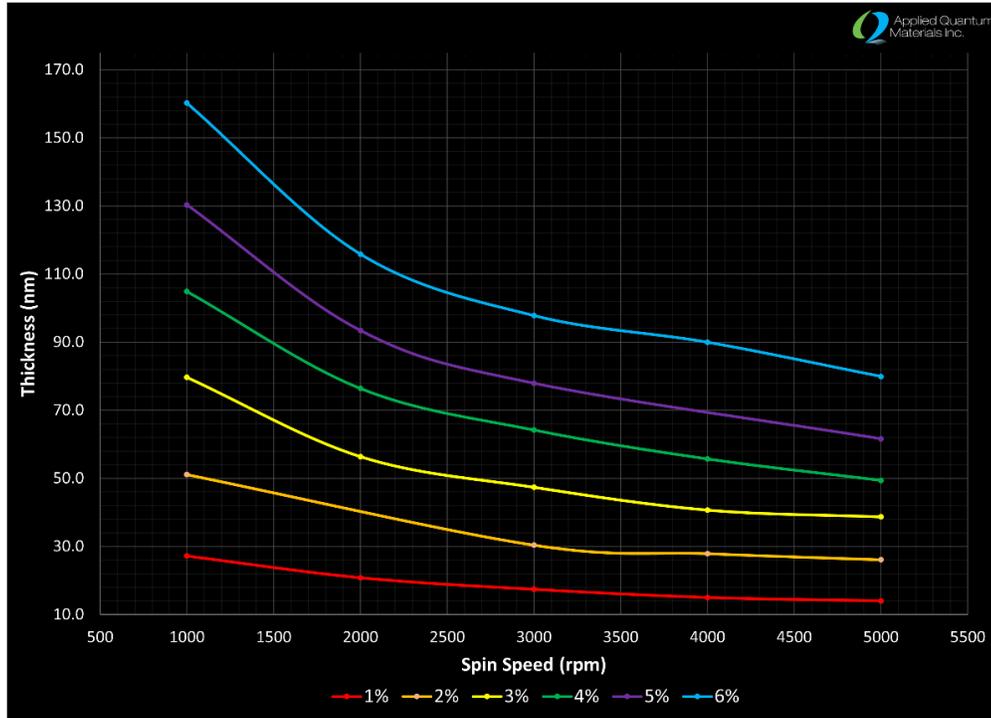
### Shelf-Life and Storage:

- Solid: at least 1 year when stored in a vacuum (≤30 mmHg) at ambient temperature in HDPE.
- Dissolved in *n*-butyl acetate: greater than 3 months (estimate: at least 9 months) when contained in a sealed HDPE bottle, kept in a low-moisture environment.
- Dissolved in MIBK: at least 3 months when contained in a sealed HDPE bottle, kept in a low-moisture environment.
- Solutions: Shelf-life may be extended by storing at significantly lower temperatures. Storing in a fridge is not significantly colder than room temperature. Containers should never be opened below room temperature.

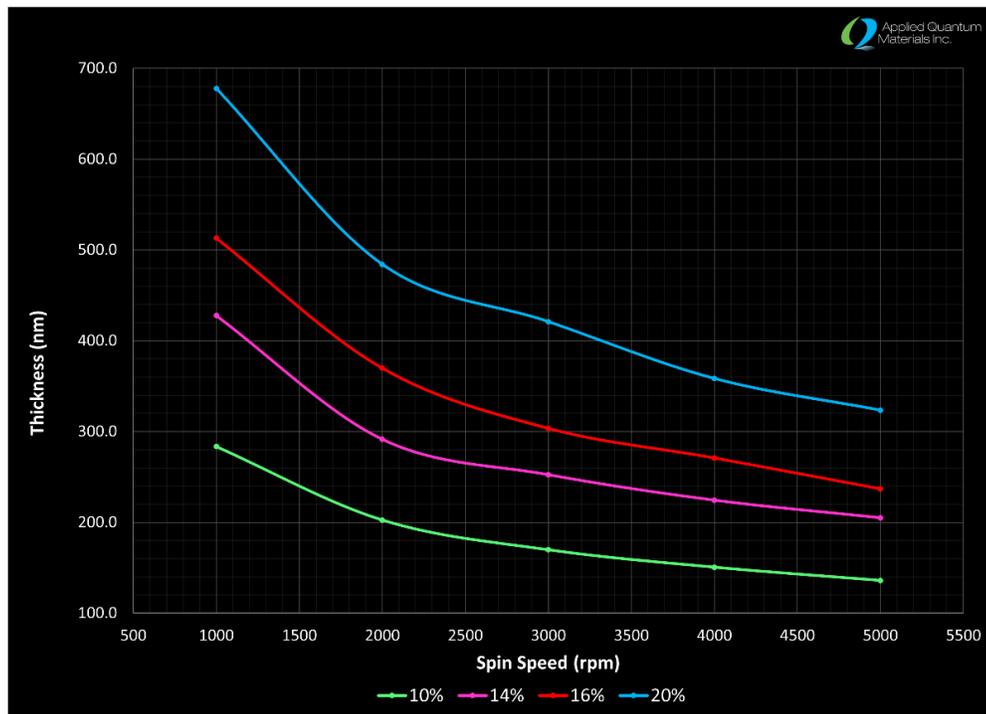
## Spin Curves:

### *H-SiO<sub>x</sub>-15 in n-butyl acetate:*

Spin curves were constructed using 10 x 10 mm P-Type Silicon wafers. The wafers were centered on a Laurell Model WS-400B-6NPP Spin Coater. Using a sterile syringe, 1-2 drops of H-SiO<sub>x</sub> in *n*-butyl acetate was added; the spinner lid was closed, and the wafer spun at the desired speed for a total of 60 seconds (1 second ramp to speed). Thicknesses were measured on a Filmetrics F50-UV.



**Figure 2.** Spin Curves for H-SiO<sub>x</sub> at 1, 2, 3, 4, 5 and 6% (w/w) in *n*-Butyl acetate.



**Figure 3.** Spin Curves for H-SiO<sub>x</sub> at 10, 14, 16, and 20% (w/w) in *n*-Butyl acetate.

### H-SiO<sub>x</sub>-15 in MIBK:

Spin curves were constructed using freshly piranha cleaned 10 x 10 mm P-Type Silicon wafers. The wafers were prebaked at 180 °C for >3 minutes, transferred to a Brewer 200X Spin Coater, and centered. Using a sterile syringe, 1-2 drops of H-SiO<sub>x</sub> in MIBK (passed through a 0.1 μm PTFE filter if specified) was added; the spinner lid closed, and the wafer spun at the desired speed for a total of 60 seconds (1 second ramp to speed). A post-apply bake at 80 °C for 3 minutes was performed before measuring the thicknesses on a Filmetrics F50-UV.

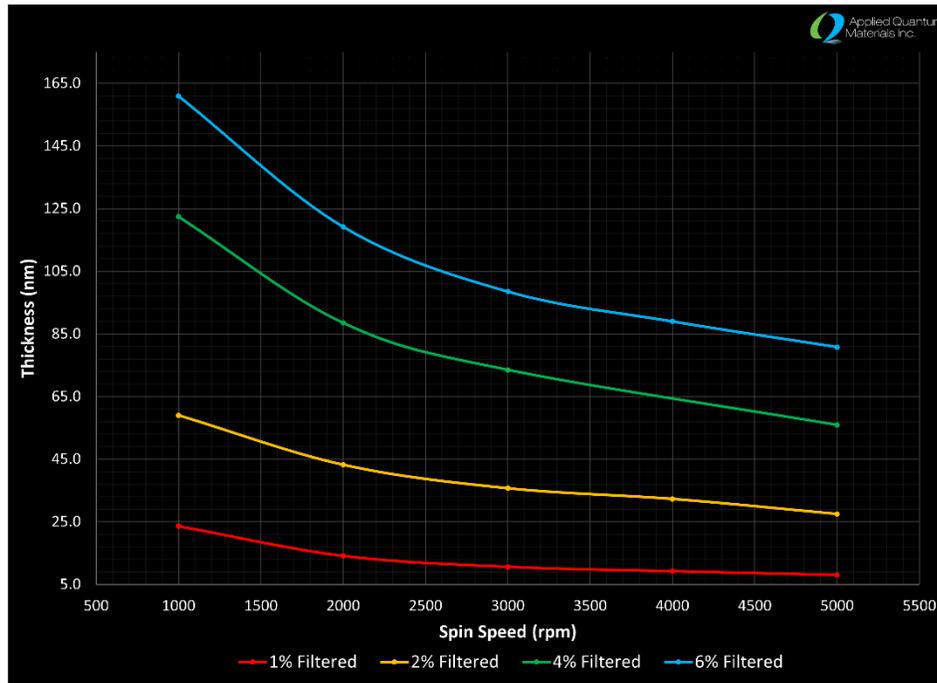


Figure 4. Spin Curves for H-SiO<sub>x</sub> at 1, 2, 4 and 6% (w/w) in MIBK.

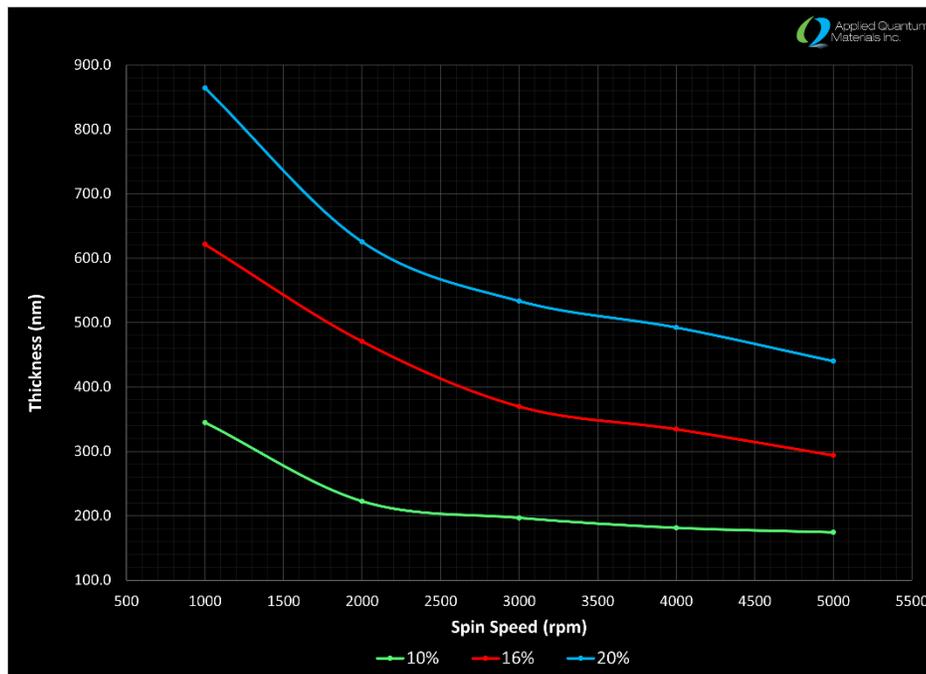
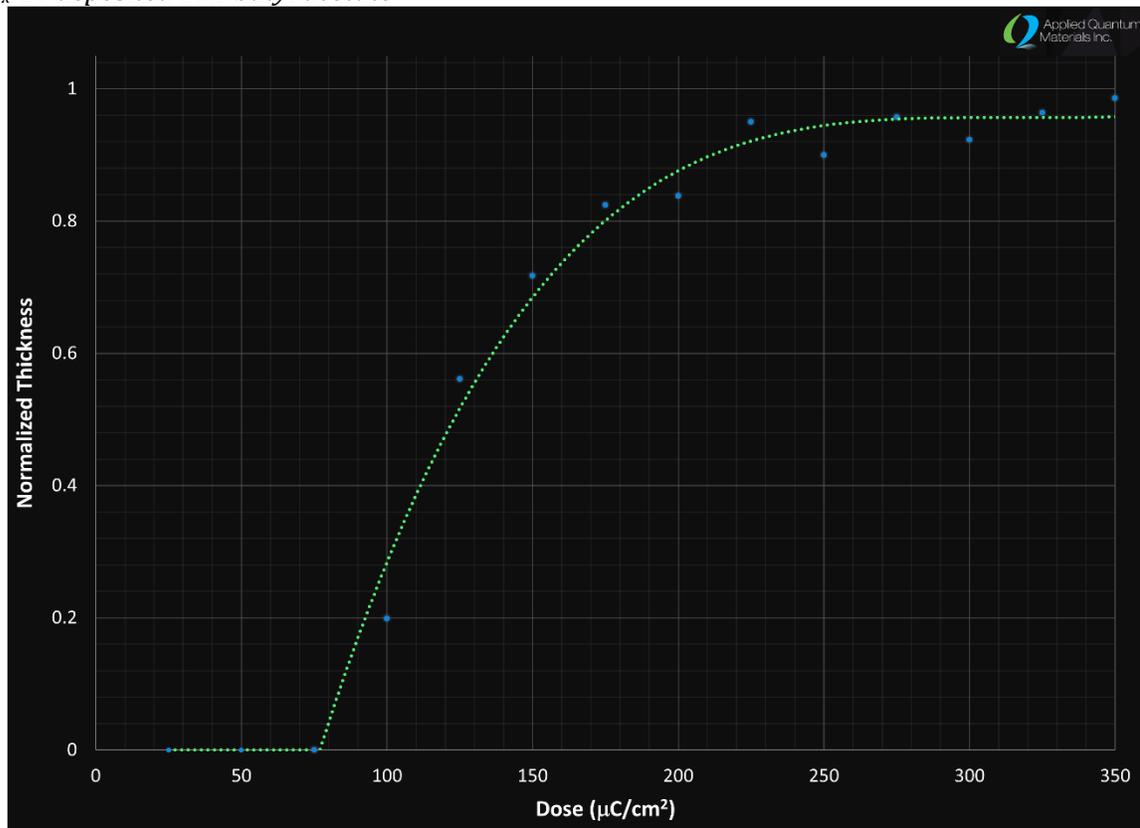


Figure 5. Spin Curves for H-SiO<sub>x</sub> at 10, 16, and 20% (w/w) in MIBK.

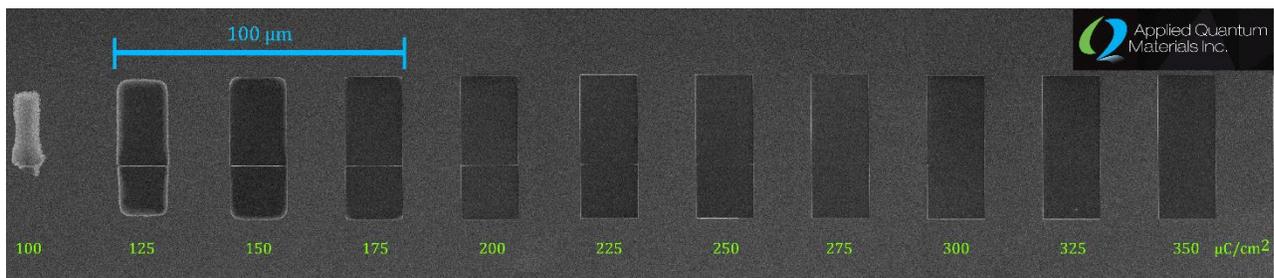
## Contrast Curves:

*H-SiO<sub>x</sub>-15 deposited in n-butyl acetate*



**Figure 6.** Contrast Curve of ~100 nm H-SiO<sub>x</sub>. Developed in MF-319 for 90 sec.

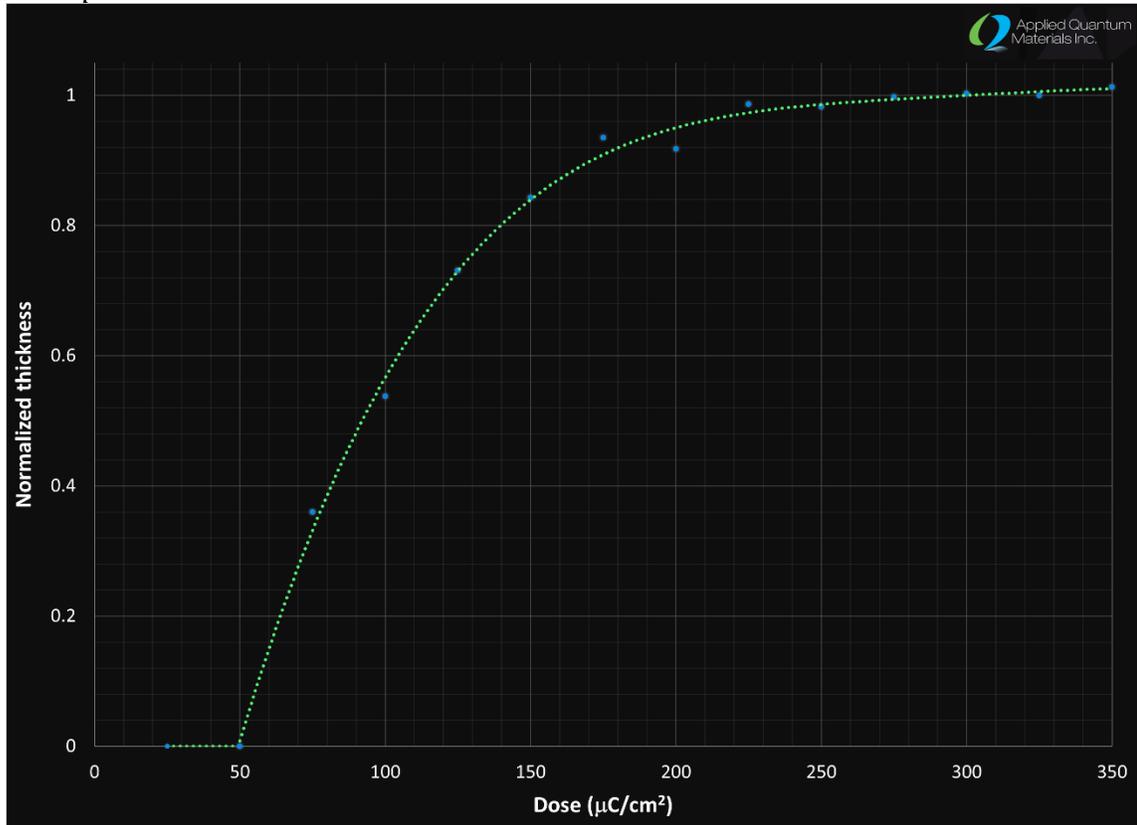
Note: Contrast curve was constructed using the thicknesses of rectangles (supposed to be 20 x 50 µm) formed on an ~100 nm thick layer of H-SiO<sub>x</sub> (spin process above). The H-SiO<sub>x</sub> was exposed using a RAITH150 Two at 30 kV, developed in MF-319 for 90 seconds, and rinsed with deionized water for 60 seconds. The dose rectangles thicknesses were determined using an Alpha-Step IQ.



**Figure 7.** SEM image of dose rectangles formed on an exposed (RAITH150 Two at 30 kV, 15 µm aperture) and developed (MF-319, 90 seconds, deionized H<sub>2</sub>O 60 seconds) ~100 nm thick layer of H-SiO<sub>x</sub>. The dose rectangles are supposed to be 20 x 50 µm.

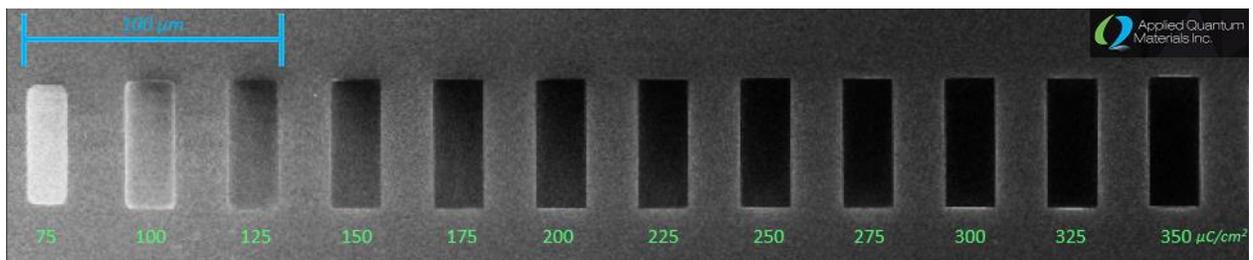
Note: For smaller features (e.g., lines smaller than 100 nm) higher doses (>500 µC/cm<sup>2</sup>) are required for an ~100 nm thick layer developed in MF-319 for 90 sec.

## H-SiO<sub>x</sub>-15 deposited in MIBK



**Figure 8.** Contrast Curve of ~80 nm H-SiO<sub>x</sub>. Developed in MF-319 for 90 sec.

Note: Contrast curve was constructed using the thicknesses of rectangles (supposed to be 20 x 50 μm) formed on an ~80 nm thick layer of H-SiO<sub>x</sub> (spin process above). The H-SiO<sub>x</sub> was exposed using a RAITH150 Two at 30 kV, developed in MF-319 for 90 seconds, and rinsed with deionized water for 60 seconds. The dose rectangles thicknesses were determined using an Alpha-Step IQ.



**Figure 9.** SEM image of dose rectangles formed on an exposed (RAITH150 Two at 30 kV, 15 μm aperture) and developed (MF-319, 90 seconds, deionized H<sub>2</sub>O 60 seconds) 80 nm thick layer of H-SiO<sub>x</sub>. The dose rectangles are supposed to be 20 x 50 μm.

Note: For smaller features (e.g., lines smaller than 100 nm) higher doses (>400 μC/cm<sup>2</sup>) are required for an ~80 nm thick layer developed in MF-319 for 90 sec.