

AQM H-SiOx/HSQ

H-SiOx 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., acetone, 2-propanol, ethyl acetate, methyl isobutyl ketone [MIBK], toluene, hexanes). H-SiOx is commonly dissolved in MIBK and used as a negative tone resist to fabricate thin-films using electron beam (EB), extreme ultraviolet (EUV), and nanoimprint lithography (NIL) systems.

Sufficiently dosed and exposed H-SiOx forms a low dielectric constant (low-κ) silicon rich oxide that is resistant to removal in developers, such as Salty Developer (NaOH/NaCl) and tetramethylammonium hydroxide (TMAH).

AQM is constantly synthesizing, testing, and improving its H-SiOx to meet and exceed customers' expectations. AQM has sold H-SiOx since 2017 and has distributors located in the United States (DisChem Inc.), Europe (EM Resist), and China (GermanTech).





Lithography features:

- Thin uniform films (5 nm 2 µm thickness)
- High resolution ($\leq 10 \text{ nm patterning}$)
- Excellent line edge roughness
- Good dry-etch resistance

Other Applications:

- Photoresist for mask making.
- Mask for etching, e.g. Si, SiO_2 , Si_3N_4 or metals.
- Silicon-based photonics waveguide components, grating couplers and photonic crystals.
- Generation of stamps with nanopatterns.

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Figure 1. SEM image of an AQM logo (~13.5 μ m wide, ~80 nm thick layer) exposed at 400 μ C/cm² (RAITH150 Two at 30 kV) and developed in MF-319 for 90 seconds, then rinsed with deionized H₂O for 60 seconds. Taken at the <u>UofA NanoFAB</u>.

Purchasing:

AQM H-SiOx powder can be purchased separately or in a resist kit containing H-SiOx powder, HPLC grade 99.5% MIBK, syringes with 0.1 μm PTFE filters.

H-SiOx dissolved in MIBK can be made into standard 1, 2, 4, 6% or custom pre-mixed solutions. For best results it is recommended that purchased MIBK be used or open for no more than 3 months. AQM is currently developing our own line of developer solutions: Salty developer (NaOH/NaCl); TMAH (various concentrations). Contact us for details and pricing.

H-SiOx	MIBK	w/w solution
1.002 g	5.0 mL	20%
0.2560 g	5.0 mL	6%
1.024 g	20.0 mL	6%
0.0405 g	5.0 mL	1%



Shelf Life and Storage:

- Powder Form: at least 1 year when stored in a vacuum (≤30 mmHg) at ambient temperature in low light.
- Dissolved in MIBK: at least 3 months when contained in a sealed PP bottle, kept in a low moisture environment.
- Shelf-life may be extended by storing at lower temperatures (Bottles should never be opened below room temperature).

Testimonials:

"A side-by-side EBL comparison of standard HSQ and AQM H-SiOx (both 6% w/w in MIBK) showed that AQM H-SiOx can be readily used as a direct replacement for many existing EBL process flows." (2017) Aaron Hryciw, Ph. D, P.Eng. Fabrication Group Manager



Spin Curves:

Spin curves were constructed using 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. Then 2-3 drops of H-SiOx in MIBK (passed through a 0.1 μ m filter if specified) were added; the spinner lid was closed, and the wafer was spun at the desired speed for a total of 60 seconds (1 second ramp to speed). A post-apply bake at 80 °C for exactly 3 minutes was performed before measuring the thickness on a Filmetrics F50-UV.



Figure 2. Spin Curves for H-SiOx at 1, 2, 4 and 6% (w/w) in MIBK.



Figure 3. Spin Curves for H-SiOx at 10, 16, and 20% (w/w) in MIBK.

Contrast Curve:



Figure 4. Contrast Curve of 80 nm H-SiOx. Developed in MF-319 for 90 sec.

Note: Contrast curve was constructed using the thicknesses of rectangles formed on an 80 nm thick layer of H-SiOx (spin process above). The H-SiOx was exposed in a RAITH150 Two at 30 kV, developed in MF319 for 90 seconds, and rinsed with deionized water for 60 seconds. The dose rectangles thicknesses were determined using an Alpha-Step IQ.



Figure 5. SEM image of dose rectangles formed on an exposed (RAITH150 Two at 30 kV) and developed (MF-319, 90 seconds, deionized H_2O 60 seconds) 80 nm thick layer of H-SiOx. The dose rectangles are supposed to be 20 x 50 μ m.

Note: For smaller features (e.g., lines smaller than 100 nm) higher doses (\geq 400 µC/cm²) are required for an 80 nm thick layer developed in MF-319 for 90 sec.