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Protocol for Ultrathin Nitride Membranes

Materials:

Wafer 4" or 100mm silicon wafer, 200-300 microns thick, double side polished, Orientation <100>
Shipley 1813 photoresist (S1813)
726 MIF developer
40% KOH
PMMA e-beam resist

CNF machines used:

MOS clean hood
LPCVD Nitride – B4
FilMetrics Film Measurement System (F40)
Wet/Dry Oxide –B2 (Furnace)
Resist Spinners
90 degree bake oven
ABM Contact Aligner
Optical Microscope
Oxford 81 Etcher
Hot Plate with Temperature Probe
Nanostrip Bath hood
AFM (can also use AFM at Syracuse)

For Electron beam thinning-
E-beam Resist Spinner
JEOL 9300 or JEOL 6300

Introduction:

This protocol gives an outline of how to make thin, free-standing, low-stress nitride windows. This protocol assumes familiarity with the equipment listed under "CNF machines used". Do not attempt this protocol without first having had training from a CNF technician.

Growth of films:

Wash the wafers in the MOS clean directly prior to using the LPCVD furnaces

The first goal is to grow oxide on the wafer as an insulation layer. Use the LPCVD furnace Wet Oxide growth works well. Grow between 300 and 2000 microns of oxide on the surface (my advice is 400 microns)

Use the Filmetrics to measure the thickness of the film at multiple points on the wafer.

Rewash wafer with MOS clean.

Grow low stress nitride using the different LPCVD furnace. Use a protocol that gives a thickness of about 50 nm.

Measure the film thickness with the FilMetrics. Use the value obtained in the first scan as the thickness of the oxide layer.

Patterning of TEM grid pattern:

-Coat the wafer using S1813 photoresist using one of the spin coaters.
Use a 3000 RPM protocol and the flat (non-vacuum) shielding holding chuck.

-Bake the wafers in the 90 degree Celsius oven for 30 minutes

-Use the Filmetricts to get a thickness value of the coating.

-Use the ABM in contact lithography mode to expose the pattern.
Use the "TEMgridThinning" quartz mask.
14 seconds is the typical exposure time. Actual time will depend on resist thickness and can be looked up on the ellipsometer in the resist spinning room.

-Develop the photoresist pattern using MIF 726 developer (Do this in the fume hood rather than with the automatic developer. Should be 60 seconds development and then place in water. Rinse well and dry completely.)

-Spin coat the opposite side of the wafer. Be sure to use the falt (non-vacuum) shielding chuck.

-Paint an extra layer of S1813 on the rim of the wafer. This will protect it from under etching while you are etching the nitride.

-Bake the resist again for 90 min.

-Repeat the resist development step using MIF 726

-Use the Nitride etch protocol in the Oxford 81 to remove the nitride mask.

The etch rate for low stress nitride is listed under “LSnitride” in the Oxford 81 log book under “nitride etch”. It is typically 72.1nm/min. Etch for at least 50% longer than necessary to remove the nitride layer.

-Use the CHF3/CF4/Ar oxide removal protocol to remove the oxide. Do 8 minutes on, then vent check and redo (give the chamber 10 minutes to recuperate) until you see that it has reached the silicon.

-Do an oxygen plasma clean for 10 minutes to remove excess photoresist.

-Use the Nanostrip bath hood system to remove the remaining photoresist.

Use the YES asher to remove any persistent photoresist on the wafer (if needed)

Etching

-Heat 40% KOH to 90 degrees C and etch wafers. Etch rate is ~2.1 um per minute. Watch the wafers to see when silicon is completely etched.

You will know when the silicon is etched because the grid pattern (small squares) will now allow light to pass from one side of the wafer to the other. These squares are free-standing oxide and nitride.

The oxide layer underneath the nitride must be removed. There are several ways to do this. I have found the following to be most effective.

-Use the Oxford 81 to remove about half of the oxide layer by placing the wafer **nitride-side down** so that the plasma can only reach the oxide layer.

-Continue the 40% KOH etch. Until the oxide layer is removed. (KOH etches oxide at about 200nm per hour. You will tell when the oxide has been removed because the bottom layer of the wafer will start to etch. Remove the wafer immediately after this starts.)

-Use Buffered oxide etch for a few seconds remove the residual oxide. A room temperature etch rate is ~1.2 μm per minute.

Note: I use KOH to remove the oxide because KOH does not etch nitride. I found BOE to etch the nitride slowly.

Ebeam thinning

-Spin coat PMMA onto the wafer and bake for 60 seconds at 115 degrees.

-Load into JEOL 9300.

-Use outline to pattern.

-Alignment should be done by using the TEM windows. 3 windows in the small 5 grid pattern should be used. Pattern should be aligned to the middle window using 3 edges of 5x5 pattern. This should be repeated for 3 edges of the total pattern.

-Dose should be 1.3 maxium. For free standing nitride. Pattern will have 100 nm squares.

-Remove and thin nitride using Oxford81 (goto **Thinning**).

Photolithography Thinning (Alternative)
-Use photolithography mask with small pattern windows 1000 nm square.

-Spin coat the wafer using high RPM (5000).
-Use ABM in contact lithography mode to pattern wafer, following the same development protocol given in the "**Patterning TEM grids**" section.

-Thin the grids using the Oxford 81 (see **Thinning**)

Thinning

-Use the Oxford 81 nitride etch protocol to thin the wafers.

-I suggest making several test runs to get proper thickness. As of 2/14/12 etch rate for photolithography thinning was ~2.4 nm per second. Membranes less than 5nm in thickness break very easily.

-Remove PMMA (or S1813) using oxygen clean.

-Be careful. Membranes thinner than 10 nm are very fragile.

-Test thickness of the membrane using AFM. Alternatively you can image pores with the TEM and estimate the thickness by conductance.