Nanonex NX-2500 Imprint Tool Documentation

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January 2013

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1. Introduction

The NX-2500 from Nanonex Inc is a nanoimprint lithography (NIL) tool that can perform thermal NIL (T-NIL) or photocurable NIL (P-NIL). Specially developed resists from Nanonex are available, but other resists of your choosing may also be used.

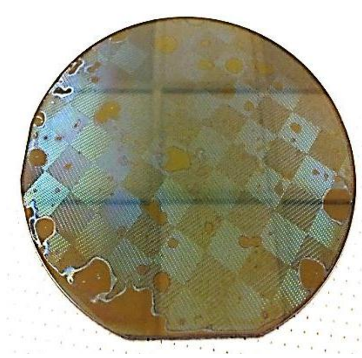
T-NIL is the simplest process as it uses a silicon template and single layer resist (NXR-1025).

P-NIL requires a quartz template that is transparent in the UV so that UV-curing can take place during the imprint. A bilayer resist is used consisting of an under layer with improved etch resistance (NXR-3022) and a photocurable upper layer (NXR-2030). The advantage of P-NIL over T-NIL is that, with the bilayer resist, features can be etched a great depth into the substrate.

Alignment is possible using the alignment module on the right of the NX-2500. This puts yet greater restrictions on the template: it must fit into the 5" square photomask holder of the alignment module and be transparent so that alignment marks on the substrate can be seen through it. Once aligned the substrate and template are brought into contact and sufficient pressure applied to stick them together, but not perform imprint. The substrate/template pair must then be removed from the alignment module and loaded into the imprint module. In my experience this was a delicate and frustrating process that often took several repeats. Also, even once loaded into the imprint module there was sometimes a shift in their relative position after closing the draw but before imprint – resulting in skewed and un-aligned imprints.

Resolution is effectively determined by the template fabrication process. If you can create small features on the template they will most likely be reproduced in the imprint resist on the substrate. In my experience, and using templates fabricated using the ASML I was able to reach 200 – 300nm features. Work is ongoing with e-beam fabricated templates to push the resolution of this tool to smaller dimensions.

Wafer Coverage is greatly affected by the cleanliness of the substrate and template. Even a tiny piece of grit can be enough to keep the template and substrate separated leaving a patch about $1cm^2$ of un-imprinted resist. There are often a few millimeters around the edge of the wafer that also do not imprint. Considering these two effects I found that a good imprint would typically result in coverage of about 90% of the wafer.



2. Template Fabrication

Silicon Template Fabrication Recipe (for Thermal Nanoimprint) – *untested by CN*

1. Substrate Clean – solvent clean, acetone, IPA
2. PECVD oxide – GSI 200nm
3. AS200/620-7i lithography module/ASML-DUV
    a). HMDS vapor prime
    b). BARC apply, bake
    c). 620-7i/deep UV resist apply
    d). soft bake, hotplate
    e). exposure
    f). develop – Hamatech
4. BARC etch – Oxford 82
5. Oxide RIE – Oxford 80, CHF3/O2
6. Photoresist removal – Oxford 80
7. Silicon RIE – PT770 or PT720 Cl2 chemistry timed etch
8. Profilometry/interferometry
9. Oxide removal – Buffered oxide etch
10. Anti-stiction application – FOTS – MVD tool

Quartz Template Fabrication Recipe (for UV nanoimprint)

1. Substrate clean – Hot Piranha Hamatech Wafer Clean (Prog 1)
2. Sputter deposit 20nm chrome – CVC sputter system, use rotating chucks for improved uniformity
3. Lithography
    a. **either** ASML (for features down to 300nm)
        i. HMDS vapor prime – YES oven
        ii. apply BARC – AR3-0.6, 4500rpm, 45sec, bake at 205^{o}C 90sec
        iii. apply resist – UV210-0.6, 4500rpm, 45sec, bake at 135^{o}C 60sec
        iv. expose – 25mJcm^{-2}, bake at 115^{o}C 60sec
        v. develop – Hamatech Prog 1 (MIF726, 60sec)
        vi. ARC etch – Oxford 80, “AR3 ARC etch” recipe, 70sec
    b. **or** AS-200 (for features down to 800nm) – *untested by CN*
4. Cr etch – Trion
    a. Clean - 10min O_2 with sapphire wafer in
    b. Clean - 10min N_2/Cl_2 with sapphire wafer in
    c. Season - 10min using “Standard Cr etch” recipe with CR target in
    d. Etch template - 3min Cr etch using “Standard Cr etch” recipe (check mask has cleared)

    e. *Immediately after etch:* DI H2O rinse, N2 dry (to prevent damage by Cl from etch)
5. Inspection – optical microscopy or SEM
6. Photoresist removal – Oxford 80, 2min O_2 clean
7. Quartz etch – Oxford 82,
    a. Clean – 10min O_2
    b. Clean – 10min N_2/Cl_2
    c. Season – CHF_4, 10min
    d. Template etch – CF_4, 3min (-> about 80nm depth)
8. Profilometry measurement
9. Inspection – optical microscopy or SEM
10. Cr strip – wet etch (use 10min Cr stripper in chem hood, Hamatech does not work)
11. FOTS (anti-stiction) application – MVD 100, 2min preclean, 10min reaction time, 10 purge cycles.

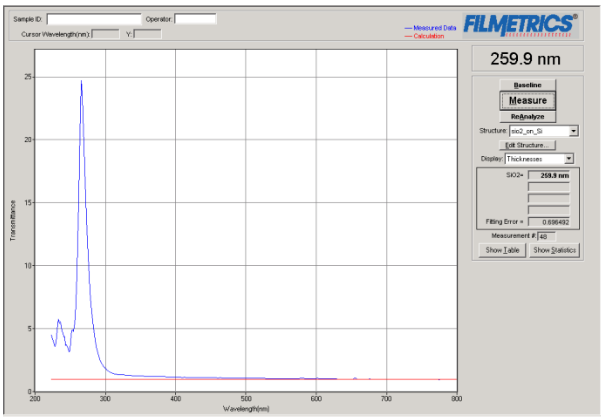
Template cleaning

1. Oxford 81, 3min O_2 clean with 5sccm CF_4, both sides
2. Hamatech Hot Piranha – Prog 1 (Wafer clean) or Prog 2 (Mask clean – more thorough)
3. Reapply FOTS as necessary – check with contact angle measurement

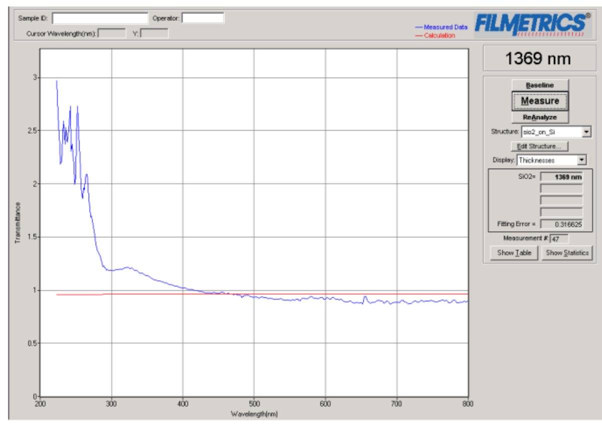
Troubleshooting

<u>Substrate Material</u>

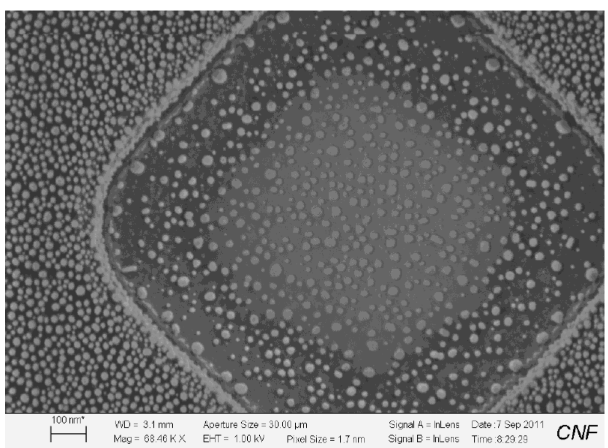
Transparent wafers may be one of many materials – borofloat glass, silica, quartz. It's important to verify that your substrate is indeed quartz for two reasons, the etch chemistries in the Template Fabrication Recipe above are designed for quartz and may have strange effects on other materials… The template must be transparent in the UV so that the photosensitive resist can be cured during imprint. Quartz is transparent in the UV, so this can be used to test whether your materials is quartz (using the Filmetrics in Duffield 224).



Quartz reference (Telic Quartz Mask)



Non-quartz substrates (probably Borofloat)

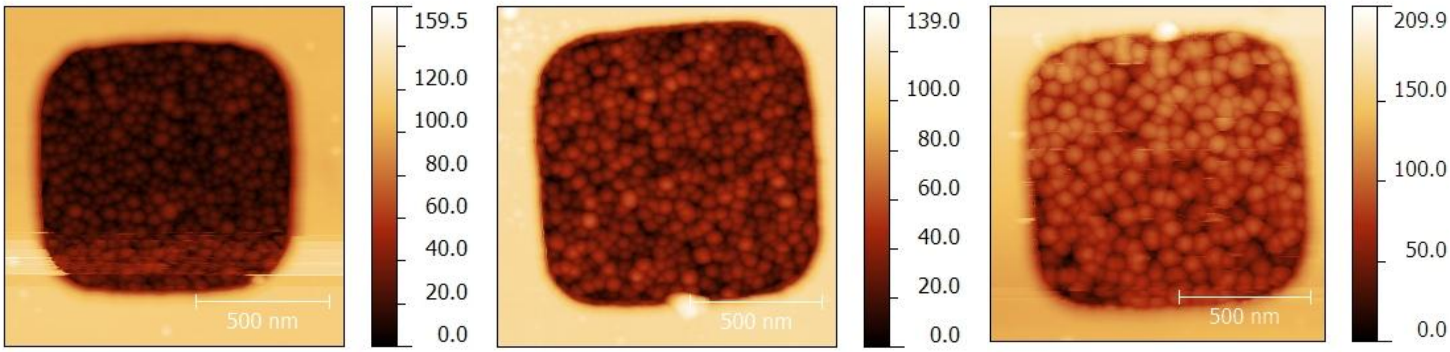


Non-quartz substrate after quartz etch

The non-quartz substrates do not etch as expected. This SEM shows a 1µm square on a non-quartz substrate after a 3 min CF_4 etch on the Oxford 81. (The distinctive spherical blobs shouldn't be there.)

Quartz etch

Several different chemistries are used to etch quartz. Here is a comparison made using the tools in CNF:

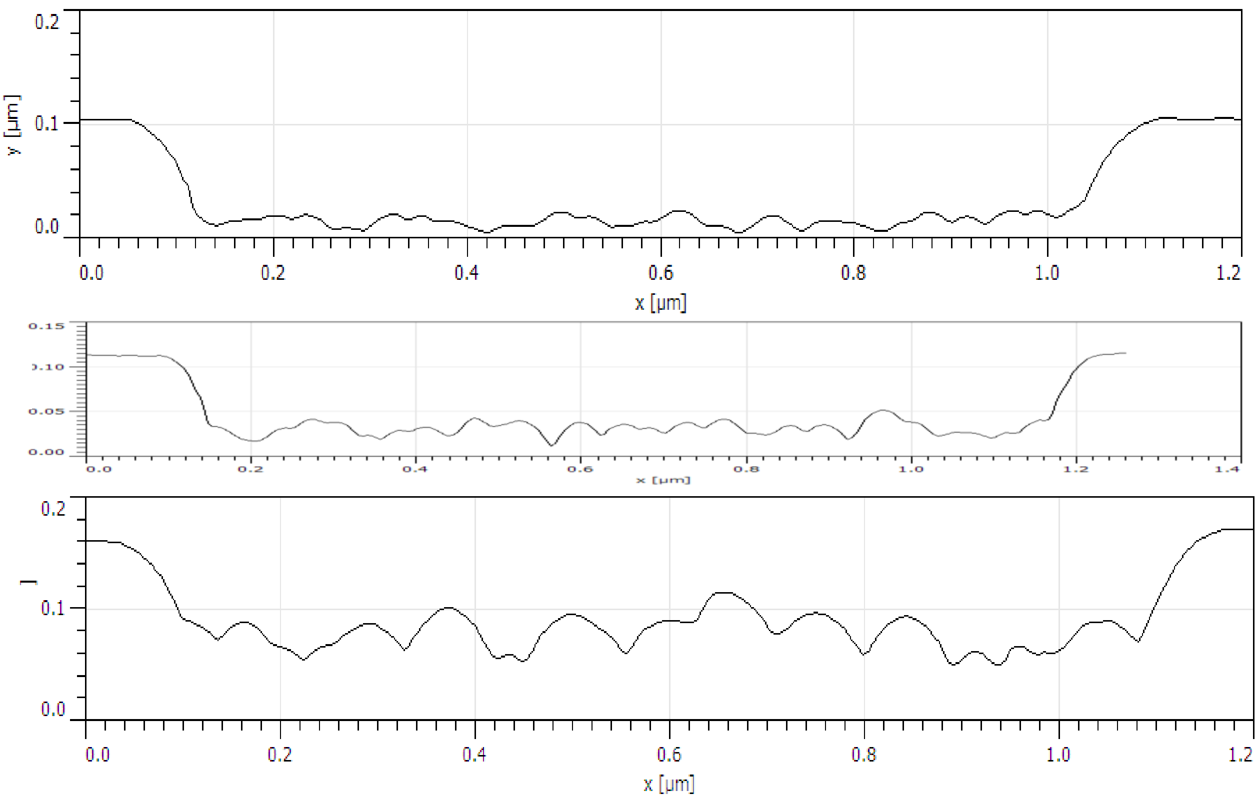


CF_4 **CHF_3/Ar** **CHF_3/O_2**

CF_4

CHF_3/Ar

CHF_3/O_2



Vertical scales have been adjusted so all profiles can be compared. It can be seen that the template fabricated using a CF_4 etch results in the lowest roughness so this is recommended for use in CNF.

3. The software

<u>To log on to the computer:</u>
Account: Engineer
Password: NX2500

<u>To log on to the software:</u>
User name: Engineer
Password: NX2500

The NX-2500 comes with two programs, one for alignment and one for performing NIL. The software is in general somewhat unstable and crashes occasionally.

The two programs cannot be run at the same time. When you are finished working with one program you must log out of it in order to be able to open the other program. If you do not do this, when you go to open the second program a dialog box will inform you the program is already running. Close everything and go to the desktop. Open the file called ???.txt. It is a blank document with either a 1 or a 0 at the top. If it's a 1 the program will not open. Change the 1 to a 0, save it and close it. You should then be able to open the program you wish to.

Once the program is open you must then log in with the user name and password above.

If you are doing P-NIL make sure the UV lamp indicator is green.

To enter the parameters of your NIL process go to Recipe -> User Set. In the window that opens enter your parameters and click OK. Check that they are correct in the top right of the control screen.

When you have loaded your template and substrate in the correct order (see below) you can hit Start.

At any time during the process you can hit Abort. This will immediately release the pressure then unlock the draw for you to unload.

<u>Warning:</u> Sometimes the parameters you enter can have unexpected effects during the NIL process. For example, if you set the imprint parameters to be 50 psi at 100C for 2 mins and the vent temperature to be 55C, this means that the 2 min imprint will be conducted, then the 50 psi pressure *<u>will be maintained</u>* until the sample temperature drops to 55C. This means that the total time your sample is subjected to 50 psi could be considerably longer than 2 mins!

4. The Nanoimprint Process

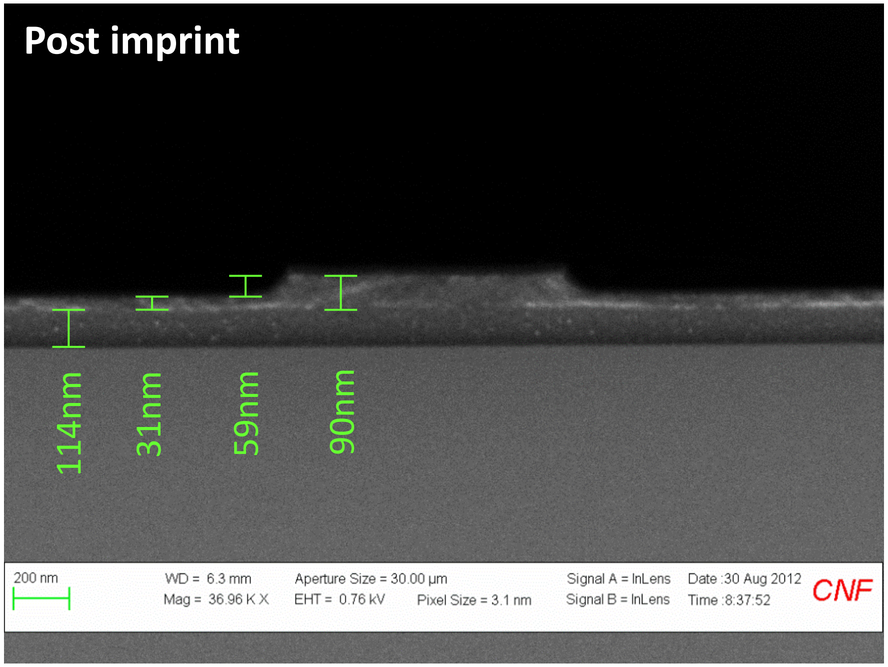
Thermal Nanoimprint Process – *untested by CN*

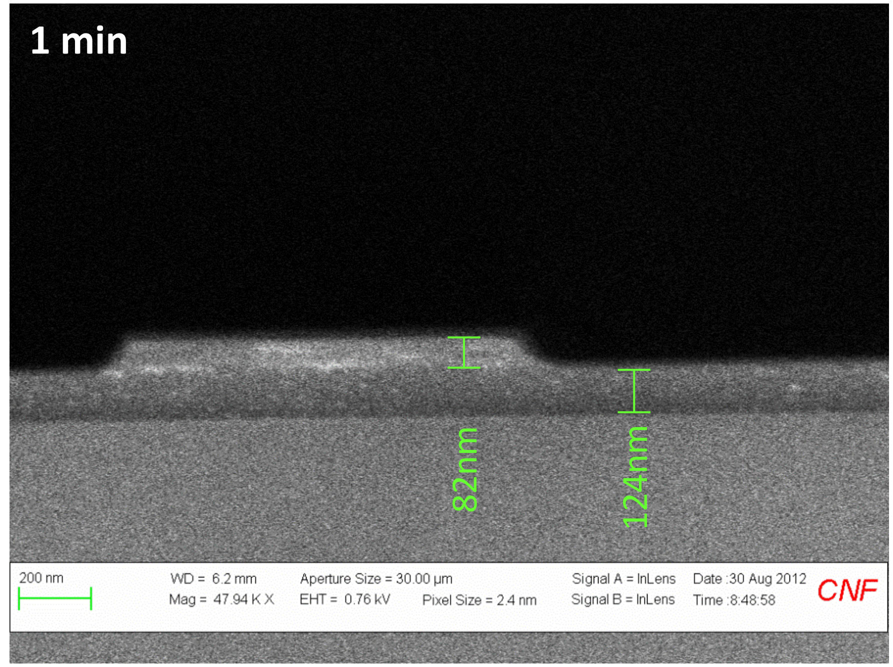
1. Imprint substrate clean – Hamatech
2. Apply NXR-1025 thermoplastic resist – 1500rpm, 60sec, 250nm (thickness should exceed maximum protrusion height of the template)
3. Bake at 150C for 1 min. on hotplate
4. Imprint – NX-2500, 200psi, 120C.
5. Mold removal
6. Pattern transfer – RIE of residual layer- Oxford 80, O_2 chemistry
7. Substrate etch – TBD
8. SEM/AFM inspection/measurement of CDs, bias, etc.

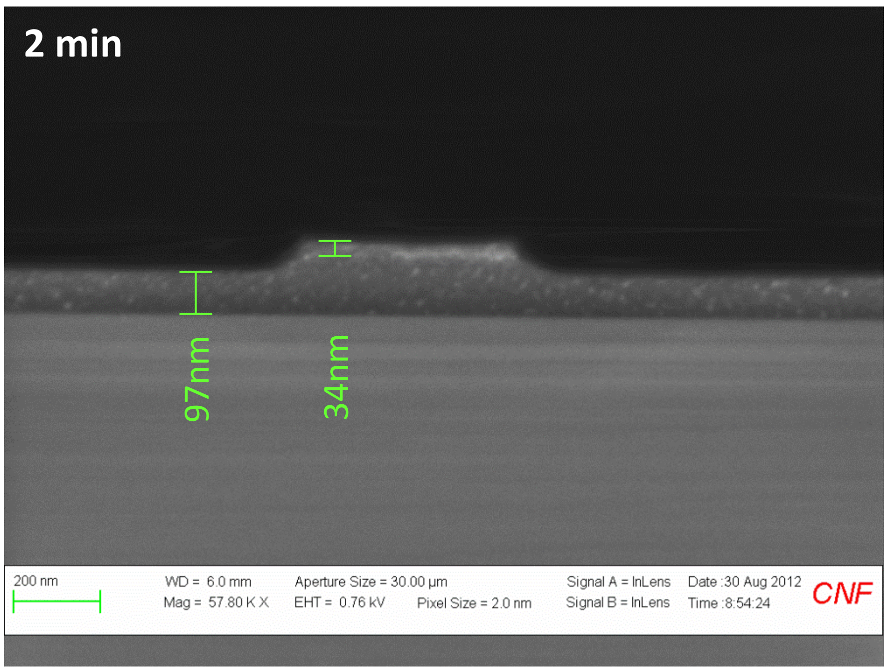
UV Nanoimprint Process

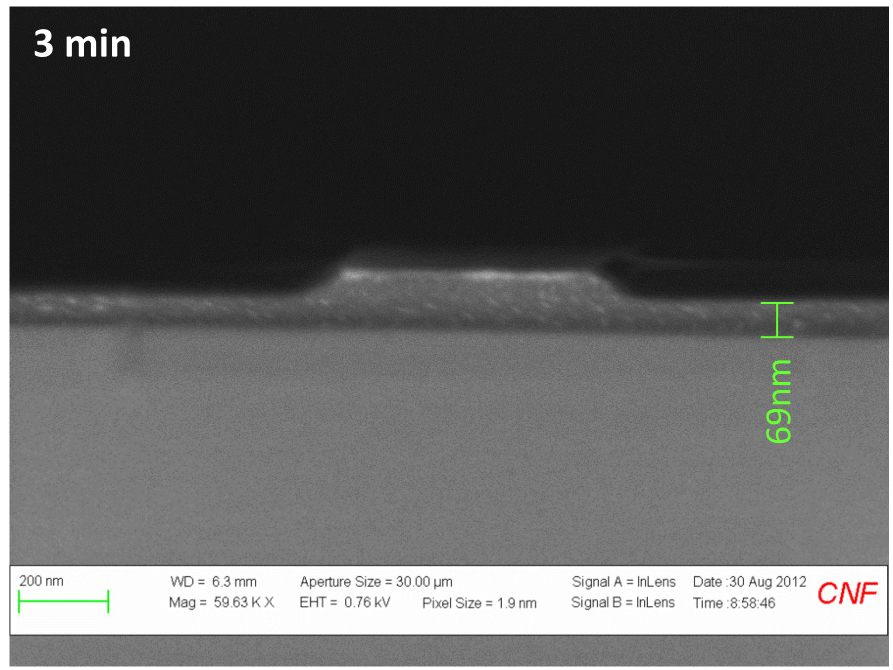
1. Imprint substrate clean – Hamatech Hot Piranha Prog 1
2. Spincoat NXR-3022 underlayer resist – 3000 rpm, 60sec (-> 200nm)
3. Bake at 190^{o}C for 90sec
4. Spincoat NXR-2030 UV curable resist – 3000 rpm, 60sec (-> 90nm)
5. Imprint
    a. Clean silicone disks (both sides) with IPA an let dry. – *greatly affects reproducibility!!*
    b. Stretch large silicone disk across metal ring, fix with magnets – *make as flat as possible.*
    c. Load: small disk (on the bottom), substrate, template (on top), large disk on ring.
    d. Set imprint parameters: 15-50 psi, 3min imprint time, 25^{o}C, 10sec UV exposure
    e. Hit start.
    f. Note: Pump time should be 2 mins or more, pressure should drop to -14psi at this time.
6. Mold removal – can be firmly stuck together, use razor blade with caution as quartz chips easily.
7. Pattern transfer into resist underlayer:
    a. NXR-2030 descum etch – Oxford 82, 30sccm CF_4, 15mT, 50W, 1min (rate: 34nm/min)
    b. NXR-3022 underlayer etch – Oxford 82, 50sccm O_2, 15mT, 50W, 2.5min (rate: 86nm/min)
8. Substrate etch – Unaxis, Photonics Etch, (NICKZOR1) (rate: 166nm/min, selectivity: ~4:1)
9. Inspection – optical, AFM or SEM

Descum Etch of NXR-2030 (Ox82, 30sccm CF4, 15mT, 50W) Rate calculated: 34nm/min

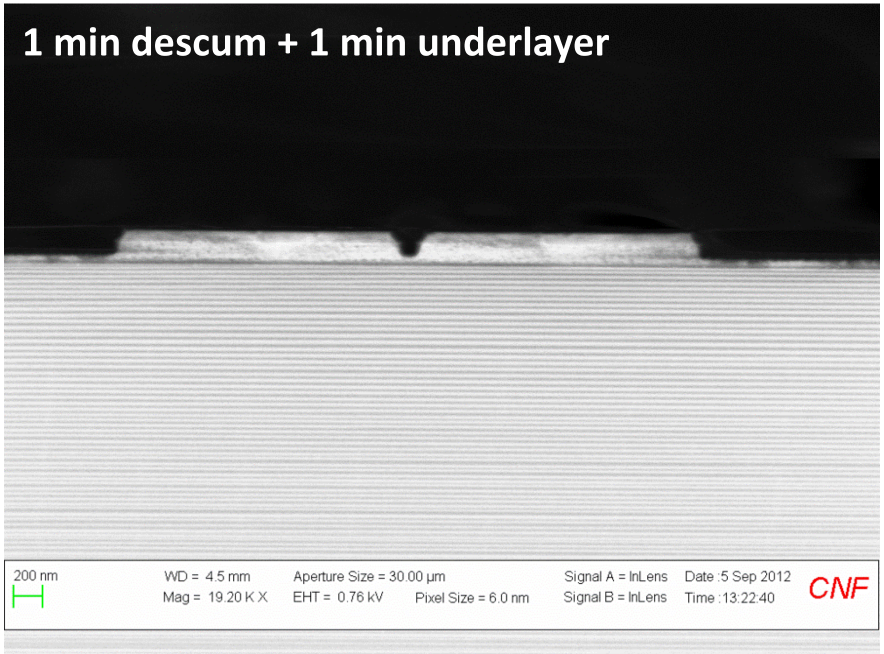


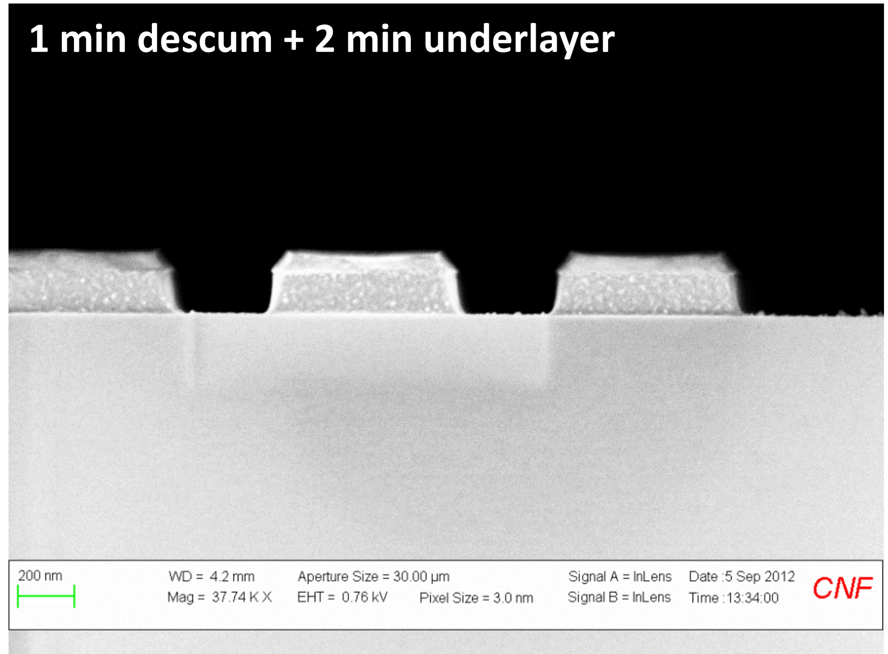


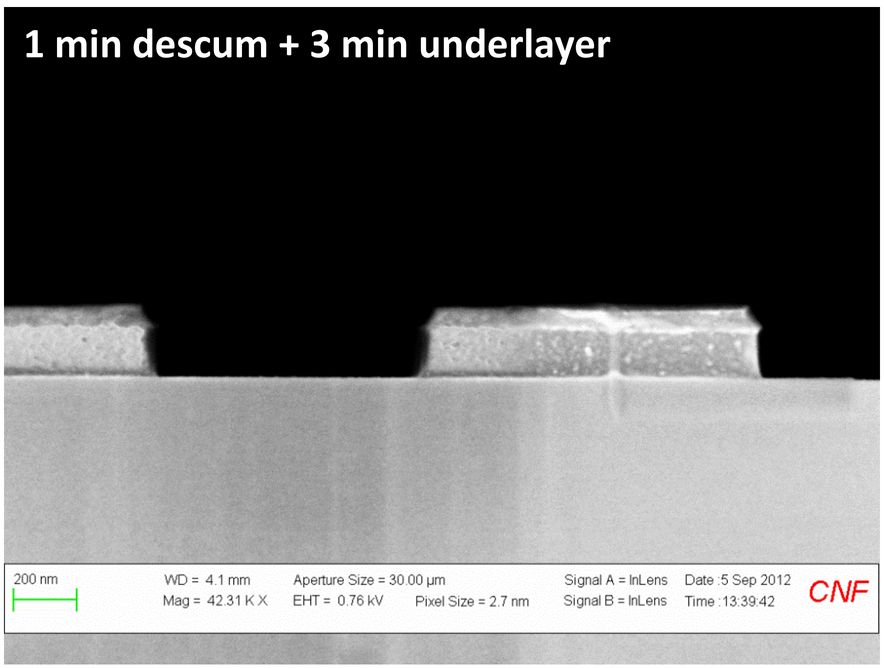


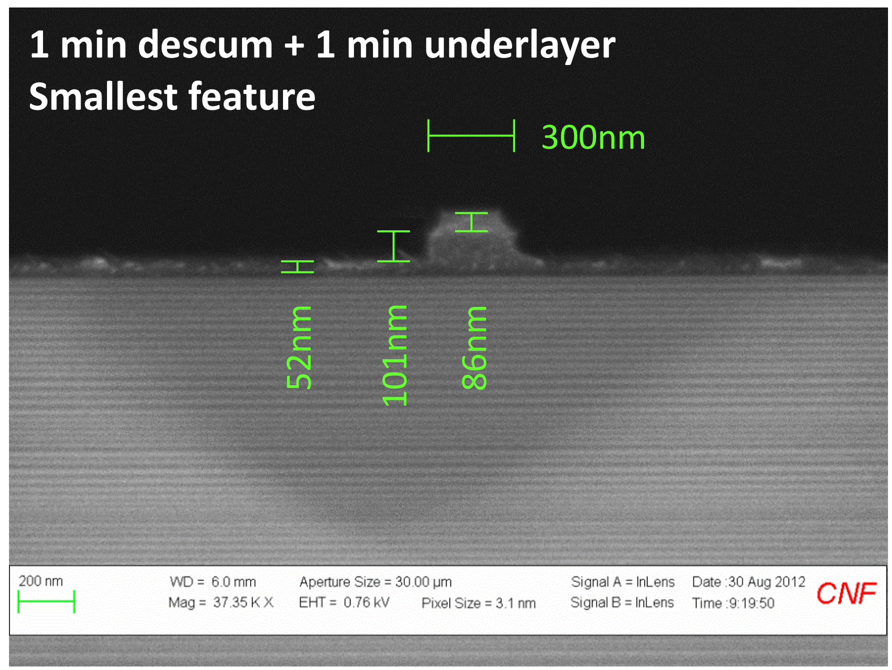


Underlayer Etch of NXR-3022 (Ox82, 50sccm O2, 15mT, 50W) Rate calculated: 86nm/min

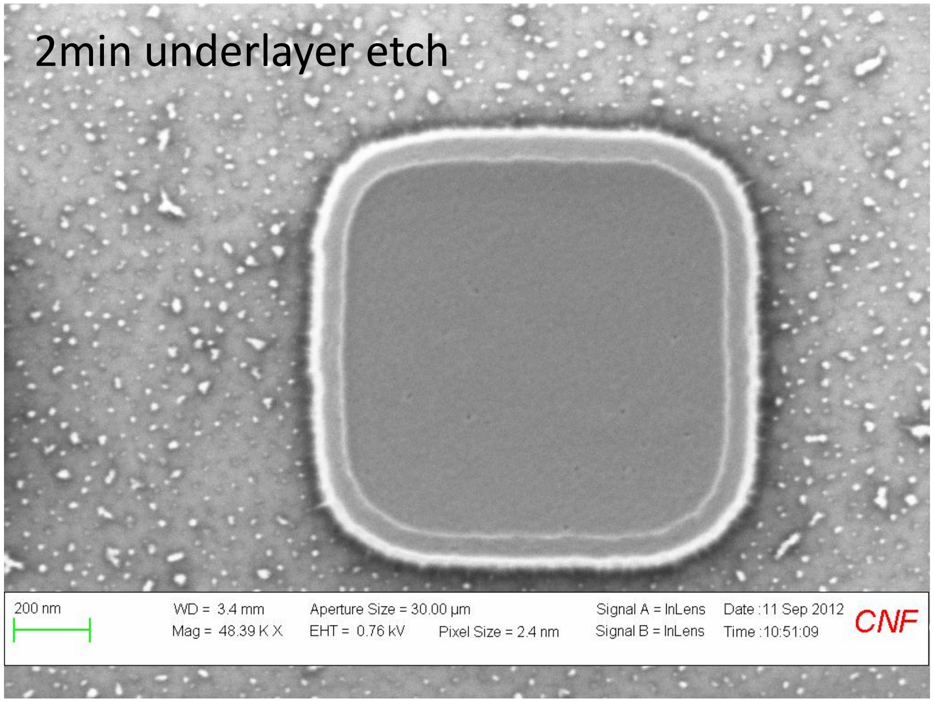


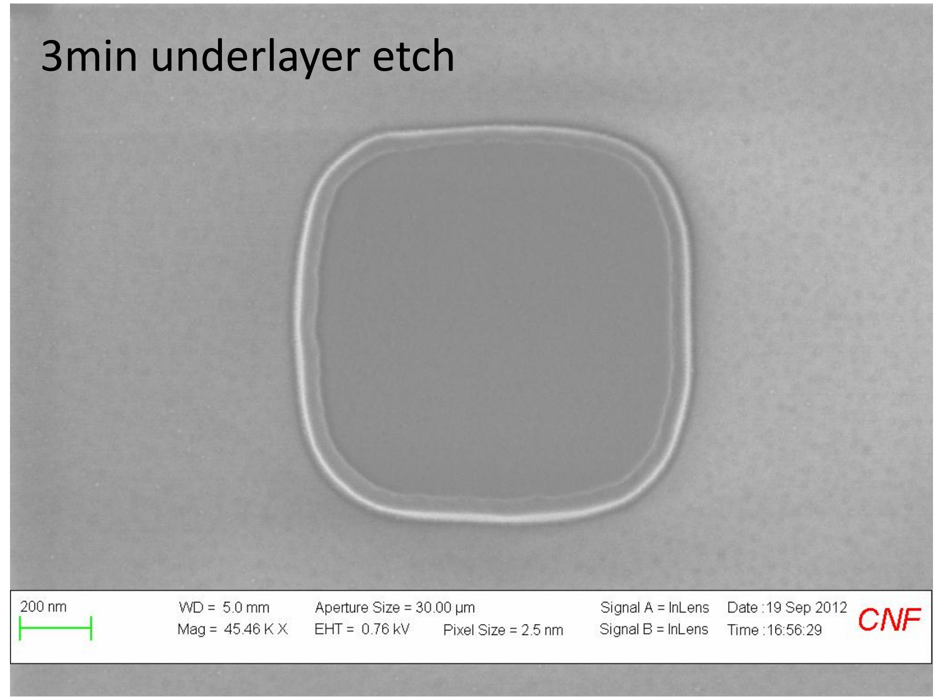


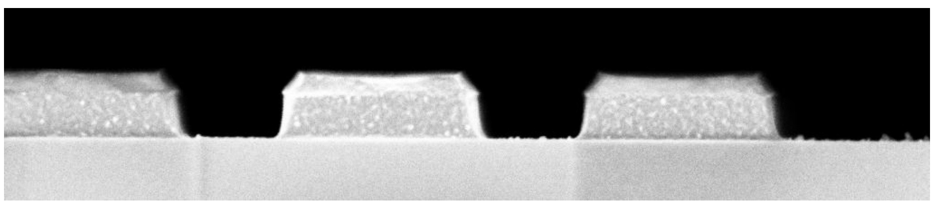


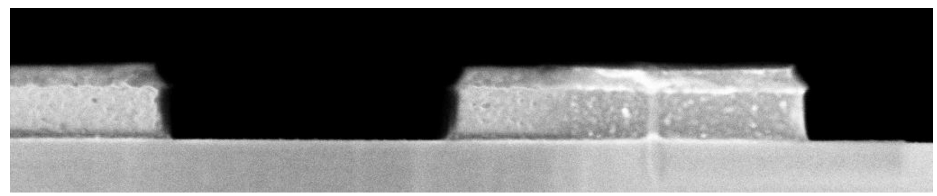


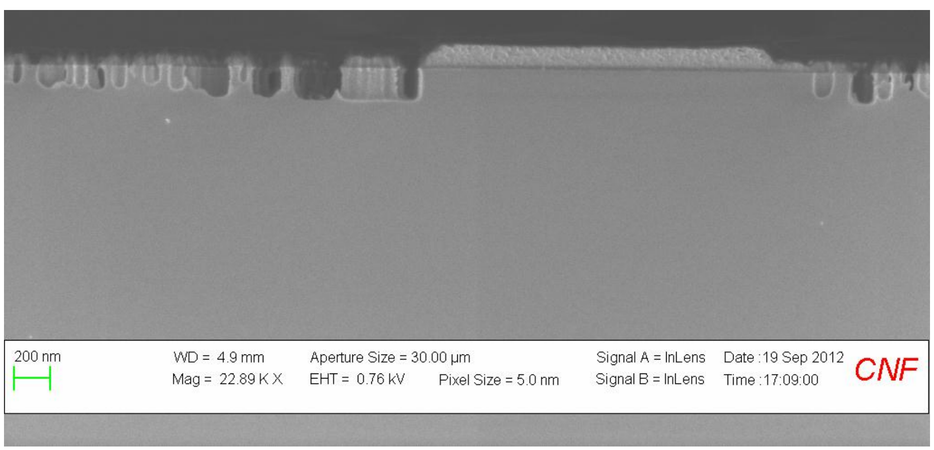
Micromasking may be caused by incomplete underlayer etch…



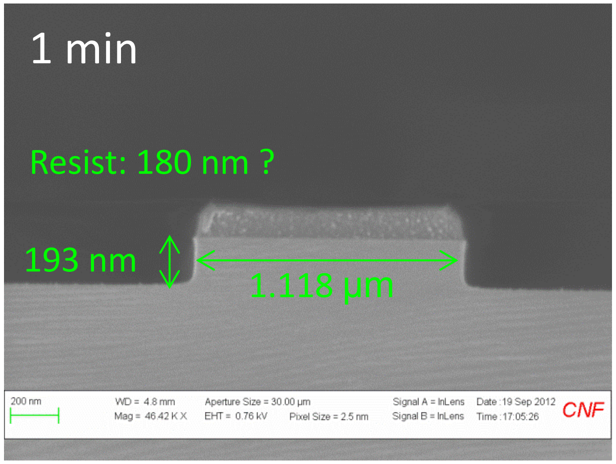


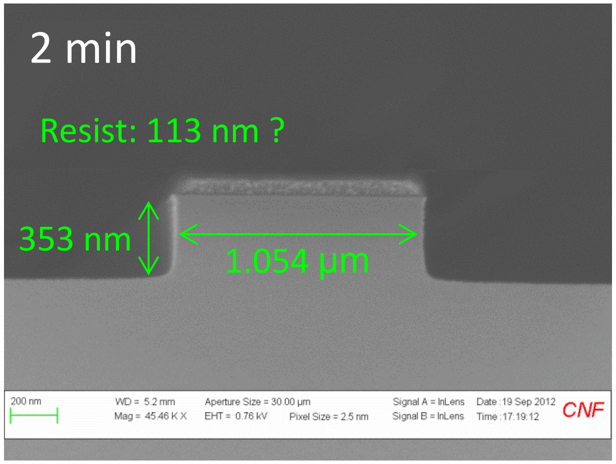


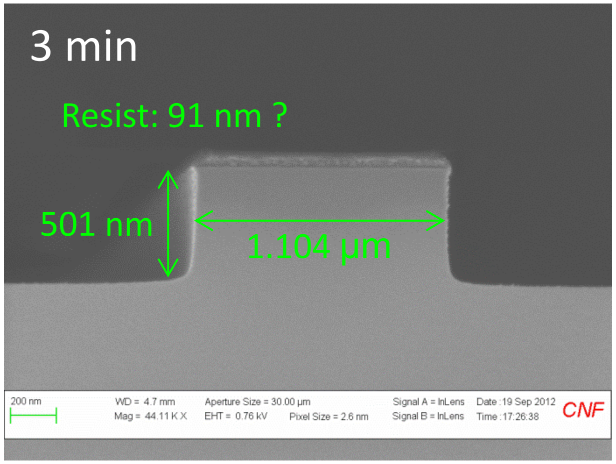


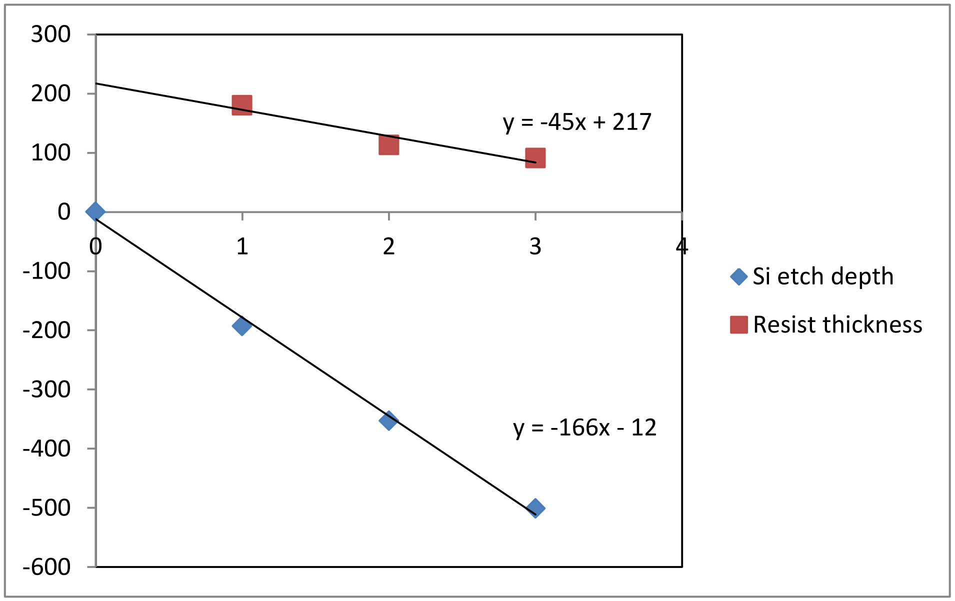


Imprint – 1 min 02 descum – 2 min CF4 underlayer etch – Unaxis Photonic Etch





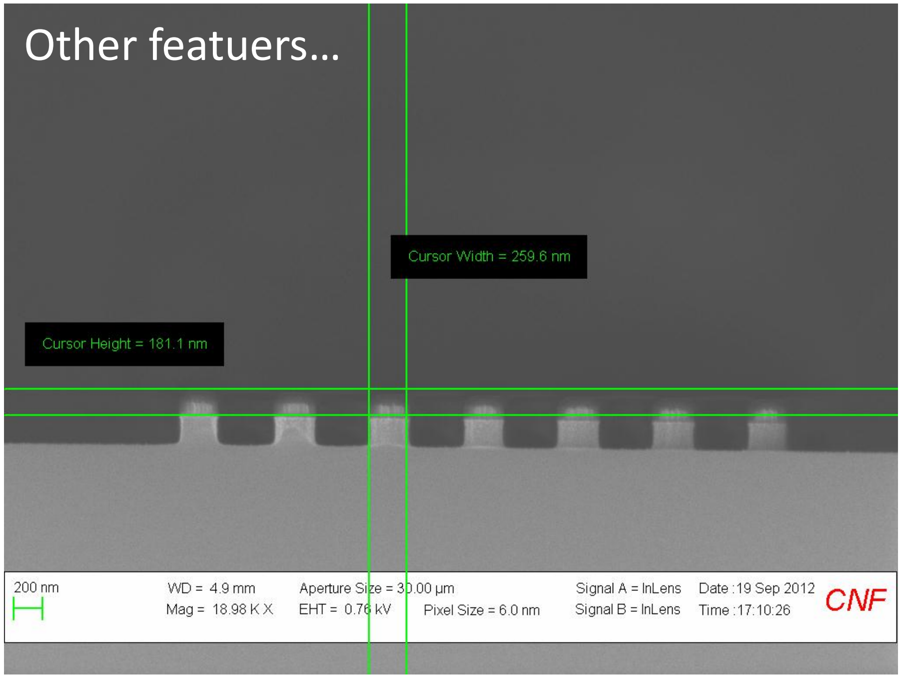


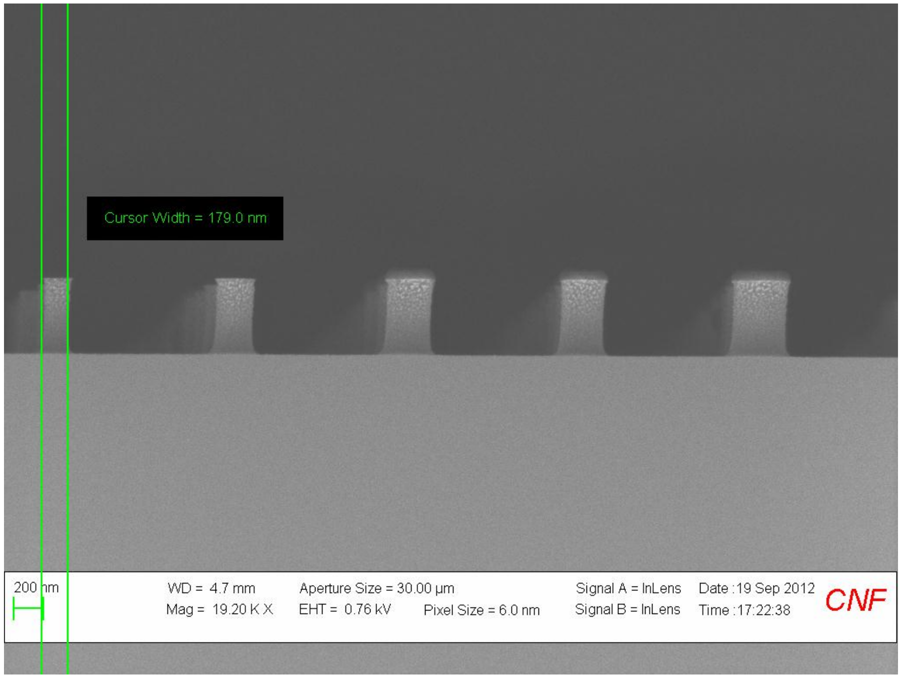


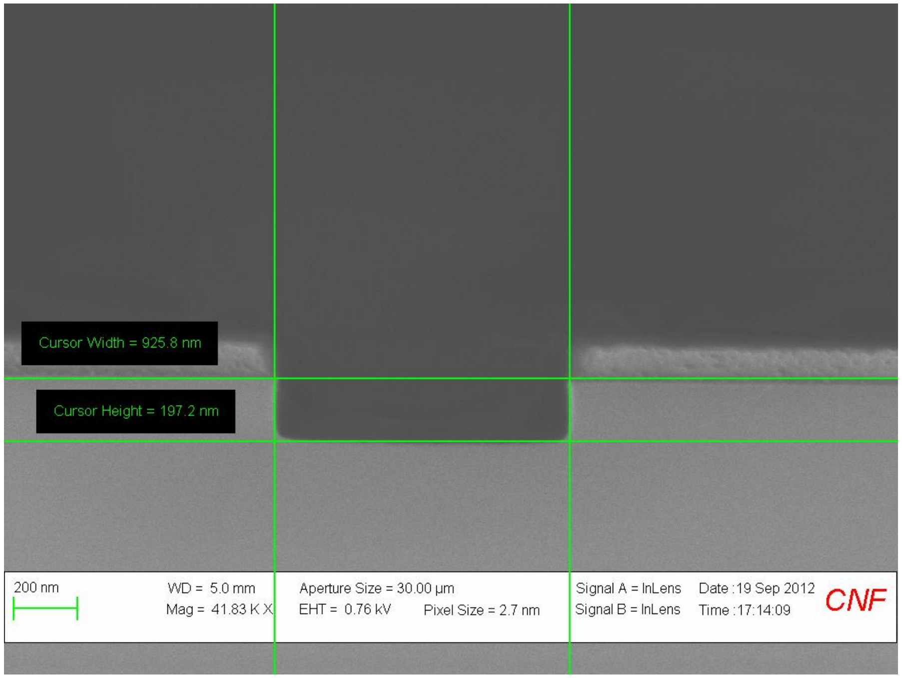
Si etch rate: 166 nm min^{-1}

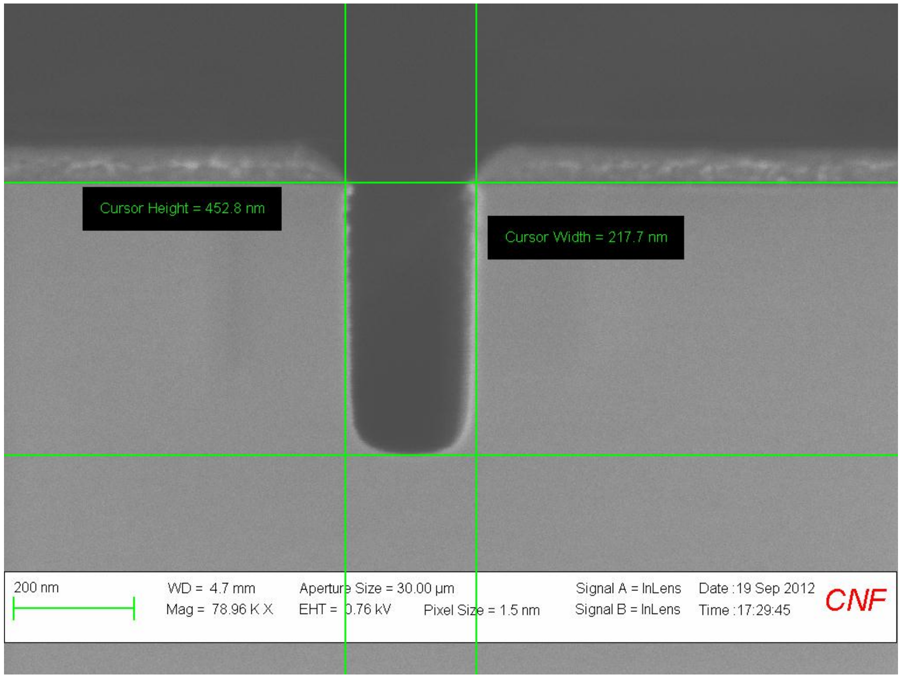
Resist etch rate: 45 nm min^{-1}

Other featuers…

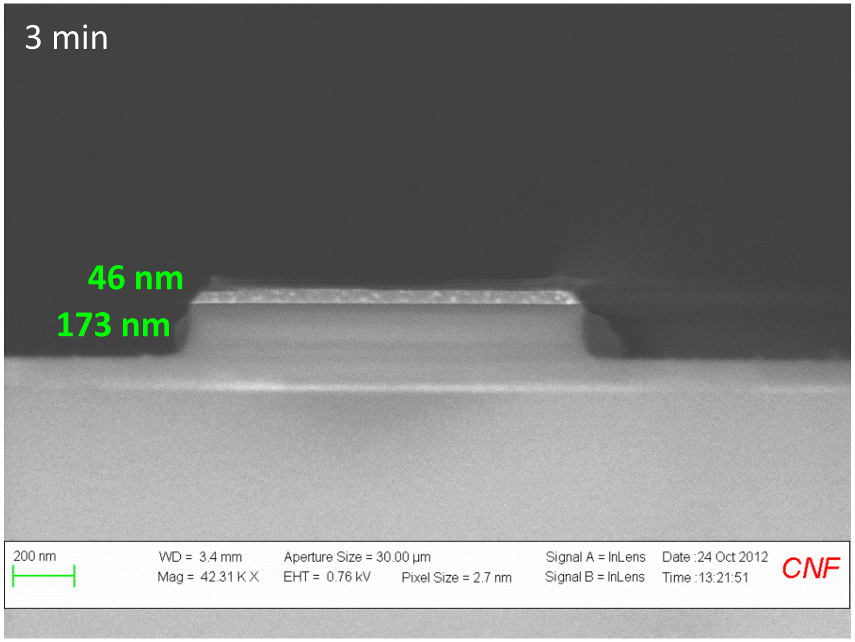


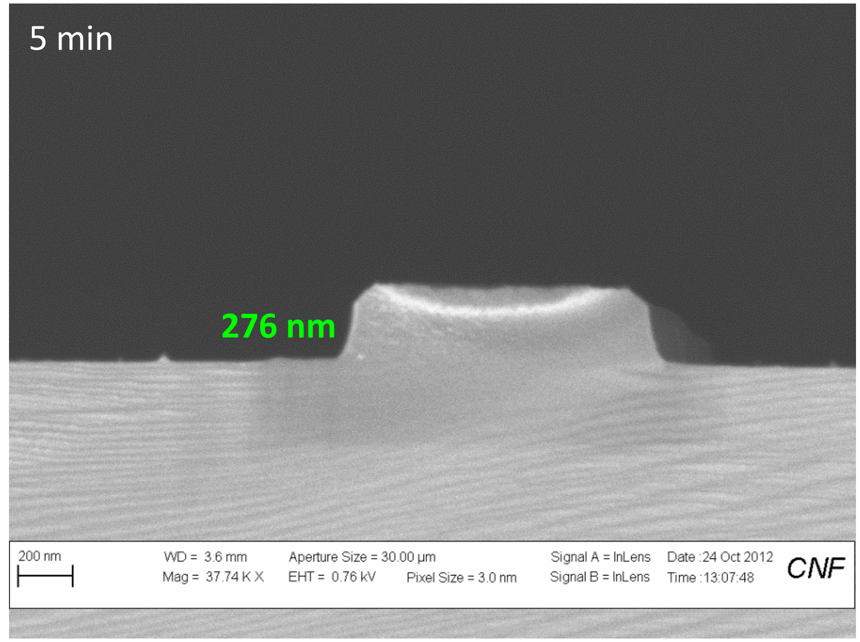


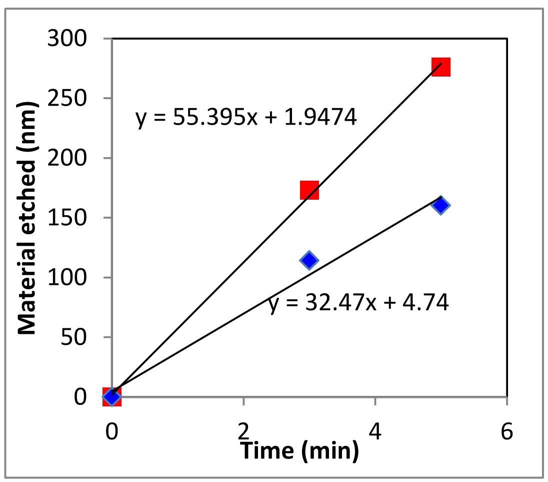




Nitride Ox82 Etches (50/5sccm CHF3/O2, 150W, 55mT)



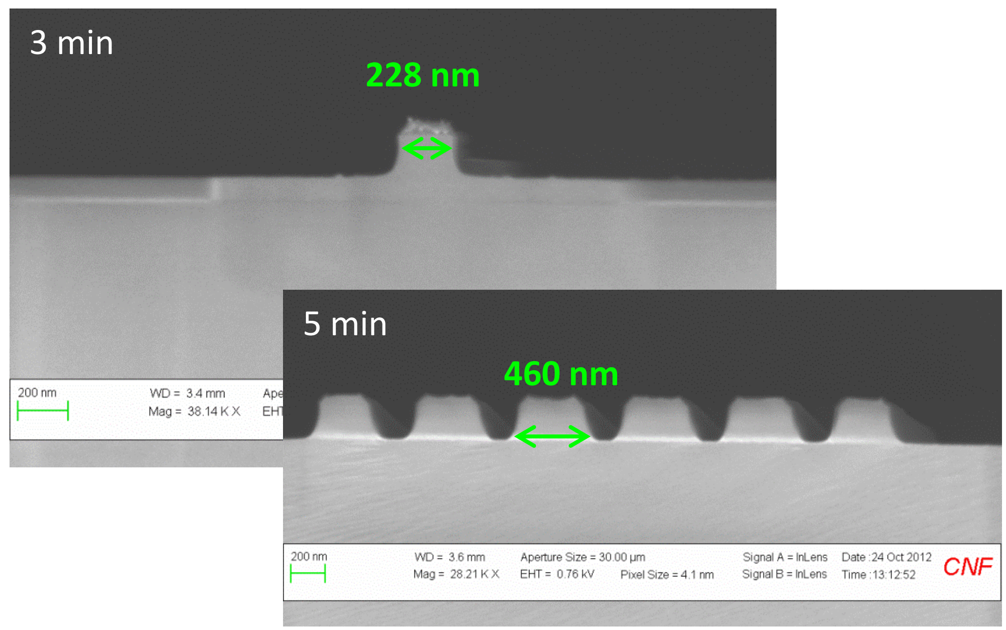




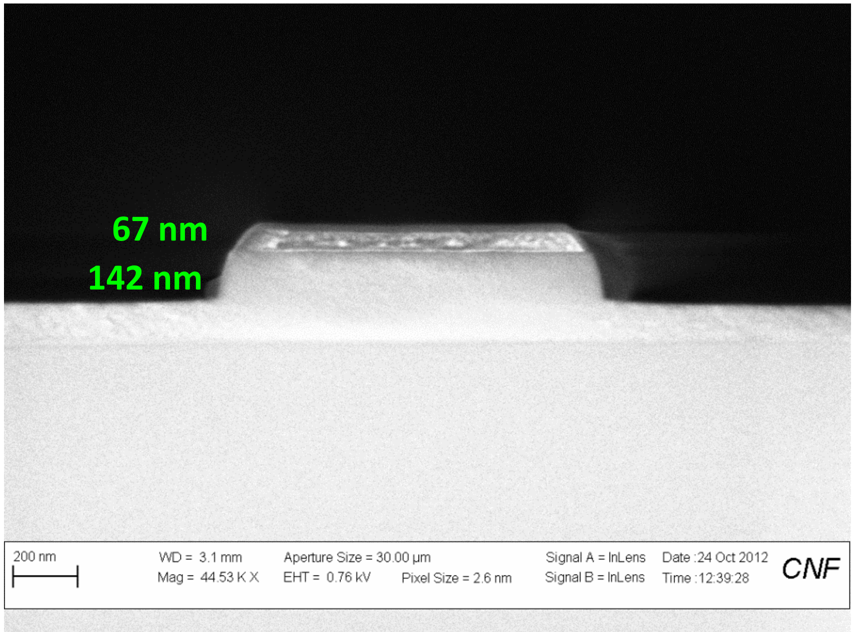
Nitride etch rate: 55nm/min

Resist etch rate: 32nm/min

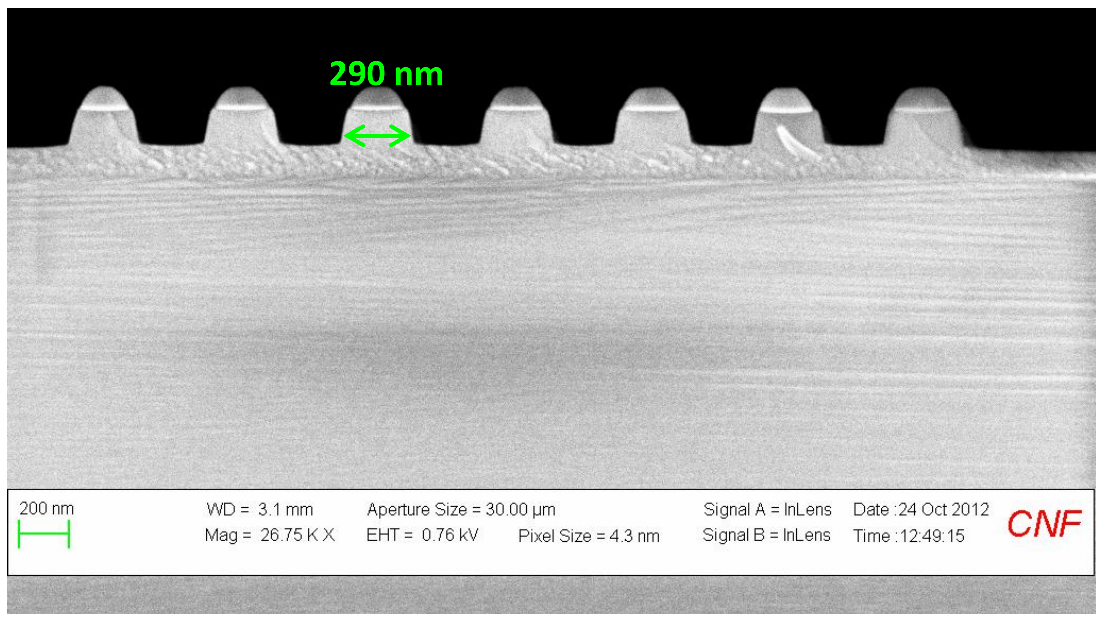
Selectivity: 1:1.7



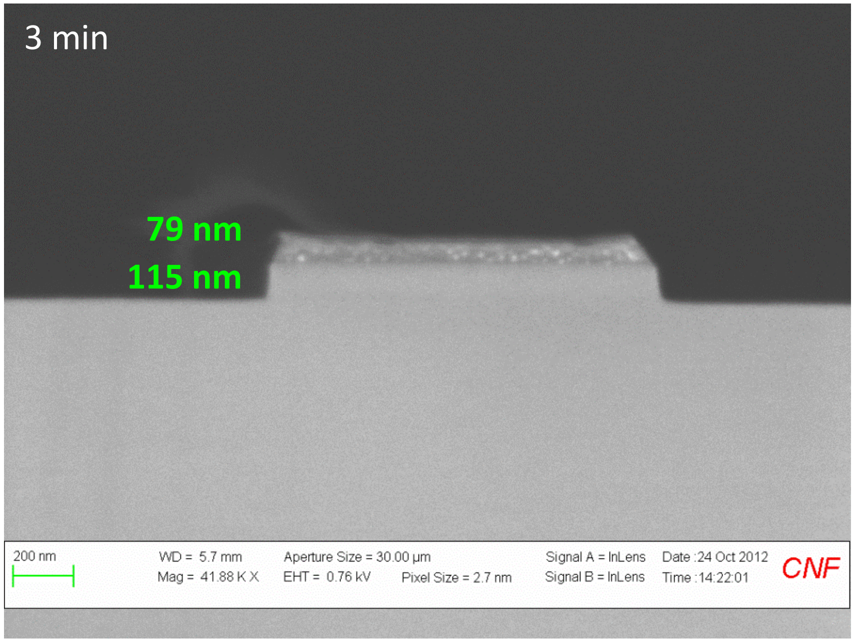
Nitride Ox100 Etches (1 min)

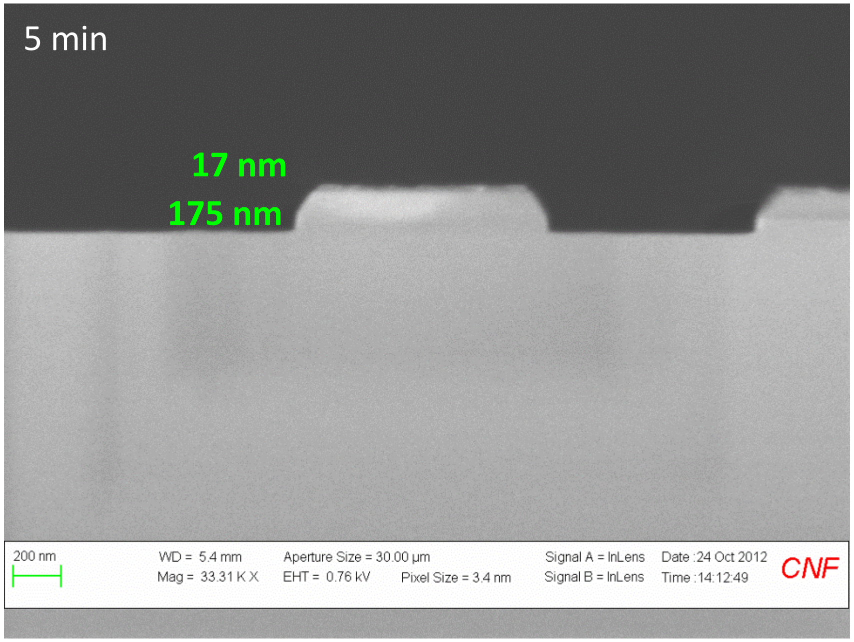


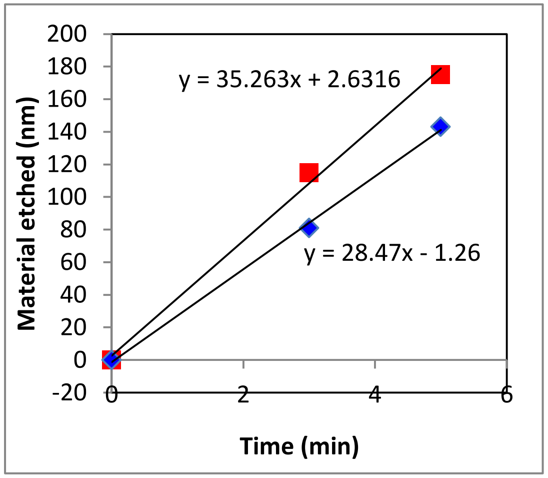
Nitride etch rate: 142nm/min
Resist etch rate: 94nm/min
Selectivity: 1:1.5



Oxide Ox82 Etches (50/2sccm CHF3/O2, 240W, 40mT)

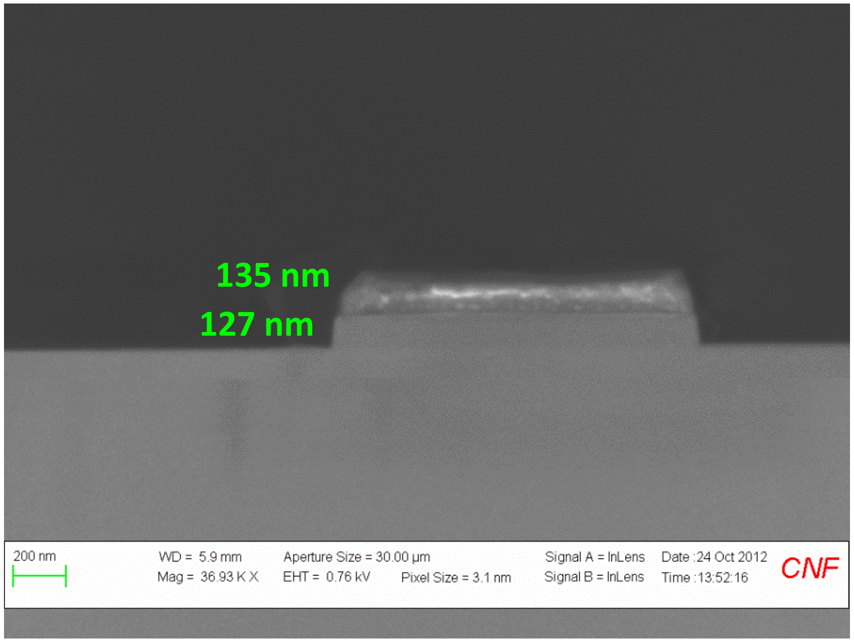




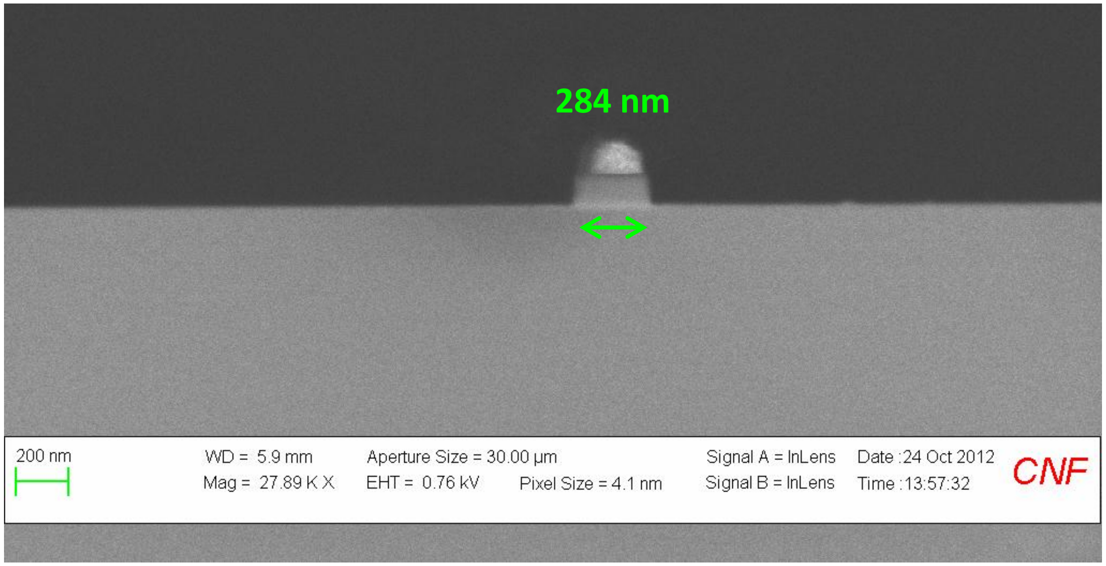


Nitride etch rate: 35nm/min
Resist etch rate: 28nm/min
Selectivity: 1:1.3

Oxide Ox100 Etches (1 min)



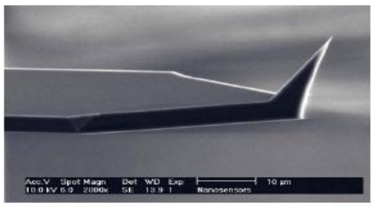
Nitride etch rate: 127nm/min
Resist etch rate: 25nm/min
Selectivity: 1:5



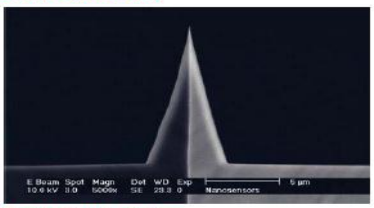
5. Characterization techniques

AFM

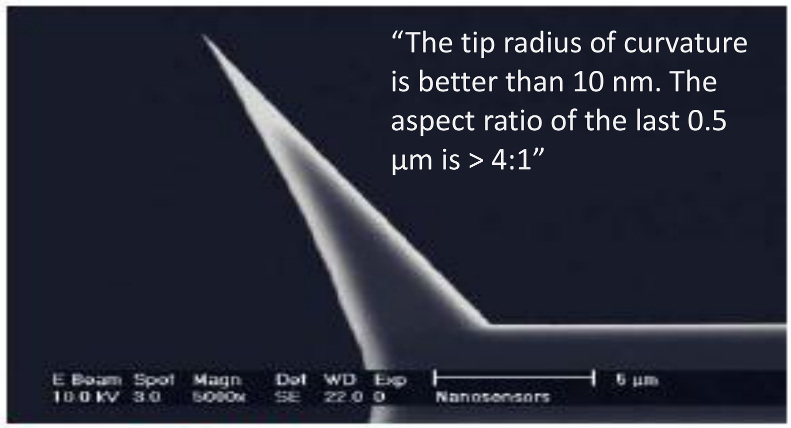
There was initially some question about whether the features seen in the AFM were really representative of the template, or whether the size and angle of the tip was having a detrimental effect. This is particularly important when examining the steepness of the side walls of the template features. The information below is about the non-contact (NC) AFM tips sold from the CNF store.



AdvancedTEC 3D View



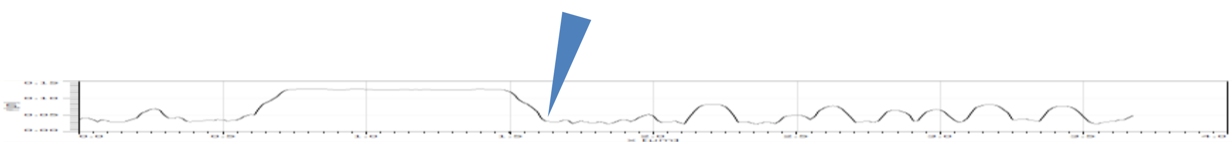
AdvancedTEC Front View

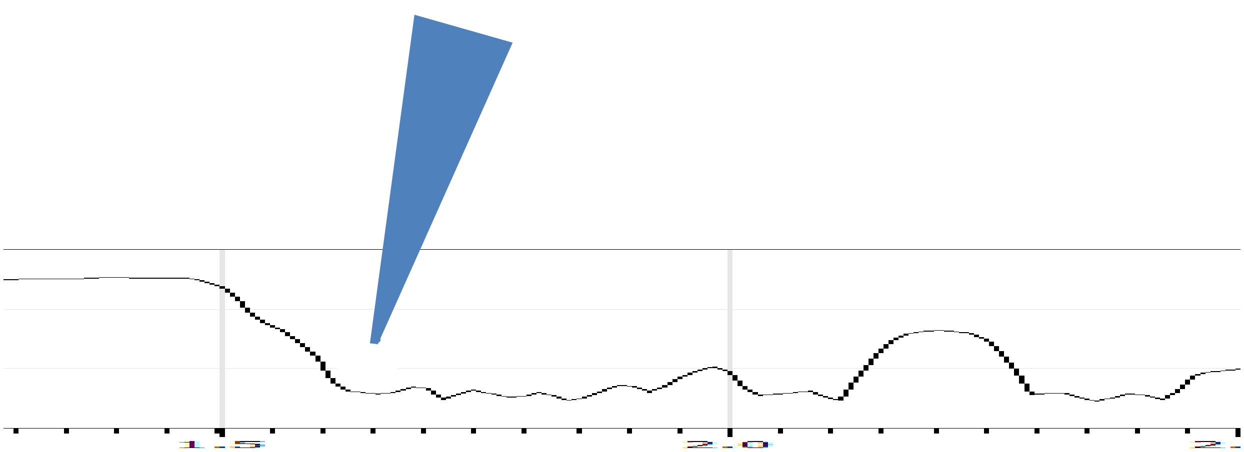


AdvancedTEC Side View

Non Contact	ATEC-NC	Non Contact / Tapping Mode™	45	335

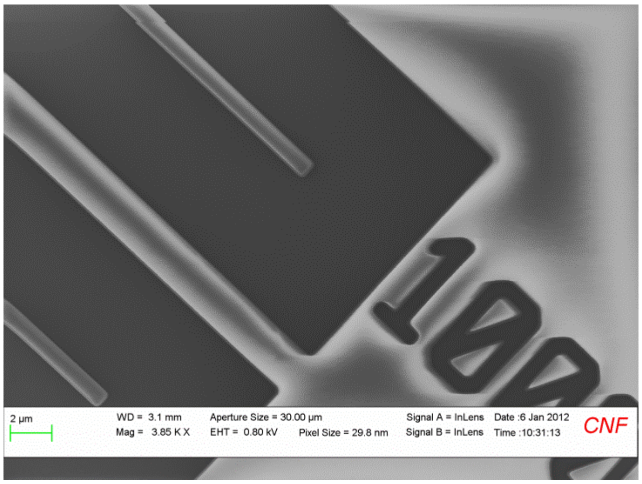
Given this information, below is a diagram showing the AFM tip to scale compared to an AFM trace of a template (assuming the tip is not damaged or broken). It shows that the AFM tip is really very small compared to the template features so it is probably having little effect on the appearance, and that the side wall angles are to be trusted – definitely in the <- scan direction, but maybe not the -> direction.





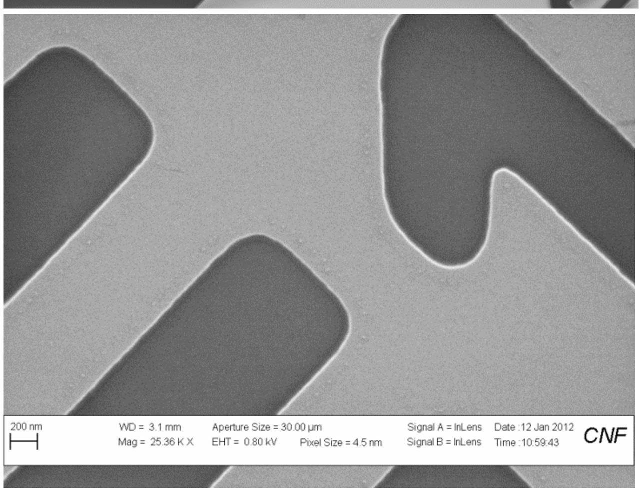
SEM

Imaging the template when the Cr is still on is easiest. It can also be imaged when the resist is still on – but the working distance must be very short and the beam current has to be reduced to about 0.8kV. There may still be significant charging but image quality is sufficient to determine whether features have developed well. It is not possible to image the final template which is all quartz.



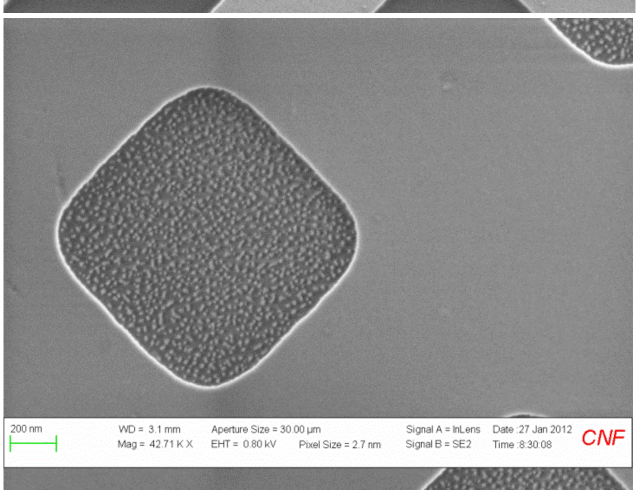
Template after photoresist development.
Before ARC etch.

Light = photoresist, dark = ARC



Template after Cr etch and photoresist strip.
Before quartz etch.

Light = Cr, dark = quartz

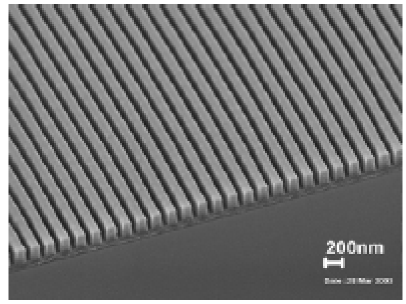


Template after quartz etch.
Before Cr strip.

Light = Cr, dark = quartz

Product Information

NXR-1025 Nanoimprint Resist (Thermoplastic)



NXR-1025 nanoimprint resist is designed not only for sub-10 nm patterning in nanostructure engineering in the near future, but also for today's micro- and nano-patterning. NXR-1025 nanoimprint resist offers ease of handling and processing, good flow characteristic at imprint temperature, and good thermal stability at room temperature. It has been thoroughly tested on our imprint machines.

Film casting:

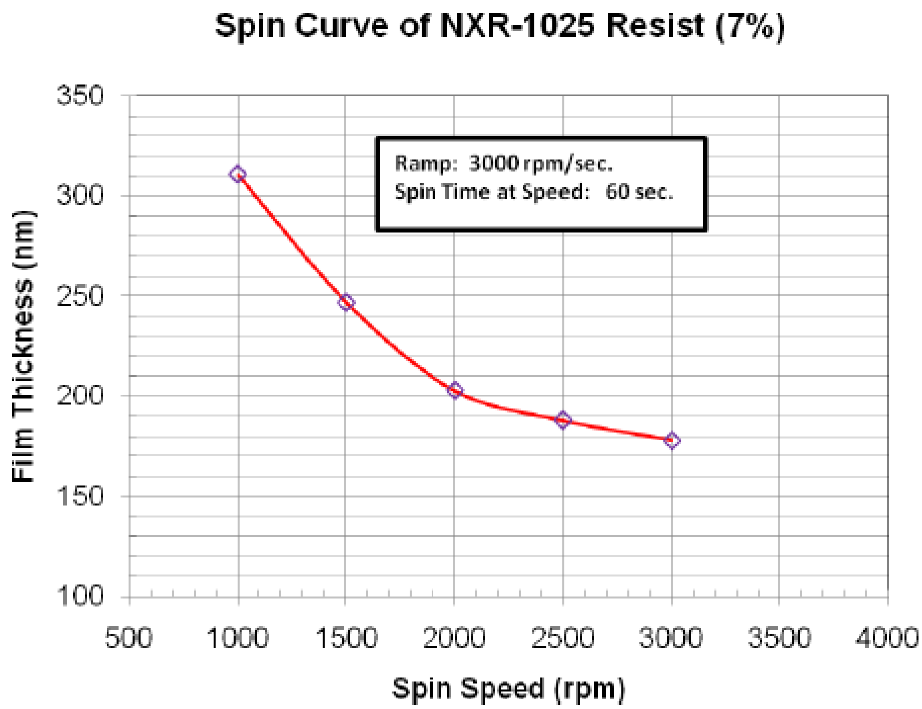
NXR-1025 nanoimprint resist can be spin-coated using a standard spinner. Uniform thin films can be formed on a substrate by spin-coating using a standard spinner. Filtering through a 0.2-µm filter is recommended when applying the resist to wafers. Residual solvent in the resist film can be further driven out by baking on a hotplate at 150°C for 1 minute, or at 80°C for 30 minutes in a vacuum oven. The resist film can be prepared up to microns in thickness, depending on resist concentration and spin-coating conditions. We recommend keeping the resist solution in a refrigerator when it is not in use.

Imprinting:

NXR-1025 nanoimprint resist is typically imprinted at 120°C and 200 psi (or 15 bar) with high resolution and excellent pattern transfer fidelity. It can be etched in oxygen plasma.

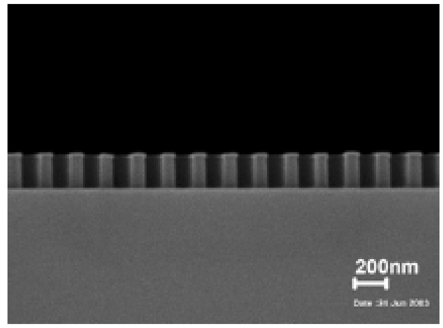
Stripping:

NXR-1025 nanoimprint resist can dissolve in acetone before oxygen plasma etching, and NH_4OH:H_2O_2:H_2O (1:1:5 volume ratio, 70-80°C) after oxygen plasma etching.


Spin Curve of NXR-1025 Resist (7%)
Ramp: 3000 rpm/sec.
Spin Time at Speed: 60 sec.
Film Thickness (nm)
350
300
250
200
150
100
500
1000
1500
2000
2500
3000
3500
4000
Spin Speed (rpm)

Product Information

NXR-3022 Underlayer Resist



NXR-3022 underlayer resist is designed not only for sub-10 nm patterning in nanostructure engineering in the near future, but also for today's micro- and nano-patterning. NXR-3022 underlayer resist offers ease of handling and processing, good thermal stability, and ease for lift-off process. It has been thoroughly tested on our nanoimprinters.

Film casting:

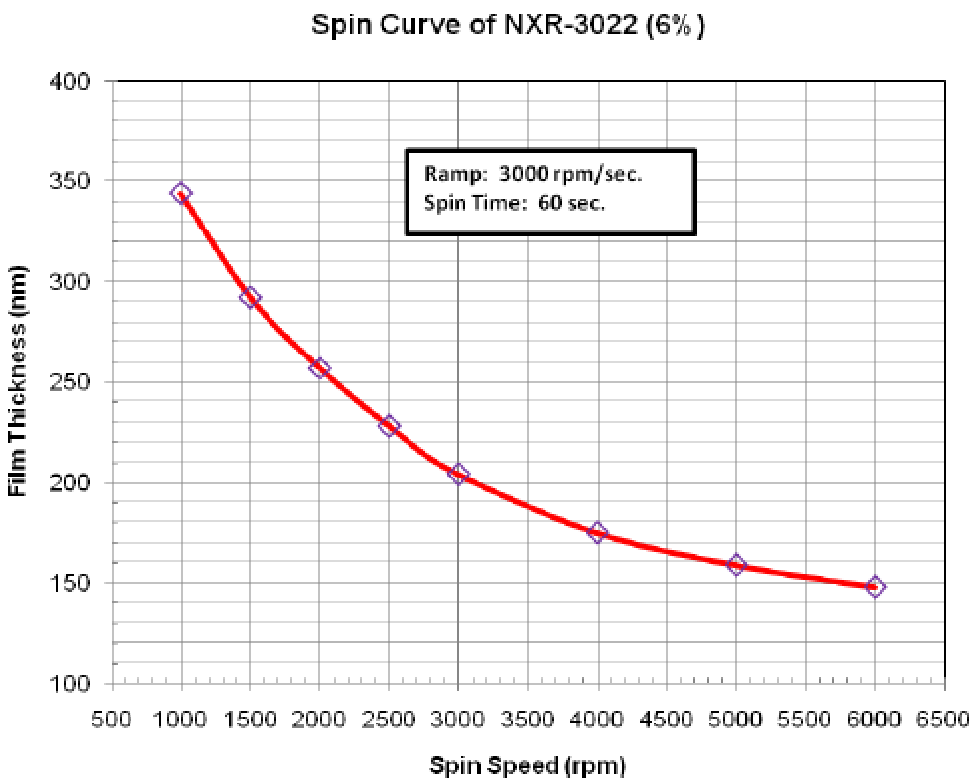
NXR-3022 underlayer resist is a polymer material solution with a safer solvent. Uniform films can be formed on a substrate by spin-coating using a standard spinner. Filtering through a 0.2-µm filter is recommended when applying the resist to wafers. Residual solvent in the resist film can be further driven out by baking on a hotplate at 190°C for 60 to 90 seconds, or in a vacuum oven at 140°C for 30 minutes. The resist films can be prepared of thicknesses from a few tens of nanometers up to microns, depending on resist concentration and spin-coating conditions. The resist solution should be kept in a dark and cool place when not in use.

Imprinting:

NXR-3022 underlayer resist is typically used together with NXR-2030 (or NXR-2010) UV-curing imprint resist. The NXR-3022 underlayer serves as a pattern transfer layer between the imprinted UV-curing resist on top and the substrate material. Imprinted UV-cured resist patterns can be transferred to the NXR-3022 underlayer by oxygen RIE, and the resist template thus created can then be used for metal lift-off or as the resist for further etching process.

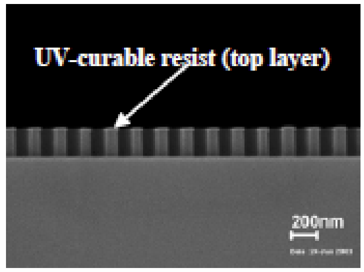
Stripping:

NXR-3022 underlayer resist dissolves easily in either methanol or water. It can also be removed by oxygen plasma.



Product Information

NXR-2030 UV-Curable Nanoimprint Resist



NXR-2030 UV-curable resist is designed for fabricating from sub-10nm nanometer scale to micron scale structures and patterns. NXR-2030 nanoimprint resist offers ease of handling and processing, room-temperature and low pressure imprinting, and high etching selectivity related to NXR-3000 series under-layer resists in oxygen plasma. It has been thoroughly tested on Nanonex imprinters.

Film casting:

NXR-2030 UV-curable resist can be spin-coated using a standard spin coater in an environment with ultraviolet light filtered out. A 35% to 65% relative humidity of the environment is also generally required for coating quality films. A filter (0.2 µm) is recommended to use when applying the resist onto wafers. Spin curve(s) are provided in this document for some standard concentration(s) of this material. As the film thickness may vary from equipment to equipment, and may be affected by the atmospheric conditions, a precise desired film thickness can be achieved by fine adjusting the spin speed. Resist films can be prepared in thicknesses from sub-10nm up to microns, depending on resist concentration and spin-coating conditions. To preserve the resist and protect underlayer materials, no soft bake is recommended after spin coating of this resist. When not in use, the resist solution is recommended to be stored in refrigerator and with light blocked.

Imprinting and post processing:

To be used as a top imaging layer, NXR-2030 UV-curable resist is typically used together with a NXR-3000 series underlayer resist (such as NXR-3032, NXR-3022, and NXR-3010). Images and structures of cured NXR-2030 material are made by room temperature UV imprint process. The resist is cured using 200-410nm UV light in an oxygen-free environment. On 4-inch wafer level, the liquid UV resist can be imprinted at

Nanonex

about 30psi and cross-linked at a dosage of about 40mJ/cm^2. Solid resist structures/patterns with high resolution and excellent fidelity can be made.

After an appropriate residual layer removal by a suitable plasma etching, the patterns of NXR-2030 can be transferred to the NXR-3000 series underlayer material by oxygen plasma.

NXR-2030 resist can be etched by fluorinated plasma. In oxygen plasma, it provides etching selectivity of greater than 10 against NXR-3000 series underlayer materials.

Cleaning and Stripping:

Uncured NXR-2030 UV resist can be rinsed away by acetone or other organic solvent. If on top of a NXR-3032 or NXR-3022 under layer, which are dissolvable materials, whether the UV resist is cured or uncured, the whole layers can be removed away by thoroughly rinse with appropriate solvent that dissolves the under layer. Cured NXR-2030 UV resist can also be removed in fluorinated plasma or hot piranha solution.