The University of Western Ontario

# NanoWestern

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## THIS ISSUE

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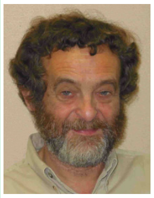
## NEXT ISSUE

- ♦ Microscale Bioactive Patterning
- ♦ Leo 1530 Calibration Improvements

# New Tool: Variable Angle Spectroscopic Ellipsometer

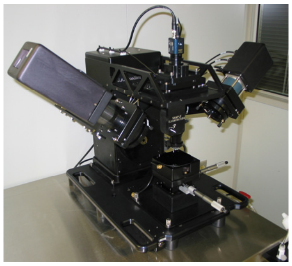


*Rick Glew*
*Laboratory Manager*
*Nanofabrication Laboratory*
*University of Western Ontario*

Anyone using the Nanofab recently may have noticed a new tool occupying the bench where the old Ellipsometer used to sit. In order to replace the old Gaertner L116C Ellipsometer, a M2000V automated angle Variable Angle Spectroscopic Ellipsometer (VASE) was purchased from J. A. Woollam Co. Inc, a leading Ellipsometer tool manufacturer with excellent after sales support that will answer any ellipsometric measurement questions.

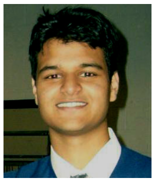
The VASE instrument collects information over 580 wavelengths spanning from 370 nm to 1700 nm. This is a big improvement over the old instrument which only measured at two wavelengths (6328 nm and 13150 nm). In addition, the instrument also has focussing capability; we can now focus down to a 150 micron diameter spot and position the spot on the sample with a camera.

It is simple and straightforward to obtain measurements with the new tool, but unlike the Gaertner instrument, it is more complicated to analyze the data. With a bit of practice the user will be rewarded by obtaining quality data. The software, WVASE32, has six windows for data acquisition and analysis and is a large improvement over the Gaertner analysis software. The data analysis requires the operator to build a model of the material which is then fitted to the data. A major feature which I like is the statistical analysis function which gives a goodness of fit and confidence. It is good to know whether the results are reliable or not.



As with the old ellipsometer, one cannot expect the tool to measure an unknown sample; the material and structure must be roughly known. Contact Rick Glew if you are want to be trained on the new tool.

# Surface and Sidewall Profile Control for DRIE in Silicon



*Sarvesh Varma*
*2nd Year, Nanotechnology Engineering*
*University of Waterloo*

This project was a continuation and extension of my previous co-op term research (supervised by Dr. Todd Simpson) at the Western Nanofabrication Lab. My work focused mainly on enhancing the existing capabilities of the Alcatel (Axiden) 601e Deep Reactive Etching (DRIE) system to suit specific requirements for deep and sub-micron etching of silicon.

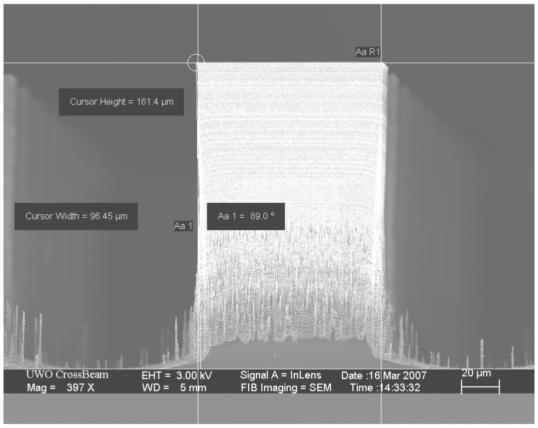


*Figure 1: SEM cross-sectional image of improved threshold for deep etching.*

Deep Reactive Ion Etching (DRIE) is a bulk-micromachining process used to etch highly anisotropic and high aspect ratio holes and trenches into a substrate. This process is essential in the semiconductor industry for fabrication of micro-electro-mechanical-systems (MEMS) devices, sensors, actuators, as well as in the biotechnology field by assisting in the fabrication of micro-fluidic channels. In the cryogenic mode, the substrate is cooled to -90°C and the sidewalls are passivated by oxidation. The pulsed Bosch process alternates between passivating (protecting) and selectively etching the substrate. Initially, the $C_4F_8$ plasma passivates (coats) the with inert polymers. Subsequently, the $SF_6$ plasma etches silicon vertically (due to the substrate bias). Both these processes were tested under different recipes to etch samples fabricated with different techniques.

*(Continued on page 4)*

# FABRICATION OF NOVEL PINHOLES FOR DIGITAL IN-LINE X-RAY HOLOGRAPHY BY FOCUSED ION BEAM MILLING

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In-line holography experiments require pinholes with diameters in the submicron range. The fabrication of such tiny apertures is not trivial. A superior technique for manufacturing structures in these dimensions is Focused Ion Beam (FIB) milling. With the Leo 1540 FIB/SEM CrossBeam instrument, Western's Nanofabrication Laboratory is in possession of an exceedingly powerful tool to machine nanoscale materials like these pinholes. Why we need pinholes at all and how they were produced at the Nanofab will be explained here.

Common imaging techniques give either only two-dimensional images or get the depth information by scanning the focus of the imaging system through the sample. This scanning might be disadvantageous when observing dynamic three-dimensional (3D) processes since the speed of the scanning is limited. A technique with an intrinsic 3D character is holography. Holography can be accomplished with any kind of coherent radiation. Indeed, its "inventor", the Hungarian physicist Dennis Gabor, developed it for electrons while trying to improve electron microscopes in 1947. The adaptation of the classic Gabor setup for in-line holography *[i]* to photons instead of electrons includes a pinhole which provides a diverging photon beam and a detector, e.g. a CCD-camera, to record the so called hologram. This scattering pattern results from the interference of the unscattered coherent radiation ("primary wavefront" or "reference wave") with the part of the wave which is scattered off the object present between pinhole and detector ("secondary wavefront", see figure 1). Due to the divergence of the photon beam, the interference pattern is magnified and therefore in-line holography acts as a coherent microscope. Since in the hologram both the amplitude as well as the phase of the object wave are recorded all 3D information is preserved and thus three dimensional real space information can be retrieved either optically or digitally ("reconstruction") *[ii,iii]*. As the resolution is determined by the wavelength of the photons and the numerical aperture of the detection system, it is straightforward to increase the photon energy in order to enhance the performance of the technique. Two years ago we used vacuum-ultraviolet (VUV) synchrotron radiation with a wavelength of 14 nm to image dried rat embryonic fibroblast (REF) cells (see figure 2) and so implemented a digital Gabor microscope in that wavelength range *[iv]*.

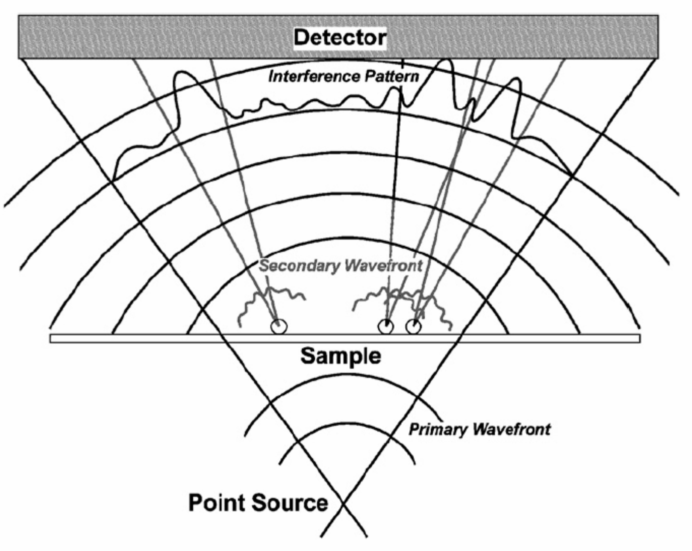

*Figure 1: Schematic geometry of a digital in-line holography experiment. The hologram is formed by interference of the undisturbed primary wave front and the scattered, secondary wave front.*

The theoretical lateral and depth resolution depend on the wavelength and the numerical aperture only. The latter is given by the sine of the half angle under which the CCD chip is seen from the pinhole. But not always can the full chip size be used. If the diameter of the pinhole is larger than the wavelength of the light, it produces not a perfect spherical wavefront but an intensity distribution in the far field (as depicted in figure 3) which is known as Airy pattern. The size of the central Airy maximum, which is in turn inversely dependant on the diameter of the pinhole, limits the effective numerical aperture. In order to access shorter wavelengths it is therefore desirable to have small apertures with sizes of the order of the wavelength.

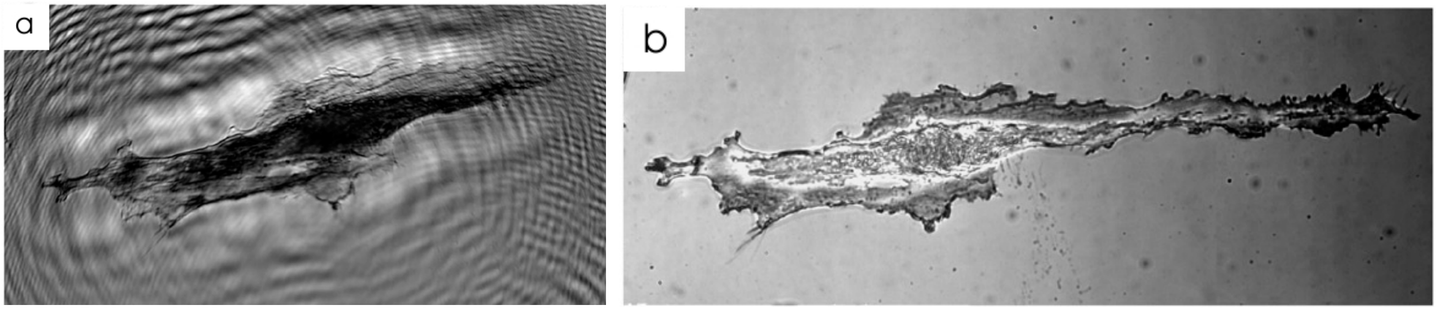

*Figure 2: Dried rat embryonic fibroblast cell. (a) Numerical reconstruction of a hologram recorded at $l = 13.8$ nm, NA = 0.014, NAeff = 0.012, image size 210 x 100 $\mu m^2$, (b) optical microscopy picture, Zeiss Axioplan 2, Zeiss 40x Neofluar objective, NA = 0.75, image size 290 x 100 $\mu m^2$.*

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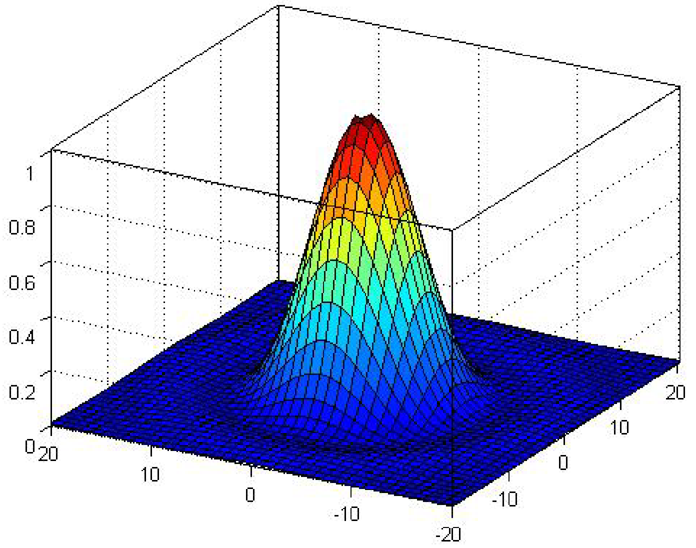

*Figure 3: Intensity distribution in the far field behind a pinhole. The normalized intensity in dependence on the position on the detector is plotted.*

In collaboration with the Nanofab, these pinholes are produced in a hybrid design: Larger guard pinholes are used to trim the direct synchrotron beam, and a gold foil is glued onto this backing. Into this gold foil the final pinhole with the desired size of 250 to 400 nm is milled by Focused Ion Beam. Since the gold foil has to be of a certain thickness – in our case 2 µm – in order to block the remaining direct synchrotron beam the pinholes have an aspect ratio of about 1:5 to 1:10. The challenge lies in the fact that the walls should not be tapered but parallel. To get such holes, first the area dose that has to be applied to punch through the material has to be found. For this purpose, an array with varying beam parameters is milled (figure 4, left). Then the sample is flipped to see which of the holes have punched through (figure 4, right), and to derive the corresponding pinhole diameter and milling dose. Afterwards, the line dose and milling radii to ream the aperture to the chosen diameter have to be determined. Instead of turning the test sample over, one can always cut open the membrane by milling a rectangle of appropriate size to get the cross section of the milled structure. With the technique described above, pinholes with a diameter of the exit hole ranging from 400 nm down to 250 nm have been fabricated (compare figure 5).

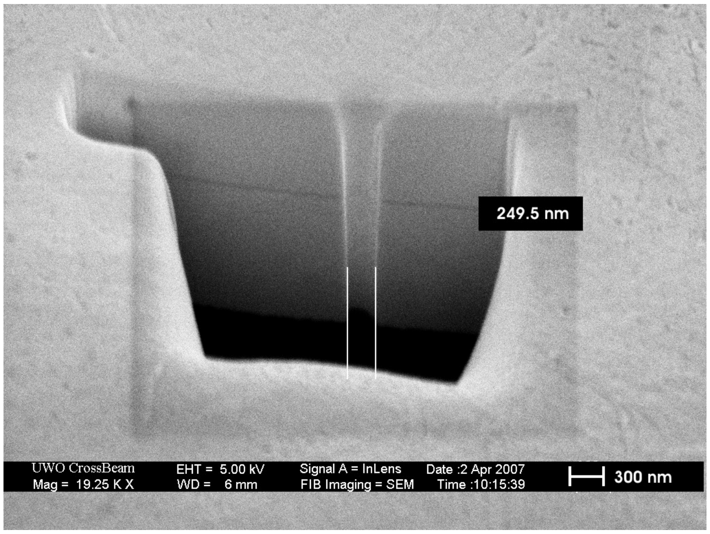

*Figure 5: SEM image of a cross section of a pinhole with 250 nm diameter. The magnification is indicated by the scale marker on the image.*

Those pinholes were used in our experiments during the beamtime at the synchrotron source BESSY in Berlin, Germany, in June. As expected, the 2 µm gold membrane attenuated the direct synchrotron beam completely, so that a beamblock or numerical suppression of the bright spot are no longer necessary, and due to the smaller pinhole diameter a larger energy range was accessible compared to the previous beamtimes. Although the results are not yet fully analyzed, they look promising with respect to enhanced resolution, and it is already certain that the new pinholes were a major improvement for our setup.

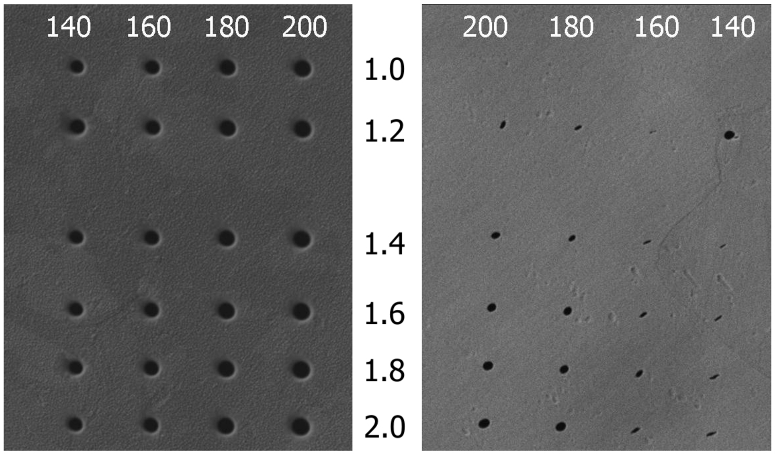

*Figure 4: SEM images of the array for finding the punch through dose, UWO Cross Beam, magnification 5.86 K. The pinhole diameter is varied from left to right (white numbers, size in nm), the exposure scale increases from top to bottom (black numbers). Left: Entrance side of the gold foil. Right: Corresponding exit holes.*

My personal thanks go to Prof. Dr. Silvia Mittler, who invited me to come to London and supported me in various ways, to Dr. Todd Simpson for his instruction and advice, and the rest of the Nanofab team for the excellent working conditions. This work was supported financially by the Landesstiftung Baden-Württemberg and The Ontario Photonics Consortium.

## Acknowledgements

[i] D. Gabor, Nature 1948, 161, 777-778
[ii] W. Xu, M.H. Jericho, I.A. Meinertzhagen, and H.J. Kreuzer, PNAS 2001, 98 (20), 11301
[iii] W. Xu, M.H. Jericho, I.A. Meinertzhagen, H.J. Kreuzer, Applied Optics 2002, 41, 5367
[iv] A. Rosenhahn, R. Barth, X. Cao, M. Schürmann, M. Grunze, S. Eisebitt, Ultramicroscopy 2007, doi:10.1016/j.ultramic.2007.01.010

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The objective was to determine optimal conditions for best surface and sidewall profile control. One of the main targets of the project was to extend the current limitations of the DRIE machine for both deep and sub-micron etching. Sensitivities of parameters such as etch rates and mask selectivity towards changing etching parameters such as the substrate power, gas flow rate and substrate temperature were examined in order to reach ideal conditions. In this process, the goal was understand which conditions minimize surface roughness and sidewall angles. This information was needed not only to gain a better understanding on the performance of the DRIE system, but also help save costs and efforts involved with etching a sample.

It was concluded that the etch conditions are dependant on the desired depth etch and its applications. Additionally, it was discovered that depending on the focus of the study, different conditions can be used to optimize sidewall angles, surface roughness, mask selectivity as well as the etch rates.

Deep etching required using an optimized aggressive high powered Bosch etch process with higher substrate power to extend the existing threshold from 80 to 160 microns in terms of the etch depth. The surface and sidewall profiles illustrated an unexpected dependence on feature geometries along that on the etching conditions. The high power settings of the cryogenic process were unsuitable for deep etches comparable to that by the Bosch process as it was more susceptible to surface roughness.

To minimize surface roughness due to micro-masking from non-volatile residue, an oxide mask was found to be much better than a photoresist mask. When patterning a one or two micron masked oxide, it was preferred to use a wet HF Buffered Oxide Etch (BOE) process over the Reactive Ion Etching Process (RIE), considering total process time and the non-vertical sidewall profiles. The average etch rate of the BOE process is 96.2 nm/min. If one has to use the RIE process for other considerations, $CHF_3$ chemistry proved better than the $CF_4$ chemistry due to superior selectivity to the photoresist (3.4:1).

Optimizing source power and temperature under the optimized high powered cryogenic etch conditions led to the optimal conditions for sub-micron etching with etch rates close to 100 nm/min. In this process, the selectivity to the photoresist was 5:1, e-beam resist ZEP520A: 10:1 and that to the oxide was about 100:1. Under the Bosch process, selectivity to the photoresist was 20:1, e-beam resist PMMA-A4: 7:1 and that of the oxide was about 500:1. The low powered shallow cryogenic process had a minor footing at the etched surface due to crystallographic preference of the etching ions. Cryogenic sub-micron etching used the optimized conditions from the deep cryogenic etching and worked significantly better than the low powered Bosch process, especially due to the controlled slow etch rate.

For best results, it is advised to use the optimized cryogenic process for shallow and sub-micron etching and the Bosch process for deeper (larger than 100 microns) etching for best control over surface and sidewall profiles. Specific detailed recipes can be obtained from the Nanofab.

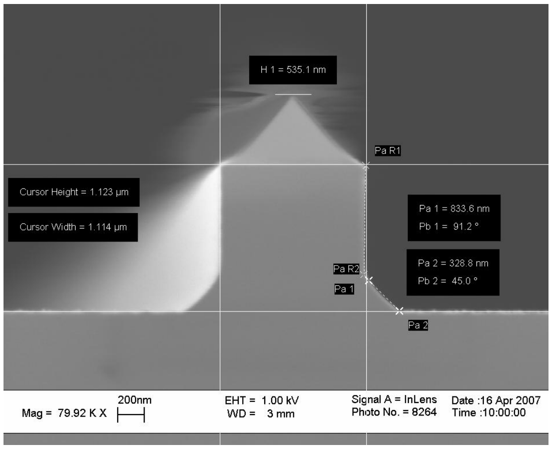


*Figure 2: SEM cross-sectional image of optimized sub-micron cryogenic etch.*

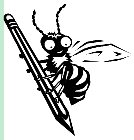
# 3RD ANNUAL OPC SHOWCASE

The 2007 OPC Showcase took place on May 24th, 2007 at the University of Waterloo. Building on the two previous events at McMaster and University of Western Ontario, the Showcase grew again in both attendance and poster count. A record 94 posters were presented in the areas of photonic devices, optical communications, quantum optics, photonic materials, nanophotonics, biophotonics, lasers and non-linear optics, and sensors and detection.

OPC Director Peter Mascher commented, "It is wonderful to see the explosive growth in the number of graduate students participating in this all-Ontario photonics event. It was very difficult to choose the top presentations. The wide topical range of winning posters, from biophotonics to nanophotonics to quantum communication is an indication of the breadth of excellence in the field of photonics within the Ontario Photonics Consortium."

Ruth Rayman
Business Development
Ontario Photonics Consortium

# CALLING ALL AUTHORS....



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