

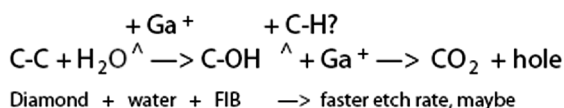
Surface Rippling & Ion Etch Yields of Diamond Using a Focused Ion Beam: With or Without Enhanced-Chemistry, Aspect Ratio Regulates Ion Etching

W. J. MoberlyChan, T. E. Felter, & M. A. Wall,
Lawrence Livermore National Lab., Livermore, CA
moberlychan2@llnl.gov

I) Motivation & Background: Micromachining & Ripples

The Focused Ion Beam (FIB) instrument, originally designed for semiconductor circuit modification and repair, has found considerable utility as a tool for specimen preparation in several microscopy disciplines and for micromachining small parts. Essentially, a FIB makes very small and precise cuts into a target sample, which implies well-controlled etch rates and close tolerances of surface finish. However, redeposition can affect etch rate and final surface topographies. This work quantifies this redeposition as it modifies yields for different parameters of etching; models the influence of redeposition as applicable to all ion beam processing; and optimizes FIB processing parameters for enhanced yields.

The FIB, especially in conjunction with an SEM for live metrology (*i.e.* a DualBeam FIB/SEM), is effective for producing a wide range of small objects. Although the FIB may be slow and costly for micromachining millions of parts, it is ideal for prototyping and for making tools and templates that in turn can make millions of parts. As an example, the FIB can nanomachine a diamond tool-bit, which can then micromachine (microlathe) many small parts. However, FIBing even a single diamond tool-bit can be slow, and the enhancement of the ion etch rate of diamond is desirable [1, 2]. Etch rate can be increased by angle-of-incidence and/or chemistry. The ion yields of all materials are increased when the incident angle of sputter etching is increased from normal (traditionally defined as 0°) to grazing. Chemical gases can be judiciously and site-specifically introduced to enhance (and/or selectively enhance) FIB ion etching. The combination of angle and chemistry have provided >10 times enhancement of FIB etch rates for diamond [2, Fig. 1]. In the case of diamond, an effective chemical addition is simply water. A non-finalized chemical equation allows the hardest material on earth to lose its sp^3 bonding, albeit only for the top carbon atomic layer, leaving a more readily eroded surface for FIBing:



(The details and sequence of the reaction mechanism of carbon plus water plus Ga^+ remains unclear.) The modern FIB provides versatile control of sample angle, as well as numerous options for gas chemistries (using GIS-Gas Injection Systems). In the present work, FIB processing parameters are investigated to optimize etching.

Preliminary reproduction of the research of [2], finds that a variety of etch rates might be achieved by FIBing at 70° angle of incidence. The colored data-spread overlaid in Figure 1 indicates our FIB yields may be twice (or only half) what others have accomplished [2]. This wide spread of data at a single inclination is disconcerting from a metrological view of process replication. Figure 2 plots how yield drops as aspect ratio increases for all cases of processing variables. The data of Figure 2 shows that a 1nm diameter hole can not be drilled indefinitely; that all etch rates will decay and eventually go to 0; and that this decay with increasing aspect ratio is worsened by angle and worsened by water vapor or another chemical addition. This decay in yield starts impacting FIB work at surprisingly small aspect ratios.

Ion angle of incidence not only influences yield but also influences the resulting surface morphology. Generally the FIB is requested to

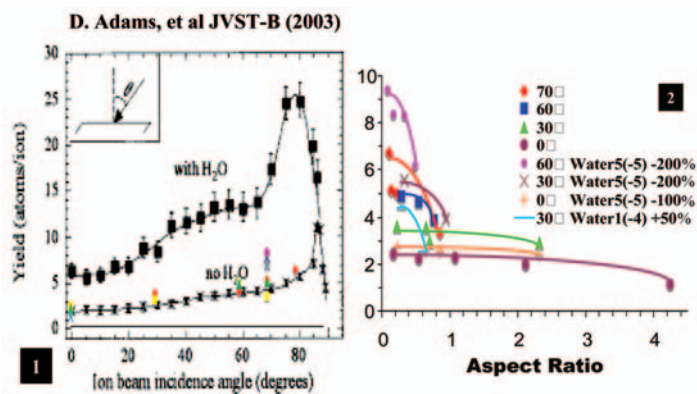


Figure 1. Plot of yield enhancement with angle of incidence and with water for chemical enhanced etching, by permission from D. Adams [2]. The present data (overlaid color for the no-water condition) indicates yield can be more, or less.

Figure 2. Although yield (etch rate) varies with angle and chemistry, yield always decays with increased aspect ratio (depth/length) and more so with angle and chemistry. (As yield drops, the redeposition is increasing.)

process surfaces as smoothly as possible, although there are potential applications where roughened surfaces are desirable. For example, ion beam “rippling” [4] and/or quantum dot formation are being considered as self-assembly templates [5]. Rippling is often considered in the presence of a broad, static ion beam, and for its potential application to a full wafer substrate. However, devices requiring site-specific ripples will require FIB processing. If broad beam ripples are processed in conjunction with photolithography patterns, the ripples within these patterned pits will have similar boundary conditions as FIB-processed ripples. The FIB also provides a versatile platform for changing experimental variables. A FIB can produce several different sets of ripples in seconds, where a static beam may take days; and the SEM in the DualBeam provides instantaneous diagnostics of the process. Thus studies of processing parameters that provide faster etch rates, also identify the parameters that influence the ripple formation.

Figure 3a presents an SEM image of a broken diamond, which has been exposed to modest ion sputter etching. (Fig. 3c depicts a sharpened tip after hours of FIB processing.) A close-up view (Fig. 3b) shows “ripples” develop simultaneously on all of these inclined slopes. At a lower magnification these ripples resemble terraced slopes on hillsides such as Machu Picchu, or erosion steps on the great pyramids, or even cow-grazing paths (Fig. 3d). Such macroscopic terracing occurs due to “erosion” and/or “redeposition”. Erosion, redeposition, and the formation of ripples are also observed in fusion reactors studies [3].

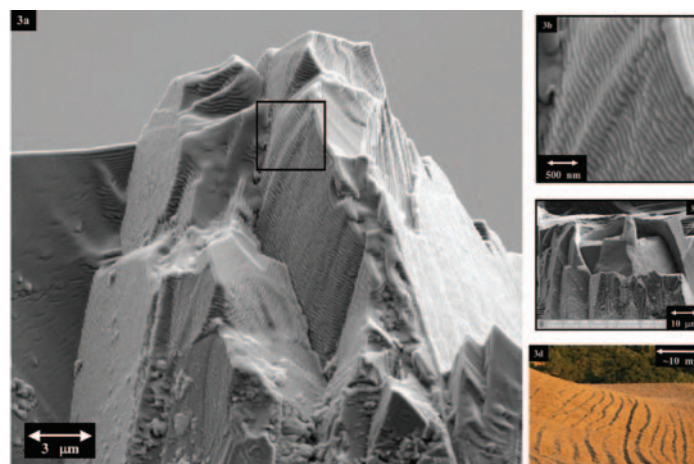
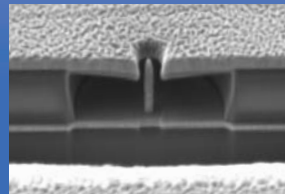


Figure 3. A single crystal diamond is FIB-micromachined to make a sharp tip (Fig. 3c). However, the many facets and inclinations make an ideal platform for testing ripple formation (Fig. 3b) from different angles and crystallography. Ripples appear similar to erosion and redeposition patterns in nature, such as cow paths on a steep hillside (Fig. 3d).

ENVISION A NEW WAY OF COMBINED COMPETENCIES

ZEISS NVision 40

The new 3D CrossBeam® Workstation
that combines FIB and GEMINI® SEM columns
to a unique system enabling a variety of
outstanding nanotech capabilities



Enabling the Nano-Age World®

NVision 40 – First-Class Efficiency and Convenience – Envision the Value of Precision



Carl Zeiss SMT Inc.
Tel. +1914/747 7700 • Fax +1914/681 7443
info-usa@smt.zeiss.com • www.smt.zeiss.com/nts

SII NanoTechnology Inc.
www.siint.com
Tel. +81 / 3 62 80 00 66



Earlier studies of FIBing diamond have observed ripples [2] and have correlated angle of inclination and water vapor to the resulting topographies [2, Fig. 4]. The present work determines that the etch rate decreases with aspect ratio (Fig. 2); and this decay becomes more dramatic when the angle of inclination and the pressure of water vapor increase. Thus the two parameters (angle, water) used to increase the etch rate also cause the etch rate to decay faster with aspect ratio. In turn, the three factors (angle, water, and aspect ratio) also alter the formation of the ripples. Since the purpose of FIBing is often to create local geometrical shapes, the interdependence of chemistry and aspect ratio need to be measured and considered both for etch rates and ripple formation.

II) FIB Ripples on Diamonds

Ripples can be produced either by a broad, static ion beam or by a Focused Ion Beam tool that scans a beam over an area [4, 5]. However, when the ion beam impinges the surface between normal incidence and ~40° (Fig. 5a and 5b), the resulting etched surface is quite smooth [2, 4]. In fact, with a scanning FIB, the surface can even be made smoother than it originally was if original surface imperfections are not too large. When the angle of incidence increases beyond ~40°, ripples are achieved (Fig. 5c); and at the highest angles the ripples are broken into “steps” (Fig. 5d), by the intersection of compound ripples perpendicular to each other [4-6]. The surface morphology of these three regimes is

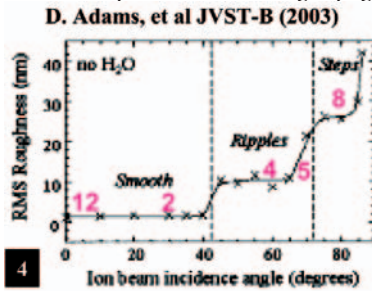


Figure 4. Angle of inclination provides 3 regimes of topography for ion bombardment of diamond (by permission from D. Adams [2]). Low angle (normal=0°) makes surfaces smoother; midrange produces a single wavelength of ripples; and high (grazing) angles produce steps or terraces in 2D.

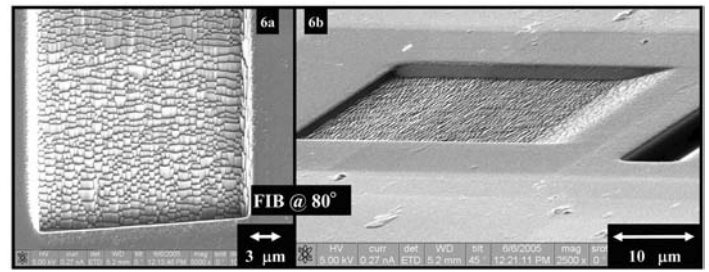


Figure 6. SEM provides in situ metrology of ion etching and development of ripple wavelengths. SEM measure of depths can be misleading (see Fig. 15); however controlled rotation and tilt establishes ripple amplitudes can be >200nm, a reliable measure of the amplitudes. However, 90° rotations coupled with 45° tilt (Fig. 6b) indicate the amplitude of the ripples are >200nm after ion etching to a depth >2microns. These depths represent orders of magnitude greater fluence than are usually applied to observe ripples [6]. The fluence utilized in the present work ranged from 10(exp18) to 10(exp20) ions/cm2.

Figure 7 exhibits wavelength increasing from 280nm to 310nm as fluence increases from 1.3 to 2.1 x 10(exp19) ions/cm². The averaged ripples plotted in Figures 7b and 7d also provide metrology that larger ripples (420nm) exist at the boundary (trailing edge) of the FIB crater. Most reports of ion-induced ripples (both theory and experiment) indicate a saturation wavelength of the ripples for specific processing angle and ion energy. However, experiment often exhibits a rippling wavelength 10 times more than theory [6], with wavelengths >100nm commonly observed. Saturation wavelengths of 100-250nm have been reported on diamond [2, 7], depending on angle and added water vapor. Theories for rippling involve surface diffusion [4-6, 8-9], as well as Sigmund sputtering theory [10]; however, most reports discuss limits to the theory that must avoid nonlinear effects and boundary conditions [4]. All reports indicate the amplitudes of ripples are well below their wavelength. As the amplitude grows, nonlinearity develops that causes the saturation wavelength to increase. Redeposition is not treated in most models, although mentioned as a concern [4]. However, redeposition appears to enable the present ripples to grow larger than other reported saturation values.

III) Effects of Water Vapor on FIB Etching of Diamond

Modern FIBs enable versatile addition of chemistry via Gas Injection Systems (GIS). The chemical vapor pressure at the sample may reach 10(exp-2) Torr, while the majority of the vacuum chamber is lower than 10(exp-5) Torr, and the FEG-SEM retains 10(exp-10) Torr. Water vapor can be added to enhance sputter yield of diamond, however, the addition of chemistry has a strong interdependence with many of the FIB process controls. Chemically enhanced FIB etching may result in smoother

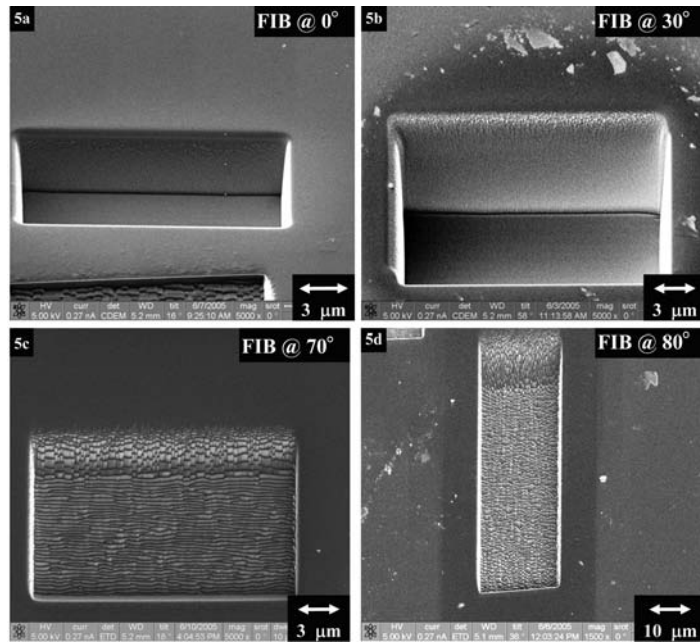


Figure 5. SEM images, acquired at tilt, of FIB etch pits produced with ion incident angles of 0° (normal), 30°, 70°, and 80°; producing flat, rippled and stepped surfaces. Particles on the surface alter local angles for small ion dose conditions but can be ignored for high dose.

neatly documented in Fig. 4 (from ref [2]). The present work observes the same regimes of smooth, rippled, and stepped surfaces. However, the RMS reported in Fig. 4 is nearly an order of magnitude less than the metrological observation in the present work. Although SEM imaging normal to the surface (Fig. 6a) measures wavelength, it does not provide

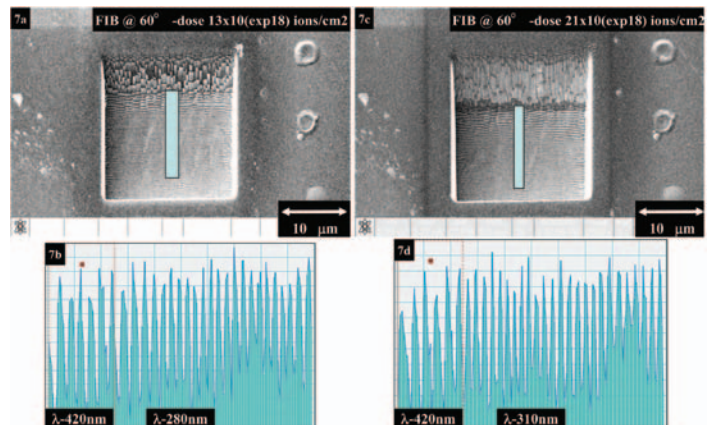


Figure 7. SEM data is averaged to measure ripple wavelengths that grow larger than reported saturation values [2, 6, 7, 9]. FIB etch pits invoke boundary conditions [5], where the first 6 ripples have a larger (420nm) wavelength.

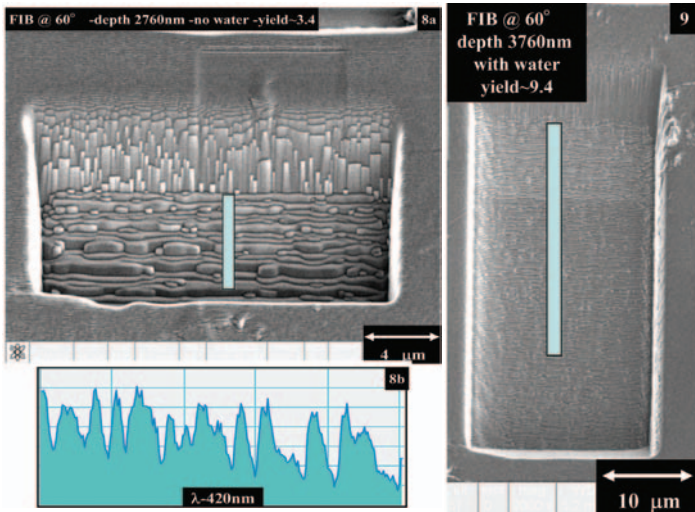


Figure 8. Change from ripples to steps can occur with increased aspect ratio, contrary to predicted regime control by angle and/or water-enhanced etching.

Figure 9. Ripples persist without steps even though this area was FIB water-enhanced-etched deeper than Fig. 8; however, the aspect ratio of this area was less.

surfaces, depending sensitively on the combination of pressure and processing parameters. Conflicting results regarding surface roughness have been obtained with water-enhanced FIB etching of diamond [1, 2].

The addition of chemistry can make the reliability and reproducibility of FIB processing more difficult. Ripples tend to be destroyed by errors in FIB processing. For example, if the sample drifts too much, the ripples may be lost. The added parameter of chemistry makes possible more “errors” in the FIB experiment, and potentially more chances to lose ripples.

Figures 8 and 9 present etch craters produced with similar FIB processing parameters except without and with water vapor, respectively. Both produce ripples but the addition of water vapor triples the sputter yield. The region of Figure 8 has not been etched as extensively as the area of Figure 7c (dose of 1.4×10^{19} and 2×10^{19} ions/cm², respectively); however, the ripple wavelength is substantially larger (420nm versus 310nm). In addition, Figure 8 represents a condition where “ripples” have begun to change to “steps” [2, 4]. This crossover regime makes metrology of ripple wavelength noisier in Figure 8a compared to Figure 7. Furthermore, the steps in Fig. 8 were produced at 60° ion incident angle, which based on Fig. 4 should have been ripples. Conversely, Fig. 4 also plotted ripples at 70° as achievable in the present data-set rather than the steps from reference [2]. Although the water vapor caused more extensive (deeper) etching in the area of Figure 9 as compared to Figure 8, the ripples have a shorter wavelength for the wet condition. These apparent inconsistencies will be discussed and explained by including consideration of aspect ratio and redeposition while optimizing FIB processing parameters.

The sputter yield is independent of many computer-controlled FIB processing parameters, except when chemistry is employed. Generally, sputter yield is considered to be fairly independent of current (i.e. double current and the number of atoms sputtered is doubled, and yield remains constant). Typically dwell time also does not influence yield when no water is present (Figure 10); however with water one may observe either an up or down change of yield. Other computer-controlled parameters besides dwell time can also exhibit this apparent lack-of-control when chemistry is added. A major component of this work is to sort out the interdependency of parameters for chemical-enhanced FIB processing.

A dramatic increase in yield can be achieved in the presence of water, if the dwell time is reduced (Figure 11). Chemical enhanced etching occurs because water reacts with the surface atoms to break the strong sp³ diamond bond. The subsequent FIB ion can more easily sputter this carbon. However, if the FIB dwells too long, then the water-affected layer

Yield vs Dwell (& vs. Water)

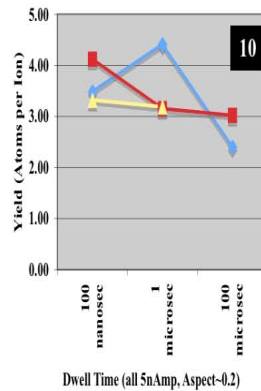


Figure 10. Water-enhanced FIB should enhance the yield for diamond (Fig. 1 [1,2]); however, a more-complex interdependence of FIB etching parameters, such as water pressure and dwell times, means yields sometimes do not increase. (Reported water pressures are for entire vacuum chamber; pressures at sample are ~1000 times greater using GIS systems.)

Yield vs Water Pressure & Dwell & Current

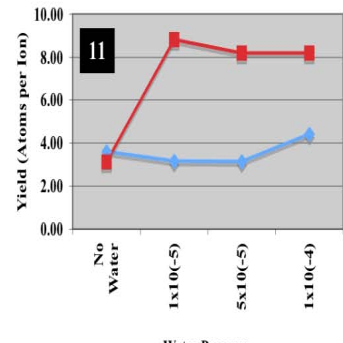


Figure 11. Chemical-enhanced FIB etching is optimized for the shortest dwell times and minimal chemistry. If the FIB beam dwells at a pixel too long, the 2nd impinging ion does not see a chemical-affected surface and does not etch faster.

is removed and the subsequent ions in the rest of the dwell period have to sputter true diamond bonds. While the FIB scans away to other areas, water vapor can reabsorb onto the next exposed layer of diamond. It is desirable to reduce the dwell time, so that all FIB ions sputter only surfaces that have had enough time to absorb water. However, modern FIBs are limited to dwell times in excess of 100nanosec, which is 1000 times too

Yield vs Current (with&w/o Water)

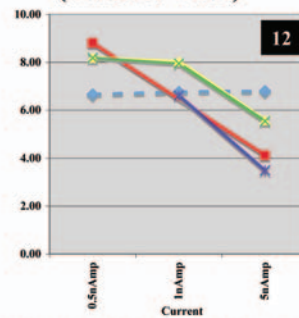


Figure 12. Chemical-enhanced FIB etching is optimized for lower current, and minimal chemistry (for same reason as Fig. 11). Note yield enhancement can only be compared when aspect ratio is constant. The plotted no-water data was acquired at a higher angle; and would have produced a flat yield of ~3.5 at 30°.

Yield vs Overlap (& vs. Water)

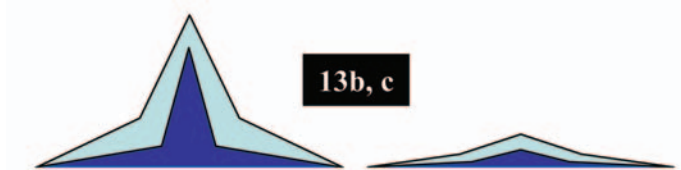
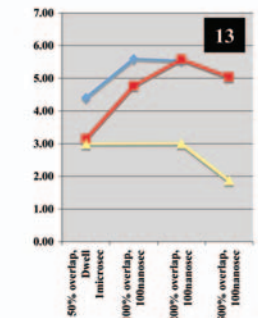


Figure 13. Digital FIB enables variable pixel overlap, and gaps between pixels (negative overlap) provide the most chemical-enhanced yield. The tails of the FIB beam are location of lower current (dark blue profile of FIB intensity), which relatively experience the most chemical enhancement (light blue); and lower currents (smaller FIB spot sizes) and larger gaps take advantage of these tails.

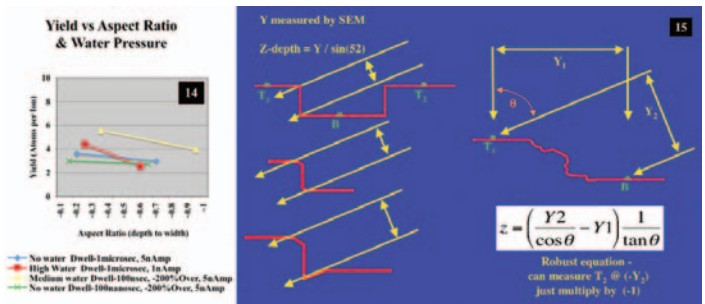


Figure 14. Aspect ratio causes yield decay, and more so for chemically enhanced yields. The higher vapor pressure enhances redeposition. Only ideal chemistry may make redeposition molecules become more volatile.

Figure 15. Metrology of hole depths is difficult by all techniques. The use of FIB spot burns (Fig. 16), and two SEM tilts improves metrology.

long. Increasing the water pressure does not enable water to reabsorb faster during this 100nanosec, and higher water pressure disrupts the vacuum system enough to hinder FIB focusing and etching.

As an alternative to reducing dwell time, the current can be reduced, thereby eliminating double-ion hits during an individual dwell. Figure 12 plots different currents, showing the water enhancement of yield is greater as the current is lessened. However, the percent yield enhancement is less than the percent reduction of current, which means the total volume of material etched is reduced for a given time. Thus, FIB manufacturing processes that require fast turnaround, may not always be achieved via chemical enhancement.

Increasing the spacing between digital pixels (a more-negative overlap) can have an effect akin to reducing dwell time (or reducing current exposure), as plotted in Figure 13. However, if there is too much separation between pixels (< -500% overlap), etching becomes inefficient and yields decay, both with and without water. Figure 16a depicts a case where an overlap of -200% causes the scan pattern to be imprinted into the etched surface. These are not natural ripples, but rather poor FIB processing that eventually is a detriment to yield. When water vapor is present, however, the ion beam is defocused and increases the overlap. Negative overlap can have a positive impact on chemically enhanced yields, because a digitally controlled FIB does not have a square shaped ion beam akin to pixels. The beam profile is primarily Gaussian, but with extensive tails. These tails become a more significant component of the beam shape for low current (small spot size). Figures 13b and 13c are schematics of the cross-section intensity profile of a high current and a low current beam (dark color). Without water-enhancement, the etch rates will be proportional across these beam profiles. (Note yield would be a constant across the beam profile.) With chemical enhancement, the effective yield enhancement across the profile of the beam is greater at the lower intensities (plotted in light blue). As noted earlier, a lower current facilitates a higher yield enhancement by chemical addition. Thus the tails of the beam (spatial regions of lower current) will exhibit a greater yield enhancement. In typical FIB scanning, the pixels overlap each other sufficiently to where the beam tails have minimal impact. With a negative overlap, however, there is a gap between pixels. This gap region is only exposed to beam tails and in turn experience the higher yield enhancement. The more the beam tails are utilized in the overall process, the more the chemical enhancement is realized. However, the maximized effect of negative overlap is interdependent with chemistry pressure, angle, dwell time and current.

Having optimized conditions of a short dwell and negative overlap, the effect of aspect ratio versus water-enhanced yield is plotted in Figure 14. As noted in Figure 2, the yield drops with aspect ratio. Figure 14 directly compares the same aspect ratios to show that this drop is worse for water (chemistry) enhanced etching. All data in Figure 14 is at the same angle of 30° and the same 5nA current. When dwell is long or no water present, the negative overlap becomes a nonfunctioning parameter. Although water increases the overall yield, a combination of high water

pressure and high aspect ratio can become a detriment.

IV) Metrology

Metrology of the yield is necessary in order to understand these cumulative effects. Thus, depths of etch pits must be measured. Modern DualBeam FIBs provide an SEM for *in situ* observation and software tools for measuring pit depths. However, errors in depth measurement worsen as the hole deepens. Such software assumes an orthogonally well-shaped pit (Fig. 15), however, such is never the case as rounding exists both at the top and bottom of pits. Moreover, the roughness of ripples, the defocusing caused by water vapor, the near-sidewall-depressions caused by inefficient computer-controlled scanning algorithms and physics of edge-scattering, and the pit shapes altered by high angle of incidence; all add complexity to pit profiles. A number of additional metrology methods have been developed, including rotating the pit 90° and tilting 45° (Fig. 6b).

Since the DualBeam is designed to tilt with two coincident beams and eucentric stage, it becomes a few-second operation to observe features at the bottom and the top of the pit in stereo and triangulate the depth (Fig. 15). However, to enhance this metrology, small spot burns can be FIB-induced at the bottom and the top of pits (see arrows in Fig. 16). With digital images limited to 1000 pixels, this metrology method is good to tens of nanometers accuracy and precision. But metrology can be worse than ±100nm for holes of aspect ratio >4. Improvement in metrology helps establish the reproducibility of modern FIB processing tools. Plots such as Figure 2 indicate the decay of the etch rates with even small aspect ratios.

V) Redeposition

Redeposition can become distinctly visible in SEM imaging of FIB craters of high aspect ratios, and especially at high angles. The angular pit produces a lip above the leading edge, eventually making it very difficult for sputtered atoms to exit. This redeposited material exhibits a lighter contrast at the leading edge of 30°-pits in the SEM images of Figure 16. When water vapor is added for chemically enhanced etching (Fig. 16b), the redeposition is dramatically increased. In general, the higher pressure above the surface represents increased probability for sputtered atoms to redeposit. In the case of an ideal chemical addition, the vapor could react with sputtered atoms to create a more volatile species and thus decrease

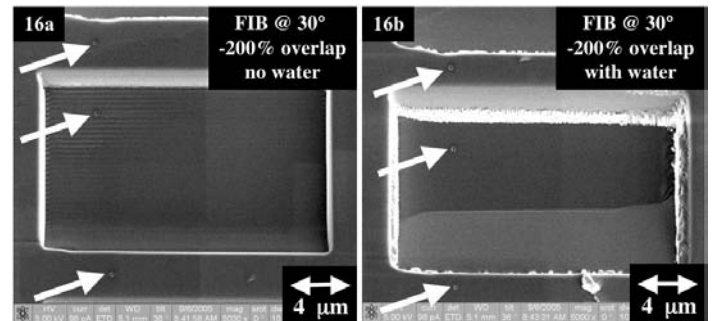
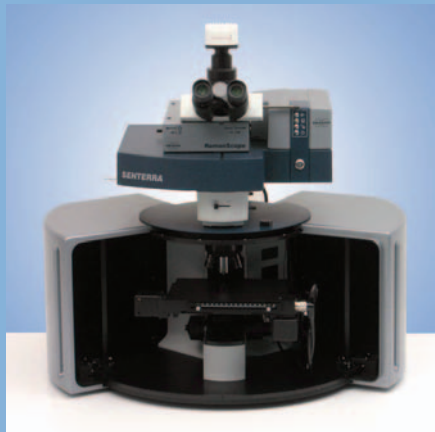


Figure 16. Negative overlap artificially roughens the surface for 30° FIB, but is mitigated and desirable (Fig. 13) when water vapor is used for chemically enhanced etching. Redeposition is enhanced by chemical vapor, especially as seen at pit leading edge. (Arrows indicate FIB spot burns used for metrology.)

redeposition. Since redeposition is observed in micron-scale FIB pits, especially as a consequence of incident angle and aspect ratio, then the trough between two ripples may be modeled as a nanometer-scale pit with redeposition occurring at the nanometer scale. Redeposition is increased at higher angles, and may help ripples become steps at these higher angles (Fig. 4, 5). Similarly, if an angle of 60° without water normally produces ripples, then increasing water vapor pressure can increase redeposition thereby causing the ripples to become steps at this lower angle. Although the water can both enhance etching and enhance redeposition, it is the aspect ratio that dominates control of redeposition. Thus the higher aspect ratio allows the reverse conditions of steps without water at 60° (Fig. 8), and lower aspect ratio retains ripples with water (Fig. 9).



FT/Dispersive Raman Microscopy



- All-in-one, compact, confocal design
- Multiple wavelegths; 1064nm, 785nm, 532nm and/or 633nm.
- Sure_Cal[®] automatic continuous calibration
- Spectral imaging utilizing sample stage mapping
- Confocal depth profiling with FlexFocus[™]
- Automatic fluorescence rejection using SERDS for 785nm

Isn't it time to make peace between two technologies ?

Introducing the **SENTERRA[™]** Dispersive Raman Microscope with the new integrated 1064nm FT-Raman technology

Bruker Optics introduces the first Raman microscope system to combine the long wavelength benefits of 1064nm excitation with the scattering benefits of shorter wavelength Raman excitation. The new 'hybrid' platform accommodates the RamanScope fluorescence-free Fourier transform Raman system and the SENTERRA grating based dispersive Raman technology with fluorescence rejection tools. This combination provides full spectroscopic characterization and optimizes the strengths of the both techniques for your complex micro-analysis samples.



for more information please visit:
www.brukeroptics.com/microscopy
 1-888-4BRUKER | microscopy@brukeroptics.com

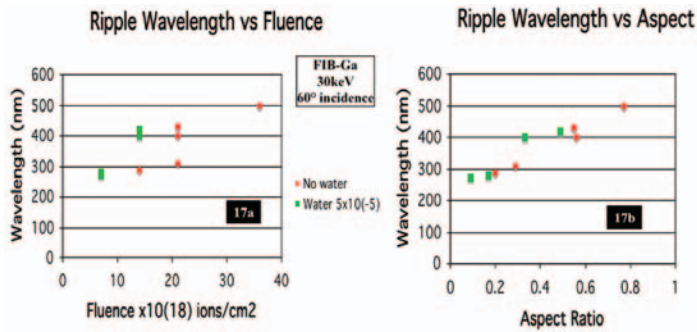


Figure 17. Attempts to rationalize the larger than predicted wavelengths by high fluence (a) actually show ripple wavelength increases with aspect ratio independent of fluence or chemical enhancement (b).

VI) Discussion

Why does the FIB etch rate (yield) vary as a function of etch depth? And why does the present study exhibit ripples with larger wavelengths (>400nm) for high dose, $>1 \times 10^{19}$ ions/cm², than have been reported earlier for saturation wavelengths? “Redeposition” appears to be the answer for both questions. Figure 2 indicates the yield decreases as the aspect ratio increases; etch rate drops as a hole deepens. However, it is not the depth, nor fluence, but rather the aspect ratio that is the controlling factor. Figure 17a attempts to plot ripple wavelength versus fluence; however, several conditions with the same fluence give a wide range of wavelengths. In Figure 17b the ripple wavelengths plot fairly linear with aspect ratio, independent of fluence, chemistry or yield. Reduced yield and disturbance of ripples are not unexpected for the bottom of a FIB pit, however, it is somewhat surprising to see this detrimental effect kick in at such low aspect ratio (Fig. 2). The etch rate already drops just as the hole starts to form. Since holes are a basic fundamental shape of FIB production, the etch rate for many FIB structures will not be a constant. It is also critical to note that this redeposition effect is much greater when ion sputtering at high angles. Thus yields for ion etching at high angles are far from constant.

Redeposition is an accepted culprit for preventing a FIB from drilling a 1nm-diameter hole to an indefinite depth. Aspect ratio provides an upper limit, as a consequence of redeposition. Even when chemistry enhances the etch rate, that same chemistry may also increase the redeposition, especially at higher aspect ratio. Angle of incidence can enhance etch rate as well, but this advantage is also strongly affected by aspect ratio. When a FIB processes a micron size hole, the limits caused by redeposition are recognized; however, redeposition is not commonly accepted in models for formation of nanometer size ripples.

If sputtered atoms are not getting out of a FIB hole, but rather being redeposited, then this should be treated as an additional source in the equations for defining ripples formed by sputtering. If yields decay at high angles due to redeposition even when the aspect ratio of the micro-pit is <0.1 , then the same occurs at the nanometer scale when the amplitude of a ripple is 10% of a wavelength. If increasing redeposition within a micron-size FIB pit causes the wavelengths of ripples to grow, then when a ripple achieves a height of several nanometers, its wavelength must grow to tens of nanometers due to redeposition within itself even if no macroscopic FIB pit exists. Atoms diffusing on the surface add to the ripple growth (becoming dominant at higher temperatures [4-6]). It may be possible to model the redeposition atoms as “diffusing” in a boundary layer just above the surface. The aspect ratio (both at the nanometer scale of amplitude and wavelength of ripple and at the micron scale of FIB pit depth and width) becomes a necessary boundary condition for increasing the redeposition flux term [4]. The value of this flux is plotted as an increasing source (of redeposition versus aspect ratio) when Figure 2 is plotted upside-down.

Controlled processing of ripples may not be as much a holy grail as is the formation of ordered quantum dots. Not only small is good,

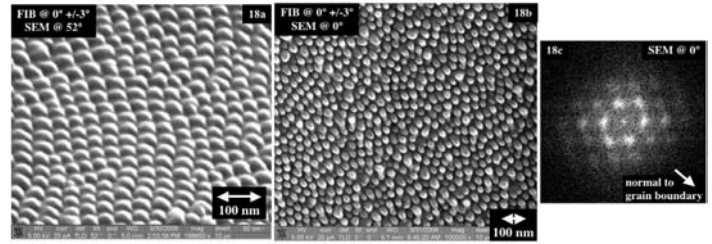


Figure 18. FIB of polycrystalline beryllium produces small ripples (<40nm) of high aspect ratio. Original machining scratches on surface do not dominate ripples but cause a small change in surface angle. (Crystal orientation of grain does affect ripples on Be.) The Fourier transform (b) establishes a non-hexagonal pattern influenced (as is wavelength) by a nearby grain boundary.

but high aspect ratio will be desirable. Figure 18 depicts FIB-induced roughness on a polycrystalline Be surface. (Favorably, the FIB has actually smoothed a very rough and contaminated Be surface prior to the 2-Dim array developing.) Unfortunately, the next grain with a different crystal orientation provided a somewhat different topography. Also the Fourier transform (Fig. 18c of image acquired normal to surface) quantifies variation in 2D wavelength due to minor variations in surface inclination due to polishing within a single grain of this Be; as well as influence of boundary conditions such as nearby grain boundary. Furthermore, the chemistry of the rippled surface contains Ga from the FIB; therefore these rippled topographies may have utility only to template these topological patterns onto another substrate via imprinting. Ripples at the bottom of a FIB pit will have more function when imprinted to become the highest topographies on the imprinted surface.

VII) Conclusions

- 1) Ripples are influenced by redeposition, which is influenced by aspect ratio, both at the micron scale of the FIB pit depth and width and also at the nanometer scale of the ripple amplitude and wavelength.
- 2) Aspect ratio causes a drop in yields, becoming more pronounced for FIB etch rates enhanced by angle and/or chemistry; and this extra redeposition material becomes an increasing source term in the model that causes ripples to grow. ■

Acknowledgements

This work was performed under the auspices of the United States Department of Energy by the University of California, Lawrence Livermore National Laboratories under contract of No. W-7405-Eng-48. UCRL-IM-337258.

References

- [1] Russell, P.E., T.J. Stark, D.P. Griffis, J.R. Phillips, and K.F. Jarausch, “Chemically and geometrically enhanced focused ion beam micromachining,” *J. Vac. Sci. Tech. B* V16(4), p2494-2498, 1998.
- [2] Adams, D.P., M.J. Vasile, T.M. Mayer, and V.C. Hodges, “Focused ion beam milling of diamond: Effects of H₂O on yield, surface morphology and microstructure,” *J. Vac. Sci. Tech. B* V21(6) p2334-2343, 2003.
- [3] Brooks, J.N., “Modeling of sputtering erosion/redeposition - status and implications for fusion design,” *Fusion Engin. And Design* V60 p515-526, 2002.
- [4] Bradley, R. M. and J.M.E. Harper, “Theory of ripple topography induced by ion bombardment,” *J. Vac. Sci. Tech. A* V6(4) p2390-2395, 1988.
- [5] Cuenat, A., and M.J. Aziz, “Spontaneous pattern formation from focused and unfocused ion beam irradiation,” *Mat. Res. Soc. Symp. Proc.* V696 pN2.8.1-N2.8.6, 2002.
- [6] Brown, A.D., J. Erlebacher, W.L. Chan, and E. Chason, “Transient Topographies of Ion Patterned Si(111),” *Phys. Rev. Lett.* V95 (5) p056101-04, 2005.
- [7] Mayer, T.M., D.P. Adams, M.J. Vasile, and K.M. Archuleta, “Morphology evolution on diamond surfaces during on sputtering,” *J. Vac. Sci. Tech. A* V23(6), p1579-1587, 2005.
- [8] Castro, M. R. Cuerno, L. Vazquez, and R. Gago, “Self-Organized Ordering of Nanostructures Produced by ion-Beam Sputtering,” *Phys. Rev. Lett.*, V95 (1) p016102-05, 2005.
- [9] Makeev, M.A., and A.L. Barabasi, “Ion-induced effective surface diffusion in ion sputtering,” *Appl. Phys. Lett.* V71(19) 2800-2802, 1997.
- [10] Sigmund, P., “A Mechanism of Surface Micro-Roughening by Ion Bombardment,” *J. Matls. Sci.*, V8, p1545-1553, 1973.

Upgrading your EDX detector? Buying a new EDX system?

Consider the SiriusSD advantage:

- no liquid nitrogen
- analytical quality performance at high count rates

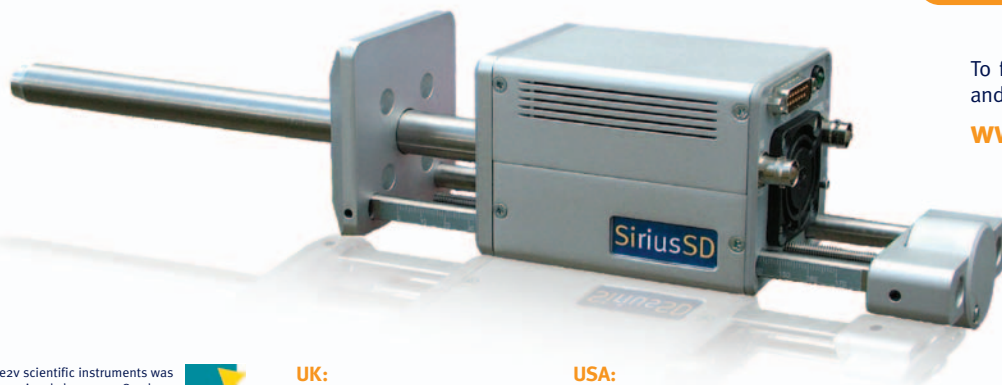
SiriusSD is a silicon drift detector designed to make short work of X-ray analysis. Its industry-standard restored preamplifier output provides some compelling benefits.

e2v scientific instruments is the only independent EDX detector company offering true worldwide support, with repair and upgrade facilities based in both Europe and the USA.

SiriusSD range

Silicon drift detector technology for EDX applications

- High rate capability
- Excellent resolution
- Stable peak position and resolution over a broad range of count rates
- Flexible system integration



To find out more about our products and new facilities, please visit:

www.e2vsi.com

e2v

e2v scientific instruments

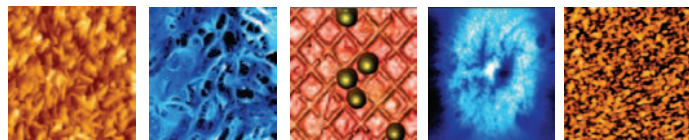
see what we're made of

e2v scientific instruments was previously known as Gresham Scientific Instruments Ltd



UK:
T: +44 (0)1628 533060
E: e2vsi@e2v.com

USA:
T: +1 914 592 6050, Ext 892
E: e2vsi-na@e2v.com



Nano-R₂TM AFM

- High Performance
- Easy to Use
- Versatile
- Proven



The Pacific Nanotechnology Nano-R₂TM AFM is now used in hundreds of labs throughout the world. It is the only AFM that is versatile enough for educating students as well as for high demand research projects.

Pacific Nanotechnology, Inc. • 3350 Scott Blvd., #29 • Santa Clara, CA 95054-3105 • 800-246-3704
NanoParticles.PacificNanoTech.com • ProbeStore.com • PacificNano.com

