Self-Organized Nanostructural Pattern Formation under Ion Beam Irradiation

Michael Hovish

Nanoscale Science, College of Nanoscale Science and Engineering, SUNY Albany

NNIN REU Site: Center for Nanoscale Systems, Harvard University, Cambridge, MA
NNIN REU Principal Investigator: Prof. Michael J. Aziz, School of Engineering and Applied Sciences, Harvard University
NNIN REU Mentor: Joy C. Perkinson, School of Engineering and Applied Sciences, Harvard University
Contact: mhovish@albany.edu, aziz@seas.harvard.edu, joyp@seas.harvard.edu

Abstract:

Ion bombardment is a diverse laboratory technique with many applications including reactive ion etching, focused ion beam milling, ion implantation, sputter deposition, and ion beam characterization techniques such as Rutherford backscattering spectrometry (RBS). Of particular interest are the nano-topologies that form under the presence of ion irradiation. Periodic nanoscale ripples, dots, and high-aspect ratio structures can self-organize under ion bombardment. However, the relative importance of various mechanisms that underlie the self-organization process are poorly understood.

This project aimed to investigate the importance of ion beam-injected stress as a mechanism influencing the dynamics of nanoscale ripples. It has previously been observed that ripples will propagate along the surface of certain materials during ion bombardment, and a recently-developed theory attributes this phenomenon to beam-injected stress. Our study aimed to test this theory by quantitatively measuring ripple propagation velocity at a variety of incidence angles for comparison with the functional form of velocity vs. angle predicted by theory. A 30 keV Ga^+ focused ion beam was used to irradiate Si <001>. *In situ* scanning electron microscopy (SEM) was used to directly record the ripple dynamics.

Additionally, a methodology for measuring the propagation of ripples with respect to fluence (ions•cm^{-2}) was developed and preliminary measurements were taken.

Introduction:

There has been much attention granted to self-organized nanostructures that propagate under the presence of ion bombardment [1]. Nanoscale features at and below 100 nm have been shown to form under both focused and unfocused ion beams, a process known as "sputter patterning." In a recent study, a regular array of nanodots with diameters as small as 7 nm were fabricated [2]. However, there is little understanding on how to control the ion induced self-organization process. A cohesive theory by which we can understand the governing mechanisms of ion induced self-organization could lead to a high level of control and manipulation of nano-pattern formation.

In this work, we investigated the propagation velocity at which nano-ripples propagated along the surface of Si <001> under the raster of a focused ion beam. Parallel mode ripples were observed to propagate in a direction anti-parallel to the incoming gallium ions.

Note, the term "propagation velocity" used in this report does not refer to the traditional concept of velocity. Rather, "propagation velocity" was measured in $^{nm}/_{fluence}$, i.e. how far a ripple would propagate under a given amount of ion impingement.

Experimental Procedure:

A 30 keV beam of focused Ga^+ ions was produced using an NVision 40 Dual Beam focused ion beam (FIB) outfitted for *in situ* SEM. FIB and SEM guns were fixed at an angle $\Theta = 54°$ from each other, while the specimen stage had the freedom to tilt. The angle between the specimen stage and the FIB gun is denoted Θ_{FIB} and was held constant at 30°. Although we were ultimately interested in varying the angles of incidence, Θ_{FIB} was held constant while beam current, dwell time and other tool specific parameters were optimized. Beam currents of 80 pA and 150 pA were utilized to expose a 100 μm^2 area of p-type Si <001>. We minimized dwell time, set pixel overlap to 53%, and passed the ion beam over the exposed area many times, thereby approximating uniform radiation. This was done to ensure ripples were not directly written by the FIB.

Real-time Fast Fourier Transform was used to observe the evolution of periodicity while post-mortem Fast Fourier Transform was used to ascertain final periodicity of the ripples. In order to measure propagation velocity, measurements were made on how long it took for the Raith FIB software to execute one exposure loop. Using this metric, we correlated the amount of ions impinging on the surface between SEM frames.

Results and Conclusions:

Figure 1 exhibits the relevance of dwell time. At sub-microsecond dwell times (1a), nanoscale ripples were apparent. As we increased dwell time, the ion beam was able to mill deeper into the surface, thereby directly writing nano-topologies. Dwell time should therefore be minimized in order to ensure that features are not directly written onto the surface.

In situ SEM was employed to record the evolution of ripples shown in Figure 2. We incrementally increased the total fluence, measuring the time to deliver the total fluence. Using this trend, we correlated the amount of ions impinging the surface between video frames and obtained a preliminary propagation velocity of 35.8 nm/10^{17} ions•cm^{-2} anti-parallel to the beam direction. This falls within an order of magnitude reported by [3] earlier this year.

It has been observed that there are inconsistencies in the functioning of the Raith software, and so further characterization of the ion flux is required to ensure repeatability of the metric.

Future Work:

Obtaining the measure for propagation velocity requires further characterization. Once fully characterized, measurements will be used in comparison to what theory predicts. With the aid of a verified theory, we hope to research more complex material systems and nanostructures.

Acknowledgements:

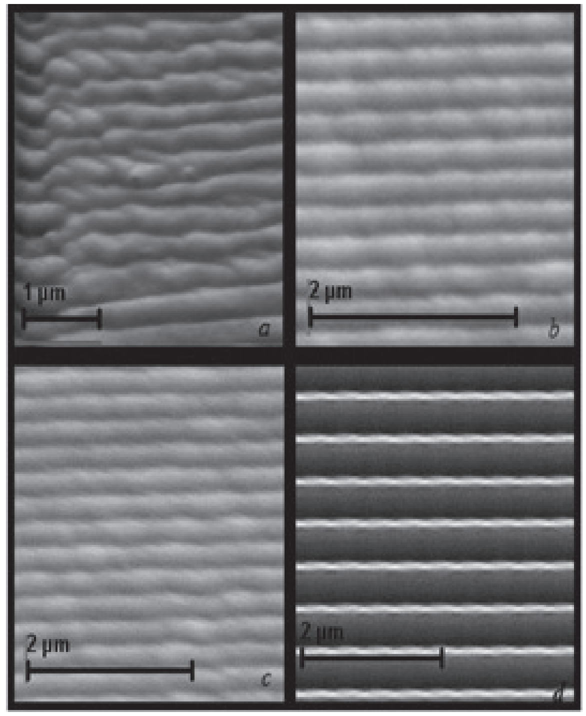
Prof. Michael J. Aziz, Joy C. Perkinson, Nicholas Antoniou, National Nanotechnology Infrastructure Network Research Experience for Undergraduates (NNIN REU) Program, National Science Foundation.

References:

[1] P. Sigmund and M.-fysiske Meddelelser, "Ion Beam Science: Solved and Unsolved Problems," pp. 1-5, 2006.

[2] Q. Wei, J. Lian, S. Zhu, W. Li, K. Sun, and L. Wang, "Ordered nanocrystals on argon ion sputtered polymer film," Chemical Physics Letters, vol. 452, no. 1–3, pp. 124-128, Feb. 2008.

[3] H. Gnaser, B. Reuscher, and A. Zeuner, "Propagation of nanoscale ripples on ion-irradiated surfaces," Nuclear Instruments and Methods in Physics Research Section B: Beam Interactions with Materials and Atoms, vol. 285, pp. 142-147, Aug. 2012.



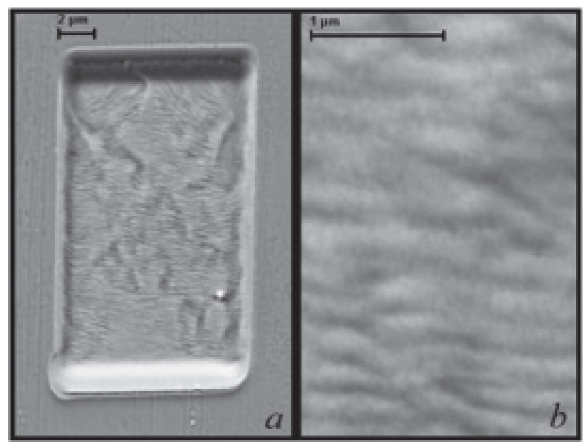


Figure 1, top: Dwell Time varied: 0.1 µs (a), 6.4 µs (b), 12.8 µs (c), 25.6 µs (d). Scale bar index: 1 µm (a), 2 µm (b), 2 µm (c), 2 µm (d).

Figure 2, bottom: Post-ion bombardement: a. 11.12 µm × 19.49 µm milled area, magnification 2.03 kx.
b. High magnification (15.05 kx) of nanoscale ripples. Scale bar index: 2 µm (a), 1 µm (b).