Grayscale Lithography and Its Application to Screw Dislocations in Colloidal Crystals

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Abstract:

In nature, screw dislocations cause atomic crystals to grow in upward spiral patterns [1], which is distinctly different from their typical island-like epitaxial growth patterns. However, the small size of these atomic crystals makes study of the growth of this dislocation-driven crystallization very difficult. Colloidal crystal growth, on the other hand, occurs on a scale that is visible using traditional microscopy techniques, and direct observations of such crystals allow an unprecedented glimpse of the particle-scale growth mechanisms.

The goal of this project was to study the spiral growth of colloidal crystals by providing the core of a screw dislocation and observing the growth of the crystals at the dislocation site. In order to accomplish this, grayscale lithography techniques were used to create a feature similar to a spiral staircase. Grayscale lithography is unique in that, unlike typical photolithography, some thickness of the photoresist remains after developing. For this application, we used a binary mask method, in which five different masks were created, each mask being assigned an exposure that is twice as long as the previous mask. Utilizing the additive properties of exposure doses, one wafer can be exposed using all five masks in order to create up to 32 different variations in height in the photoresist. Using this technique, we have achieved up to 24 steps in several variations of a spiral design and have begun to collect data regarding the colloidal crystal growth.

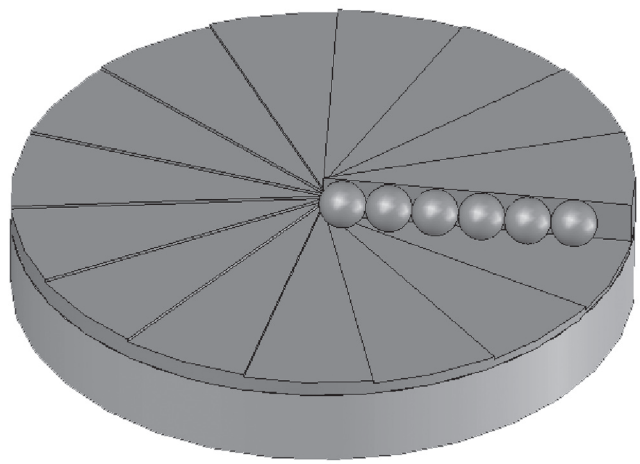

Figure 1: A cartoon of the 15 step spiral staircase design, with the crystal beginning to nucleate at the step edge.

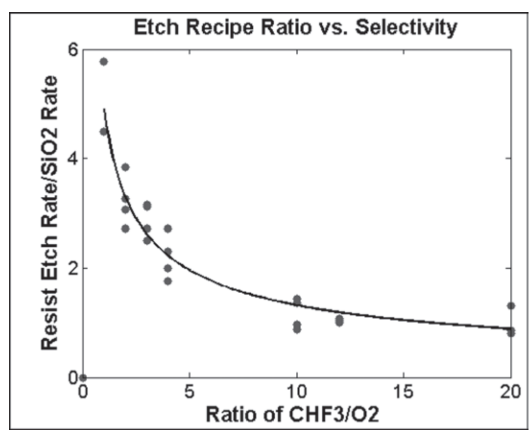

Figure 2: The relationship between the CHF_3/O_2 ratio in the plasma etching recipe to the etch selectivity. The ratio of CHF_3/O_2 used for this experiment was 30/25 ppm.

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Introduction:

The mechanisms of dislocation-driven growth in crystals are largely unknown, but have the potential to provide many insights into the natural world. By using grayscale lithography techniques to mimic the core of a screw dislocation, we hope to induce dislocation-driven growth in colloidal crystals to gain a better understanding of what causes this unique growth pattern.

This research could lead to a more reliable process to grow thin films with less polycrystallinity. Currently, the manufacture of many circuitry devices relies on the epitaxial growth of atomic thin films, which can be difficult to produce without many grain boundaries and other dislocations. In dislocation-driven growth, however, the nucleation site at the dislocation is energetically favorable compared to nucleation sites at

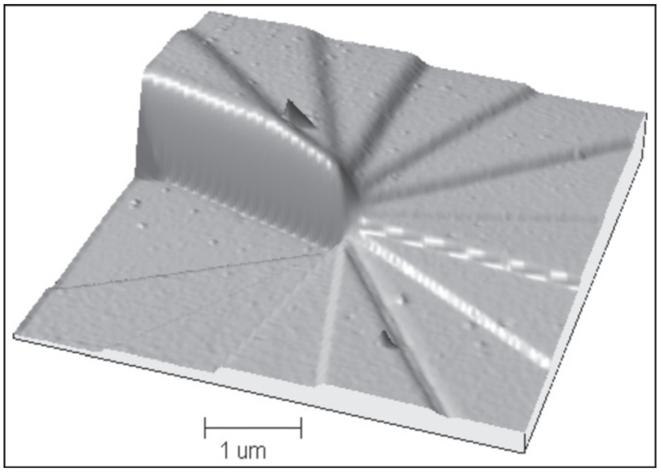

Figure 3: AFM of the 15 step spiral staircase design etched into oxide. The highest step is ~ 0.93 µm taller than the lowest step.

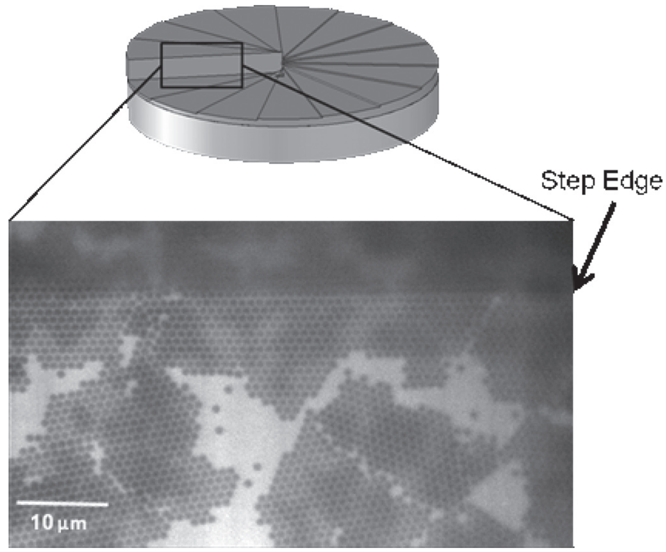

Figure 4: Colloidal crystals aligned with the step edge.

other points on the surface, which may help to minimize grain boundaries and other dislocations.

Experimental Procedure:

A binary mask method was utilized to create a feature of varying heights. We used five masks, each of which was assigned a different dose ranging from 0.02 to 0.32 seconds, to create spiral staircase patterns of 15 different step heights in SPR 220-7 resist. Next, the resulting 4 μm step height in the resist was transformed into a 1 μm step height in a SiO_2 layer on top of a glass coverslide. We did this by varying the selectivity through changing the ratio of CHF_3/O_2 in the plasma. Because O_2 etches photoresist quickly, but hardly etches oxide at all, increasing the amount of O_2 in the plasma increases the rate at which the photoresist etches relative to the oxide. In this way we achieved a selectivity of 4:1, etching the photoresist 4 times more quickly as the oxide and changing the 4 μm step in the photoresist to a 1 μm step in the oxide.

With a 12 × 12 array of this pattern etched into an oxide layer on the glass coverslide, we built a simple flow cell and deposited a colloidal suspension of 1 μm silica particles dispersed in an index-matched solution of DMSO and water. In order to create an attractive force between these colloidal particles, we used polystyrene particles as a depletant. While allowing the particles to settle onto the coverslide over a period of 12 hours, we observed the crystallization using an inverted confocal microscope.

Results and Conclusions:

As Figure 4 shows, the colloids are attracted to the step edge and heterogeneous nucleation has occurred at multiple locations along the step edge. In addition, these heterogeneously nucleated crystals seem to align with the step edge, which could lead to larger single-domain regions and less polycrystallinity.

Homogeneous nucleation also occurred throughout the system. Until the depletion effect is properly tuned in order to eliminate the homogeneous nucleation, it is unlikely that the heterogeneous nucleation will be able to drive crystal growth in the type of spiral pattern that is seen in natural atomic systems.

Future Work:

There are many exciting directions to explore related to the interactions between crystals and dislocations. First, we hope to examine the effects of using a different spiral pattern with two full revolutions and 24 steps, which we believe will more closely mimic the natural atomic system. We would also like to explore the interaction between two spirals in close proximity and the effects of applying shearing forces to the system. Examining the effect of these parameters on the polycrystallinity of the resulting crystal will help to elucidate whether dislocation-driven epitaxial growth can improve film quality, as would be desirable for such devices as microprocessors.

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References:

[1] Kodambaka, S, S. V. Khare, W. Swiech, and K. Ohmori. "Dislocation-driven surface dynamics on solids." Nature. 429 (2004): 49-51.