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13. ABSTRACT (Maximum 200 words)  In this final report, we describe our efforts in exposing self-assembled molecular monolayers to a beam of neutral sodium atoms, and chemically etching the resulting substrate, and characterization of the resulting surface. We also discuss our development of a rubidium magneto-optical trap.				
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## Neutral Atom Lithography Project Summary (06/15/2003 -- 06/14/2006)

In this final report, we will discuss the progress we have made in the program DAAD19-03-1-0138 entitled "Neutral Atom Lithography with Multi-Frequency Laser Fields" during the program period of June 15, 2003 to June 14, 2006. Our efforts in this program have been directed along two avenues, including (1) studies of surfaces of Self-Assembled Molecular (SAM) mono-layers after being exposed to a beam of neutral sodium atoms, and (2) development of a neutral atom trap.

### Advances in Lithographic Techniques with Atomic Sodium

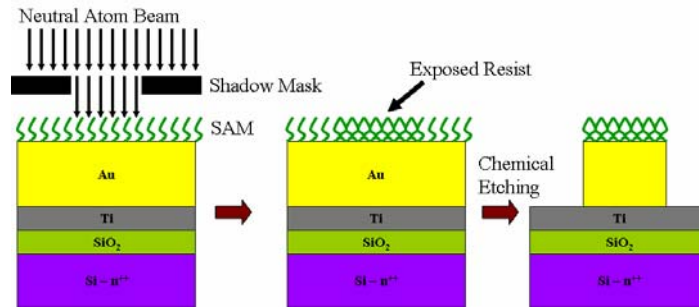
We completed a series of experiments designed to explore issues that must be resolved in order to improve the quality of the exposed surface: i) testing variations among different self-assembled molecules (SAMs) on the Au-coated silicon substrates, ii) changing the thickness of the gold metal layer, iii) varying the time of exposure of the substrate to the atomic beam, and iv) trying a hydrogen flame annealing process to improve the morphology of the gold surface.

In the first experiments, we have explored a variety of SAMs to determine which were well suited to lithographic processing after exposure to a beam of sodium atoms. Very little is currently understood regarding the mechanism that takes place during the collision between an incident atom and the SAM. There has been speculation regarding chemical effects, intertwining of adjacent molecular chains, or bond breakage as the atoms smash into the surface, but not much progress has been reported that helps sort these processes out. In addition, neutral atom lithography (NAL) using sodium atoms has received only passing notice in previous reports. Therefore, our first efforts have been to establish the conditions and parameters for useful exposures, and to begin studies that will help us understand the physical or chemical mechanisms involved.

We tested eight SAMs for their ability to pattern gold metal through NAL. For this experiment, we prepared silicon substrates with the eight molecules listed in the following table.

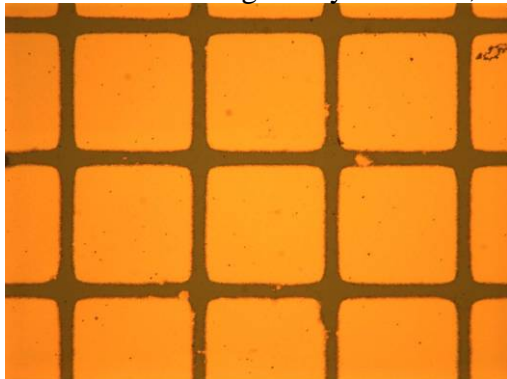
Designation	Name	Molecular Weight
C6	Hexanethiol	118.24
C8	Octanethiol	146.29
C9	Nonanethiol	160.32
C10	Decanethiol	174.35
C12	Dodecanethiol	202.40
C14	Tetradecanethiol	230.45
C16	Hexadecanethiol	258.51
C18	Octadecanethiol	286.56

We show a diagram outlining the preparation and processing of a substrate to the right. We first prepared the substrate by depositing a 5 nm layer of titanium and a 30 nm layer of gold on a silicon substrate. We then deposited one of the SAMs listed in the table above on the substrate, attached a

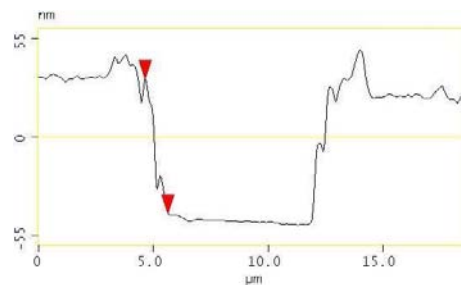


TEM copper grid which served as a physical mask, and exposed the substrate to the neutral atom beam. (A TEM copper grid is a square crossed pattern of 5  $\mu\text{m}$  wires spaced by 50  $\mu\text{m}$ .) Each of the eight SAMs exhibited negative resist behavior, i.e. the gold layer remained in regions that were exposed to the sodium atoms, and was removed from regions that were blocked. From this comparative study, we determined that the C18 (ODT) SAM provided the best behavior with the cleanest separation between exposed and unexposed regions, and sharp transitions between these regions.

In the second experiment, we fabricated NAL samples with four different thicknesses of the gold layer: 30 nm, 50 nm, 80 nm, and 100 nm. We exposed each sample to the sodium beam for 60 min. Each sample showed negative resist behavior and we recorded excellent patterns (50 x 50  $\mu\text{m}$  patterns and 5  $\mu\text{m}$  gaps, respectively). The figure to the left shows the etched substrate using a C18 (ODT) SAM grown over a 6 nm thickness of Ti and an 80 nm gold layer. These results show that if samples are exposed sufficiently, any gold thickness can be etched and have fine patterns.



Since the primary goal of any lithographic process is to minimize the dimensions of structures on a substrate, an important parameter is the resolution of the structure, or the transition distance between the gold coated surface and the etched surface. We have therefore spent considerable effort measuring profiles of our surfaces, and show an example obtained through Atomic Force Microscopy (AFM) to the right. The 350 nm resolution in this image is likely due to a shadow effect from the TEM grid, but we will continue to measure this as we move to optical focusing of the atomic beam.



With the results of these two experiments, we next turned to optimization of the exposure time using ODT SAMs on samples. For exposure times under 45 min, we could not observe any pattern on the samples. The exposed areas of a sample exposed for 45 min

were also etched partially. Samples exposed for more than 60 min gave excellent Au patterns. Thus, we conclude that exposure times of more than 60 min result in sufficient Na coverage of the surface. Using these results, we concluded that gold thickness was not dominant factor, but that the exposure time was critical for generation of fine patterns.

Our final investigation for processing was to determine the effect of using a hydrogen flame annealing process to improve the gold surface. Our hope was that the annealing process would improve the flatness of the gold surface, leading to improved lithographically-generated structures. Our observations, however, were that contamination of the gold surface during the annealing process prevented good deposition of the SAM on substrates, and caused unexpected etching on unexposed areas. With better sample cleaning after annealing, better results should be possible.

More recently, we focused our attention on understanding the physical mechanism that leads to the pattern transfer. For this, we used X-Ray Photoemission Spectroscopy (XPS) and Secondary Ion Mass Spectroscopy (SIMS) to study the atomic and chemical composition of the surfaces before and after etching. The SIMS studies were carried out at Northwestern University. Our conclusion from these studies is that the sodium atoms appear to accumulate on top of the SAM layer, or perhaps close to the top of the layer, but that they do not chemically or physically alter the thiol molecules in any significant way. This result was somewhat surprising, as it was generally expected that the incident atoms had a stronger impact. During the etch process, then, the sodium atoms seem to shield the thiol molecules from the etchant, such that the SAM and gold are removed from regions where the sodium atoms do not collect, but remain in regions exposed to the sodium.

### **Magneto-Optical Trap**

In a parallel effort, we have been developing a neutral atom source for these studies. Our original goal was to generate a monochromatic rubidium beam source. This source included a two-dimensional magneto-optical trap as the starting point. We recently shifted our apparatus to create a 3-D MOT, which seems easier to set up and meets our immediate needs as well or better than the original design. We have constructed an array of external cavity diode lasers, each of narrow bandwidth, highly controlled, and frequency-locked to an atomic transition in rubidium. These lasers form the basis of the MOT, as well as performing spectroscopic measurements in the rubidium. We cycle the MOT periodically, turning on the MOT lasers and inhomogeneous magnetic field to collect atoms from the background vapor, and then turning off the MOT lasers and magnetic field to allow measurements in a clean ultra-cold vapor of atoms. The capture time is on the order of 1 second.

We are progressing toward measurements in the rubidium “lambda” system, consisting of two lower levels (two hyperfine components of the ground state) and the  $5p\ ^2P_{3/2}$  upper state. Our first measurements here will be of the gain/absorption spectrum of a weak probe beam interacting with the rubidium atoms when driven by a pair of laser

fields in a Raman coupling scheme. Measurements to date in this system include observations of Electro-magnetically induced transparency and a precise spectrum of the hyperfine components of the  $5p^2P_{3/2}$  state. Ongoing work of the gain spectrum is to investigate theoretical predictions of Paul Berman at the University of Michigan.

### **Publications and Reports**

Publications and conference proceedings that resulted from the supported work include:

- (b) Conference Presentation, with Proceedings  
"Self-Assembled Monolayer Resists for Electron Beam Lithography,"  
S.O. Koswatta, A.D. Scott, S. Bhattacharya, and D.B. Janes, presented at  
IEEE-NANO 2004, August 2004. Published in IEEE-NANO 2004  
proceedings.
- (c) Conference Presentation, without Proceedings  
"Self-assembled monolayer resist for electron/neutral atom beam  
lithography," S. O. Koswatta, D. B. Janes, A. K. Mills, D. S. Elliott,  
presented at the 46th Electronic Materials Conference (EMC), June 2004.
- (d) Manuscripts submitted, but not published  
"Negative Resist Behavior of Neutral Sodium Atoms Deposited on Self-  
Assembled Monolayers," Sanghyun Ju, A. K. Mills, Qingling Hang, D. S.  
Elliott, and D. B. Janes, submitted to the Journal of Vacuum Science and  
Technology.

### **Scientific Personnel:**

Principal Investigators: Daniel S. Elliott and David B. Janes

Qingling Hang, Post-doctoral research associate

Graduate Students:

Arthur K. Mills, Ph.D. expected December 2006

Sanghyun Ju, Ph.D. student

Siyu Koswatta, MS awarded, August 2004.

We have also been able to provide valuable research experience for eight undergraduate students through this program: Mesfin Getaneh during the summer of 2004; six students during the summer of 2005; and one additional student in the summer 2006. Mesfin was an undergraduate student at the University of New Orleans, and is now in the graduate program at Northwestern University. He investigated properties of diode lasers during his undergraduate research experience. The 2005 students were:

Caleen Markland (University of Arkansas, Pine Bluff); Stephen Pace (Prairie View A&M University); Miguel Vargas (University of Texas, El Paso); John Lorenz (University of Louisville); Mercedes Mane (Purdue University); and Sana Qamar (Purdue University). Working in teams, these students built a traveling wave Michelson interferometer wavemeter, a temperature controller for a diode laser, a feedback system for stabilizing the frequency of diode lasers, and a frequency control unit for a diode laser. They also set up a saturated absorption measurement using a rubidium cell, and recorded several spectra of the D2 line of rubidium. John Lorenz has since enrolled in the graduate program in Physics at Purdue. The student who joined us in the most recent summer was Blake Conley, a sophomore in Physics from Clarkson University. Blake worked on a variety of projects, including a switching circuit for the magnetic field driver for the MOT and improvements to the electronics for the Michelson wavemeter. Overall, the students each made valuable contributions to our project while they were here.