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VARIABLE TEMPERATURE SCANNING TUNNELING MICROSCOPY

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FINAL REPORT

for

Grant No. N00014-88-K-0416

Office of Naval Research
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800 North Quincy Street
Arlington, Virginia 22217

Prepared by

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19 ABSTRACT *(Continue on reverse if necessary and identify by block number)*

Variable temperature STM/STS has been used over the 4.2 K to 400 K temperature range to study CDW formation and spatial variations of the superconducting energy gap in Pb films. Topographic variations of the superconducting energy gap in Pb films show an intriguing transition from superconducting to normal behavior over distances much less than the bulk coherence length. More recently, effort has been focused on the development of a multi chamber UHV STM system that will couple a wide range of STM/STS capability with sample and tip preparation and characterization facilities. The first chamber of this system is fully functional and is being used to study and modify semiconductor surfaces. Other chambers will be specialized for STM nanolithography and cryogenic UHV STM. This system accommodates UHV sample and tip transfer between chambers in addition to independent chamber operation modes. STM modification of H passivated silicon surfaces has been accomplished using techniques developed by John Dagata at NIST. A unique STM lateral coarse translation system has been developed and used to locate and modify Si MOSFET structures fabricated for this project. Gratings written by STM within the gate region are being evaluated for the creation of finished devices that will exhibit quantum interference effects at higher temperatures than previously observed. Work is also underway to fabricate planar tunnel junction structures that utilize coulomb charging effects to realize logic functions

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Abstract

Variable temperature STM/STS has been used over the 4.2 K to 400 K temperature range to study CDW formation and spatial variations of the superconducting energy gap in Pb films. Topographic variations of the superconducting energy gap in Pb films show an intriguing transition from superconducting to normal behavior over distances much less than the bulk coherence length. More recently, effort has been focused on the development of a multi-chamber UHV-STM system that will couple a wide range of STM/STS capability with sample and tip preparation and characterization facilities. The first chamber of this system is fully functional and is being used to study and modify semiconductor surfaces. Other chambers will be specialized for STM nanolithography and cryogenic UHV-STM. This system accommodates UHV sample and tip transfer between chambers in addition to independent chamber operation modes. STM modification of H-passivated silicon surfaces has been accomplished using techniques developed by John Dagata at NIST. A unique STM lateral coarse translation system has been developed and used to locate and modify Si-MOSFET structures fabricated for this project. Gratings written by STM within the gate region are being evaluated for the creation of finished devices that will exhibit quantum interference effects at higher temperatures than previously observed. Work is also underway to fabricate planar tunnel junction structures that utilize coulomb charging effects to realize logic functions.

Key Words: Scanning Tunneling Microscopy, Scanning Tunneling Spectroscopy, Low Temperature Ultra-high Vacuum STM, Superconductivity, Superconducting Spectroscopy, Semiconductor Nanostructures, STM Nanofabrication, Nanolithography, Coulomb Blockade, Quantum Interference Effects

I. Introduction

The following list represents the events and work affiliated with this grant over the past three years.

- Variable temperature STM design and operation
- STM studies of 1D CDW systems
- STM studies of organic adsorbates on Au(111)
- Move into the Beckman Institute
- Acquisition of Ardent graphics supercomputer
- STM nanoscale modification of graphite surfaces
- STM studies of DNA
- Energy gap topology in superconducting Pb films

The items listed above have been described in previous semiannual reports and will not be discussed in detail here. The items listed below, however, represent recent activities and accomplishments and form the basis for continued ONR-supported work over the next few years.

- Status of multi-chamber UHV-STM system
- Development of 2D coarse translation system for the STM
- Nanolithography on semiconductor surfaces and devices
- Real-Time supercomputer processing of STM data

These items will now be discussed in detail.

UHV-STM System

Considerable progress has been made recently on the construction of a multi-chamber UHV-STM system in our laboratory at the Beckman Institute. Figures 1 and 2 show a diagram and photograph of this system. The overall objective of the UHV-STM system is to provide wide ranging sample preparation, STM/STS, and nanolithography capabilities without compromising UHV conditions. Although the three main chambers, depicted as A, B, and C in figure 1, have separate loadlock and pumping systems to enable independent operation, they are interconnected to accommodate UHV sample and tip transfer between them. At present, chamber A is fully operational, with a base pressure of ~3 x 10^{-11} Torr, and is being used to conduct UHV-STM experiments on semiconductor surfaces. Figure 3 shows atomic resolution images of Si(111) 7x7 and Si(100) 2x1 surfaces prepared and imaged in chamber A. The STM stage in chamber A is outfitted with a UHV-STM capable of coarse lateral translation over mm dimensions without compromise to atomic resolution operation. Provisions have been made to install a second STM on the same stage, while future plans call for the construction of a second UHV-STM side chamber housing an STM designed specifically for the capture and analysis of photons emitted from the tunnel junction. This electroluminescence-STM (EL-STM) is being designed in collaboration with Professor Steve Bishop at Illinois, and preliminary test measurements will be obtained using our existing UHV-STM.

A major priority in our laboratory is the construction of a cryogenic UHV-STM that is coupled directly to chamber A, as shown in figure 1. At present, the loading and transfer chamber is nearly complete and the STM head is under construction. Tests will begin shortly to determine the proper compromise between vibration isolation and thermal contact between the STM and the helium dewar. Since we are able to scan at atomic resolution with very little vibration isolation in the room temperature UHV-STM, we anticipate successful operation of the cryogenic UHV-STM system.

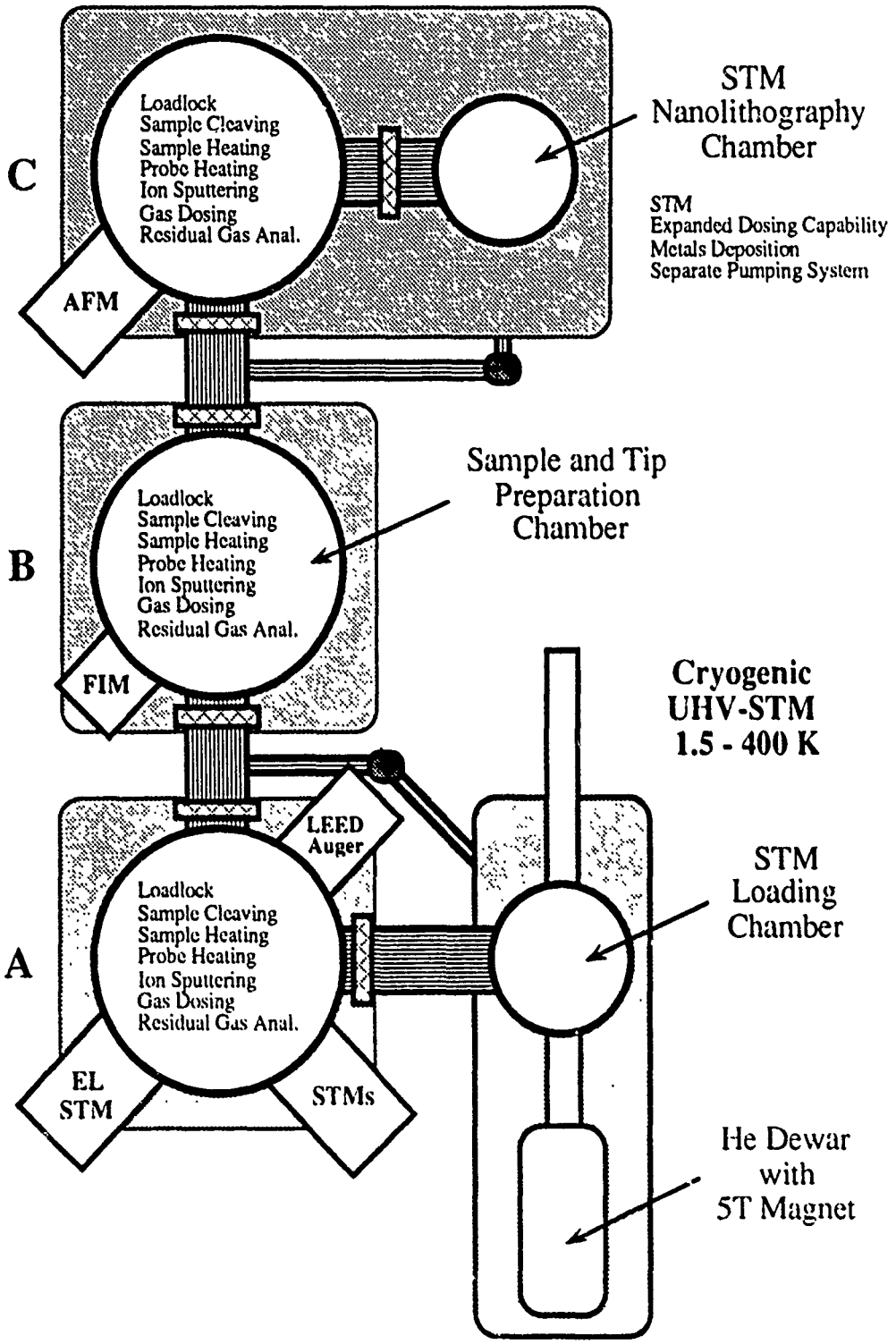


Fig. 1 Diagram of the new UHV-STM system under construction at the Beckman Institute.

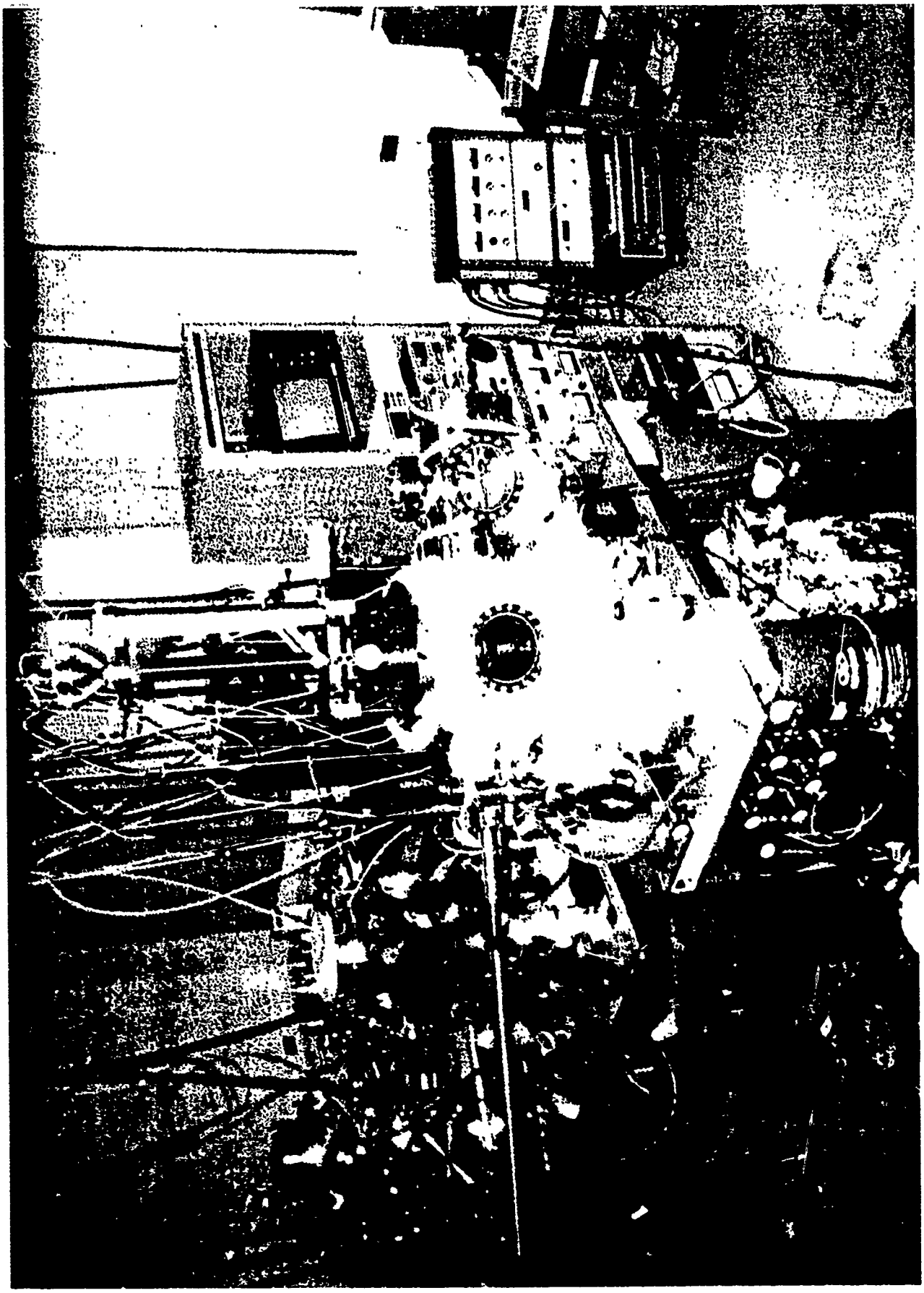
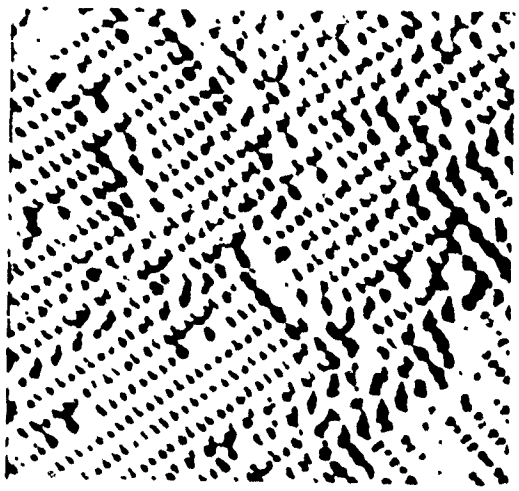


Fig. 2 Photograph of the UHV-STM system.

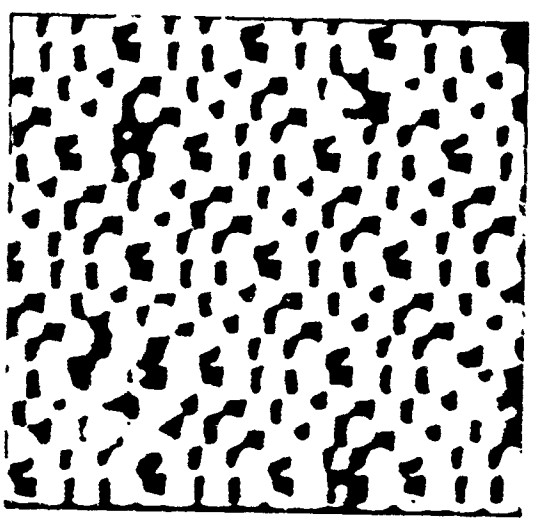
Si(100)



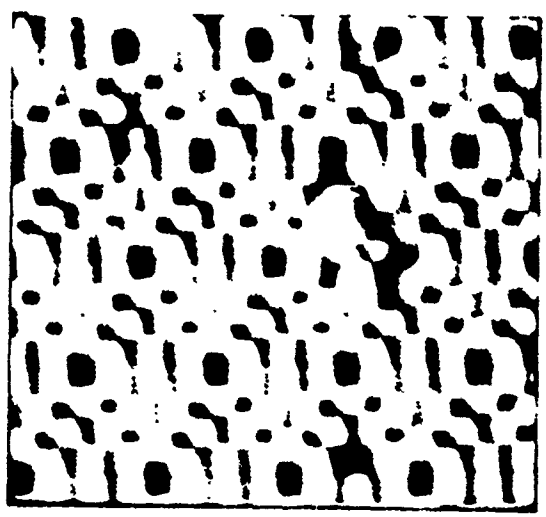
Occupied States

Unoccupied States

Si(111)



Occupied States



Unoccupied States

Fig. 3 STM images showing occupied and unoccupied states for Si(100) 2x1 and Si(111) 7x7 surfaces prepared in chamber A of the new UHV-STM system.

At present, the STM loading and transfer chamber is nearly complete and the STM is being built. Room temperature tests will begin shortly so as to determine the proper compromise between vibration isolation and thermal contact between the STM and the helium dewar. Since we are able to scan at atomic resolution with very little vibration isolation in the room temperature UHV-STM, we anticipate successful operation of the cryogenic UHV-STM system.

STM Coarse Translation System

Nearly every STM research group is motivated to extend the scanning range of their STMs to macroscopic dimensions. In order to provide a useful link between complementary technologies such as crystal growth and device fabrication, the STM must have controlled access to large surface areas in order to locate features of interest. Although commercial STMs are available with > 100 μm scan range, they use long piezoelectric elements having several thousand Å/V deflection, making atomic resolution operation virtually impossible. Another route to large area access is to provide coarse translation that moves the relative position of the probe and the sample without consuming the dynamic range of the scanner. Various schemes have been used such as X-Y sample stages moved by micrometers, stepper motors, or inchworm motors, however, these are generally bulky, inducing vibration sensitivity, thermal drift, design complexity, and operational difficulties to the microscope operation. The IBM Zurich group has used the 'louse' of the original Binnig and Rohrer STM design to translate III-V heterolayer samples in 1D to locate the superlattice layers. 2D translation would be very useful in that, for example, one could translate along superlattice layers to locate device structures. Recently, we have successfully tested a 2D coarse translation system on both our air and UHV-STMs. The details of this system will be disclosed in the very near future, once patent considerations have been handled, and this grant along with funding from the NSF-ERC supported Microelectronics Laboratory will

be acknowledged as the sources for its development. The NSF-ERC supported work is for STM of optoelectronic device structures fabricated in III-V heterolayer substrates.

Shown in figure 4 is a series of four STM images of a source/drain well of a silicon n-channel MOSFET that has been partially fabricated on a silicon wafer. With the STM coarse translation system it is easy to locate and study device structures such as this. At present, our coarse translation system has access to 3mm of the sample surface in any direction without compromising atomic resolution, low vibration, low thermal drift capability, as the images in figure 3 demonstrate. We view this technology as essential for the successful pursuit of nearly all of our future STM work.

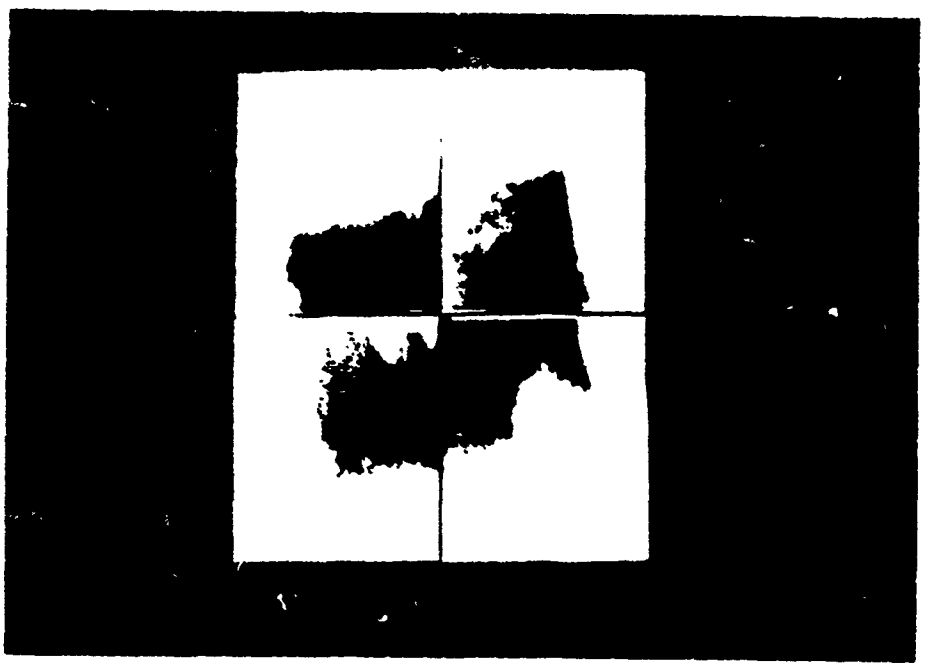


Fig. 4 STM images (8μm x 8μm) demonstrating the use of the 2D coarse translation system to locate the corners of one of the source/drain wells of a Si-MOSFET structure.

Nanolithography

The development of 2D coarse translation has made it possible to apply techniques of STM nanofabrication to device structures fabricated by conventional means. We have begun a multi-faceted program to integrate STM nanofabrication with conventional technologies including electron beam lithography (EBL), reactive ion etching (RIE), and molecular beam epitaxy (MBE) crystal growth, as illustrated in figure 5. The first test

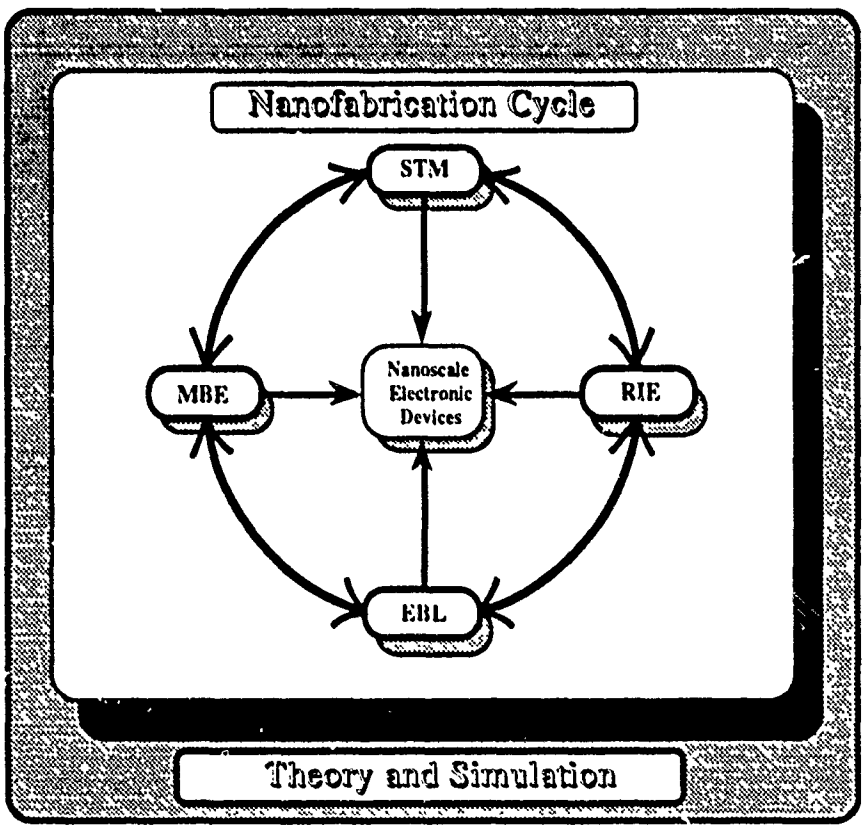


Fig. 5 Diagram illustrating the integration of STM-based techniques with conventional semiconductor growth and patterning technologies.

vehicle of this effort is to modify the gate region of a silicon n-channel MOSFET. The objective here is to create a periodic modulation in the gate oxide thickness, resulting in modulation of the electric field intensity at the Si/SiO_2 interface of a finished device. At sufficiently low temperatures the modulation period would be comparable to the mean free

path of channel electrons, giving rise to quantum interference effects that would modify the channel conductance and be controllable by the applied gate potential, as predicted by Hess et al[1]. Using conventional EBL technology Warren et al[2] have fabricated a buried periodic gate structure having 2000 Å periodicity that showed interference effects at < 4.2 K. In order to raise the temperature scale for these effects the grating period must be reduced. We intend to use STM nanolithography coupled with coarse translation to accomplish this goal. Our first approach is to pattern H-passivated silicon surfaces in the manner developed by Dagata et al[3], in which the STM probe voltage is elevated sufficiently to break down the passivation layer, creating a locally oxidized (or oxynitrided) region. Shown in figure 6 are examples of a grid of 200 Å wide lines on a 1000 Å pitch and a square patterned in this manner. The next step is to locate and pattern the gate region of a partially fabricated MOSFET. To this end we are collaborating with Prof. Ted Higman at the University of Minnesota to obtain suitable device structures. Ted received his Ph.D. at Illinois and is now

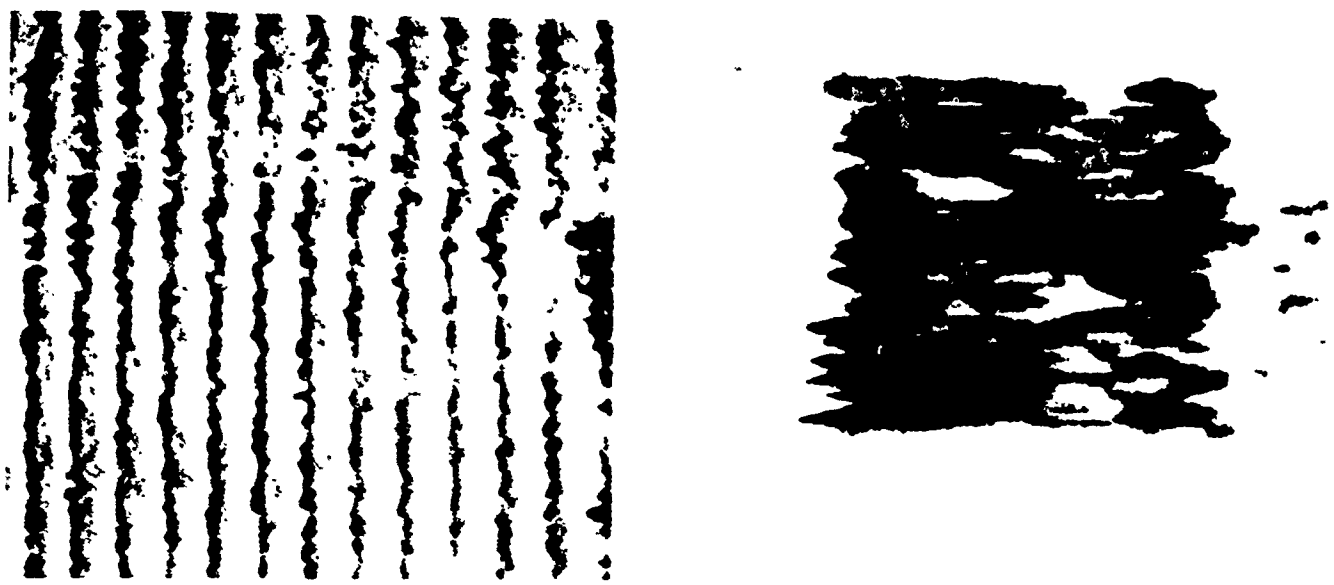


Fig. 6 STM patterned features on a H-passivated silicon surface a) grid with 1000 Å period, and b) a 1μm x 1μm square.

working within the state-of-the-art silicon fabrication facility at Minnesota. Figure 7 shows an SEM image of one of Higman's MOSFETs, the corresponding STM image of the gate region, and an STM-patterned grid written in the gate region after H-passivation of the surface. We are now evaluating various means to transfer these patterns into finished devices. An interesting approach is based on work by Dagata's group[4] in which they observed selective MBE nucleation of GaAs outside of STM-patterned areas on silicon substrates. Apparently the patterned areas withstood the 800°C treatment normally used to remove SiO_2 in preparation for MBE growth. This intriguing result indicates that perhaps an oxynitride is formed by STM-patterning. It is well known that silicon nitride is more durable than SiO_2 with regard to thermal treatments such as oxidation. This suggests the interesting possibility of performing selective oxidation using the STM-generated pattern as a mask. If, for example, the oxidation rate is a factor of two or so lower for the patterned areas, then there would be a considerable modulation of gate oxide thickness for the 100 Å thick oxide needed for one of Higman's MOSFETs. We are now testing the possibility of selective oxidation by performing dry oxidation of silicon substrates that have been pre-patterned by STM and we plan to publish this work in the near future.

Another approach to modifying silicon surfaces is to work with atomically clean surfaces under UHV conditions. Although direct surface modification of silicon has been accomplished by mechanical indentation[5] and more recently by field induced motion of clusters of atoms[6], these methods are limited to the top few layers of atoms and would provide little thickness modulation if a subsequent oxidation is performed. On the other hand, with the gas dosing array of our UHV-STM system it should be possible to control the adsorption of potential etchants or deposition species onto clean surfaces and then use the high electric field of the STM to activate the chemical process. A specific reaction we are interested in is the dissociation of silane (SiH_4) to deposit silicon. It may be possible to grow features to any desired thickness in the manner used by McCord and Awschalom[7] to grow high aspect ration iron pillars by STM dissociation of $Fe(CO)_5$.

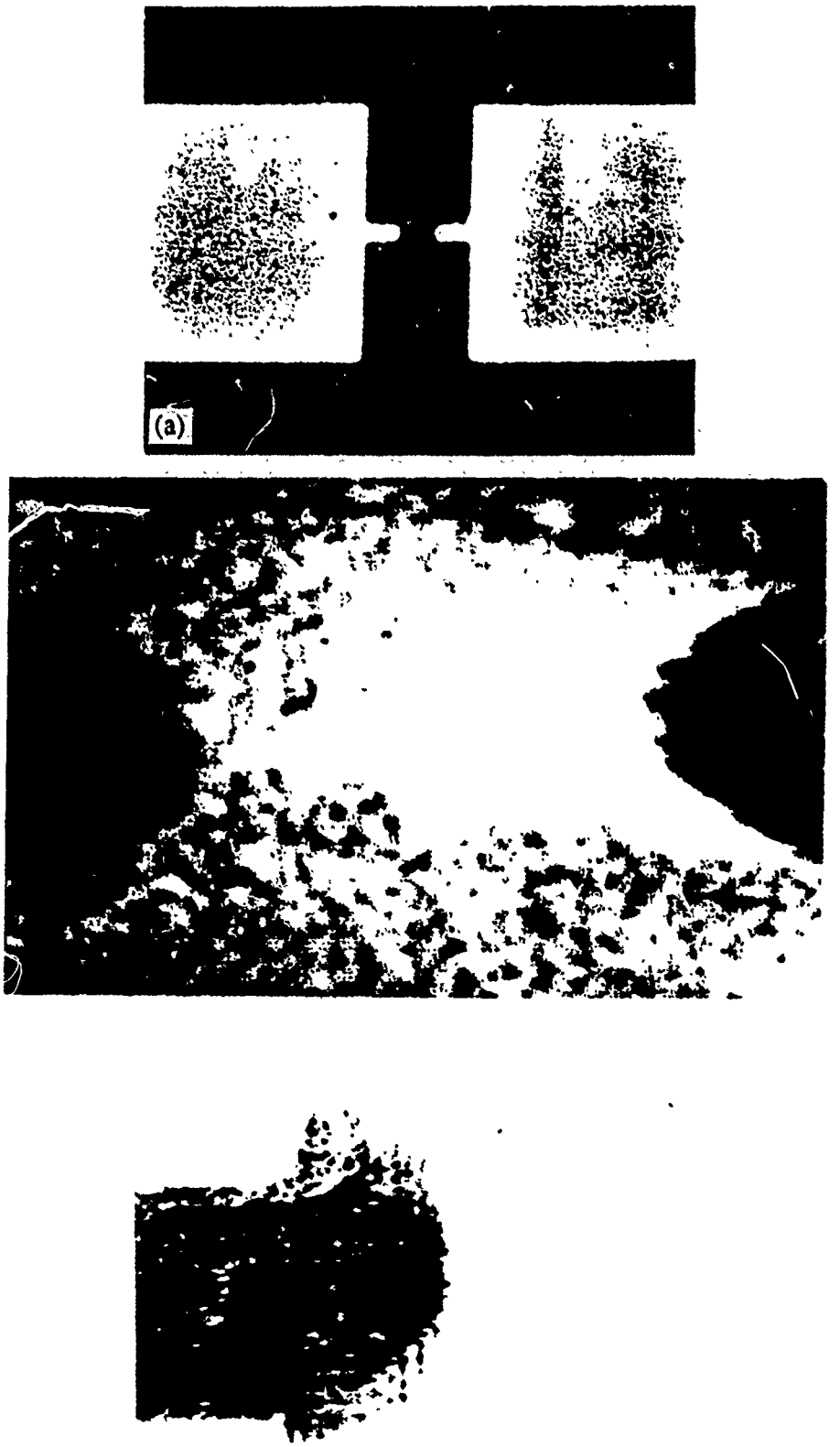


Fig. 7 a) SEM image of a Si-MOSFET fabricated for STM nanolithography, b) an STM image of the gate region, and (c) an STM-patterned grid in the gate region.

To avoid interface roughness scattering of the MOSFET channel electrons it is desirable that the Si/SiO_2 interface be relatively unperturbed. Therefore, gate thickness modulations accomplished by selective oxidation or deposition are preferable to etching or direct mechanical modification of the interface. We are assessing the possibility of growing a thin oxide on Si(100) surfaces under UHV conditions followed by silicon deposition from silane as described above in order to preserve interface quality. To date we have successfully tracked the oxidation of Si(100) with atomic resolution and will be submitting a paper on this subject in the near future. It will also be interesting to attempt direct nitridation of atomically clean silicon by adapting Dagata's techniques for passivated silicon. We have spoken to Dagata about collaborating on this experiment and have invited him to work with us in our laboratory.

As device dimensions shrink below ~1000Å, electron interference effects can play an important role as discussed above. In addition, however, the near continuum of electron energy levels existing in most present day device structures becomes more discrete as feature sizes shrink, and, in fact, the coulomb charging energy $e^2/2C$ due to individual electron tunneling can dominate both the thermal energy k_BT and the energy scale $\hbar/RC$ of quantum fluctuations. This effect has led to the proposal of a 'single electronics' technology by Likharev[8] in which, for example, individual electrons would be information bits in a logic array of single electron devices. Although the prospect of logic devices based on single electrons is intriguing, there are associated practical considerations such as fanout and the possibility of high bit error rates for single electron operation. In view of these potential problems, Prof. John Tucker at Illinois has proposed a robust logic scheme[9] utilizing coulomb charging effects to achieve CMOS logic functionality without the obvious pitfalls of single electron operation. Tucker's scheme translates directly into planar technology in the simplest possible form in that the basic circuit element is a double junction structure in which a small metal island is separated from adjacent electron reservoirs by planar tunnel junctions. In practice this could be achieved by using STM nanolithography to cut small

gaps across metal lines that have been patterned by EBL. In this planar double junction structure electrons would tunnel through the reduced barrier of the metal/substrate system onto and off of the island. A nearby gate electrode would control the Fermi level of the central island and hence the tunneling probability that controls current flow. A suitable system for this would be epitaxial aluminum that is MBE grown onto GaAs. Prof. K.Y. Cheng at Illinois is an expert at MBE growth and is preparing such structures for this purpose. We have already begun work with Cheng's group on the technologically important issue of transferring samples from their MBE systems to our UHV-STM. Although future plans call for a 'vacuum suitcase', it should be possible to protect the surface by As-capping as was done by Pashley et al[11]. The transfer of As-capped samples into our UHV-STM has been successfully tested as shown by the atomic resolution image in figure 8. This image shows the GaAs(001) c-8x2 reconstruction which we created by flashing off the As while simultaneously monitoring the LEED screen. Good STM of this reconstruction has

180x180 Å^2

Fig. 8 STM image of the GaAs(001) c-8x2 reconstructed surface prepared by flashing off a protective As capping layer in the UHV-STM system. The

sample was grown in Prof. K. Y. Cheng's MBE system at the University of Illinois.

not been reported yet so we are preparing a paper in collaboration with Cheng's group. The same experiment will be performed in the near future for As-capped epitaxial Al on GaAs in hopes of obtaining atomically clean Al surfaces for nanolithography work.

We are also working with Prof. I. Adesida at Illinois to develop STM nanolithography schemes that are compatible with his extensive EBL and RIE capability. One approach we are exploring is to see whether suitable passivation schemes can be developed to protect aluminum surfaces for air and UHV-STM work. We are about to test the P_2S_5-based scheme that Dagata et al[10] have used to passivate AlGaAs/GaAs heterolayer structures for air operated STM work. As with H-passivated Si they found that the STM can locally modify the passivation resulting in local oxidation suitable for RIE processing. If this works well for aluminum surfaces then we envision Adesida's group prepatterning planar circuit structures in which STM and RIE will be used to fabricate the small lateral tunneling junctions. Again we intend to collaborate with Dagata's group and also C. R. K. Marrian and R. J. Colton at NRL, as they have considerable experience with STM nanolithography and especially RIE pattern transfer of STM-generated features.

Real-Time Supercomputer STM Data Processing

The Beckman Institute is ideally suited for high-speed distributed computing and is the home for the National Center for Supercomputer Applications, under the direction of Prof. Larry Smarr. NCSA has taken an active interest in providing real-time image analysis and high end 3D graphics to experimental research efforts and has developed large suites of software to this end. Working with Clint Potter at NCSA, our STM experiments are now online with this capability. In practice, the experimental operator can analyze STM data sets immediately after they are acquired, without leaving the experiment or having to know de-

tails of the data transfer protocol. Analysis options are presented in mouse selectable menu format. Figure 9 demonstrates this capability by showing a Si(111) 7x7 STM data set and its corresponding 2D FFT calculated online by NCSA's Convex computer and then displayed on our experimental monitor.

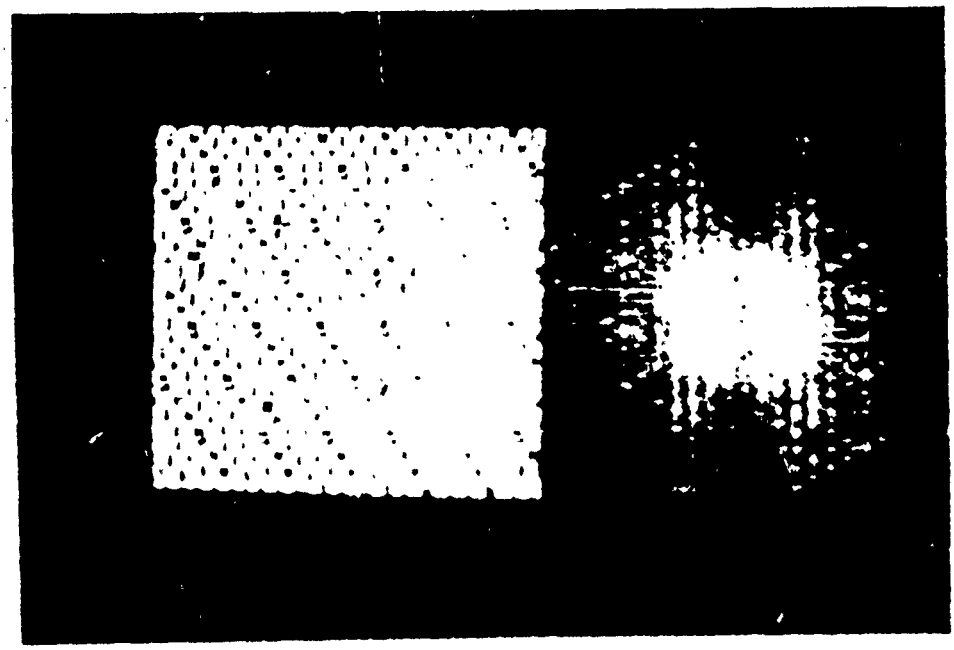


Fig. 9 STM image of the Si(111) 7x7 surface and its corresponding 2D FFT calculated and displayed online with the experiment.

In addition to tying into NCSA's Cray and Convex machines, this scheme will also include our Ardent graphics supercomputer acquired with ONR funds from a DURIP grant in 1988. NCSA has ported their image processing software to run under the application visualization system (AVS) developed by Ardent (now Stardent). In addition, we have recently received shipment of a high end graphics workstation from Hewlett Packard via a proposal submitted to their educational grants program ($160,000 award). This system is also supported by NCSA and will also become an integral part of our network.

Undergraduate Research

We continue to involve undergraduates in various aspects of our STM research effort. Even the best undergraduate students need considerable experience in a 'real' environment before they are capable of contributing in a meaningful manner to research and development. The following is a list of undergraduate projects and students involved with us over the last year.

Design, construct, and test an entirely new STM control electronics box: Geoff Maxson and Thomas Tomazin, both Electrical Engineering.

Build a digital integrator for the STM feedback loop: Kyle Drewry, Electrical Engineering.

Write an AutoLisp program to automate the AutoCad design of UHV-STM chambers: Alfred Pierce (minority), Mechanical Engineering.

Design a 32-bit interface board for the EISA bus specification: Jim Janninck, Electrical Engineering.

Figure 10 shows a photograph of students at work testing the new STM control electronics. Geoff Maxson is now a graduate student with us and will obtain his Masters Degree for this project.

Fig. 10 Graduate students Jerome Hubacek (foreground) and Geoff Maxson testing our new modular STM control electronics.

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