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Chapter 1. Submicron Structures Technology and Research

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1.1 Submicron Structures Laboratory
The Submicron Structures Laboratory at MIT develops techniques for fabricating surface structures with linewidths in the range from nanometers to micrometers and uses these structures in a variety of research projects. These projects of the laboratory, which are described briefly below, fall into four major categories: (1) development of submicron and nanometer fabrication technology; (2) nanometer and quantum-effect electronics; (3) crystalline films on non-lattice-matching substrates; and (4) periodic structures for x-ray optics, spectroscopy and atomic interferometry.

1.2 Microfabrication at Linewidths of 100 nm and Below
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A variety of techniques for fabricating structures with characteristic dimensions of 0.1 μm (100 nm) and below are investigated. These include: x-ray nanolithography, holographic lithography, achro-
matic holographic lithography, electron-beam lithography, focused-ion-beam lithography, reactive-ion etching, electroplating, and liftoff. Development of such techniques is essential if we are to explore the rich field of research applications in the deep-submicron and nanometer domains.

X-ray nanolithography is of special interest because it can provide high throughput and broad process latitude at linewidths of 100 nm and below. Figure 1 shows the replication of a 100 nm period grating (40 nm linewidths) using the C\textsubscript{k} x-ray at 4.5 nm. We are developing a new generation of x-ray masks made from inorganic membranes, primarily Si\textsubscript{N\textsubscript{x}}, in order to eliminate pattern distortion and avoid mask breakage during handling. Figure 2 shows our most recent mask architecture. The mesa rim is composed of Si (what remains of a Si wafer that has been etched away). The Si\textsubscript{N\textsubscript{x}} membrane is under moderate tension and is optically flat to better than 0.25 \(\mu\text{m}\).

To achieve gaps of 5 \(\mu\text{m}\) and below, we can use spacer studs on the mesa rim. Such gaps are routinely achieved and allow us to replicate sub-100 nm features using the Cu\textsubscript{L} line at 1.34 nm. To achieve multiple-mask alignment we currently use a dark field optical imaging system. In the future, in order to produce alignments compatible with 50 nm linewidths, we will fix the mask-sample gap at 4 \(\mu\text{m}\), translate the mask piezoelectrically, and detect alignment to < 10 nm by a dual-grating interferometric scheme.

Phase-shifting x-ray masks should permit us to achieve sub-50 nm linewidths at gaps ~ 4 \(\mu\text{m}\). In previous studies, we showed that a pi-phase-shifting mask improves process latitude by increasing the irradiance slope at feature edges. For linewidths below 50 nm, we bring the mask membrane into soft contact with the substrate by electrostatic means.

A variety of techniques are used to pattern the x-ray masks including e-beam lithography, focused-ion-beam lithography (FIBL), holographic lithography and sidewall shadowing. Figure 3 shows the process used, and figure 4a shows the result of e-beam lithography in a collaborative effort with S. Rishton of IBM. Using a single-layer resist, 250 nm thick, we were able to expose 50 nm lines and spaces of a quantum-effect device pattern and subsequently electroplate 200 nm of gold, suitable for the Cu\textsubscript{L} x-ray at 1.34 nm. Reduced electron back-scattering from the 1
**E-Beam Lithography (Single Resist Layer)**

E-beam Exposure (50keV)

- Au plating base
- SiNx membrane
- PMMA

Au Plating (200 nm-thick) - 50 nm

200 nm of Au→10 dB atten. @ 1.34 nm

**Figure 3.** Schematic depiction of process used to make x-ray masks of 50 nm lines and spaces. The e-beam lithography depicted (left) achieves fine pitch by virtue of the thin (1 μm) substrate which reduces backscattering. The electroplating (right) is done under conditions that produce zero stress.

μm-thick SiNx membrane played a crucial role in achieving such a result. This mask was then replicated with x rays and the “opposite polarity” pattern obtained (figure 4b).

We have further developed the achromatic holographic lithography (AHL), which enables us to achieve 100 nm period gratings (50 nm nominal linewidth). New anti-reflection resists have been developed and tested. This technology will be used to make gratings for x-ray spectroscopy and atom beam interferometry, and to fabricate new classes of quantum-effect electronic devices.

### 1.3 Improved Mask Technology for X-Ray Lithography

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- Semiconductor Research Corporation
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- U.S. Navy - Naval Research Laboratory
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In order to utilize x-ray lithography in the fabrication of submicron integrated electronics, distortion in the x-ray mask must be eliminated. Distortion can arise from stress in the absorber, which is usually gold or tungsten. Tungsten is preferred because it is a closer match in thermal expansion to Si, SiC, SiN, and other materials used as mask membranes. However, W is usually under high stress when deposited by evaporation or sputtering. Earlier, we demonstrated that for a given type of substrate, zero stress (i.e., less than 5 x 107 dynes/cm²) can be achieved by controlling the sputtering pressure to within one-tenth of a millitorr. This year we have developed a computer-controlled system for monitoring *in situ* during deposition, the stress in sputtered W on x-ray mask membranes. Stress is determined from the resonant frequency of the membrane. By monitoring the membrane resonant frequency during deposition and taking into account the mass...
loading and temperature shifts, we can achieve zero stress (i.e., below 5 x 107 dynes/cm$^2$).

We are also investigating mask membranes including: SiN$_x$, SiC, and laminates of SiO$_2$/Si$_3$N$_4$. The strongest membranes were Si rich Si$_3$N$_4$. A 1.2 $\mu$m thick membrane of this material can sustain a full atmosphere pressure differential across a span of 20 mm. Because of its unusual strength we now use SiN$_x$ as a vacuum window, 20 mm in diameter. We have also investigated the radiation hardness of SiN$_x$ (in collaboration with the University of Wisconsin) and found very minor changes in resonant frequency as a result of over one million equivalent x-ray exposures.

1.4 Study of Electron Transport in Si MOSFETs with Deep-Submicron Channel Lengths

Sponsor
Joint Services Electronics Program
Contract DAAL03-89-C-0001

Project Staff
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We have continued to use x-ray lithography to fabricate NMOS devices with effective channel lengths down to 50 nm. As channel lengths decrease below about 150 nm, velocity overshoot has been observed both at room and liquid nitrogen temperatures. It appears that a necessary condition for this phenomenon is high surface mobility which we have achieved in our devices by utilizing a sharp, retrograde doping of the channel. Our initial devices used a single, moderately deep boron ion implant followed by a very short thermal activation step that also grew the gate oxide. More recently, we have used indium implants to achieve improved results. Indium, by virtue of its heavier mass, gives much sharper retrograde doping than boron. Also, it is a slower diffuser than boron, allowing more flexibility in subsequent thermal processing. Finally, it tends to segregate and diffuse through silicon dioxide, and thus it is better suited to give a low interface doping concentration.

Record saturated transconductances (710 mS/mm) were obtained with the new In-doped devices. This underscores the achievement of increased surface mobility with the steeper retrograde channel doping. Velocity overshoot and reduction of impact ionization rate with channel length reduction, which were observed earlier in boron-doped NMOS devices, were also observed in the In-doped devices.

During this reporting period we have also developed a technology for self-aligned silicided NMOS device fabrication. We have used cobalt deposition on the exposed silicon of source/drain and gate electrodes, with a subsequent two-step (450°C and 750°C) rapid thermal annealing, to form CoSi2 self-aligned to the exposed silicon. Thin oxide or silicon nitride spacers around the gate electrode have been used to prevent shorts between sources/drains and gates. These process improvements were tested first with conventional lithography where the short gate was achieved by resist erosion in an O2 plasma after resist exposure and development. More recently, an inorganic x-ray mask technology was developed and has allowed us to use x-ray lithography for the definition of the gates. Fabrication of devices with the new technologies is now in progress.

Work is also in progress to develop a corresponding deep-submicron self-aligned PMOS process. This should give us 100 nm-channel-length CMOS circuits fabricated by an x-ray lithography technology compatible with commercial mass production.

### 1.5 Studies of Electronic Conduction in One-Dimensional Semiconductor Devices

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**Project Staff**

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Sophisticated processing techniques and advanced lithography have allowed us to enter what we believe is a fundamentally new regime in the study of electronic conduction in one-dimensional systems. A slotted-gate MOSFET structure (figure 5) was used to produce an electron gas at the Si/SiO2 interface beneath the gap in the lower-gate. This was done by biasing the upper gate positively, while keeping the slotted gate just below threshold. Fringing fields around the lower gate confined the electron gas to a width substantially narrower (~ 25 nm) than the distance separating the two halves of the slotted gate (~ 70 nm). The slotted gate was produced using x-ray nanolithography and liftoff. It was composed of refractory metals to allow a subsequent high temperature anneal. This anneal removed damage created by the e-beam evaporation of the refractory metal, so that the electron gas had a mobility of 15,000 cm²/V-sec at 4.2K. The electrical conductance of the 1-D gas was measured as a function of the upper gate voltage for temperatures less than 1K, and a surprising series of periodic oscillations was seen in the conductance (figure 6).

Changing the gate voltage can be thought of as changing the number of electrons per unit length of electron gas. Since the conductance is thermally activated, the oscillations reflect a periodic change in the activation energy of the electron gas as the electron density is changed. Computer simulations solving Poisson's equation and the single particle Schrodinger wave equation strongly suggest that the electron gas is dynamically one-dimensional when the oscillations are most strongly seen. That is, the electrons are in the lowest quantum energy level of the potential well created by the fringing fields of the slotted gate.
The period of the oscillations varies randomly from device to device. Additionally, the period changes when the same device is heated to room temperature and then cooled. This suggests that impurities are responsible for the conductance oscillations. The impurities delimit a segment of the channel that is capacitively coupled to the gates of the device. The electrostatic energy required to move an electron into this segment (and hence carry current) changes periodically with the gate voltage. This so-called Coulomb Blockade model of the oscillations is strongly supported by recent experiments in similar GaAs devices. It should be stressed that these oscillations are seen with no magnetic field, implying that the phenomenon is fundamentally different from phenomena requiring Landau quantization (such as the Quantum Hall Effect).
1.6 Lateral-Surface-Superlattice and Quantum Wire Arrays in Si

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Project Staff
Professor Dimitri A. Antoniadis, Phillip F. Bagwell, Professor Terry P. Orlando, Professor Henry I. Smith

We have been studying quantum mechanical effects in electrical conduction using the silicon grating gate field effect transistor (GGFET). The Si GGFET is a dual stacked-gate MOS type structure in which the gate closest to the inversion layer (bottom gate) is a 200 nm period grating made of refractory metal. A SiO2 insulating layer separates the grating gate and the inversion layer from a second continuous aluminum gate (top gate). Using this dual gate structure, we can gradually vary the electron geometry in the inversion layer from many narrow wires in parallel, to a superlattice, and to a two-dimensional electron gas.

Electron weak localization becomes much more pronounced as the device is electrostatically pinched from a 2D inversion layer into many narrow 1D wires in parallel, proving that the wire width can be reduced below the electron phase coherence length. For fixed intermediate magnetic field of 1-10 Tesla, there is a large drop in the current of 90% or more, which persists to room temperature as electrons are added to the device, so that it opens electrostatically from many narrow inversion layers in parallel into a 2D electron gas.

Chapter 1. Submicron Structures Technology and Research

1.7 Study of Surface Superlattice Formation in GaAs/GaAlAs Modulation Doped Field-Effect Transistors

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We have used the modulation-doped field-effect transistor (MODFET) as a test vehicle for studying quantum effects such as electron back diffraction in a GaAs/AlGaAs material system. In a conventional MODFET, the current transport is modulated by a continuous gate between source and drain. In our studies, we have used Schottky metal gratings and grids for the gate, as illustrated in figure 7. Such gates produce a periodic potential modulation in the channel.

The grid was produced by x-ray nanolithography and liftoff. The x-ray mask of the grid was produced by two successive x-ray exposures at 90 degrees to one another, using a master mask that was fabricated via holographic lithography. The latter yields coherent gratings over areas several centimeters in diameter. A new technique was developed that yields grating and grid patterns only in the channel region between source and drain. This has simplified the overall process and enhanced its reliability.

The MODFET is normally on; that is, a negative gate bias of about −0.2 V must be applied to pinch off conductance from source to drain. As the gate bias is raised above this threshold point, the height of the periodic potential modulation is reduced and, simultaneously, the Fermi energy is raised (or, equivalently, the electron wavelength is reduced) in the 2D electron gas residing at the AlGaAs/GaAs interface. When the electron wavelength phase-matches the periodic potential, electron back-diffraction occur provided the inelastic length (i.e., the coherence or phase breaking length) is longer than the grating-period. Such back diffraction is manifested by a drop in the conductance. A stronger back diffraction effect is observed in the case of a grid because true mini-gaps are formed. The measurements of conductance modulation of grating and grid-gate MODFETs agrees with the theoretical predictions. In the grid gate devices it was also possible to
observe negative differential resistance which might be due to sequential resonant tunneling.

We plan to decrease the periodicity of the gratings and grids by a factor of two, to 100 nm period. For devices with such fine grating periodicity, the superlattice effect might become more pronounced and observable at higher temperatures. We will also conduct magnetotransport measurements with devices of 100 and 200 nm periodicity.

We will also take advantage of a back-gate technology, illustrated in figure 8. This will give us the ability to independently control the confining potential experienced by the electrons, as well as the electron density in the channel. This will allow for much more quantitative understanding of device operation.

Figure 7. Schematic cross section of a grid-gate MODFET device. Contacts to the grid are made by pads off to the sides of the conduction channel.

Figure 8. Schematic showing a cross section of our new configuration for studying grid-gate or grating-gate MODFETs and arrays of quantum wires. The substrate is n⁺ doped, allowing us to apply a back bias to sweep the Fermi energy while keeping the potential modulation constant.
1.8 Study of One-Dimensional Subbands and Mobility Modulation in GaAs/AlGaAs Quantum Wires

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Project Staff
Professor Dimitri A. Antoniadis, Phillip F. Bagwell, Dr. Keith Evans, Reza A. Ghanbari, Dr. Khalid Ismail, Professor Terry P. Orlando, Professor Henry I. Smith

In order to study one-dimensional conductivity in the AlGaAs/GaAs modulation-doped structure, but without the conductance fluctuations normally associated with single microscopic systems, we previously fabricated arrays of 100 parallel quantum wires (MPQW) by etching the wires into the MODFET structure. The devices were then ground thin from the back side so that the charge concentration in the quantum wires could be increased by applying a positive bias to a backside contact or by illumination. The devices were not optimal because the degree confinement was set by the etch and not electrostatically. Also, thinning the samples was haphazard at best.

To overcome these difficulties, we have developed a technology that allows us to electrostatically confine the electrons to QID channels. In parallel, in collaboration with K. Evans at the Wright-Patterson Air Force Base, we are developing the technology to give us the backside gating by growing the epitaxial layers on n+ GaAs instead of the traditional semi-insulating GaAs. A schematic of the device is shown in figure 8.

Using this approach, we can explore the regime from a regular 2D gas \( V_{TG} \approx 0 \) to weakly coupled QID wires \( V_{TG} \approx 0.5V \) to strongly isolated QID wire \( V_{TG} < 1V \), while at the same time using the backgate to sweep the electron density and hence, “probe” the confining potential.

1.9 Arrays of Field-Effect-Induced Quantum Dots

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A metal grid on a modulation-doped AlGaAs/GaAs substrate (depicted in figure 9a) produces a two-dimensional periodic potential modulation at the AlGaAs/GaAs interface via the Schottky effect. If a gate electrode is attached to the grid, the potential can be further modified with an external voltage source. By changing the gate voltage from positive to negative values, the potential seen by the electrons located at the AlGaAs/GaAs interface can be varied from uniform (in which case the electrons behave as a 2-D electron gas), to weakly coupled zero-D quantum wells (figure 9b), to isolated zero-D quantum dots (figure 9c). We have made such structures with spatial periods of 200 nm in both orthogonal directions using technology similar to that described in Section 1.7, but now the grid gate occupies an area of several square millimeters. The isolated quantum dots and the attendant zero-dimensional electronic subbands were examined in collaboration with D. Tsui at Princeton University using far-infrared (FIR) cyclotron resonance. Transitions between the discrete energy levels in the quantum dots were observed as a function of magnetic field. Results were in agreement with a theoretical model.

Currently, we are continuing our study using extremely high quality samples prepared by M. Shayegan’s group at Princeton. With typically greater than \( 10^{6} \text{cm}^2/\text{Vsec} \), the resolution of the experiments should improve dramatically.

We are currently fabricating a new set of grid-gate MODFETS, using an improved fabrication process and will study their transport, capacitance, and absorption properties as a function of magnetic field.
Chapter 1. Submicron Structures Technology and Research

1.10 Planar-Resonant-Tunneling Field-Effect Transistors (PRESTFET)

Sponsor
U.S. Air Force - Office of Scientific Research
Grant AFOSR 85-0154

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Previously, we reported on the performance of a planar-resonant-tunneling field-effect transistor (PRESTFET) depicted in figure 10, in which the gate electrodes were 60 nm long and separated by 60 nm. Clear evidence of resonant tunneling through the bound states in the well between electrodes was observed, as shown in figure 10b. In order to reduce the electrode separation while retaining a large process latitude, we have chosen to pursue a new technology for making the PRESTFET. In collaboration with S. Rishton of IBM, a high-performance e-beam nanolithography system was used to write PRESTFET patterns on SiN x-ray mask membranes, 1 µm thick. Reduced backscattering from the thin membrane allows finer linewidths to be obtained, as shown in figure 3. The written masks are then processed and replicated at MIT. We have succeeded in making and replicating masks with PRESTFET patterns of 50

Figure 9. (a) Metal grid gate on a modulation-doped AlGaAs/GaAs substrate; (b) Depiction of potential seen by electrons at the AlGaAs/GaAs interface for weakly coupled quantum dots; (c) Potential for the case of isolated quantum dots.

Figure 10. (a) Layout of a 4-terminal double-barrier planar-resonant-tunneling field-effect transistor (PRESTFET). (b) Plot of source-drain current versus gate voltage for a PRESTFET with 60 nm well width.
nm linewidth. The masks can be aligned to GaAs substrates using an adapted deep-UV aligner and exposed with the CuL x-ray (1.3 nm). Lift-off of appropriate Schottky electrodes will complete the device fabrication.

1.11 Submicrometer-Period Transmission Gratings for X-Ray and Atom-Beam Spectroscopy and Interferometry

Sponsors
Joint Services Electronics Program
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X-OPT, Inc.

Project Staff
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Transmission gratings with periods of 0.1-1.0 nm are finding increasing utility in applications such as x-ray, vacuum-ultraviolet, and atom-beam spectroscopy and interferometry. Over 20 laboratories around the world depend on MIT-supplied gratings in their work, and this project constitutes the sole source for these diffractors. For x-ray and VUV spectroscopy, gratings are made of gold or tungsten and have periods of 0.1-1.0 µm and thicknesses ranging from 0.1-1 µm. They are most commonly used for spectroscopy of the x-ray emission from high-temperature plasmas. Transmission gratings are supported on thin (1 µm) polyimide membranes or made self supporting (“free standing”) by the addition of crossing struts (mesh). (For short x-ray wavelengths, membrane support is desired, while for the long wavelengths a mesh support is preferred in order to increase efficiency.) Fabrication is generally performed by holographic lithography, x-ray lithography and electroplating. Progress in this area tends to focus on decreasing the period and improving the yield and flexibility of the fabrication procedures.

Another application is the diffraction of long-de Broglie-wavelength (0.17Å) neutral sodium beams by mesh-supported gratings. Professor Pritchard’s group at MIT has clearly demonstrated atomic diffraction with these gratings, and work is in progress to use these gratings to divide and recombine an atomic beam coherently, thus realizing an atom wave interferometer. Because good spatial coherence (low distortion) of the grating is critical to ensure measurable interference of the beams, efforts are concentrating on fabrication with low stress and high stiffness materials such as tungsten, silicon nitride, and silicon oxide.

1.12 High-Dispersion, High Efficiency Transmission Gratings for Astrophysical X-Ray Spectroscopy

Sponsor
National Aeronautics and Space Administration
   Contract NAS8-36748

Project Staff
Professor Claude R. Canizares, Dr. Mark L. Schattenburg, Professor Henry I. Smith

This work involves a collaboration between the Center for Space Research and the Submicron Structures Laboratory (SSL), providing transmission gratings for the Advanced X-ray Astrophysics Facility (AXAF) x-ray telescope, currently scheduled for launch in 1998. Many hundreds of low-distortion, large area transmission gratings of 0.2 µm period (gold) and 0.6 µm period (silver) are required. These will provide high resolution x-ray spectroscopy of astrophysical sources in the 100 eV to 10 keV band.

Because of the requirements of low distortion, high yield, and manufacturability, a fabrication procedure involving the replication of x-ray masks has been selected. Masks are made of high-stiffness silicon nitride membranes to eliminate distortion. Masks are patterned using a process involving holographic lithography, reactive-ion etching, and electroplating. The masks are then replicated using soft x-rays (10 - 15 Å), and the resulting patterns electroplated with gold or silver. An etching step then yields membrane-supported gratings suitable for space use. Flight prototype gratings have been fabricated and continue to undergo space-worthiness tests. Progress in this area focuses on increasing the yield and flexibility of the fabrication procedures and perfecting various mask and grating evaluation tests.
Chapter 1. Submicron Structures Technology and Research

1.13 Epitaxy via Surface-Energy-Driven Grain Growth

**Sponsor**
AT&T Bell Laboratories

**Project Staff**
Jerrold A. Floro, Professor Henry I. Smith, Professor Carl V. Thompson

Epitaxial grain growth (EGG) in polycrystalline thin films on single crystal substrates is being investigated as an alternative process for obtaining and studying epitaxy. EGG can produce smoother ultra-thin epitaxial films than those produced in conventional epitaxy and may yield lower defect densities as well. In addition, EGG can produce unique non-latticed-matched orientations not observed in conventional epitaxy.

The mechanism of epitaxial grain growth is simple. The anisotropic film/single-crystal substrate interfacial energy selects one film crystallographic orientation as having lowest total free energy. Grains in this orientation have the largest driving force for growth and will predominate as the system coarsens.

We have continued our work on model materials systems, i.e., metals on mica and alkali halides. We made extensive use this year of x-ray pole figure analysis for quantitative measurement of texture and epitaxy. Using this technique we measured the epitaxial fraction transformed versus film thickness, verifying the rate of EGG increases with decreasing film thickness as predicted by theory.

In order to achieve perfect epitaxy, the EGG process must be highly orientation selective, with a small fraction of iso-orientation grains in the initial population growing extremely large. We have shown that by proper treatment of a mica substrate surface prior to deposition, the selectivity can be greatly increased, and the final grain size can be as large as 50 μm, an order of magnitude larger than previously obtained. This increased selectivity is apparently due to modification of the mica surface chemistry.

We have performed extensive numerical analysis of EGG using mean field coarsening theory, we are trying to determine under what conditions (interface energy, boundary pinning, etc.) significant selectivity in grain growth rates can occur.

1.14 Publications

**Journal Articles**


Chapter 1. Submicron Structures Technology and Research


Published Meeting Papers


Meetings Papers Presented


Chapter 1. Submicron Structures Technology and Research


Theses


Chapter 1. Submicron Structures Technology and Research

Professor Henry I. Smith explains the development of an alignment system for x-ray nanolithography that should be capable of 100-angstrom precision.