

Chapter 4. Submicron and Nanometer Structures Technology and Research

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4.1 Submicron Structures Laboratory

The Submicron Structures Laboratory at MIT develops techniques for fabricating surface structures with feature sizes in the range from nanometers to micrometers and uses these structures in a variety of research projects. These projects, 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.

4.2 Microfabrication at Linewidths of 100 nm and Below

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Project Staff

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A variety of techniques for fabricating structures with characteristic dimensions of 100 nm (0.1 μm)

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and below are investigated. These include: x-ray nanolithography, holographic lithography, achromatic 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. As discussed in section 4.3, we have established a reliable mask technology, based on 1 μm -thick SiN_x membranes, as shown schematically in figure 1. Mask blanks are patterned by several techniques including electron-beam lithography, focused-ion-beam lithography, holographic lithography, and transfer from other x-ray masks.

For high-quality x-ray lithography, the mask-to-sample gap, G , must be precisely controlled. The mesa-rim architecture of figure 1 yields optically flat membranes enabling us to control gap down to about 2 microns by means of spacer studs. For 100 nm features a gap of 10 microns is suitable. However, for 50 nm features a gap of 2.5 microns is required. Figure 2 shows the replication of a planar-resonant-tunneling field-effect transistor pattern having 50 nm minimum features. The gap in this case was 2.7 microns. This gap is significantly larger than predicted by simple Fresnel diffraction formulae which neglect diffraction inside the mask absorber. Taking this into account, using vector electromagnetic calculations and

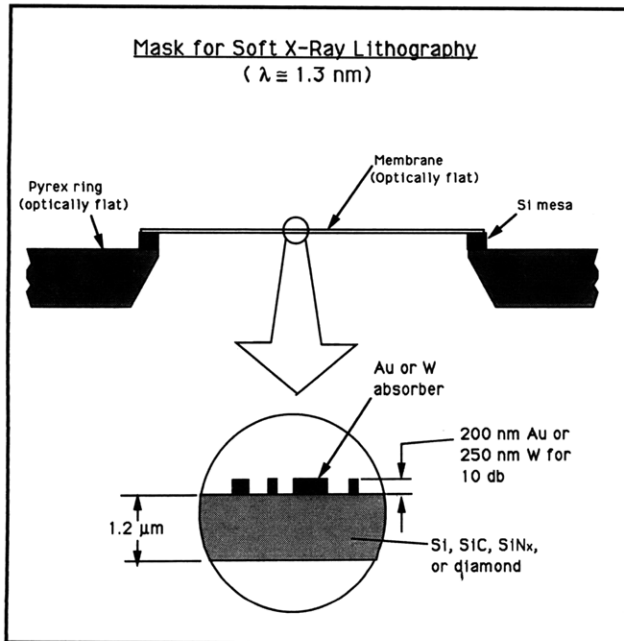
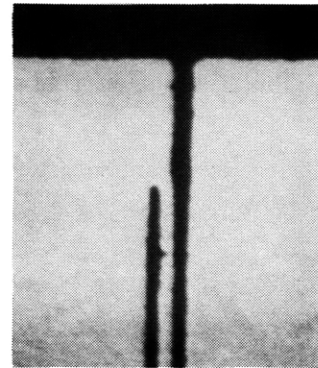


Figure 1. Structure of the new standard, 31 mm diameter, mesa-rim, x-ray mask suitable for lithography from 1000 to 20 nm feature sizes. Membranes are flat to $< 0.25 \mu\text{m}$.

Proximity X-ray Nanolithography



Electrode Pattern PRESTFET *

50 nm

- 50 nm lines and spaces
- $\lambda = 1.32 \text{ nm}$ $G = 2.72 \mu\text{m}$
- $G = \alpha W^2 / \lambda$
- $\alpha = 1.44$
- Implication: if $W = 0.1 \mu\text{m}$, $\lambda = 1 \text{ nm}$
- $G = 10 \text{ to } 15 \mu\text{m}!$

* PRESTFET \rightarrow Planar Resonant-Tunneling Field-Effect Transistor

Figure 2. Replication, using the Cu_L x-ray (1.3 nm) and a mask-substrate gap of 2.7 microns, of 50 nm line and space pattern.

accounting for the finite source size, we have developed a simulation that predicts image contrast as a function of feature size and gap. In order to increase the allowable gap for features of 70 nm and below, we are investigating the feasibility of near-field in-line x-ray holography.

Using the figure 1 mask architecture, patterns are readily transferred onto standard Si wafers. However, in research on GaAs or other III-V devices, substrates are often much smaller than the 31 mm-diameter mask membrane. We have developed an apparatus that allows us to bring the membrane into contact with such substrates during exposure, without risk of mask damage. Resolution down to 10-20 nm is feasible under such exposure conditions.

Alignment of x-ray masks to patterns pre-existing on a substrate is currently done with an optical imaging system equipped with a video camera. We are investigating electronically enhanced moiré imaging methods in order to achieve sub-10 nm alignment precision. In this approach the gap is

set at ~ 4 microns and the mask translated piezoelectrically.

The achromatic holographic lithography system in conjunction with an anti-reflection coating (ARC) specially formulated for the 193 nm wavelength is now producing high quality images, as shown in figure 3. The ARC consists of PMMA and a bis-azide, 4, 4'-diazidodiphenyl sulfone.

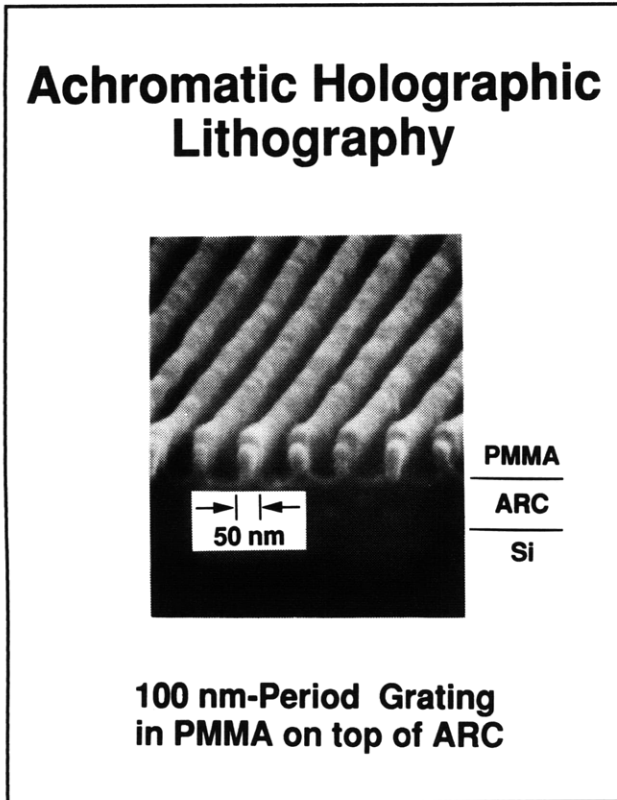


Figure 3. A 100 nm-period grating exposed in PMMA on top of an anti-reflection coating (ARC) by achromatic holographic lithography using the ArF laser at 193 nm.

4.3 Improved Mask Technology for X-Ray Lithography

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Project Staff

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The x-ray mask architecture shown in figure 1 is compatible with both our research in the sub-100 nm domain and the commercial fabrication of submicron integrated electronics. For the latter application especially, distortion in the mask must be eliminated. Distortion can arise from stress in the absorber, which is usually gold (Au) or tungsten (W). We have developed means of sputtering W so that the stress is below 5×10^7 dynes/cm², and the distortion induced by stress is below the level of detectability in a Linnik interferometer. The corresponding in-plane pattern distortion is calculated to be less than 1 nm. We are currently developing an interferometer to directly measure in-plane distortion.

Membranes of SiN_x are preferred at the present time because they can be made by conventional LPCVD methods and are extremely rugged and resistant to breakage. (SiN_x membranes only 1 micron thick can even be used as vacuum windows, 20 mm in diameter.) Membranes are characterized by their optical transmission, resonant frequency and bulge under pressure. From these measurements we extract index of refraction, optical attenuation, thickness, stress, and Young's modulus. We have also made radiation hardness measurements on membranes by subjecting them to the equivalent of one million exposures on the University of Wisconsin synchrotron. Effects of x radiation on membrane stress are extremely small, and we believe this can be eliminated by reducing the oxygen content of the SiN_x. To this end a special purpose LPCVD reactor for low-stress SiN_x will be set up within the Integrated Circuit Laboratory at MIT.

We are collaborating with the Naval Research Laboratory (NRL) in using their JEOL JBX5D11 e-beam lithography system to pattern x-ray masks. The patterns are generated using the CAD tools at MIT, converted to an acceptable binary intermediate format, transferred to NRL via ARPA Internet, converted there to the JEOL format, and written onto mask blanks that we send in advance to NRL (by express mail). Once the masks are written, they are shipped back to MIT for further processing. This collaboration has been fruitful for a number of projects including short-channel MOSFETs (section 4.4), multiple-parallel one-dimensional conductors (section 4.6), and the electron waveguide device (section 4.7).

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

Sponsors

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Project Staff

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We have continued using x-ray lithography to fabricate both N and P channel MOSFET devices with effective channel length down to 80 nm. As channel lengths decrease below about 150 nm, electron velocity overshoot, and thus an increase of device transconductance has been observed both at room temperature and liquid nitrogen temperature. It appears that the necessary conditions for this phenomenon are high carrier mobility close to the Si/SiO₂ interface, and low parasitic resistance. The former is achieved in both our NMOSFET and PMOSFET devices by utilizing a super steep retrograde doping of the channel. In the NMOSFET case, indium, by virtue of its heavier mass and desirable doping profile in silicon, has been used to give retrograde threshold control doping with low surface concentration and yet high peak concentration. In the PMOSFET case, arsenic has been used to achieve the same goals.

Record saturation transconductance of over 1S/mm in deep submicron NMOSFET with channel length down to 100 nm was obtained with the indium doping technique. This underscores the achievement of increased surface mobility with the steeper retrograde channel doping. A transconductance of 0.23 S/mm was achieved in deep-submicron PMOSFET.

As shown in figure 4 we have developed a technology for self-aligned silicide N/PMOSFET device fabrication for the purpose of significantly reducing parasitic resistance. We have used cobalt deposition on the exposed silicon of source/drain and the exposed polysilicon of the gate electrode with a subsequent two-step rapid thermal annealing to form cobalt silicide (CoSi₂). Thin oxide spacers around the gate electrode have been used to prevent shorts between gate and source/drain. These process improvements were

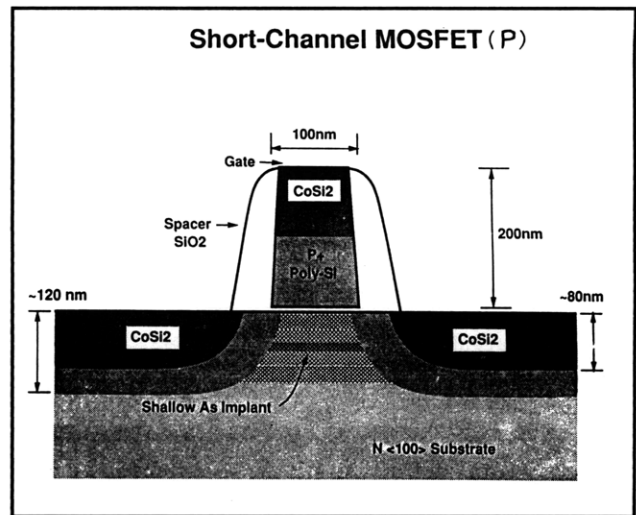


Figure 4. Configuration of self-aligned, 100 nm-channel-length, P-MOSFET.

tested first with conventional lithography where the submicron gate was achieved by resist erosion in an O₂ plasma after resist exposure and development. The first ultrashort channel self-aligned silicide MOSFET devices with channel length down to 0.12 μm for PMOS and 0.15 μm for NMOS and parasitic resistance down to 400 ohms per micron width have been successfully fabricated.

More recently, SiN_x-membrane x-ray mask technology has allowed us to use x-ray lithography for the definition of the gates. X-ray masks were patterned by electron-beam lithography at the Naval Research Laboratory, as shown in figure 5. Fabrication of both NMOSFET and PMOSFET devices with the new lithography technology and new cobalt silicide shallow junction source/drain technology should give us 100 nm channel length CMOS circuits compatible with commercial mass production.

4.5 Studies of Coulomb Charging in Ultrasmall Semiconductor Devices

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Project Staff

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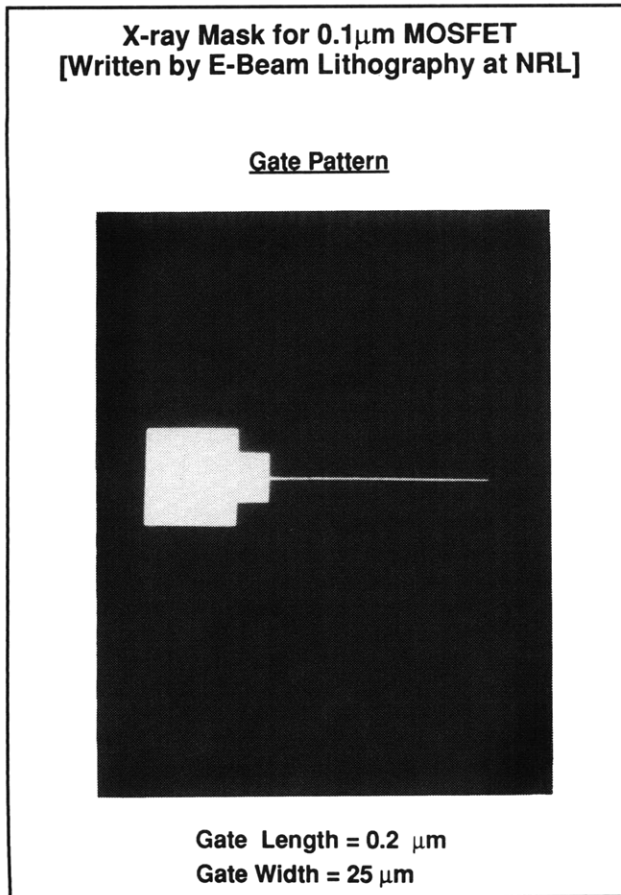


Figure 5. Scanning electron micrograph of x-ray mask for the 100 nm channel-length MOSFET. The e-beam patterning was done at the Naval Research Laboratory, the plating and x-ray lithography was done at MIT.

In previous studies on very narrow silicon MOSFETs fabricated using x-ray lithography a fundamentally different mechanism to modulate the low-temperature electrical conduction was found. When the devices were cooled to the milli-Kelvin range, the electrical conductance exhibited a series of sharp oscillations which were almost perfectly periodic in the voltage of the gate used to control the electron density. This periodic modulation of the conductance was thought to be caused by presence of two dominant impurities in the electron channel which resulted in an isolated segment of the electron gas. In order to test the two-impurity hypothesis, the structure illustrated in figure 6(a) was fabricated using electron beam lithography and liftoff. The structure, fabricated on an inverted GaAs/AlGaAs heterostructure, consists of two Schottky gates used to squeeze the electron gas laterally, while the n⁺ substrate serves as a bottom gate to independently modulate the channel electron density. The two sets of constrictions were intended to emulate the potential due to the two impurities in the MOSFET device,

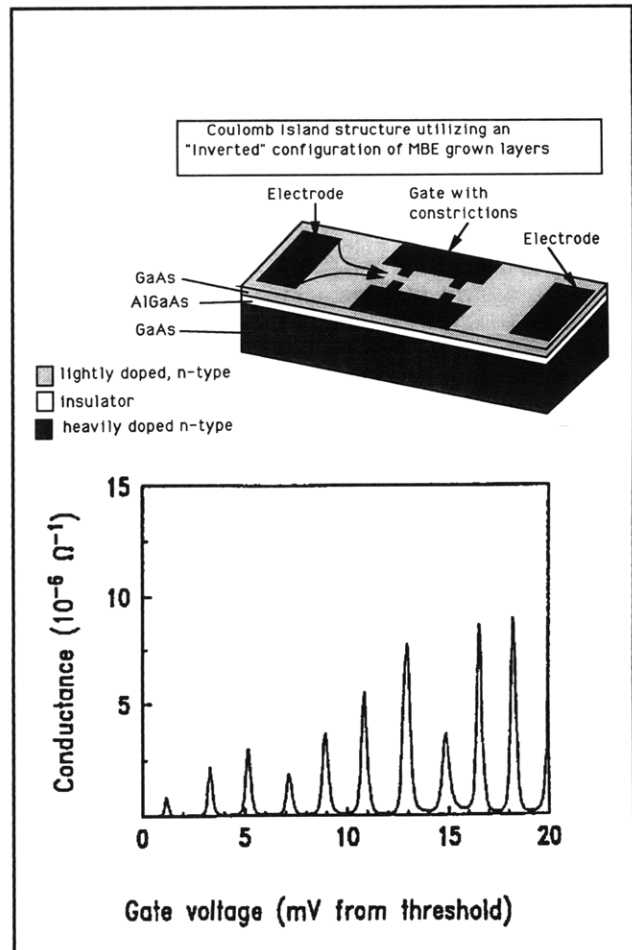


Figure 6. (a) Device structure used to study Coulomb blockade effects. A negative voltage on the two Schottky gates on the top depletes the electron gas at the lower GaAs/AlGaAs interface below, forming a narrow electron channel interrupted by two potential barriers at the constrictions. The n⁺ GaAs substrate serves as a bottom gate to modulate the channel electron density. (b) Periodic oscillations in the conductance as a function of bottom gate voltage for the device shown in (a).

while the mobility was sufficiently high to reduce the probability of unwanted accidental impurities.

When cooled to milli-Kelvin temperatures, conductance oscillations periodic in the bottom gate voltage were again observed, as shown in figure 6(b). Moreover, the voltage separation between successive conductance peaks was found to depend on the separation between the two potential barriers. To better understand these structures, we performed semi-classical calculations of potential profiles and electron densities for the structure in figure 6(a). Equipotential contours near the AlGaAs/GaAs interface are shown in figure 7 for a typical bias point with the shaded regions indicating areas occupied by inversion electrons.

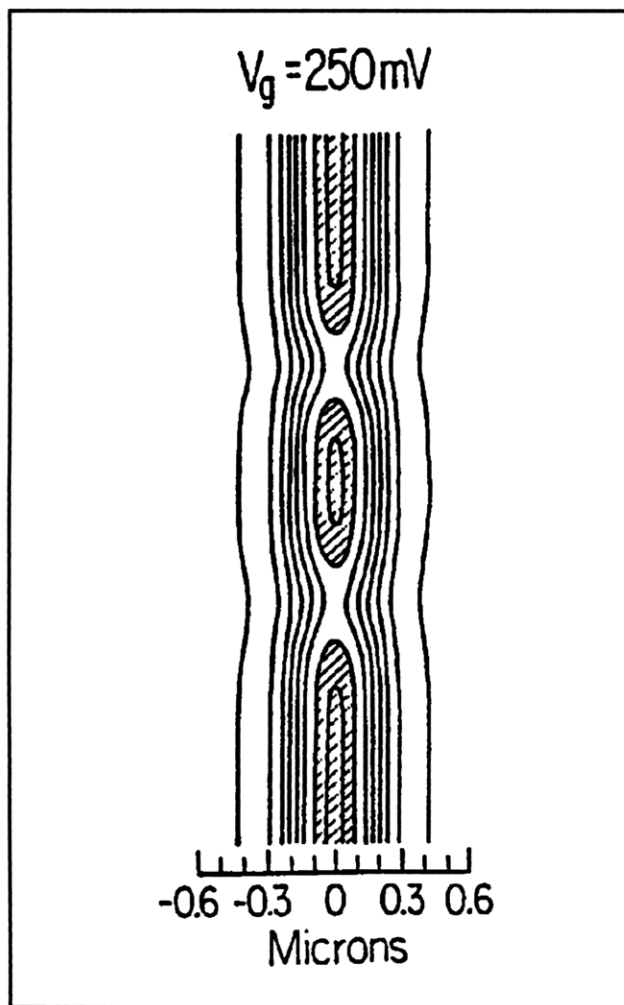


Figure 7. Equipotential contours for the device in figure 6(a) near the lower GaAs/AlGaAs interface at a typical bias point. The shaded regions indicate areas occupied by electrons. The two sets of constrictions result in an isolated electron segment, or "island," through which transport can be thermally activated.

We calculated the integrated electron charge in the isolated electron segment, or "island," between the sets of constrictions as a function of bottom gate voltage and found that it is approximately linear in bottom gate voltage, and that the voltage required to add one electron to the island is very nearly the spacing between successive conductance peaks. Thus, each conductance peak corresponds to the addition of a single electron to the island. This modulation of the conductance by changing the charge by a single electron can be understood in terms of a theory known as Coulomb blockage. According to the Coulomb blockade theory, a conductance valley corresponds to the electrostatic energy of the island being minimized by an integer number of electrons, so that addition of another electron costs some finite charging energy and transport is thermally activated. A conductance peak, on the other hand, corresponds to a state of

the island where the electrostatic energy is minimized by a half-integer number of electrons, so that there is no energy barrier to adding another electron. These two states repeat cyclically in bottom gate voltage, resulting in the observed periodic modulation of the conductance.

We are presently enhancing our understanding of Coulomb blockade effects both experimentally and theoretically. We first plan to reproduce the effect in a conventional modulation-doped GaAs/AlGaAs heterostructure, which is much more widely available than the inverted heterostructure. Secondly, we are working to understand all the capacitance components to the island in addition to the bottom gate capacitance described above. This is important because the observability temperature for Coulomb effects depends inversely on the total capacitance of the island, so that reducing the island capacitance is key to observing these effects at temperatures above the milli-Kelvin regime. Finally, we plan to study experimentally transport through more than one dot as well as the coupling of dots in parallel.

Because the modulation of the conductance is so strong, Coulomb blockade is among the most promising of novel effects in ultrasmall structures for long-term applications.

4.6 Study of Quasi-One-Dimensional Wires and Superlattice Formation in GaAs/AlGaAs Modulation Doped Field-Effect Transistors

Sponsor

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Project Staff

Professor Dimitri A. Antoniadis, Martin Burkhardt, William Chu, Professor Jesús A. del Alamo, Reza A. Ghanbari, Professor Terry P. Orlando, Professor Henry I. Smith

We have continued to use the modulation-doped field-effect transistor (MODFET) to explore electron back diffraction effects and reduced dimensionality effects in the GaAs/AlGaAs system. Because such devices have critical dimensions on the order of 100 nm, the use of x-ray lithography for fine feature definition has been central to our strategy. Recent results in our laboratory indicate that there may be a significant advantage in using x-ray lithography over direct-write e-beam lithography to fabricate quantum-effect electronic

devices because of significantly less mobility degradation in x-ray exposed samples.

Quasi-one-dimensional (Q1D) wires are defined on modulation-doped GaAs/AlGaAs material by first aligning the substrate and x-ray mask optically and then exposing. The region between the resist lines is then etched away chemically. After

etching, the areas under the etched regions are depleted of electrons, leaving behind many parallel quasi-one-dimensional channels between source and drain. A continuous Schottky gate is then deposited over the wires, as depicted in Figure 8, to allow control of the electron density in the wires. A scanning-electron micrograph of a typical device is shown in Figure 9.

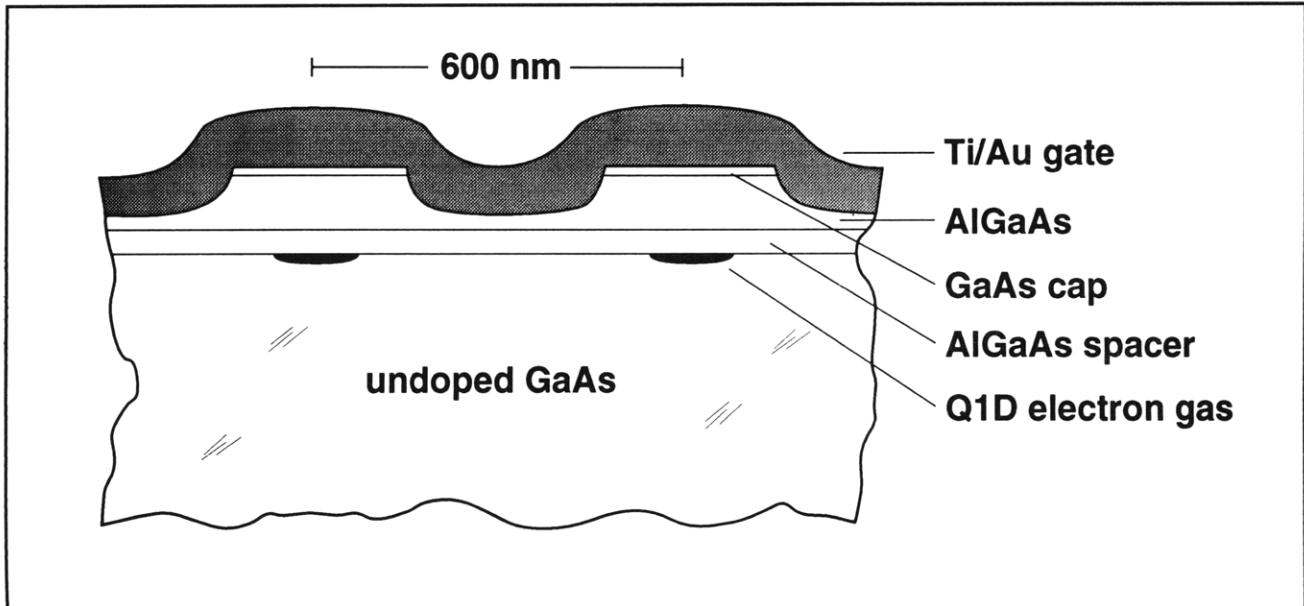


Figure 8. Cross-sectional schematic of device for studying conduction in multiple parallel quasi-one-dimensional quantum wires.

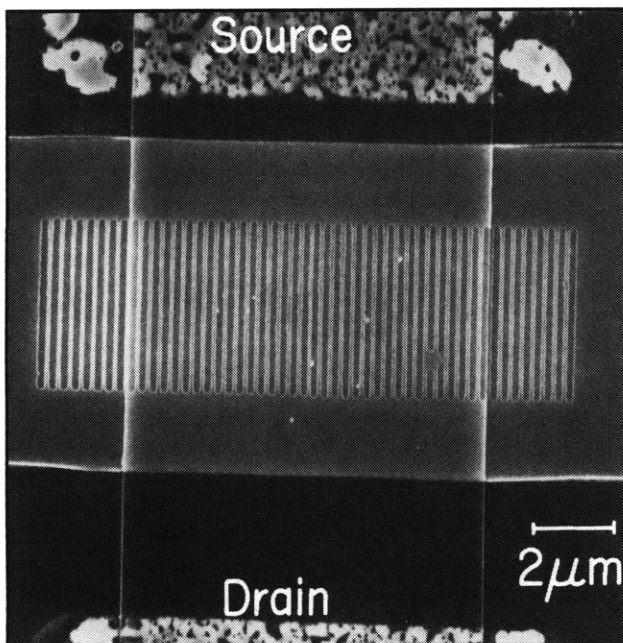


Figure 9. Scanning electron micrograph of device depicted in figure 8.

We have also fabricated MODFETs in which rather than having a large continuous gate over the channel, we have a grid gate (periodicity of 200 nm) as shown schematically in Figure 10. As a negative bias is applied to the gate, the regions immediately under the metal lines deplete more quickly than the regions under the open areas of the gate. Thus, electrons travelling between source and drain see a periodic potential. When the electron wave-length matches the periodic potential, coherent back diffraction occurs. Such back diffraction manifests itself as a drop in conductance between source and drain. The stronger the potential modulation, the more pronounced the back diffraction ought to be. We are in the process of optimizing our device design to increase the strength of the potential modulation.

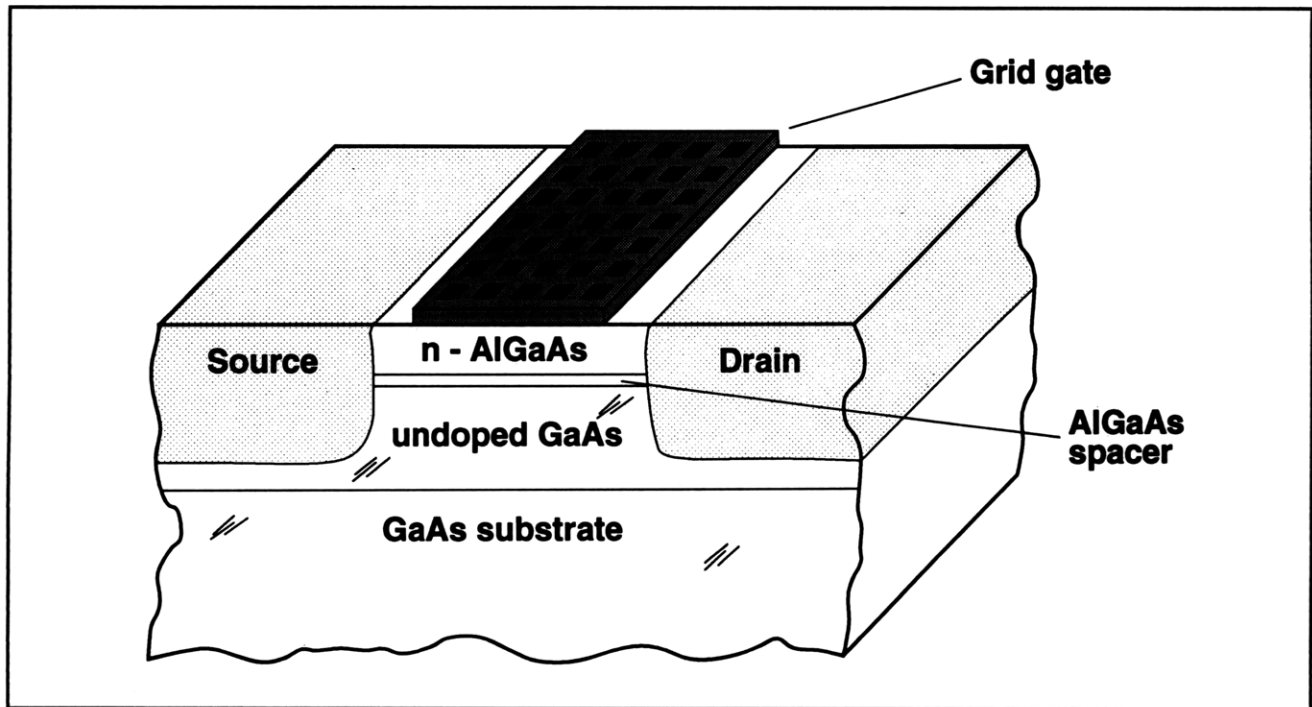


Figure 10. Schematic of grid-gate MODFET for study of surface superlattice formation.

4.7 GaAs Electron Waveguide Devices Fabricated by X-Ray Lithography

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Project Staff

Professor Jesús A. del Alamo, William Chu, Christopher C. Eugster, Alberto M. Moel, Euclid E. Moon, Dr. Mark L. Schattenburg, Professor Henry I. Smith.

We have successfully fabricated electron waveguide devices using x-ray lithography. As shown in figure 11, a split-gate structure constricts the electron gas of a high-mobility AlGaAs/GaAs heterostructure in the region between the gate fingers (length L and width W). If electron wavefunctions are coherent over the length of the device, then transport through this region is considered ballistic, and the structure can be considered an electron waveguide. A clear signature of ballistic transport is quantized conductance as a function of the electron density in this constricted region. Thus, the quality of conductance steps serves as a measure of the quality of the material and the

ability of the processing steps to maintain this quality. The longer the length of devices in which this effect can be observed, the higher the quality of the material.

We have fabricated electron waveguides of length $0.1 \mu\text{m}$ up to $2 \mu\text{m}$ using x-ray lithography. An x-ray mask (parent mask) is fabricated by using e-beam lithography to define the split-gate pattern on a PMMA-coated silicon-nitride (SiN_x) membrane. After development, the absorbers are formed by electroplating 200 nm of Au. A scanning-electron micrograph of the parent mask is shown in figure 12. In order to reverse the mask polarity, the parent mask is replicated by proximity x-ray lithography ($\lambda = 1.3 \text{ nm}$) to generate a replica (daughter) mask. The daughter mask is then aligned to a PMMA-coated AlGaAs/GaAs sample and x-ray exposed.

The finished devices are bonded and then tested at liquid-helium temperatures. Using standard lock-in techniques, the conductance of the electron waveguides is measured as a function of the split-gate bias which controls the electron density in the waveguide. Sharp $2e^2/h$ conductance steps were observed in a $0.75 \mu\text{m}$ -long device at $T=2\text{K}$, as shown in figure 13. The features in the conductance remain visible up to temperatures of 15K. Conductance features were observed in devices as long as $2 \mu\text{m}$.

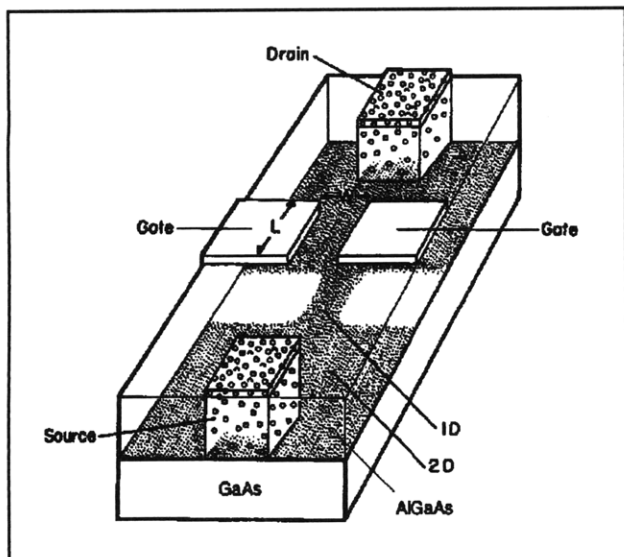


Figure 11. Schematic of an electron waveguide device defined by the split-gate configuration. The shaded region represents the electron concentration at the AlGaAs/GaAs interface.

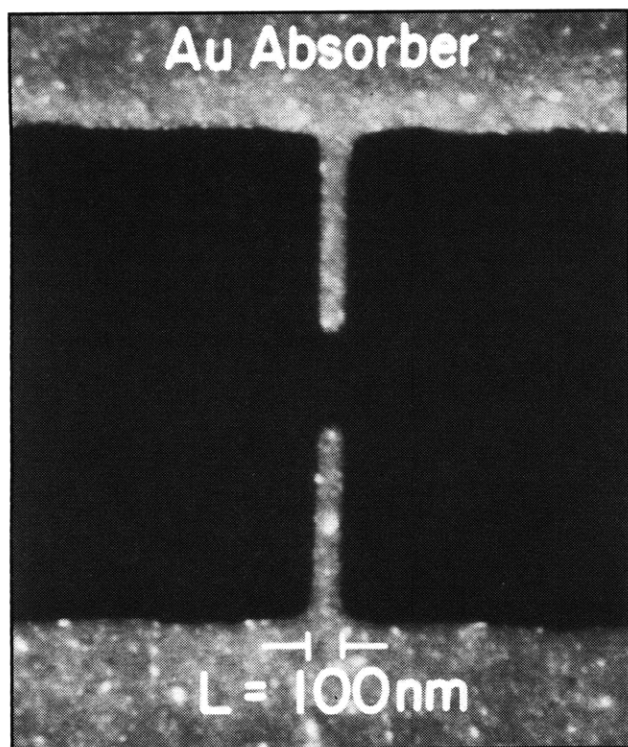


Figure 12. Top view of a parent x-ray mask showing the pattern for a 0.1 μm -long waveguide device.

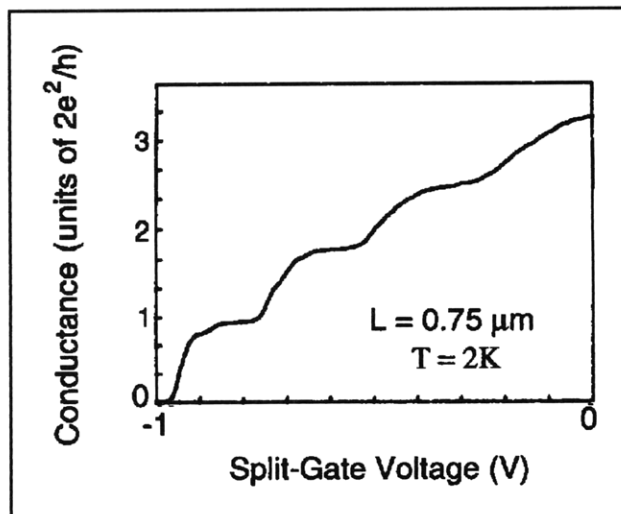


Figure 13. Conductance of a $L = 0.75 \mu\text{m}$ electron waveguide device as a function of split-gate voltage.

4.8 Arrays of Field-Effect-Induced Quantum Dots

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Project Staff

Professor Dimitri A. Antoniadis, Martin Burkhardt, Reza A. Ghanbari, Professor Terry P. Orlando, Professor Henry I. Smith, Professor M. Shayegan,⁴ Professor Daniel Tsui,⁴ Yang Zhao⁴

A metal grid on a modulation-doped AlGaAs/GaAs substrate (depicted in figure 14a) 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 14b), to isolated zero-D quantum dots (figure 14c). We have made such structures with spatial periods of 200 nm in both orthogonal directions and covering

⁴ Princeton University, Princeton, New Jersey.

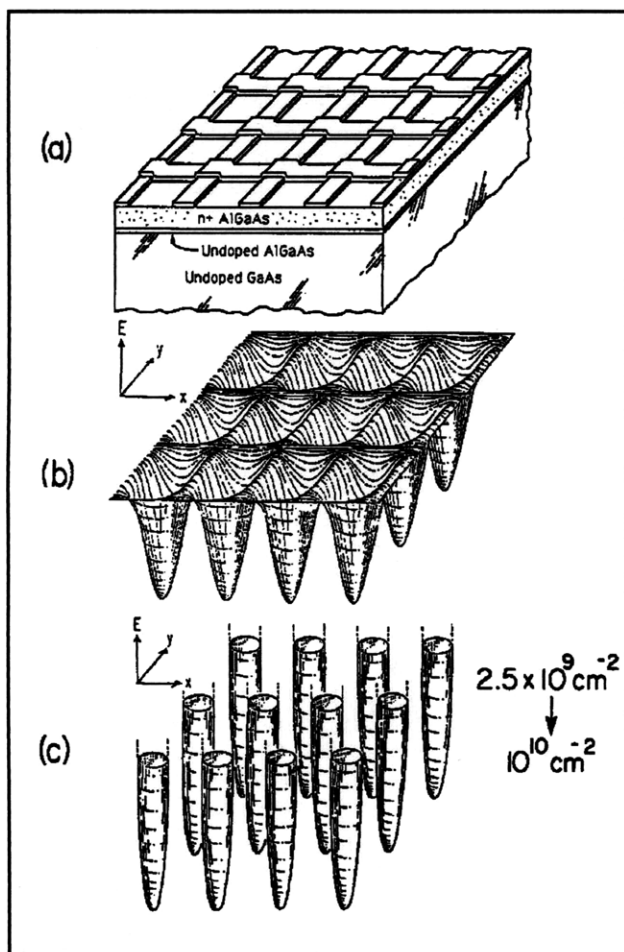


Figure 14. (a) Metal grid gate on a modulation-doped AlGaAs/GaAs substrate; (b) Depiction of potential seen by electrons at the AlGaAs/GaAs interface for weakly coupled quantum dots; (c) Potential for the case of isolated quantum dots.

areas of several square millimeters. The isolated quantum dots and the attendant zero-dimensional electronic sub-bands 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 mobilities typically greater than $10^6 \text{ cm}^2/\text{Vsec}$, the resolution of the experiments should improve dramatically.

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

Sponsor

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Project Staff

Professor Dimitri A. Antoniadis, Mike T. Chou, William Chu, Professor Henry I. Smith

Previously, we reported on the performance of a planar-resonant-tunneling field-effect transistor (PRESTFET) depicted in figure 15, 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 15(b).

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 x-ray mask membranes, 1 μm thick. Reduced backscattering from the thin membrane has permitted us to achieve 50 nm lines and spaces. We hope to get even finer features in the future. The written masks are processed at MIT, replicated using the Cu_L x-ray (1.3 nm), and Schottky electrodes formed by liftoff. Masks are aligned to GaAs substrate using a specially built alignment and exposure station.

4.10 Fabrication of Distributed-Feedback Lasers and Channel-Dropping Filters

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Project Staff

James M. Carter, Woo-Young Choi, Jay N. Damask, Professor Clifton G. Fonstad, Jr., Professor Herman Haus, Professor Leslie A. Kolodziejcki, Professor Henry I. Smith, Vincent V. Wong

We are developing techniques to fabricate large area, spatially-coherent gratings for both quarter-wave-shifted distributed-feedback (DFB) lasers

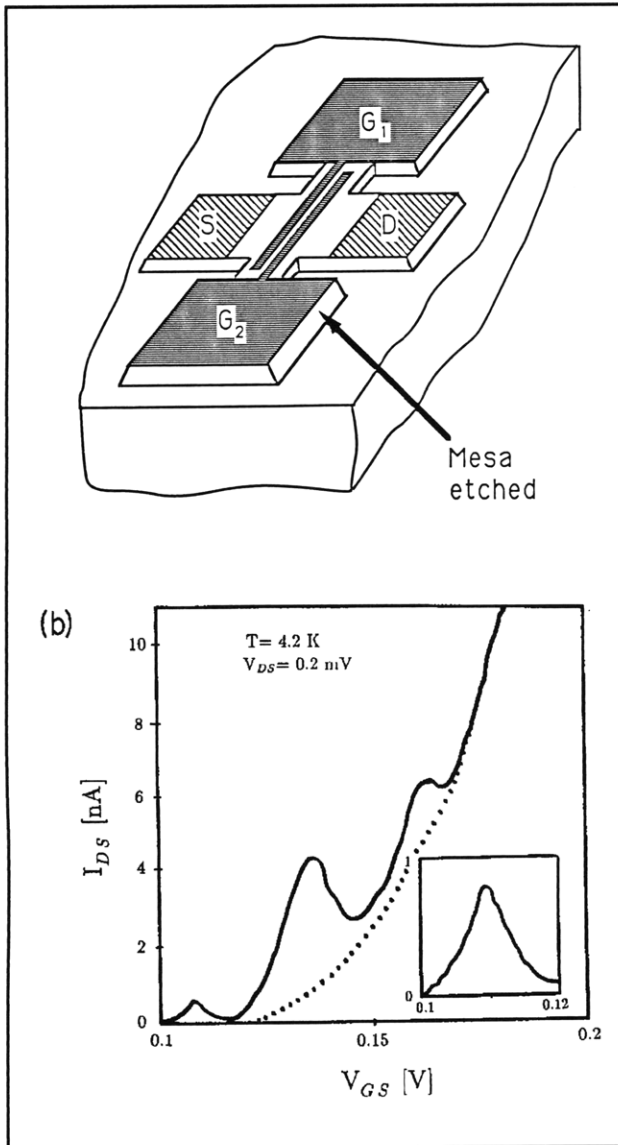


Figure 15. (a) Layout of a 4-terminal double-barrier planar-resonant-tunneling field-effect transistor (PRESTFET). (b) Plot of source-drain current versus gate voltage for a PRESTFET with 60 nm well width.

and channel-dropping filters (CDFs) on InP-based materials. The quality of these gratings is the most important factor determining the performance of these devices. Typical grating periods will be ~ 200 nm, and grating lengths will range from ~ 500 μm to several millimeters. Much of the nanofabrication technology developed in our lab over the years is directly applicable to the fabrication of these devices. SiN_x x-ray masks will be patterned using both holographic lithography and e-beam lithography, as described below. X-ray

lithography will then be used to define the device patterns on the InP-based material.

Through the use of holographic lithography, large-area, spatially-coherent grating patterns can be defined. Fine control of the grating period is also possible. However, the incorporation of the quarter-wave shift into the grating structure is not easily accomplished by holographic means. We are pursuing a technique that takes advantage of a unique aspect of x-ray lithography: the phase of a segment of a grating can be shifted by pi-radians by transferring it onto the back surface of the mask membrane. Grating structures containing single and multiple quarter-wave shifts, which are necessary for both DFB lasers and CDFs, will be fabricated using this technique.

The incorporation of quarter-wave shifts into a grating structure is straightforward with e-beam lithography. However, precise control of the grating period, and large-area, spatially-coherent grating patterns are not easily obtained with e-beam lithography. For precise control of the grating period, a continuously-variable field size is necessary. To achieve spatial coherence in the written gratings one must eliminate the stitching errors, which occur when writing adjacent fields. We are pursuing a technique based on a global-fiducial grid scheme which potentially can solve both of these problems. Once developed, this technique will be incorporated into the e-beam lithography process to define the appropriate DFB laser and CDF grating patterns onto a SiN_x x-ray mask.

4.11 Novel Superconducting Tunneling Structures

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Project Staff

Professor John M. Graybeal, Dr. Bernard S. Meyerson,⁵ George E. Rittenhouse, Professor Henry I. Smith

In this program we seek to examine the behavior of short-channel hybrid Josephson devices, whose geometry is designed to display quantum electronic interference behavior. These devices represent the first attempt to observe Josephson

⁵ IBM Corporation.

coupling via resonant tunneling. The characteristic energy scale for such a device would be set by quantum confinement, and consequently may provide a new avenue for obtaining finite gains in Josephson three-terminal devices.

These novel structures consist of an ultrathin vertical Si membrane (the quantum well) sandwiched between two superconducting Nb counterelectrodes. These structures are fabricated using a combination of x-ray and optical lithography, anisotropic wet chemical etching, planarization and reactive dry etching techniques. The basic geometry of these new structures has now been fully validated using low-mobility uniformly doped Si wafers, where we find Josephson behavior consistent with the best published work on "conventional" Nb/Si/Nb junctions. However, no resonant tunneling effects were expected or observed in these devices due to their low mobility. Present work now centers on the fabrication of similar structures in high-mobility modulation-doped epitaxial layers grown on Si, where we expect resonant tunneling effects to appear.

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

Sponsor

Joint Services Electronics Program
Contract DAAL03-89-C-0001
Contract DAAL03-92-C-0001

Project Staff

James M. Carter, Daniel B. Olster, Dr. Mark L. Schattenburg, Professor Henry I. Smith

Transmission gratings with periods of 100-1000 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. For x-ray and UV spectroscopy, gratings are made of gold and have periods of 100-1000 nm, and thicknesses ranging from 100-1000 nm. 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.) Fabri-

cation is generally performed by holographic lithography, x-ray lithography and electroplating. Progress in this area tends to focus on improving the yield and flexibility of the fabrication procedures.

Another application is the diffraction of neutral sodium beams (de Broglie-wavelength ~ 17 pm) by mesh-supported gratings. Professor Pritchard's group at MIT has clearly demonstrated atomic diffraction and interference. Because good spatial coherence (low distortion) of the grating is critical to ensure measurable interference of the beams, efforts are concentrated on the use of holographic lithography and the reactive-ion etching of free-standing gratings in low stress and high stiffness materials such as silicon nitride. Results are shown in figure 16.

4.13 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, Robert C. Fleming, 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 200 nm period (gold) and 600 nm 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, and patterned using a process involving holographic lithography, reactive-ion etching, and electroplating. The masks are then replicated using soft x-rays (1-1.5 nm) 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 tends

Free-Standing 200-nm Grating in Si₃N₄

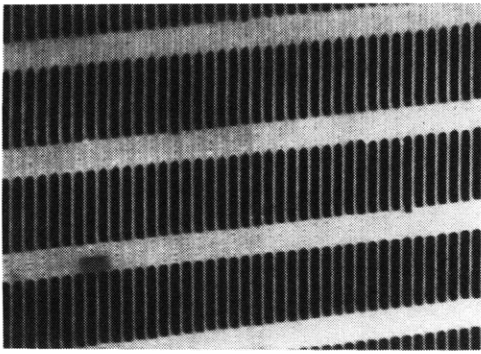


Figure 16. Scanning electron micrograph of free-standing Si₃N₄ grating fabricated for use in an atom interferometer.

to focus on increasing the yield and flexibility of the fabrication procedures, and the perfection of various mask and grating evaluation tests.

4.14 Submicron-Thickness X-Ray Window Technology

Sponsor

National Aeronautics and Space Administration
Grant NAGW-2003
Contract NAS8-36748

Project Staff

Robert C. Fleming, Seppo Nenonen, Dr. Mark L. Schattenburg, Professor Henry I. Smith

We have been investigating various schemes for fabricating leak-free, large-area, ultrathin membranes to serve as vacuum isolation windows for the transmission of x-rays. Applications include gas-filled x-ray detector windows and high-vacuum isolation windows for x-ray lithography. Current window technology uses relatively thick beryllium windows which are opaque to x-ray with wavelengths in the 0.5 - 1.5 nm band. However, this band is very useful for x-ray detector and x-ray lithography applications. Current efforts seek to perfect large-area polyimide windows which have a 1.0 micron thickness. When combined with tungsten or nickel meshes, these have been made leak free to the limit of He leak detector technology. Woven tungsten support meshes are

used, and also advanced nickel meshes made by deep-etch x-ray lithography (so-called LIGA process), in collaboration with the MicroParts company in Germany. Future efforts will seek to reduce the membrane thickness still further and also experiment with silicon nitride membranes which promise to be leak free and also bakeable for high vacuum applications. A silicon nitride isolation window of preliminary design is already being used as a vacuum isolation window in our laboratory x-ray aligner.

4.15 Epitaxy via Surface-Energy-Driven Grain Growth

Sponsor

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Project Staff

Dr. Paul D. Bristowe, Roland Carel, 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 ultrathin epitaxial films than those produced in conventional epitaxy and may yield lower defect densities as well. Compared to conventional epitaxy, EGG is a near-equilibrium transformation process that can probe the true energy-minimizing orientation of an overlayer on a single crystal substrate. In film/substrate systems that are weakly interacting or have large lattice mismatch the minimum energy orientation is often *not* that which is obtained during 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 are studying EGG in polycrystalline Ag films on single crystal Ni substrates (or vice-versa). This system is useful for fundamental study because the variation in interfacial energy with Ag/Ni crystallographic orientation can be calculated computationally using well-established embedded atom potentials. Thus, special low energy orientations can be predicted and then searched for in epitaxial grain growth experiments.

Extensive numerical analysis of EGG using mean field coarsening theory has been performed. We have examined model interface energy functions (interface energy versus orientation) and shown which of the specific aspects of the interface energy function are important in promoting bimodal secondary or epitaxial grain growth, i.e., grain growth which is strongly orientation selective.

4.16 GaAs Epitaxy on Sawtooth-patterned Si

Sponsor

Joint Services Electronics Program
Contract DAAL03-89-C-0001
Contract DAAL03-92-C-0001
Spire Corporation

Project Staff

Professor Henry I. Smith, Kenneth Yee, Dr. Khalid Ismail, Nasser Karam⁶

The growth of GaAs on Si offers the possibility of combining high-speed and optoelectronic GaAs devices with Si integrated-circuit technology. Oriented gratings of 200 nm period are fabricated in Si₃N₄ on (100) Si substrates. Anisotropic etching in KOH is then used to produce sawtooth-profile gratings in the Si. These then serve as substrates for GaAs growth by MOCVD. The dislocation density in the grown GaAs films is orders of magnitude lower than the density in films formed on planar Si substrates.

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