

18. Submicron Structures Fabrication and Research

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18.1 Submicron Structures Lab

The Submicron Structures Laboratory at M.I.T. is developing techniques for fabricating surface structures of nanometer to micrometer linewidths and using these structures in a variety of research projects. This laboratory contributes to an expansion of microsystems research at M.I.T. Submicron structures fabrication has become a distinct discipline with a world-wide and growing community of practitioners. Fabrication techniques include various forms of lithography (optical, electron beam, x-ray, ion beam), etching (aqueous, reactive plasma, sputtering), growth (oxidation, plating, epitaxy), and deposition (evaporation, chemical deposition, sputtering). With these techniques it is possible to fabricate experimental structures with minimum feature sizes smaller than certain characteristic distances important in a variety of scientific fields (e.g., coherence length, mean-free-path, wavelength, grain size, domain size, living-cell diameter). For example, metal wires with widths below 10 nm can be fabricated, and electrical conduction in such structures studied. New insights are needed to understand the behavior of matter at such small dimensions.

Although the scientific applications at small dimensions have motivated some of the research on submicron structures fabrication, the main driving force has come from the integrated circuits industry. Much activity is aimed at developing the technology and the scientific understanding that will bring integrated electronic devices into the submicron domain. The hope is that this will lead to systems of higher performance.

The main research projects of the Submicron Structures Laboratory, fall into four categories: submicron structures fabrication (nos. 2 to 3), electrical properties of submicron structures (nos. 4 to 5), device films on insulating substrates (nos. 6 to 8), and periodic structures and applications (nos. 9 to 12).

18.2 Microstructure Fabrication at Linewidths of 0.1 μm and Below

Joint Services Electronics Program (Contract DAAG29-83-K-0003)

U.S. Navy – Office of Naval Research (Contract N00014-79-C-0908)

National Science Foundation (Grant ECS82-05701)

Erik H. Anderson, Andrew M. Hawryluk, Robert F. Kwasnick, Alan C. Warren, John Melngailis, Henry I. Smith

A variety of techniques for fabricating structures with linewidths of 0.1 μm and below are under development. These include: holographic lithography, x-ray lithography using crystallographic-template masks, spatial-period-division and reactive-ion etching. A variety of holographic techniques have been developed which permit exposure of large-area (~ 5 cm diameter) patterns with improved control of line/space ratio and resist profile. In x-ray lithography with C_K sources the intrinsic resolution is ~ 100 Å. Hence, resolution depends almost exclusively on mask fabrication techniques. Crystallographic templates of triangular and square-wave cross section are etched into Si substrates using anisotropic chemical means. Following techniques developed by Flanders at M.I.T. Lincoln Laboratory, these are replicated into polyimide, which is then shadowed with W to produce the x-ray mask. Linewidth of 30 nm with minimum edge ripple have been obtained. Research on spatial-period-division has shifted from an x-ray to a deep UV approach using a combination of diffraction gratings and multilayer mirrors on SiO_2 and LiF masks. With this technique, and in collaboration with D. Ehrlich of M.I.T. Lincoln Laboratory and R. Osgood of Columbia University, we expect to expose large-area grating patterns having 400 Å linewidths. Reactive-ion etching of structures with linewidths ~ 0.1 μm and below is investigated to improve control of cross-sectional profile and determine contamination effects. This research employs new techniques we developed for preparing samples for TEM analysis.

18.3 Reactive Sputter Etching Studies

I.B.M. (PO No. 90305-QPSA-559)

Joint Services Electronics Program (Contract DAAG29-83-K-0003)

Christopher M. Horwitz

Extensive reactive sputter etching studies have been carried out. As a result, a more complete understanding of electrical properties of the plasma has been achieved, a new type of etching configuration has been discovered, and a new etch for Al and Al-Cu alloys has been demonstrated.

By careful measurements of the peak-to-peak rf voltage and the induced DC voltage on the target electrode, the ion energies at both the target and the chamber walls can be deduced. The effects of

various geometries on the ratio of these two ion energies was investigated.

The power dissipated in the rf discharge has been measured by separating out the power dissipated in the matching network. Thus, for the first time, data on power dissipation in the plasma was determined as a function of peak-to-peak rf voltage, pressure, and system geometry.

The etch rate of SiO_2 in CHF_3 was studied in order to achieve reproducibility. The dependence on gas pressure, flow rate and voltage were measured. Increasing the effective capacitance between rates.

A target configuration with two facing electrodes both at the same target potential (also called a hollow cathode), was investigated. The plasma between these two electrodes is seen to be much more intense than in the normal chamber. At a pressure of 0.3 Pa the etch rate of SiO_2 in CF_4 was found to be 60 times higher than on a conventional open target.

With SiF_4/O_2 gas mixtures, aluminum, Al_2O_3 , and Al-Cu-Si alloys were etched while the horizontal faces of other materials, such as AZ photoresist, were covered with SiO_x . This is potentially an important etch for Al since the gas contains no chlorine and thus problems of corrosion and undercut are avoided.

18.4 Electronic Conduction in Ultra-Narrow Silicon Inversion Layers

Joint Services Electronics Program (Contract DAAG29-83-K-0003)

Robert F. Kwasnick, Marc A. Kastner, John Melngailis

Silicon n-channel field-effect transistors have been fabricated with a gate cross-section of 50 nm x 50 nm and gate lengths of 10 μm or 125 μm . The narrow-gate was fabricated by evaporating aluminum at a glancing angle against a step in the gate oxide. The narrow-gate devices exhibit oscillations in their conductance as a function of gate voltage near gate-voltage threshold, at temperatures $\leq 10\text{K}$. Wide-gate devices identically fabricated on the same (100) silicon wafer exhibit no such oscillations. Detailed experiments are underway to explore the dependence of these oscillations on temperature, drain voltage, substrate bias, and magnetic field. There is evidence that ionic motion on the surface changes the detailed structure of the oscillations. This suggests that the oscillations are associated with strong disorder in the potential experienced by the quasi-one-dimensional electron gas contained in the inversion layer.

18.5 Corrugated–Gate MOS Structures

Joint Services Electronics Program (Contract DAAG29-83-K-0003)

Alan C. Warren, Dimitri A. Antoniadis, John Melngailis, Henry I. Smith

The principal aim of this work is to demonstrate and understand the effects of a periodic potential variation upon conduction electrons in Si inversion layers. MOS transistors have been fabricated with 300 nm period gratings etched in the gate insulator. Initial testing indicates a successful fusion of standard planar technology with the required submicron structures processing, yielding high-quality, well-behaved devices at room temperature. The immediate goal is to test the devices at very low temperatures (~1–3°K) and attempt to demonstrate the quantum effects due to a quasi-one-dimensional density of states (conduction parallel to grating) and due to superlattice dispersion (conduction perpendicular to grating). In the future, we anticipate using the facilities of the National Magnet Laboratory to test the devices. Characterization of the grating with regard to uniformity, line/space ratio and sidewall angle is also being initiated.

18.6 Graphoepitaxy of Si and Model Materials

National Science Foundation (Grant ECS82-05701)

U.S. Navy – Office of Naval Research (Contract N00014-79-C-0908)

Stephane S. Dana, Erik H. Anderson, Carl V. Thompson, Henry I. Smith

Graphoepitaxy, in which an overlayer film is crystallographically oriented with respect to an artificial surface pattern, has been demonstrated. However, to achieve single-crystal films on a variety of substrates, for electronic and optical devices, the mechanisms of nucleation, growth, coalescence and reorientation need to be understood so that orientation-spread and defects can be eliminated. We conduct basic studies of graphoepitaxial mechanisms in silicon, and model materials (i.e., materials that exhibit large interfacial anisotropies and easy reorientation). This research is aimed at developing means of producing crystalline films of semiconductors and optically active materials on insulating substrates. Two low-temperature approaches to silicon graphoepitaxy are pursued: SiI₄ chemical vapor deposition (CVD) and solid-state surface-energy driven secondary recrystallization. The SiI₄ CVD is carried out in closed tubes, both single and two-zone, generally with temperature oscillation to produce alternating deposition and etch-back. In the experiments on solid-state recrystallization, very thin silicon films are used in order to favor surface energy driven grain growth. Similar approaches are used with the model materials.

18.7 Zone Melting Recrystallization of Si for Solar Cells

U.S. Department of Energy (Contract DE-AC02-82-ER13019)

Harry A. Atwater, Henry I. Smith

Zone-melting recrystallization of Si on SiO₂ has produced high-quality crystalline films with (100) texture, suitable for MOSFET devices. We are investigating the feasibility of using this technique to produce low-cost solar cells. A planar constriction technique was developed that allows us to select a single in-plane orientation. Also, subboundaries were shown to terminate when solidification passed through channels about 20 μm wide. A (100) texture was achieved in Si films up to 50 μm thick.

18.8 Zone Melting Recrystallization of InSb and InP

U.S. Navy – Office of Naval Research (Contract N00014-79-C-0908)

Christopher J. Keavney, Henry I. Smith, Clifton G. Fonstad

The success of Si zone-melting-recrystallization in producing device-quality films has prompted us to investigate if similar results can be achieved with the III-V materials. These materials present special problems of adhesion and stoichiometry control. Films are prepared by ion beam sputtering and flash evaporation. Natural oxides, Si₃N₄ and SiO₂ are used as encapsulants, and zone melting is done with a heated W wire in vacuum or an Ar atmosphere. A (111) texture predominates in zone recrystallized films of InSb. Planar constriction techniques may permit single-crystal films to be produced.

18.9 Submicrometer-Period Gold Transmission Gratings and Zone Plates for X-Ray Spectroscopy and Microscopy

Joint Services Electronics Program (Contract DAAG29-83-K-0003)

Lawrence Livermore Laboratory (Contract 2069209)

Andrew M. Hawryluk, Mark L. Schattenburg², Henry I. Smith, Natale M. Ceglio³

Gold transmission gratings with periods of 0.2 and 0.3 μm and thicknesses ranging from 0.5 to 1 μm are fabricated using a combination of holographic lithography, x-ray lithography, and electroplating. These gratings are either supported on polyimide membranes or are made self-supporting by the

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addition of crossing struts. They are used to analyze the x-ray emission from plasmas produced by high-power lasers. Fresnel zone plate patterns are created on x-ray masks by electron beam lithography in collaboration with I.B.M. T.J. Watson Research Laboratory and Cornell University. These are then used to x-ray expose patterns in thick resists. After electroplating, the finished structures are used in soft x-ray imaging experiments at the Stanford Synchrotron Laboratory.

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

Lawrence Livermore Laboratory (Contract 2069209)

Mark L. Schattenburg⁴, C.R. Canizares⁴, Andrew M. Hawryluk

Gold gratings with spatial periods of 0.1 – 10 μm make excellent dispersers for high resolution x-ray spectroscopy of astrophysical sources in the 100 eV to 10 KeV band. These gratings are planned for use in the Advanced X-ray Astrophysics Facility (AXAF) which will be launched in the late 1980's. In the region above 3 KeV, the requirements of high dispersion and high efficiency dictate the use of the finest period gratings with aspect ratios approaching 10:1. We have developed a variety of techniques for high-aspect-ratio fabrication and can now produce gratings of 7:1 aspect ratios. In the region below 3 KeV the gratings must be made free-standing. The effort here is to decrease the loss in efficiency due to absorption in the support structure without sacrificing grating strength.

18.11 Switchable Zero-Order Diffraction Gratings as Light Valves

U.S. Navy - Office of Naval Research (Contract N00014-79-C-0908)

Josephine A. Stein, John Melngailis, Henry I. Smith, J.A. Rajchman⁵, Henry M. Paynter⁶

The aim of this research is to produce a simple, inexpensive, compact light valve which could serve as an element in a matrix-addressable array. The proposed light valve consists of two facing, aligned phase gratings (of period, say 1.5 to 5 μm) in transparent media. Displacing one grating 1/2 of a period with respect to the other switches the device from a transmitting to a blocking state. In the blocking state a periodic π phase shift grating is produced in the wave front of the light, thus cancelling the zero order. In the transmitting state a uniform $\pi/2$ shift occurs instead, and no

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cancellation occurs. The micromotion needed can be achieved by making the gratings out of polyvinylidene fluoride (PVF₂), a transparent, strong piezoelectric. Electrodes have been evaporated on sheets of PVF₂, lateral motion has been observed, and the appropriate piezoelectric coefficient d_{31} has been measured. A technique for embossing gratings in PVF₂ using a press has been developed. All of the fabrication steps needed are consistent with low cost.

18.12 Filters Based on Conversion of Surface Acoustic Waves to Bulk Plate Modes in Gratings

National Science Foundation (Contract ECS82-05701)

Dong-Pei Chen, John Melngailis, Hermann A. Haus

The almost-100%-efficient, narrow-band conversion of surface acoustic waves to bulk plate modes in normal incidence gratings was previously reported. Based on this phenomenon a novel filter, which promises to be low-loss and compact has been demonstrated. Two gratings of different periods are used in which k-vector-matching conditions must be precisely satisfied. Because not all parameters are well enough known for precise design, one of the gratings was chirped about 3% in order to satisfy the matching. With this structure, filters have been built on YZ LiNbO₃ and a few on ST quartz. Center frequencies of the filters were between 160 and 185 MHz and bandwidths between 0.13 and 0.36 MHz. The loss due to the two conversions in the gratings has been at best -5 dB. Only -1 to -2 dB of loss was expected. The observed loss appears to correlate with the lack of parallelism of the two crystal faces. The lowest loss was on crystals which were specially polished to have the two faces parallel to better than 0.006 degrees.

18.13 Collaborative Projects

The unique equipment and expertise of the Submicron Structures Laboratory has served as a resource for numerous researchers from other laboratories. Some examples are the following:

a) The directional and selective reactive ion etching based on SiCl₄ gas was used by S. Cabral and D. Rathman of Lincoln Laboratory to etch 0.3 μm period gratings in silicon for the permeable-base transistor.

b) S.W. Pang of Lincoln Laboratory and C.M. Horwitz have studied damage in silicon MOS devices produced by reactive ion etching. Instead of the expected damage due to ion bombardment or irradiation, the degradation of the silicon was found to be due to contamination by impurities sputtered from the chamber walls.

c) H. Bickford of I.B.M., Essex Junction, visited M.I.T. and, together with C.M. Horwitz, studied the undercutting of silicon masked with photoresist during reactive ion etching.

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