17. Submicron Structures Technology and Research

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17.1 Submicron Structures Laboratory

The objective of the Submicron Structures Laboratory at M.I.T. is to develop techniques for fabricating surface structures having linewidths in the nanometer to micrometer range, and to use these structures in a variety of research projects. This laboratory contributes to an expansion of microsystems research at M.I.T. 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 vapor deposition, sputtering). The research projects of the laboratory, which are described briefly below, fall into four major categories: submicron structures fabrication techniques (no. 2); submicrometer electronics (nos. 3 to 5); crystalline films on amorphous substrates (nos. 6 to 8); periodic structures and applications (nos. 9 to 12).

17.2 Microfabrication 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 (Contract ECS82-05701)
Erik H. Anderson, Andrew M. Hawryluk[3], Irving Plotnik, Henry I. Smith, Henri Lezec, John Melngailis

A variety of techniques for fabricating structures with linewidths of 0.1 μm and below are under development. These include: holographic lithography, spatial frequency doubling, x-ray lithography, and reactive-ion etching. Two new holographic techniques have been developed, which provide greatly improved control of resist profile over areas ~5.0 cm in diameter. In one technique, thick resist, of the order 0.5 μm, is used and a grating is produced in the top surface only. This is

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shadowed obliquely with chromium and then reactive-ion etched in oxygen. The result is a periodic structure with vertical side-walls and well-controlled line-space ratio. In a second technique, an undercut profile is achieved in the resist by positioning the standing wave maximum at the resist-substrate interface. This is achieved by tilting the substrate normal out of the plane of the holographic interferometer. In spatial-frequency doubling (in collaboration with D. Ehrlich, M.I.T. Lincoln Laboratory), an ArF laser is used in conjunction with a 0.2 μm-period parent mask. In one experiment, a multilayer dielectric mirror was deposited on the mask to suppress the zero-order beam. Gratings of 0.1 μm-period (50 nm linewidth) were exposed in PMMA. These optical techniques are seldom used to expose patterns directly on experimental substrates. Instead, x-ray lithography masks are fabricated. In most cases, a Carbon K x-ray source (λ = 4.5 nm) is used in conjunction with PMMA resist to replicate the mask pattern. Details on the mask <100 Å are reproduced. Reactive-ion etching of structures with linewidths ~0.1 μm and below is investigated to improve control of cross-sectional profiles. This research employs transmission electron microscopy.

17.3 Electronic Conduction in Ultra-Narrow Silicon Inversion Layers

Joint Services Electronics Program (Contract DAAG29-83-K-0003)
Robert F. Kwasnick, Jerome C. Licini, Marc A. Kastner, John Melngailis, Patrick A. Lee

In order to study conduction phenomena in a quasi-one-dimensional electron gas, field-effect transistors have been fabricated with a ~70 nm wide and 7 μm long gate. The conductance of such metal-oxide-silicon transistors exhibits non-monotonic variation with electron density at temperatures below 15 K. The variations are largest at low electron concentrations, where the current is thermally activated, and they are the result of variations of the activation energy E_0. We find the striking result that the current is exponentially dependent on the voltage V_D along the inversion layer when E_0 is much larger than its typical value. In fact, above 2 K both the temperature and V_D dependences of the current can be described by an activated form with activation energy $E_0 - feV_D$ where f~0.3–0.5. This dependence on V_D is too strong to be explained by either electron heating or variable-range hopping. Instead, it indicates that the current is limited by spatial barriers which contain tunneling channels at discrete energies, as in the model of Azbel. Recently, structures with improved gate uniformity have been fabricated by evaporating Al in controlled O_2 atmosphere.

17.4 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 on the conduction electrons in Si inversion layers. Toward this goal, MOS transistors are fabricated with a 0.2 μm–period chromium grating over the gate oxide. This serves as a first gate. A second gate (continuous Al sheet) is located above this, separated by 0.2 μm SiO_2. This dual–gate structure allows one to adjust the periodic potential in the Si inversion layer between source and drain. Test devices have been fabricated, and demonstrated a successful fusion of standard MOS processing and submicron structures fabrication. The immediate goal is to test devices at very low temperatures (~1–3°K) and to demonstrate the quantum effects due to a quasi–one–dimensional density of states (conduction parallel to the grating) and due to superlattice dispersion (conduction perpendicular to the gratings).

17.5 Submicron FET's in Si

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

Stephen Y. Chou, Dimitri A. Antoniadis, John Melngailis, Henry I. Smith

The techniques of carbon–K x–ray lithography and reactive ion etching are being combined with standard MOS processing to fabricate Si FET devices having gate lengths ranging from 0.05 μm to 2 μm. The DC characteristics of these devices will be compared with predictions based on device–scaling models.

17.6 Graphoepitaxy of Si, Ge, and Model Materials

National Science Foundation (Contract ECS82-05701)

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

Carl V. Thompson, Takao Yonehara, Chee C. Wong, Henry I. Smith, Erik H. Anderson

Graphoepitaxy, in which an overlayer of film is crystallographically oriented by an artificial surface pattern, was demonstrated some years ago. 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 graphoepitaxy mechanisms in Si, Ge, 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 are pursued: SiI_4–chemical vapor deposition (CVD), and solid–state secondary grain growth. In the experiments on solid–state grain growth, ultra–thin (less than 1000 Å) films are used. In this way, grain growth is driven by surface–energy anisotropy resulting in large grains of a specific crystallographic texture. The phenomena of surface–energy–driven secondary grain growth and solid–state agglomeration have been observed in both Si and Ge films and appear promising for low-temperature graphoepitaxy.

17.7 Zone-Melting Recrystallization of Si for Solar Cells

U.S. Department of Energy (Contract DE-AC02-82-ER-13019)
Harry A. Atwater, Henry I. Smith, Carl V. Thompson

Zone-melting recrystallization (ZMR) of Si on SiO_2 has produced high-quality crystalline films with (100) texture, suitable for MOSFET devices. We are investigating the feasibility of using ZMR to produce low-cost solar cells. A transparent substrate, such as a low-cost glass coated with SiO_2, would serve as the window for the solar cell module. Because the substrate is insulating, back-surface-contact cells will be required. A "vertical-constriction" technique was developed which produces (100) texture in Si films 60 μm-thick. By patterning a Si film with an array of crystallization barriers prior to ZMR, a narrow distribution of in-plane orientations was achieved, a technique we call orientation filtering. Cells were fabricated by non-optimized processes in 10 μm-thick Si films. An efficiency of 7% was achieved in this initial attempt.

17.8 Zone-Melting Recrystallization of III-V Materials

U.S. Navy - Office of Naval Research (Contract N00014-79-C-0908)
Christopher J. Keavney, Harry A. Atwater, Chee C. Wong, Henry I. Smith, Carl V. Thompson

The success of Si zone-melting recrystallization (ZMR) 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, stoichiometry control, and differential vapor pressure at the melting point. Initial efforts have used InSb, which was deposited by flash evaporation. The natural oxide of InSb was found to be the best encapsulation layer. Large-grained films with <111> texture were achieved. Grains contain subboundaries where excess Sb collected and formed a eutectic phase. Techniques of planar constriction and subboundary entrainment were demonstrated.

17.9 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[4], Mark Schattenburg[5], Henry I. Smith, Natale M. Ceglio[4]

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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 addition of crossing struts. They are used for spectroscopy of 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 IBM T.J. Watson Research Laboratory, Lincoln Laboratory and Cornell University. These are then used to x-ray expose patterns in thick resist. After electroplating, the finished structures are used in soft x-ray imaging experiments.

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

National Aeronautics and Space Administration (Contract NGL-22-009-638)
Mark L. Schattenburg[5], Claude R. Canizares[5], Andrew M. Hawryluk[4], Henry I. Smith

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 early 1990'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. To achieve this we first expose a grating pattern in 1.5 μm thick PMMA over a gold plating base using Carbon-K x-ray lithography. To date, we have worked with gratings having periods of 0.3 or 0.2 μm (linewidth 0.15 – 0.1 μm). Gold is then electropolated into the spaces of the PMMA to a thickness of 1 μm.

17.11 Switchable Zero-Order Diffraction Gratings as Light Valves

U.S. Navy - Office of Naval Research (Contract N00014-84-K-0073)
Josephine A. Stein, Deborah A. Summa, John Melngailis, Jan A. Rajchman, Henry M. Paynter

Simple inexpensive light valves, which can be fabricated in a line-addressable matrix configuration, may make possible flat back-lighted displays or optical signal processing elements for converting electronic information into spatial light modulation. Our aim is to build and demonstrate a single such switchable light valve. This light valve would operate by displacing two facing phase gratings in a transparent medium with respect to each other. In one position the zero order of diffraction would be cancelled, in another it would not. The displacements needed are on the order of 1 μm. Techniques for fabricating the gratings have been developed and used, and the optical principles have been verified. Several schemes for producing the micromotion have been tried and a chevron configuration has evolved as the most promising. The micro-motion will be produced using the very strong piezoelectricity of the transparent plastic poly-vinylidine fluoride (PVF_2). The geometry that is now being built involves motion producing elements in an open chevron configuration which, in effect, produces an amplification of the motion generated by the piezo-electric effect. Photolithography masks for fabricating this structure have been designed.

17.12 Studies of Surface Acoustic Wave Propagation in Gratings

National Science Foundation (Grants ECS80-17705 and ENG79-09980)

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

The attenuation of Rayleigh waves due to scattering into the bulk by a periodic grating has been experimentally measured and theoretically calculated. Results have been obtained for both normal incidence and oblique incidence gratings on Y-cut Z-propagating lithium niobate. The radiation causes considerable loss in up-chirp reflective array compressors which are widely used in radar and in signal processing. Since the loss can now be predicted, the design of these devices becomes more quantitative.

The reflection of surface acoustic waves from normal incidence metal overlay gratings has been calculated using the coupling of modes formalism. The reflection is due to field shorting and to mass loading. Under special circumstances these two effects can be made to cancel. This result may be of importance in the design of surface acoustic wave transducers where reflection is an unwanted effect. Experiments are in progress to test the theory.

17.13 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:

a) Very directional reactive-ion etching was used in the construction of a multilayer device called the joint-gate CMOS transistor. Collaboration with A.L. Robinson.

b) Submicron-period gratings, both topographic and metal overlay, have been fabricated on silicon optical detector structures by G. Ghavamishahidi. The gratings serve to reduce the lifetime of the optically generated carriers thus making the detector faster.

c) Surface-acoustic-wave transducers have been fabricated on InP by E. Wintner for the purpose of detecting waves generated by pulsed laser light which is incident on the surface.

Publications

Maby, E.W., H.A. Atwater, A.L. Keigler, and N.M. Johnson, "Electron-Beam-Induced Current Measurements in Silicon-on-Insulator Films Prepared by Zone-Melting Recrystallization," Appl. Phys. Lett. 43, 482 (1983).

Horwitz, C.M., "Rf Sputtering-Voltage Division Between Two Electrodes," J. Vac. Sci. Tech. A 1, 60 (1983).

Horwitz, C.M., "New Dryetch for Al and Al-Cu-Si Alloy: Reactively Masked Sputter Etching with SiF_4," Appl. Phys. Lett. 42, 898 (1983).

Smith, H.I., C.V. Thompson, M.W. Geis, R.A. Lemons, and M.A. Bosch, "The Mechanism of Orientation in Si Graphoepitaxy by Laser or Strip-Heater Recrystallization," J. Electrochem. Soc. 130, 2050 (1983).

Smith, H.I., M.W. Geis, C.V. Thompson, and H.A. Atwater, "Silicon-on-Insulator by Graphoepitaxy and Zone-Melting Recrystallization of Patterned Films," J. Cryst. Growth 63, 527 (1983).

Hawryluk, A.M., H.I. Smith, and D.J. Ehrlich, "Deep UV Spatial-Frequency Doubling by Combining Multilayer Mirrors with Diffraction Gratings," J. Vac. Sci. Technol. B 1, 1200 (1983).

Lezec, H.J., E.H. Anderson, and H.I. Smith, "An Improved Technique for Resist-Profile Control in Holographic Lithography," J. Vac. Sci. Technol. B 1, 1204 (1983).

Ceglio, N.M., A.M. Hawryluk, and M.L. Schattenburg, "X-Ray Phase Lens Design and Fabrication," J. Vac. Sci. Technol. B 1 1285 (1983).

Horwitz, C.M., "Hollow Cathode Reactive Sputter Etching — A New High-Rate Process," Appl. Phys. Lett. 43, 977, (1983).

Anderson, E.H., C.M. Horwitz, and H.I. Smith, "Holographic Lithography with Thick Photoresist," Appl. Phys. Lett. 49, 874 (1983).

Kwasnick, R.F., M.A. Kastner, J. Melngailis, and P.A. Lee, "Non-Monotonic Variations of the Conductance with Electron Density in ~70nm Wide Inversion Layers," Phys. Rev. Lett. 52, 224 (1984).

Atwater, H.A., C.V. Thompson, H.I. Smith, and M.W. Geis, "Orientation Filtering by Growth-Velocity Competition in Zone-Melting Recrystallization of Silicon on SiO_2," Appl. Phys. Lett. 43, 1126 (1983).

Atwater, H.A., H.I. Smith, C.V. Thompson, and M.W. Geis, "Zone Melting Recrystallization of Thick Silicon on Insulator Films," Mater. Lett., to be published.

Thompson, C.V. and H.I. Smith, "Silicon-on-Insulator by Surface-Energy-Driven Secondary Recrystallization with Patterning," Appl. Phys. Lett., March 15, 1984, to be published.

Geis, M.W., H.I. Smith, B.Y. Tsaur, J.C.C. Fan, D.J. Silversmith, R.W. Mountain, and R.L. Chapman, "Zone-Melting Recrystallization of Semiconductor Films," in J. Narayan, W.L. Brown, and R.A. Lemons (Eds.), Laser-Solid Interactions and Transient Thermal Processing of Materials, Materials Research Society Symposium Proceedings, Vol. 13 (North-Holland Publishing Co., 1983), p. 477.

Johnson, N.M., M.D. Moyer, L.E. Fennell, E.W. Maby, and H.A. Atwater, "Detection of Electronic Defects in Strip-Heater Crystallized Silicon Thin Films," in J. Narayan. W.L. Brown, and R.A. Lemons (Eds.), Laser-Solid Interactions and Transient Thermal Processing of Materials, Materials Research Society Symposium Proceedings, Vol. 13 (North-Holland Publishing Co., 1983), p. 491.

Atwood, D.T., N.M. Ceglio, H.M. Medecki, H.I. Smith, A.M. Hawryluk, T.W. Barbee, Jr., W.K. Warburton, J.H. Underwood, B.L. Henke, T.H.P. Chang, M. Hatzakis, D.P. Kern, P.J. Coane, W.W. Molzlen, A.J. Speth, G.L. Stradling, and D.W. Sweeney, "Current Developments in High Resolution X-Ray Measurements," in T.J. McIlrath and R.R. Freeman (Eds.), American Institute of Physics Conference Proceedings, Laser Techniques for Extreme Ultraviolet Spectroscopy, Boulder, Colorado, 1982.

Keavney, C.J., H.A. Atwater, H.I. Smith, and M.W. Geis, "Zone Melting Recrystallization of InSb on

Oxidized Silicon Wafers," Electrochemical Society Symposium on III-V Opto-Electronics Epitaxy and Device-Related Processes, San Francisco, California, May 1983.

Smith, H.I., E.H. Anderson, and M.L. Schattenburg, "Planar Techniques for Fabricating X-Ray Diffraction Gratings and Zone Plates," in D. Rudolph and G. Schmahl (Eds.), Symposium on X-Ray Microscopy, Gottingen, Federal Republic of Germany, Sept. 14–16, 1983 (Springer-Verlag, 1983).

Wong, C.C., C.J. Keavney, H.A. Atwater, C.V. Thompson, and H.I. Smith, "Zone Melting Recrystallization of InSb Films on Oxidized Si Wafers," presented at the Materials Research Society Symposium, Boston, Massachusetts, November 1983, to be published in Materials Research Society Symposia Proceedings, "Energy Beam-Solid Interactions and Transient Thermal Processing," (Elsevier Science Publishing Co.).

Yonehara, T., C.V. Thompson, and H.I. Smith. "Abnormal Grain Growth in Ultra-Thin Films of Germanium on Insulator," presented at the Materials Research Society Symposium, Boston, Massachusetts, November 1983.

Smith, H.I., C.V. Thompson, M.W. Geis, H.A. Atwater, T. Yonehara, and C.C. Wong, "Graphoepitaxy and Zone Melting Recrystallization of Patterned Films," to be published in N.M. Johnson and J.C.C. Fan (Eds.), Materials Research Society Symposia Proceedings, "Energy Beam-Solid Interactions and Transient Thermal Processing," (Elsevier Science Publishing Co., 1984).

Kwasnick, R.F., M.A. Kastner, and J. Melngailis, "Electronic Conduction in Ultra-Narrow Si Inversion Layers," presented at March 1983 Meeting of the American Physical Society, Bull. Am. Phys. Soc. 28, 322 (193).

Warren, A.C., D.A. Antoniadis, J. Melngailis, and H.I. Smith, "Submicrometer-Period Channel Modulation of a MOSFET," presented at the 1983 International Symposium on Electron, Ion, and Photon beams, Los Angeles, California, May 31–June 3, 1983.

Schattenberg, M.L., C.R. Canizares, H.I. Smith, and A.M. Hawryluk, "Submicrometer-Period Transmission Diffraction Gratings for X-Ray Astronomy," presented at the 1983 Symposium on Electron, Ion and Photon Beams, Los Angeles, California, May 31–June 3, 1983.

Anderson, E.H. and H.I. Smith, "TEM Analysis of Vertical-Slab Cross Section of Submicrometer Structures," presented at the 1983 International Symposium on Electron, Ion, and Photon Beams, Los Angeles, California, May 31–June 3, 1983.

Thompson, C.V., and H.I. Smith, "Silicon-on-Insulator by Solid State Surface-Energy-Drive Secondary Recrystallization," 1983 Electronic Materials Conference, Burlington, Vermont, June 1983.

Smith, H.I., "Graphoepitaxy to Circumvent Lattice Matching Constraints," presented at the Fourth International Conference on Integrated Optics and Optical Fiber Communication (Post-Conference Meeting), Tokyo, Japan, July 2, 1983.

Smith, H.I., "Graphoepitaxy and Zone-Melting Recrystallization of Patterned Films," presented at the VII International Conference on Crystal Growth, Stuttgart, Federal Republic of Germany, September 12–16, 1983.

Thompson, C.V. and H.I. Smith, "Silicon-on-Insulator by Surface-Energy-Driven Secondary Recrystallization with Patterning," presented at the VII International Conference on Crystal Growth, Stuttgart, Federal Republic of Germany, September 12–16, 1983.

Atwater, H.A., "Control of silicon Film Recrystallization Using Lithography with Application to Photovoltaics," S.M. Thesis, Department of Electrical Engineering and Computer Science, Massachusetts Institute of Technology, June 1983. Also VLSI Memo 83-149, July 1983.

Keavney, C.J., "Zone-Melting Recrystallization of InSb on Oxidized Silicon Wafers," S.M. Thesis, Department of Materials Science and Engineering, Massachusetts Institute of Technology, June 1983.

Ahlers, E.D., "Method of Photolithographic Projection System Characterization," B.S./M.S. Thesis, Department of Electrical Engineering and Computer Science, Massachusetts Institute of

Technology, June 1983.
Dana, S.S. "Nucleation on Surface Relief Structures by Chemical Vapor Deposition," Ph.D. Thesis, Department of Physics, Massachusetts Institute of Technology, September 1983.

Domestic Patents Filed

"Orientation Filtering," H.I. Smith, C.V. Thompson, H.A. Atwater, and M.W. Geis, filed April 1, 1983.

"Thick Crystalline Films on Foreign Substrates," H.I. Smith, H.A. Atwater, and M.W. Geis, filed May 9, 1983.