17. Submicron Structures Fabrication and Research

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17.1 Submicron Structures Fabrication

The Submicron Structures Laboratory at M.I.T. is developing techniques for fabricating surface structures of nanometer to micrometer linewidths and is using these structures in a variety of research projects.[1,2] Submicron structures fabrication is becoming a distinct discipline with a world wide and growing community of practitioners. The binding force of this new discipline acts on two levels, namely the specialized techniques used and the novel properties of the structures fabricated. Fabrication techniques include various forms of lithography (optical, electron beam, x-ray, ion beam), etching (acqeous, reactive plasma, sputtering), growth (oxidation, plating, epitaxy) and deposition (evaporation, chemical vapor deposition, sputtering). With these techniques it is possible to fabricate experimental structures with minimum feature sizes smaller than 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 (~20 atom spacings) 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 structure 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 circuits of higher complexity operating at higher speeds.

The main research projects of the Submicron Structures Laboratory, which will be described below, fall into three categories: microstructure fabrication (x-ray lithography and reactive ion etching) nos. 2 to 5, graphoepitaxy nos. 6 to 8, and device-related research nos. 9 to 11.

17.2 Submicron-Period Gold Transmission Gratings for Soft X-ray Spectroscopy

Lawrence Livermore Laboratory (Subcontract 2069209)
Joint Services Electronics Program (Contract DAAG29-C-0104)
U.S. Navy - Office of Naval Research (Contract N00014-79-C-0908)

Henry I. Smith, Andrew M.Hawryluk, John Melngailis, and Mark L. Schatterbug (in collaboration with N.M. Ceglio and R.H. Price, of the Lawrence Livermore Laboratory)

Transmission gratings are useful spectroscopic tools in the wavelength range of 0.5 nm to 200 nm and span the gap between Bragg crystals and optical reflection gratings. Gold gratings, shown in Fig.17-1 with spatial periods of 0.3 and 0.2 μm, have been fabricated in thicknesses of 0.6 and 0.25 μm, respectively and used in x-ray spectroscopy. Fabrication techniques include: holographic lithography, shadowing, x-ray lithography and gold microplating. Control of linewidth to tolerances of the order of 10 nm has been demonstrated for gratings of 0.2 μm period. A high resolution imaging spectrometer, composed of a 22x Wolter x-ray microscope in conjuction with a gold transmission grating, was tested. At a wavelength of 0.69 nm a resolving power, $\lambda/\Delta\lambda$, of 200 was demonstrated. Resolution in this case was source size limited.[3-6]

These gratings and other x-ray spectroscopic elements fabricated in our laboratory are used at Lawrence Livermore Laboratory for analysis of laser fusion plasmas. In addition, we are currently working on transmission gratings for soft x-ray astronomy.[7]

17.3 Spatial-Period-Division

Joint Services Electronics Program (Contract DAAG29-80-C-0104)
U.S. Navy - Office of Naval Research (Contract N00014-79-C-0908)

Andrew M. Hawryluk, Henry I. Smith

Another application of transmission gratings is spatial-period-division. This is a technique for halving the period of a grating. We are developing it as a means of achieving a smaller period than can be fabricated by holographic techniques. At a specific distance in the near field beyond a transmission grating of period p the intensity is modulated with a period p/2. Starting with a parent mask of 199 nm period with 40 nm wide slits in 200 to 250 nm thick gold, PMMA was exposed at a distance of 4.4 μm from the mask to produce a 99.5 nm period grating.[6] Spatial-period-division using deep UV has also been demonstrated in collaboration with R.M. Osgood and D.J. Ehrlich of Lincoln Laboratory. An ArF laser (λ = 193 nm) was used to halve the 199 nm period of an aluminum grating to produce a 99.5 nm period grating in PMMA.

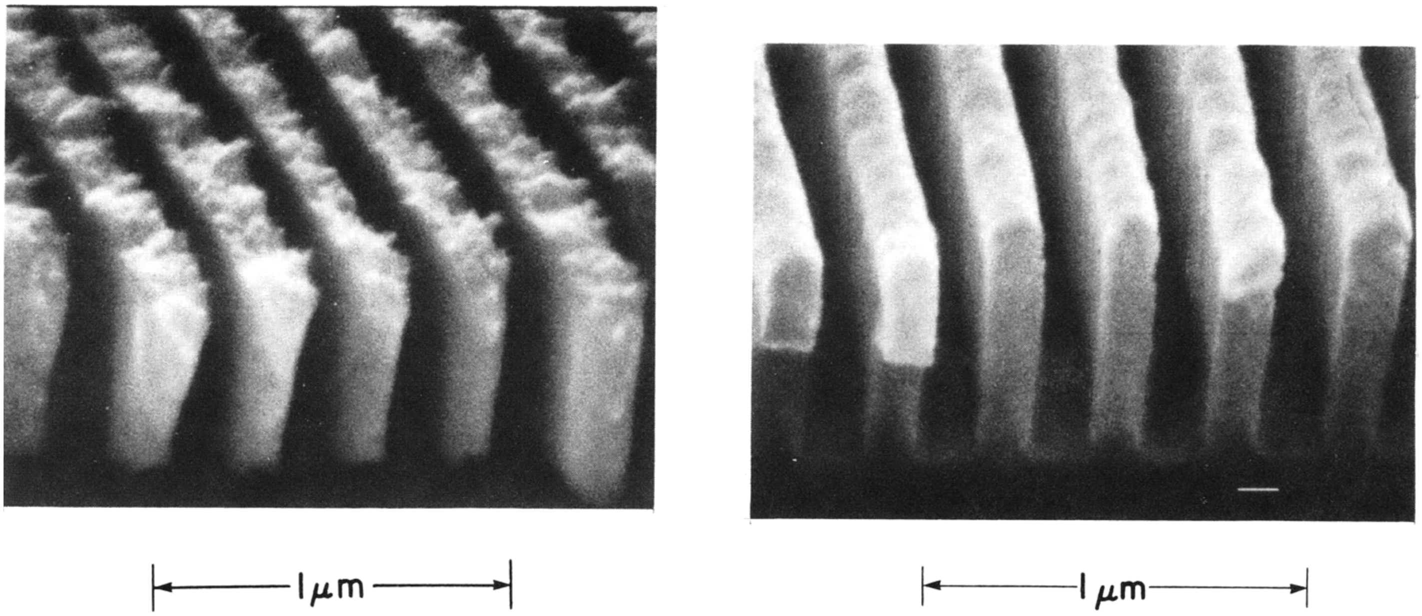

Figure 17-1: Left: End view of PMMA exposed by x-ray lithography and developed. 0.3 μm period, 0.7 μm thick.

Right: End view of gold grating. 0.3 μm period, 0.6 μm thick. The gold was plated up between the PMMA grating lines shown in the top view and then the PMMA was dissolved away.

17.4 Fabrication of X-ray Masks Using Anisotropic Etching of (110) SI and Shadowing Techniques

Joint Services Electronics Program (Contract DAAG29-80-C-0104)
U.S. Navy - Office of Naval Research (Contract N00014-79-C-0908)

Norikazu Tsumita, John Melngailis, Andrew M. Hawryluk, Henry I. Smith, Erik H. Anderson

In some of the projects described herein, items 2, 3, 6, 7, 8, 10, and 11, submicron gratings with controlled linewidths and smooth line edges are needed. However, variations in exposure intensity, resist thickness, or resist graininess are frequently serious impediments. One way around the problem is to etch a grating in a particular orientation of a silicon surface so that the edges of the grating are defined by crystallographic (111) planes. This technique was originally developed at Lincoln Laboratory[8] and consists of taking a thin-membrane polyimide mold of the crystallographically-defined grating and shadowing its edges with an x-ray absorber to form a mask for x-ray lithography. In this work, we have extended the technique by using the (110) surface of silicon and producing a grating with vertical sidewalls defined by (111) planes. Holographic lithography is used to expose grating patterns in AZ 1350 over a thin Si_3N_4 layer on the (110) Si. The Si_3N_4 is patterned by reactive ion etching and serves as the mask for anisotropically etching the square-wave-profile grooves. At the proper crystallographic orientation, the groove sidewalls are defined by (111) planes, and groove bottoms are approximately flat. The structure in Si is then transferred to polyimide which is obliquely shadowed and forms the x-ray mask. Grating patterns replicated in PMMA using the C_K x-ray (4.5 nm) show straight lines over large areas with uniform widths and edges smooth to ~ + 4 nm.[9]

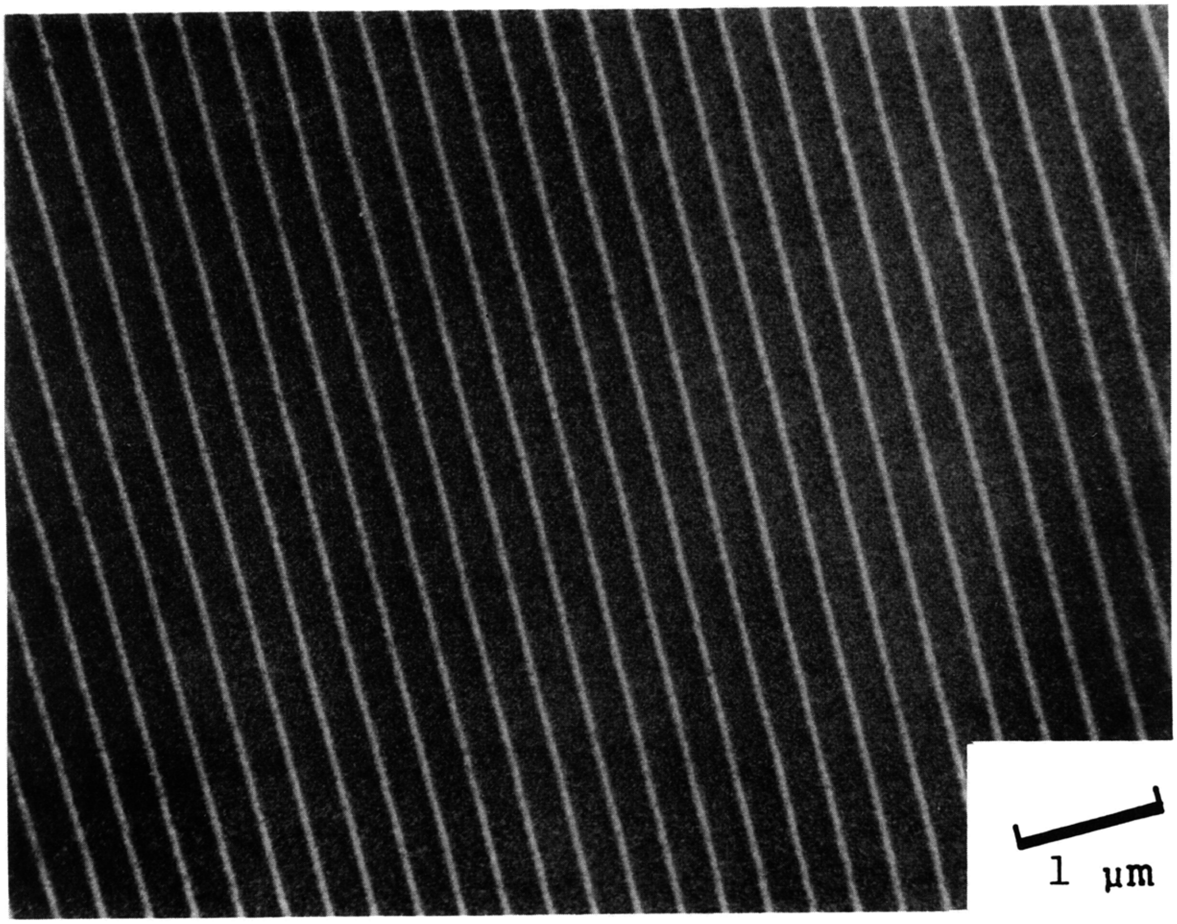

Figure 17-2: Top view of PMMAA x-ray exposed with crystallographic mask. The period of the grating is 0.3 µm and the lines are smooth to ± 4 nm.

17.5 Reactive Sputter Etching of Si, SiO_2, Cr, and Al with Gas Mixtures Based on CF_4, Cl_2, and $SiCl_4$

Joint Services Electronics Program (Contract DAAG29-80-C-0104)

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

Harkness Foundation

I.B.M.

Christopher M. Horwitz, John Melngailis

So called dry etching or plasma etching is now widely used in semiconductor manufacturing. Reactive sputter etching, also called reative ion etching, refers to the dry etching processes done in a RF sputtering type configuration at low pressure (0.65 to 6.5 Pa or 5 to 50 mtorr) where, because of a bias voltage and the longer mean free path, the ions strike the surface to be etched at normal incidence. This permits the etching of relief structures with vertical side walls, provided the reactive ion erodes the substrate much faster than the masking material. Thus, control of the relative etch rates of materials is important.

This work aims to determine which variables influence the etch rate of various materials most strongly and to measure the dependencies of etch rates of various materials as a function of these variables. At a constant gas flow of 2×10^{-2} Pa m^3/sec we use target voltage (peak-to-peak) and partial pressure of reactant species as controlled varibles rather than power density and total pressure, as is a common practice. SiO_2 and Si etch rates were found to vary as $P^{1/2}V^2$ where V is the peak-to-peak target voltage (varied between 0.6 kV and 2.9 kV) and P is the reactant gas partial pressure (varied between 0.35 Pa (2.7 μm) and 14.3 Pa (110 μm)). Cr and Al rates varied as V^3, but were almost independent of pressure. These differences enable accurate selection of etch rates and appropriate masks for directional etching. For instance, SiO_2/Cr etch rate ratios as high as 50:1 may be achieved for 3.7 Pa of CF_4 at 0.8 kV peak-to-peak target voltage. Chlorine mixed with CF_4 enables the properties of the two gases to be combined. Thus, by adjusing only the gas mixture from pure CF_4 to pure Cl_2 the Si/SiO_2 etch rate ratio can be continuously varied between 0.33 and 6 at 1.3 kV peak-to-peak target voltage, 3.7 Pa pressure, and with substrates on a Si target.[10] The etching properties of mixtures of Ar, $SiCl_4$ and O_2 and of mixtures of Ar, Cl_2 and O_2 were also studied. With these mixtures Si/SiO_2 etch rate ratios can be made arbitrarily large since with increasing oxygen concentration SiO_2 deposits on the slowly etched surfaces, e.g. the initial SiO_2 mask. In addition, highly directional etching of silicon has been observed.[11]

17.6 Studies of Graphoepitaxy by CVD and Solution Growth

U.S. Navy - Office of Naval Research (Contract N00014-79-C-0908)
Joint Services Electronics Program (Contract DAAG29-80-C-0104)
Stephane S. Dana, Henry I. Smith

Graphoepitaxy is a process in which an artificial surface pattern is used to induce or control orientation in a film. In order to exercise such control one or more dimensions of the artificial pattern must be small compared to the grain or domain size that characterizes the film. This generally implies submicron period structures, in some cases sub-100 nm. For solid crystalline films, the patterns used to date have been grating relief structures, and the objective has been to achieve uniform orientation. Although the concept of control via artificial patterns is very general, and graphoepitaxial orientation has been demonstrated for several solid and liquid crystalline films, the greatest interest has been in graphoepitaxy of Si over SiO_2 because of its impact on VLSI and three dimensional integration.

Graphoepitaxy by direct growth on submicrometer-spatial-period surface relief gratings in amorphous substrates has been investigated using Si CVD processes and aqueous solution growth of alkali halides and other ionic crystals.[12] The CVD processes used the pyrolysis of SiH_4 and $SiCl_4$ in the presence of HCl. Substrates were Si_3N_4 films into which 0.3 μm period square-profile gratings were etched. At low deposition rate, nucleation occurred preferentially at groove edges. Small islands of Si located in groove bottoms were geometrically confined by the two facing sidewalls and took on elongated shapes. Selected area electron diffraction in a STEM showed a predominance of 2- and 4 fold symmetry. Where no grating was present, islands had a hemispherical-cap morphology and a predominance of 3-fold symmetry in diffraction. Future research will emphasize CVD growth using the near equilibrium reaction

$$2SiI_2 \rightleftarrows Si + SiI_4$$

at temperatures as low as 800° C.

In solution growth of ionic crystals, surface relief gratings had a strong influence on deposit morphology. However, it proved difficult to achieve uniform deposits or to terminate experiments without loss of control of supersaturation. By analyzing the habits of small crystallite as well as crystalline sheets it is clear that orientation is induced by gratings. However, we cannot at present discriminate between the model of heterogeneous nucleation and growth, and the model of Sheftal, et al. which invokes the settling out of microcrystals. Future research will use deposition by sublimation.

17.7 Graphoepitaxy for Solar Cells

U.S. Department of Energy (Contract DE-AC02-80-E10179)
Harry A. Atwater, Krikor Bezjian, Henry I. Smith

We are investigating the use of graphoepitaxy and zone melting techniques to produce silicon films on conducting substrates for solar cells. Pyrolytic carbon on low-cost graphite appears to be the most promising substrate. Preliminary experiments have been done on SiO_2 substrates with SiO_2/Si_3N_4 encapsulation. A pit-grid technique was developed to analyze Si film orientat;ion. The technique consists of lithographically exposing and etching a grid of round, micron-size holes in a top layer of SiO_2 and then applying a directional KOH etch to the exposed silicon. Pits bounded by (111) surfaces are formed. Thus, on a (100)-oriented film (most common case) square, inverted pyramid type pits are formed. The orientation of the square reveals the orientation of the crystallites. The orientation of large areas can be analyzed by reflecting light off pit facets. This technique has facilitated the determination of mechanisms responsible for texture, orientation and linear defects in Si films. Recrystallized films on SiO_2 substrates have proven to be suitable for MOSFET devices. Surface mobilities appraoching bulk values were obtained. Means of achieving single-grain orientation and for localizing dislocations are under development. If recrystallization processes work as well on amorphous carbon as on SiO_2 then we may have a low cost process for fabricating high-efficiency solar cells.

17.8 Liquid Phase Epitaxy of InP

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

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

Following earlier experiments by S. Rotter and K. Bezjian which showed dislocation reduction in liquid phase epitaxy of PbSnTe over sawtooth relief gratings, we are investigating the phenomenon in InP. The first step was to find and test etches that would reveal dislocations. Then directional chemical etches were developed which could be used to produce sawtooth grating structures on the InP substrates.

17.9 Filters Based on the Coupling of Surface Acoustic Waves in Gratings to Bulk Plate Modes

National Science Foundation (Grant ECS80-17705)

John Melngailis, Hermann A. Haus +, Mohammed N. Islam [3], *Edward M. Garber* [4]

The very efficient and narrow band conversion of surface acoustic waves in gratings to bulk plate modes is a recently discovered phenomenon.[13,14] This phenomenon may permit one to combine the advantages of surface acoustic wave devices, such as planar fabrication and accessibility of the signal, with the low attenuation of bulk acoustic waves. One device which exploits this may be a low-loss narrow-bandwidth compact filter. In this structure two gratings of different periods are used to couple a surface wave into a bulk plate mode and then to couple it back out. These two couplings can be with almost zero loss. The grating periods for which this works are precisely determined by the plate thickness and the surface wave and bulk wave velocities. Unfortunately, some of these quantities are not quite accurately enough known to calculate the correct grating periods. Nevertheless, an attempt was made to build such a filter and to use the temperature to fine tune the parameters. The tuning range was not sufficient and the filter behavior was not observed. An alternate method would be to use one constant period grating and to build the other grating with a slowly varying period. A mask with such gratings has been fabricated.

17.10 Fabrication of Corrugated-Gate MOS Structures

Joint Services Electronics Program (Contract DAAG29-80-C-0104)

Alan C. Warren, Dimitri A. Antoniadis, John J. Melngailis

Silicon MOS transistors with periodically modulated gate oxide thickness are being fabricated. Gratings with spatial periodicities of 2000 Å or less are employed. Thus, the vertical electric field in the channel and, as a result, the surface inversion electron distribution are spatially modulated. The basic goal of the project is to study electronic transport parallel and perpendicular to the field modulation. Of particular interest is the confinement of electrons in narrow channels of 1000 Å or less which approach one-dimensional conductors. This is a condition that may be encountered as MOS devices are scaled to very small dimensions.

To fabricate the gratings the reactive ion etching apparatus was first characterized and calibrated for SiO_2 with CHF_3 as the etch gas. The emphasis was on slowing the etch rate so that several

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[3] Hertz Fellow

[4] Supported by NSF Contract No. ENG 79.09980

hundred angstroms per minute could be etched controllably. The RF p-p voltage was reduced to 400 volts, and system parameters (chamber pressure, gas flow rate, etc.) were carefully controlled so that the etch rate of SiO_2 was 108 ± 3 Å/min. over the etching times of interest (5-11 min.). In addition, the etched films have been characterized primarily through C-V analysis. Results so far have indicated that high quality films down to 350 Å thick (etched from 1200 Å) can be achieved, and the necessity of annealing and organic solvent cleaning after the etch is now being investigated.

17.11 Narrow Inversion Layers in Silicon

Joint Services Electronics Program (Contract DAAG29-80-C-0104)

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

As submicron-size electronic devices begin to be manufactured, the question naturally arises of whether the behavior of electrons is different when they are confined to structures with very small dimensions. The behavior of electrons confined to two dimensions has been studied with the inversion layer of Si MOSFET's. In the inversion layer the electric field is sufficiently strong to confine the electrons in quantum states at the surface. Because the electrons are free to move parallel to the surface, a two-dimensional electron gas results. Recent experiments have shown that the behavior of these electrons, especially when scattered by impurities and defects, is fundamentally different from that of electrons in three-dimensional systems. The theorists predict that the transition from two dimensions to one will have even more drastic consequences than from three to two. Our goal is to fabricate structures in which electrons in inversion layers will be confined to one dimension.

To do this we hope to use the electric field at the MOSFET surface to confine the electrons to one dimension. Our first attempt utilized structures in which the silicon was etched into sharp ridges parallel to the direction of current flow, between source and drain. The field was expected to confine carriers to the tips of these ridges. However, we found these devices to have highly non-reproducible characteristics, perhaps because the oxidized nonplanar Si surface was exposed to ambient gases.

A device which is expected to give much more reproducible behavior as well as narrower confinement has now been fabricated. In this structure, the metallic gate electrode is patterned into narrow wires parallel to the direction of current flow on a planar surface. The wire widths are about 50 nm and they rest on an oxide layer which is also about 50 nm thick. We expect that this will confine the inversion layer to a width of less than 100 nm. The structure is made by reactive ion etching vertical steps in the gate oxide and subsequently evaporating metal on the sides of the steps. The first measurements of the characteristics of these devices are now underway.

17.12 Collaborative Projects

In addition to the above, the Submicron Structures Laboratory has served as a resource for a number of smaller collaborative projects from other laboratories. Some examples:

(a) Gratings with square wave profiles and periods as fine as 200 nm were fabricated in quartz and used to align several liquid crystals. As expected, alignment improved with decreasing grating period. Samples of butoxybenzilidene-octylanilene (a single layer smectic A, also known as 40.8) were aligned and their properties studied. This is work of H. von Känel and J.D. Litster.[15,16]

(b) Some first-order experiments to attach organic molecules to specific sites have been done by D. Day. Amino silane molecules were attached to an aluminum surface in a patterned monolayer. This pattern was then built up into a crosslinked network by several immersions in a bifunctional epoxide and a diamine.

(c) Thick polyimide has been reactive sputter etched in oxygen with extremely high (10:1) aspect ratios. For example, 5.0 μm wide lines have been etched in 50 μm thick polyimide. This is used to fabricate Fresnel zone plates for coded imaging by the Lawrence Livermore Laboratory. The work is in collaboration between A.M. Hawryluk of M.I.T. and N.M. Ceglio and G.F. Stone of Livermore.[17]

(d) Mylar meshes consisting of 60 μm thick material with a close grid of 25 μm diameter holes have been fabricated by J. Carter and R. McDonnell for Prof. D. Frisch. They will be used as surgical implants.

(e) The project of measuring electrical signals from living cells has been advanced by the fabrication of electrode structures consisting entirely of metal and silicon dioxide. (Earlier structures consisted of metal and hard-baked photoresist). This is work of Monica Buellesbach and Prof. R. Mark.

(f) A mask for making thin (Pb) film Josephson interferometers has been fabricated. The mask has bridge electrodes 2000 Å wide and 3500 Å long. These submicron structures were made using electron beam lithography and x-ray lithography. Both single and double interferometer samples will be used in a comprehensive program to evaluate the orientational and amplitude effects of magnetic fields on these superconducting devices. Then the Josephson interferometers will be used in an Aharanov-Bohm-type experiment to determine if they can serve to detect directly the vector potential field, A, which generates the classically observable E and B-fields. This is work for P. Stöhr, Prof. J. Bostock and Prof. M. MacVicar.

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