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ANNUAL REPORT
JULY 1, 1977

J. R. SITES
L.G. MEINERS
H.A. WASHBURN

COLORADO STATE UNIVERSITY

REPORT SF12

ELECTRONIC PROPERTIES OF III-IV SEMICONDUCTOR INTERFACIAL LAYERS

Annual Report: July 1, 1977

Contract N00014-76-C-0976

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Report SF12

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Results have been obtained on the use of neutralized ion beam sputtering of Si_3N_4 on GaAs for MIS applications and on classical and quantum electrical transport in InAs epilayers.

401269

I. Introduction

This report summarizes our results of the last twelve months. The two primary research areas were (1) neutralized ion beam sputtering of insulating barriers on GaAs for MIS applications and (2) classical and quantum electrical transport in InAs epilayers. During that period the following reports were submitted for publication:

1. "Oxide Barriers on GaAs by Neutralized Ion Beam Sputtering", L. G. Meiners, R. C. Pan, and J. R. Sites, J. Vac. Sci. Technol. 14, July/August 1977. [Report SF9; Abstract in Appendix A]
2. "Electrical Characterization of the Heteroepitaxial InAs on GaAs Interface", H. A. Washburn and J. R. Sites, Bull. Am. Phys. Soc. 22, 318 (1977). [Appendix B]
3. "Multilayer Model of InAs Epilayers", H. A. Washburn, Thin Solid Films, to be published. [Report SF11; Abstract in Appendix C]
4. "Deposition of Oxides and Nitrides by Neutralized Ion Beam Sputtering", J. R. Sites, 7th International Vacuum Conf. Proc., Vienna, 1977. [Appendix D]
5. "Oscillatory Transport Coefficients in InAs Surface Layers", H. A. Washburn and J. R. Sites, submitted to Electronic Properties of 2-D Solids Conf. and Surface Sci. [Will be Report SF13; Abstract in Appendix E]

In addition we have completed relocation of our laboratory, adding in the process several pieces of electral and optical analysis equipment. Of particular note is our new ISI Super IIU scanning electron microscope purchased in conjunction with several other researchers.

We have also added two new students to the program: Ms. Lynn Bradley from Clarkson College, and Mr. Joe Bowden from the Air Force. Both are currently working on the problem of using sputtered Si_3N_4 as an encapsulent during the annealing cycle following ion implantation of gallium arsenide.

II. Insulating Barriers

Silicon nitride layers were deposited on single crystal n-type gallium arsenide substrates by neutralized ion beam sputtering, and the electrical properties of the layers were measured. The sputter depositions were done using a one inch diameter beam ion gun. The general experimental techniques for doing the depositions as well as results from other dielectric materials have been described previously[1].

The target used was a 99.999% pure polycrystalline silicon blank. The sputtering gas was a mixture of Linde UHP nitrogen and Linde UHP argon. A background pressure of at least 3×10^{-4} torr nitrogen was necessary in order to get high resistivity layers. The sputtering rate was, of course, reduced when nitrogen was introduced into the vacuum system, and a partial pressure of 1×10^{-4} torr argon was required in order to obtain deposits. The smaller mass of the nitrogen atoms reduces the sputtering yield[2]. The vacuum system was equipped

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[1] L. G. Meiners, R. C. Pan, and J. R. Sites, J. Vac. Sci. Technol., Vol. 14, No. 4, (1977). (See Appendices A and D.)

[2] Handbook of Thin Film Technology, L. J. Maissel and R. Glang, McGraw-Hill, 1970.

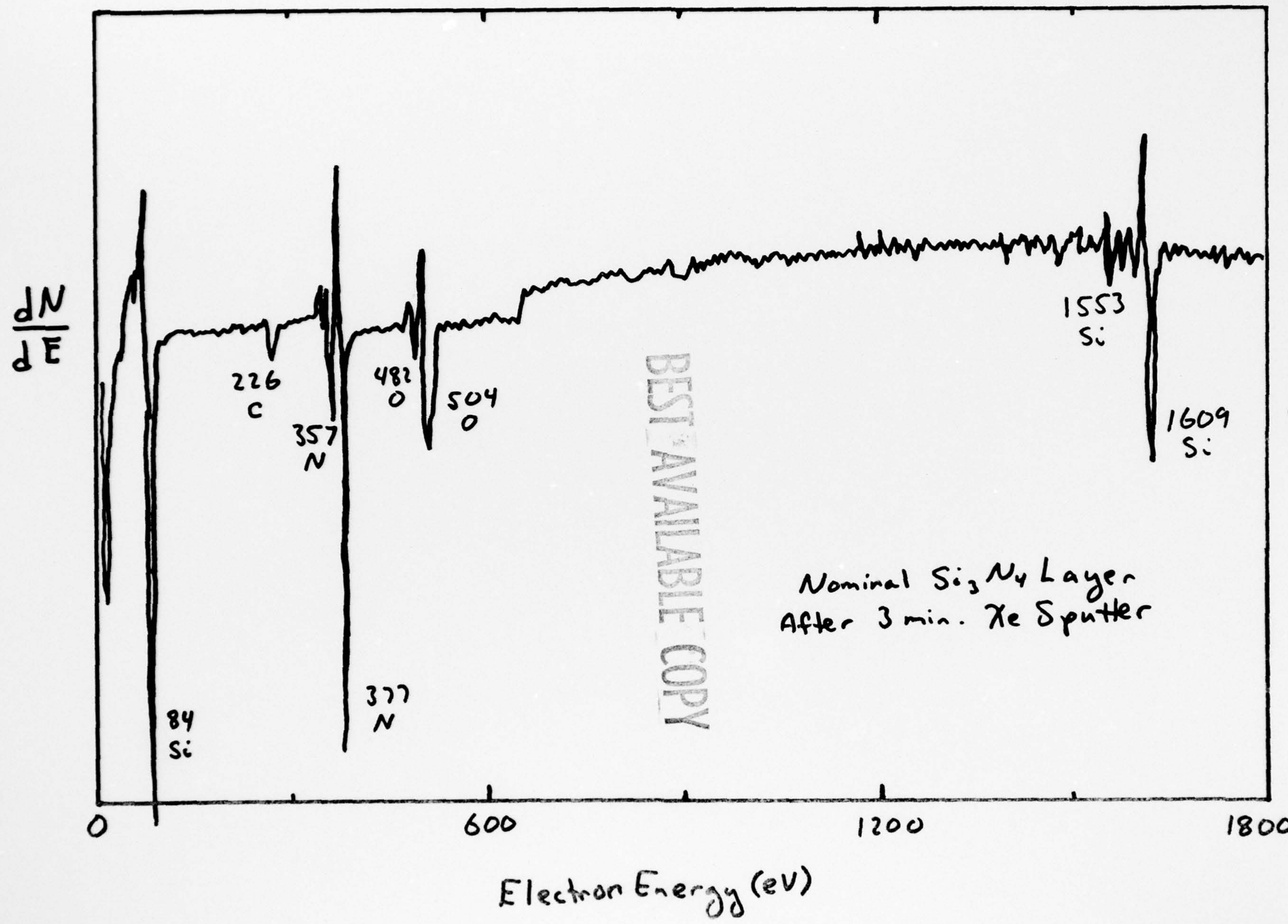
dN/dE
84 Si
226 C
357 N
377 N
482 O
504 O
1553 Si
1609 Si
Nominal Si_3N_4 Layer
After 3 min. Xe Sputter
0
600
1200
1800
Electron Energy (eV)

Fig 1

with a liquid nitrogen cold trap and the background pressure was 5×10^{-7} torr. The target and substrate were held in position with quartz fixtures in order to avoid incorporation of metal atoms into the layers.

Auger electron spectroscopy (AES) of the silicon nitride layers revealed that some oxygen contamination was present in all the layers studied. A typical spectrum, as shown in Figure 1, was obtained after sputter etching for three minutes in xenon at 1 KV. The oxygen definitely appears to be in the silicon nitride layers and is not just the adsorbed surface oxygen. Additionally there is an increased concentration of oxygen near the gallium arsenide surface. More data will be taken by Mr. Meiners, who has returned to NOSC, to obtain the percentage of oxygen concentration.

Aluminum dots were deposited on the layers and current-voltage and capacitance-voltage (CV) measurements were made on the metal-insulater-semiconductor devices. The resistivity of the layers was typically 10^{14}-10^{16} Ω cm. CV measurements were made in the frequency range 10^{2}-10^{6} Hz. Data for one of the samples is shown in Figure 2. The frequency dispersion of the curves is evidence for a distribution of surface states at the gallium arsenide surface. However, it is not yet clear that the CV data taken at 1 MHz corresponds to the theoretical "high frequency" curve, where surface states are no longer responding to the ac voltage. Further CV measurements at higher frequency are planned in order to be certain that the true high frequency curve has been obtained. In this respect the work is

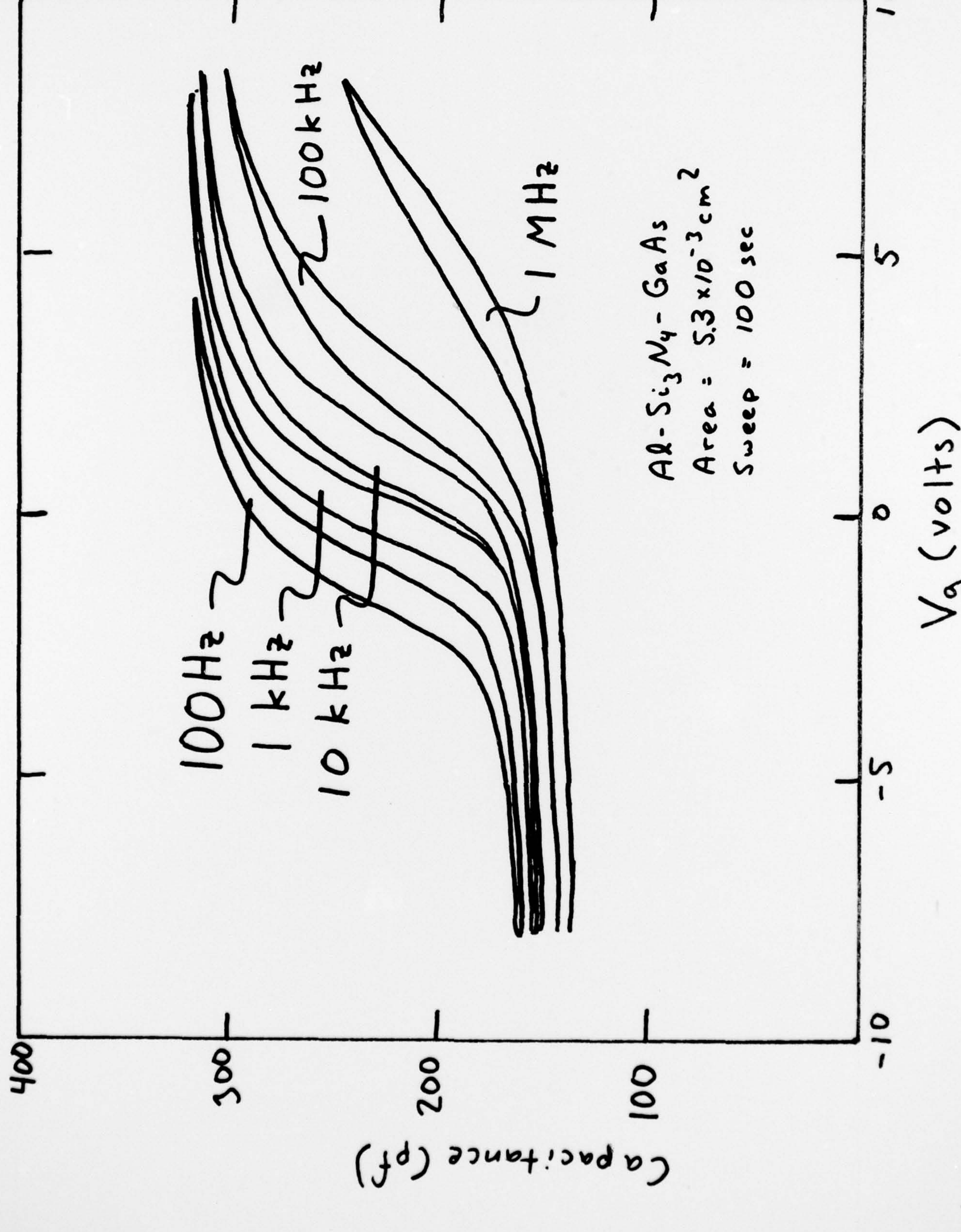
100Hz
1 kHz
10 kHz
100kHz
1 MHz
Al-Si_3N_4-GaAs
Area = $5.3\times10^{-3}cm^2$
Sweep = 100 sec
400
300
200
100
Capacitance (pf)
-10
-5
0
5
10
V_g (volts)

Fig. 2

quite similar to that of Zeisse et al.[3] on pyrolytically and anodically prepared insulators on gallium arsenide. The evidence to date points to the conclusion that on our samples the gallium arsenide surface is in depletion with zero gate voltage. Large negative gate voltages take the surface into inversion, but large positive voltages do not drive the surface past flat band. The same conclusion seems to apply to anodically and pyrolytically prepared layers. Further elucidation will be attempted by Mr. Meiners by taking high frequency (>10MHz) CV data on these samples, and by the use of saturated photovoltage measurements which give a direct measurement of the surface potential.

III. InAs Epilayer Transport

Recent measurements have emphasized the quantum regime of surface transport. Analysis of dc measurements in the higher temperature classical region were reported earlier.[4] At 4 K and below the InAs epilayers exhibited oscillations in the Hall coefficient and conductivity which can be explained by assuming the existence of quantized energy levels in the narrow surface accumulation layer. (See Appendix E) Two such levels have been observed and their position determined in terms of the gate voltage needed to produce them at zero magnetic field.

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[3] "Electrical Properties of Anodic and Pyrolytic Dielectrics on Gallium Arsenide", C. R. Zeisse, L. J. Messick, and D. L. Lile, J. Vac. Sci. Technol., Vol. 14, No. 4, (1977).

[4] H. A. Washburn and J. R. Sites, Bull. Am. Phys. Soc. 22, 318 (1977); H. A. Washburn, ONR Report SF11 [Appendices B and C]

In order to emphasize the oscillations, a small voltage modulation was added to the dc gate voltage and a lock-in amplifier was used to measure the differential voltage across the sample and Hall voltage. While this procedure complicates the analysis by shifting the phase of the oscillations, it should not introduce significant errors. Figure 3 shows a typical measurement which contains two primary components oscilltory in B^{-1}, which are identified with quantized levels. Measurements similar to Fig. 3 for other gate voltages show an orderly progression of the levels as the surface potential is varied.

While developing the above differential techniques, measurements were made at 77 K and results interesting in themselves were found. Primarily in the gate voltage region near flat-band condition and into depletion dramatic structure in the $\frac{dV_\sigma}{dV_G}$ and $\frac{dC}{dV_G}$ vs V_G curves were observed and they exhibited a dependence on frequency of the excitation voltage and on magnetic field. This structure is not reflected in the dc V_h, V_σ, and C vs V_G and it is thought that the results offer new information about the surface states and their lifetimes and positions in the InAs-oxide interface region. Analysis of the results is presently underway. Attempts have been made in the past to account for the hysteresis in the capacitance (and also seen by us in the conductance and Hall coefficient) vs. V_G by using charges inside the oxide and by using conduction through the oxide, and the current measurements may add some clarity.

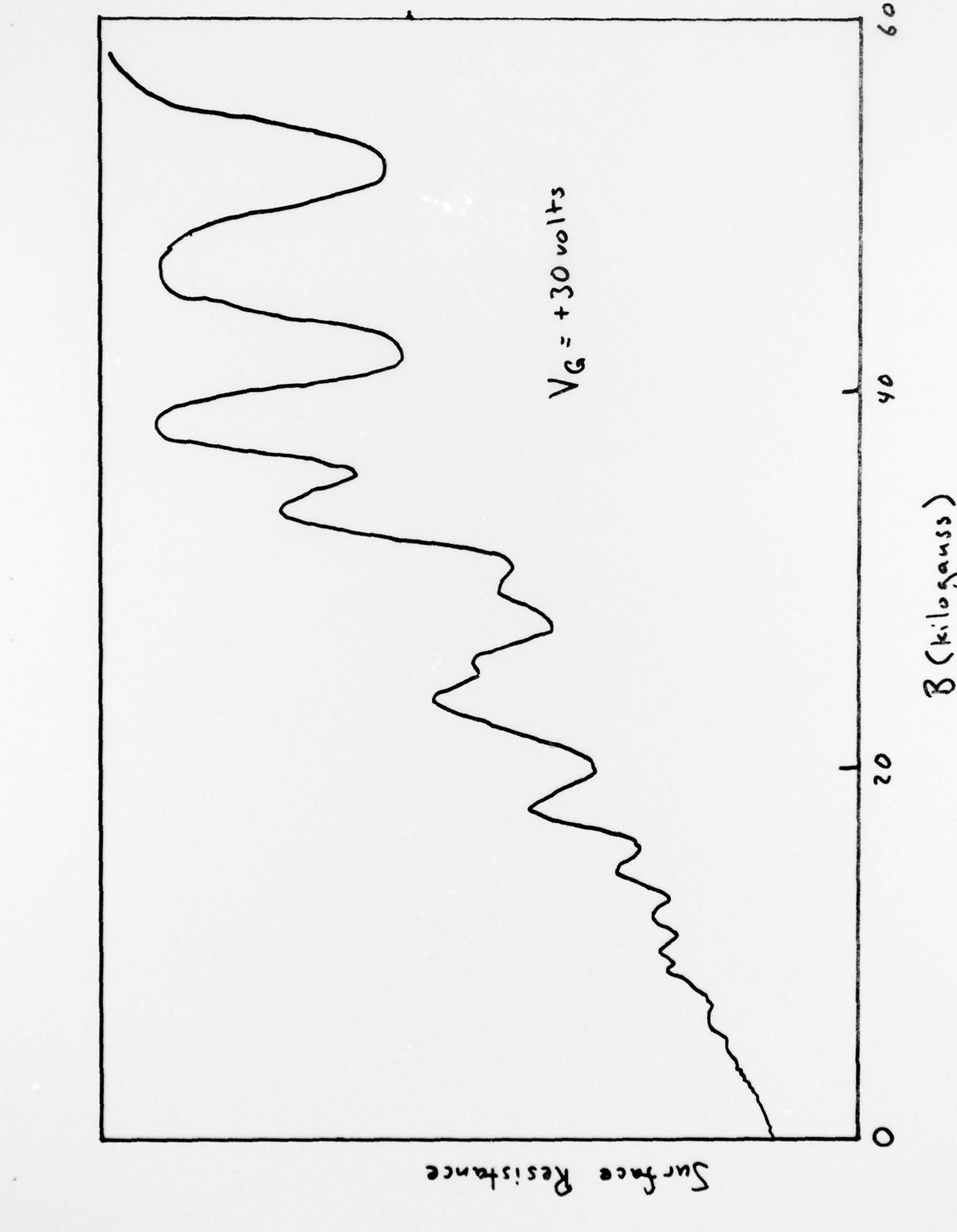

Fig. 3

IV. Appendices

Appendix A

OXIDE BARRIERS ON GaAs BY NEUTRALIZED ION BEAM SPUTTERING*

L. G. Meiners,† Ru-Pin Pan, and J. R. Sites

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ABSTRACT

Tantalum and silicon oxides have been sputter deposited onto gallium arsenide using a 500 eV beam of neutralized argon atoms. MIS devices show very low leakage and capacitances that can be varied from full accumulation to depletion with the application of modest voltages. Other measurements (breakdown field, dielectric constant, adherence, Auger profile, and photoluminescence) also suggest that these structures hold potential usefulness for insulated gate GaAs circuitry.

*Support by ONR (N00014-76-C-0976) and NASA (NSG-3086).

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Appendix B

Abstract Submitted

for the San Diego Meeting of the

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March 1977

Physics and Astronomy Classification Scheme Number 47.5	Bulletin Subject Heading in which Paper should be placed Semiconductor Transport

Electrical Characterization of the Heteroepitaxial InAs on GaAs Interface.* H.A. WASHBURN and J.R. SITES, Colorado State Univ.--Transport coefficients were measured on n-type InAs epilayers grown by VPE on semi-insulating GaAs and were found to exhibit a variation with magnetic field characteristic of an inhomogenious semiconductor. To analyze the measurements a multi-layer model was used which assumed three regions: the free InAs surface, a bulk-like layer, and the InAs-GaAs interface. By using an MOS structure for gate voltage control of the top surface and by sputter-thinning the epilayers with neutralized low-energy ion beam, the transport parameters for the interface region were deduced. Various profiles for the carrier density and mobility in the interface region were then evaluated by comparing the predicted magnetic field and thickness dependences of the Hall coefficient with measured values. A good fit was obtained using an exponential variation of the electron density with distance from the interface and an electron mobility limited by a defect density proportional to the carrier density. For the samples measured, the interface layer, at 77K, had a net electron density of about $2X10^{13}$ cm^{-2} and effective mobility of about 2000 cm^2/V-sec.

*Work Supported by ONR

Submitted by

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Appendix C

MULTI-LAYER MODEL OF InAs EPILAYERS

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SUMMARY

The charge carrier transport coefficients of an inhomogeneous, thin semiconductor in a MOS structure were obtained using a multi-layer model. The expression for the Hall coefficient of a three-layer system extended to arbitrary strength magnetic fields was used to separate the bulk transport parameters from the parameters describing transport at the two surfaces. Experimentally, a gate voltage was used to vary the surface under the oxide from depletion to accumulation and the Hall coefficient measured as a function of magnetic field. The characteristics of the back surface were obtained with the front surface held at the flatband condition. The variation of the front surface parameters with gate voltage was obtained with the front surface in accumulation. The measurements were made on a MOS structure consisting of an InAs epilayer deposited by VPE procedures on a semi-insulating GaAs substrate covered by a pyrolytic silicon dioxide insulating layer and aluminum gate.

Appendix D

DEPOSITION OF OXIDES AND NITRIDES BY NEUTRALIZED ION BEAM SPUTTERING*

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Abstract: A neutralized beam of argon ions has been used to sputter deposit conductive oxides for solar cell applications and dielectric oxides and nitrides for insulated gate structures on GaAs.

INTRODUCTION

Several types of high quality oxide and nitride layers have been sputter deposited using a neutralized ion beam source. This technique utilizes accelerated ions, generally argon, but differs from other sputtering techniques (see Fig. 1) in that all electric fields and charge separation is contained within the source chamber. The primary sputter beam is neutralized in the sense that it contains equal numbers of ions and electrons.

The basic beam source is a modified Kaufman thruster, first developed for spacecraft propulsion /1/. This type of beam has proven useful for high resolution pattern etching /2,3/, surface texturing /4,5/, and thin film deposition /6-8/. Advantages of using this type of beam for sputter deposition include (1) independent control of all parameters, (2) electrical neutrality at both target and substrate, (3) relatively low energy (80-800 eV), collimated beam, (4) ability to sputter insulating targets without special preparation, (5) straightforward in situ cleaning of both target and substrate, (6) relatively low bell jar pressure ($<10^{-4}$ torr), and (7) ease of reactive sputtering. The drawbacks are (1) the relatively slow deposition rate ($\sim 1\mu$ hour) and (2) the somewhat complex source required.

DEPOSITION

The neutralized ion beam source is shown schematically in Fig. 2. Accelerated electrons from a hot cathode collide with the source gas producing positive ions. The number of ions produced, which determines the density of sputtering particles, can be varied by adjusting the cathode current. This density is increased by lengthening the effective cathode to anode path for the electrons through an arrangement of permanent magnets with fields the order of 100 gauss. The positive ions are

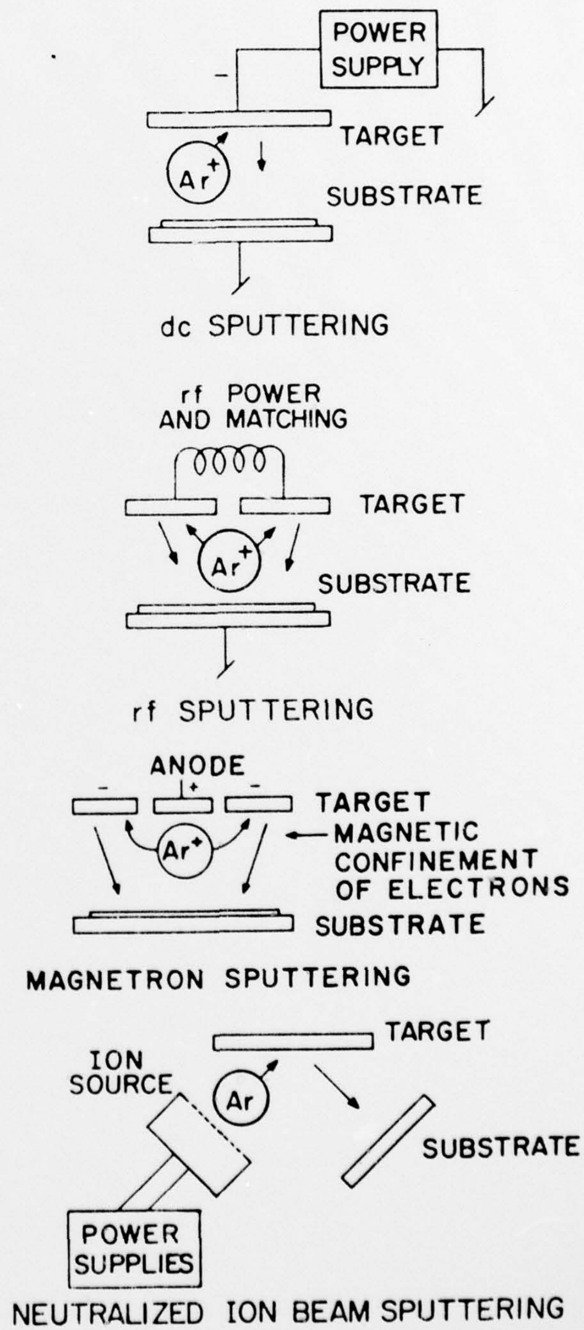

Fig. 1. Comparison of sputtering techniques.

*Supported by ONR, NASA, and ERDA.

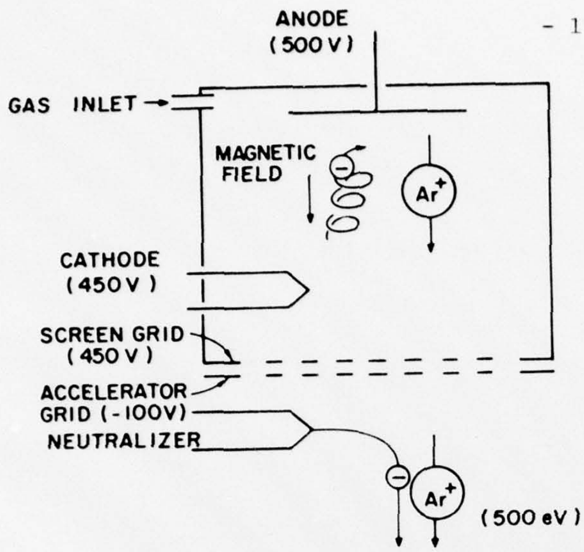

Fig. 2. Neutralized ion beam source.

accelerated by a negative voltage on the lower grid. The upper, or screen, grid is positively biased to deflect the ions so they will pass through the holes. Both grids are made of graphite sheet with the holes in hexagonal arrays aligned with one another.

Ions emerging from the source pass by a hot filament which injects sufficient electrons to neutralize the beam. The accelerator grid is kept slightly negative to prevent these electrons from backstreaming into the source. The resulting beam consists of positive ions of energy equal to the anode potential, which can be varied. These particles all have very nearly the same velocity both in magnitude and direction. The neutralizing electrons have much higher velocities, randomly oriented with an average component equal to that of the ions. (See Fig. 3.)

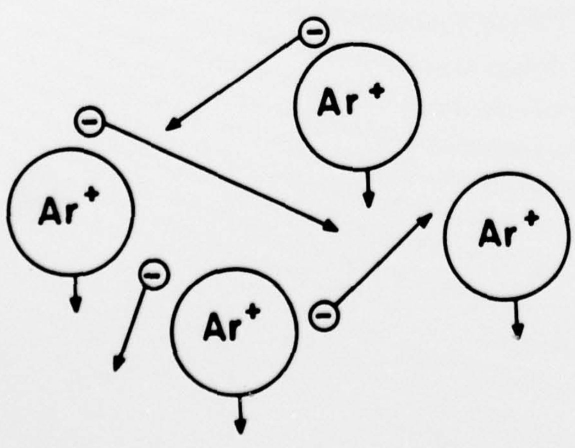

Fig. 3. Schematic of beam.

There is a probability, proportional to the residual gas pressure, of the positive ions neutralizing through charge exchange, and a much smaller probability of direct recombination to the ground state. One consequence of the higher electron velocity is that the beam tends to be slightly more negative at the edges than in the center. The beam also tends to dissipate somewhat in both energy and intensity due to collisions with residual gas atoms. By introducing the argon, or other gas, directly into the source chamber, well sealed except for the grid holes, this problem can be minimized.

The arrangement for sputter deposition is shown in Fig. 4.

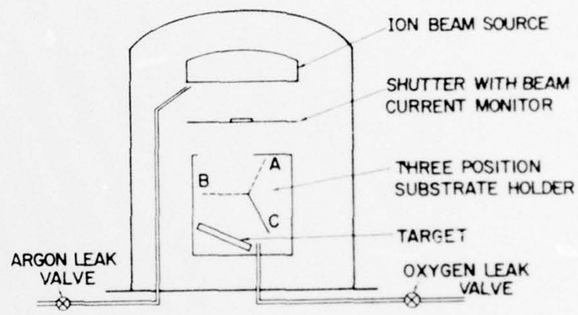

Fig. 4. Sputter deposition arrangement.

During start-up the beam is blocked by a shutter. The attached beam current monitor measures the ion current falling on a fixed area when the monitor is biased to repel electrons. By translating the shutter, it can also be used to measure the beam uniformity. Neutralization is obtained at zero net current when the monitor is at ground potential.

An aperture above the target and substrate constrains the beam cross section to be no greater than the projection of the target in its plane, hence minimizing stray sputtering. Other parts of the fixturing are constructed of materials that will not introduce disasterous impurities in deposited layers if stray sputtering should occur. The target itself can be a metal, insulator, packed powder, or essentially any physically contained, low vapor pressure material.

Pre-deposition sputter etching of the target and substrate is generally advisible. In the configuration pictured, the substrate is rotated first to position A where the beam falls on the target, but the sputtered particles are shielded from the substrate. In position B the substrate is cleaned. This step is much more critical because one wants to avoid surface damage. Our procedure has been to reduce the beam energy to about 100 eV and to etch for about five minutes.

In position C actual sputter deposition takes place. The angles and positions of the target and substrate were chosen to give reasonably uniform coverage and still keep the substrate out of the beam path. In many cases oxygen or nitrogen is introduced between target and substrate to produce a film chemically different from the target used or to fine tune the stoichiometry of the deposited layer. The substrate holder does contain a heater which is some cases improves the quality of the device being fabricated. Ambient temperatures during deposition in the absence of such heating are the order of 400°C at the target and 200°C at the substrate.

RESULTS

The oxides and nitrides that have been deposited are summarized in Table I. These depositions include both conductive oxides on silicon for solar cell applications and dielectric layers on GaAs for insulated gate structures and encapsulation. In each case, when the pre-deposition sputter etch was performed in situ, the films showed good adhesion, resistance to abrasion, and reproducible properties from run to run. Both the direct sputtering of the material to be deposited and reactive sputtering with a metallic target were successful and yielded roughly comparable films. Thicknesses were generally between 500 and 5000 Å and deposition times from ten minutes to an hour.

The conductive indium tin oxide (ITO) layers are small crystallite, degenerate, n-type semiconductors. They form good quality heterojunction diodes when deposited on p-type single crystal silicon /9/.

The attractiveness of this structure as a solar cell stems from the transparency of the ITO layer (>85% over the sun's spectrum), the built in anti-reflecting index of refraction of ITO, the relatively low resistivity of the layer (5×10^{-4} Ω-cm), the reasonable built-in voltage (0.9 volt at room temperature), and the high internal quantum efficiency (95% with the sun's spectrum).

Fig. 5 illustrates both the dark current diode curve and the solar cell curve using the sun (93 mW/cm^2) for illumination. This particular cell was 0.07 cm^2 in area and had a front layer composition of 91% In_2O_3 and 9% SnO_2. The resulting open circuit voltage (0.51 volt), short circuit current (32 mA/cm^2), and diode fill factor (70%) lead to a conversion efficiency slightly above 12%. In comparison with diffused junction silicon solar cells, this structure has a somewhat lower open circuit voltage, but a higher short circuit current due to an enhanced spectral response to the blue part of the spectrum, presumably because the diffused junction surface dead layer has been eliminated.

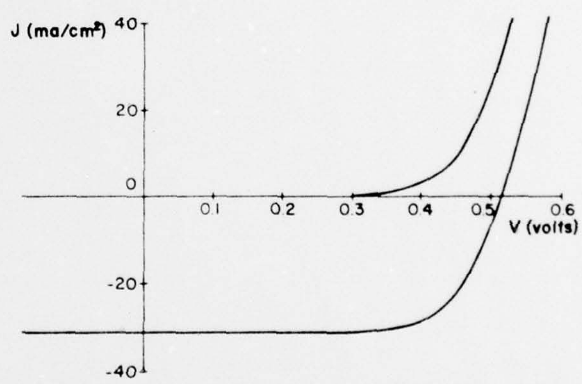

Fig. 5. ITO/Si diode curves, with and without light.

Table I. Summary of materials deposited.

Material	Target	Atmosphere	Substrate	ρ	Purpose
Indium Tin Oxides (several compositions)	ITO	Slight O_2	Silicon, 375°C	5×10^{-4} Ω-cm	Heterojunction Solar Cells
Tantalum Oxide	Ta	O_2	GaAs, 25°C	10^{12}	MIS
	Ta_2O_5	--	GaAs	2×10^{11}	
Silicon Oxide	Si	O_2	GaAs, 25°C	$>10^{15}$	MIS
	SiO_2	--	GaAs	10^{12}	
Silicon Nitride	Si	N_2	GaAs, 25°C	$>10^{15}$	MIS; encapsulation

The sputtering of dielectric layers onto GaAs has shown that it is possible to form a high resistivity ($>10^{15}\Omega$-cm), high breakdown ($2\text{-}3 \times 10^6$V/cm) film by the neutralized ion beam technique, that the GaAs surface can be driven from depletion to accumulation through the application of a gate voltage (See Fig. 6), and that

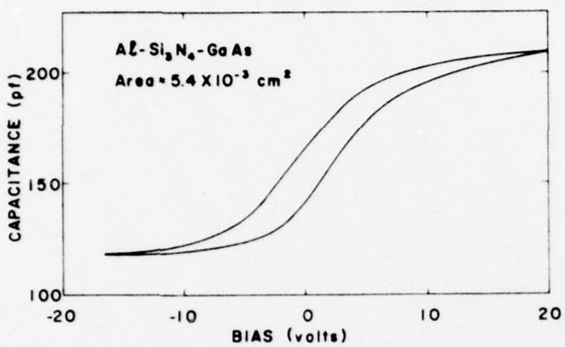

Fig. 6. Capacitance-voltage curve of MIS structure on gallium arsenide.

surface state densities have a similar distribution and magnitude to those found when insulating layers are formed on GaAs by some of the alternative techniques /10/. Best results to date have been achieved using Si_3N_4 on the <100 A> surface of GaAs. The silicon nitride films additionally form an encapsulating layer for annealing of ion implantation layers.

The critical fabrication procedure to achieve the high resistivity seems to be the removal of any impurity materials from the support fixturing. Replacement of the original stainless steel with quartz led to a three order of magnitude improvement. For minimizing surface state density, it seems most critical to perform the final sputter etch before deposition with a reduced energy beam.

ACKNOWLEDGEMENTS

I am grateful for the collaboration of Harold Kaufman in the development of ion beam techniques, Joel DuBow and Norman Chang in the solar cell development, and Larry Meiners in the MIS studies.

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Appendix E

OSCILLATORY TRANSPORT COEFFICIENTS IN InAs SURFACE LAYERS

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Magneto-oscillations have been observed in the conductivity and Hall coefficient of InAs films grown heteroepitaxially on GaAs substrates. An MOS structure was employed so that the surface layer could be driven from depletion to strong accumulation through application of the appropriate gate voltages. Differential measurement techniques were used to clarify the oscillations in the transport properties. Magnetic fields up to 6 tesla and temperatures down to 1.6K were used.

The observed oscillations are attributed to energy quantization in the surface accumulation layer. Our interpretation shows two subbands with densities at zero gate voltage of $7 \times 10''cm^{-2}$ and $3 \times 10''cm^{-2}$. The mobility of the excited subband was approximately twice that of the ground state subband.

The oscillations showed very little temperature dependence between 1.6 and 4.2K. We also observed very little temperature dependence of the flat band voltage or the bulk Hall coefficient, indicating the absence of freeze out effects. We did, however, see some splitting of the lower subband oscillations at higher magnetic fields suggesting spin splitting of the Landau levels for this subband.