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During the past year we have been able to complete the part of our ONR sponsored program concerned with InAs epilayers. At the same time we have made progress with our Si_3N_4 studies and have upgraded our capabilities for low energy ion beam sputter deposition. For the coming year we are concentrating our efforts on silicon nitride and aluminum nitride dielectric layers and on the effects of ion beam impingement on semiconductor surfaces.

1. Indium Arsenide Epilayers

We have completed our program of characterizing and profiling the electronic properties of n-type InAs epilayers. We have been fortunate to have high quality samples grown at the Naval Ocean Systems Center for these studies. Our primary conclusions are:

- (1) Although relatively defect-free epitaxial InAs can'be grown on GaAs, there is an initial layer of about one micron characteristic distance that has reduced mobility and increased carrier density.
- (2) The surface mobility of electrons in InAs is found to be quite temperature independent; it varies strongly with surface potential in a manner consistent with diffuse surface scattering.
- (3) At low temperatures surface quantization effects lead to three observable subbands for the accumulation layer electrons.
- (4) The effective mass of electrons in InAs accumulation layers is significantly larger than the band edge mass. The factor of three increase seen is consistent with that anticipated from the non-parabolicity of the conduction band.

-1-

Some of this work has been published previously,¹⁻⁵ and much of it is found in Dr. Hudson Washburn's Ph.D. thesis (Colorado State University, January 1978). A final paper (Report SF15, see Appendix A for abstract) has been recently submitted to the Journal of Applied Physics.

2. Gallium Arsenide MIS Structures

The project to use low energy ion beam sputtering techniques to deposit SiO_2 layers on GaAs substrates for metal-insulator-semiconductor (MIS) structures has been continued by Mr. Larry Meiners since his return to the Naval Ocean Systems Center. He has extended this work to a rather comprehensive study of several types of insulators on gallium arsenide. The results seem very significant and imply some rather general limitations on our ability to externally control the surface potential of GaAs. Mr. Meiners has published⁶⁻⁸ some aspects of this work, and a comprehensive report based on his Ph.D. thesis will be distributed later this winter.

3. Silicon Nitride Encapsulation

Our work during the last year with low energy ion beam sputtering has been concentrated on Si_3N_4 encapsulation applications. Ms. Lynn Bradley has

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- 8. L. G. Meiners, Appl. Phys. Lett. 33, 747 (1978).

-2-

performed a systematic study to determine the best procedures for deposition of silicon nitride layers that would withstand high temperature (> 900°C) annealing cycles. The conclusions from this study were:

- (1) Reactive sputter deposition of Si_3N_4 using an argon ion beam works well as long as the nitrogen gas is introduced in an ionized state and the nitrogen to argon ratio is about three to one.
- (2) Surface preparation was critical to mechanical adhesion during annealing. Chemical etching just before introduction to the vacuum seemed very effective. Ion beam sputter etching <u>in situ</u> was somewhat less effective.
- (3) As with many Si₃N₄ preparation techniques, a certain level of oxygen impurity in the layers seems inevitable. In our case, the oxygen content is larger in the first 100 Å of the dielectric layer.
- (4) At annealing temperatures of 600°C and above, there is a significant diffusion of silicon into the gallium arsenide. Mr. Joe Bowden has made photoluminescence measurements showing that radiative silicon complexes result from thermal annealing of the $Si_3N_4/GaAs$ structures.
- (5) A simple ellipsometric measurement of the Si₃N₄ index of refraction has proven to be a reliable means of screening poor quality samples.

This work is being distributed as Report SF14 (See Appendix B for abstract). It will be presented at the November 1978 American Vacuum Society Meeting in San Francisco and will appear in the Journal of Vacuum Science and Technology early next year. A more detailed description will be available in Ms. Bradley's M.S. thesis.

-3-

4. New Sputter System

In April 1978, we began using a new ion beam sputter deposition system, partially custom designed for the type of structures we have been fabricating. This system, shown in Fig. 1, has a stainless steel vacuum chamber with hinged doors on each and for easy access. The cabinet below contains a cryopumping system with automatic valving. The pump reaches 5×10^{-9} torr and the vacuum chamber 3×10^{-8} torr.

The Kaufman-type ion source (Fig. 2) is mounted on one door to expedite replacement of filiments. Using argon, it produces a uniform intensity two inch diameter beam which can be neutralized with a hot filament electron source. The ion beam current is variable from 0 to 50 ma and its energy from 50 to 1500 eV. The chamber pressure during operation can be maintained as low as 2×10^{-5} torr.

Also shown in Fig. 1 is the electronics console and the two high purity gas cylinders, argon and nitrogen in this case, connected to the ion source. The fixturing in the vacuum chamber is relatively straightforward. A moveable shutter blocks the beam during start up adjustments. The target holder, also moveable from the outside, allows easy interchange of target material (high purity silicon and aluminum to date). There are two substrate holders, one which can be heated to 400°C and the other which can rotate during deposition, driven by the small motor seen in Fig. 2. Either substrate holder can be turned to face the ion beam for pre-deposition sputter etching.

The new ion beam system has now run fairly smoothly for six months. In addition to the deposition of nitride dielectric layers described below, it has been used for a NASA funded program to fabricate hydrogenated amorphous silicon and for several small tasks.

-4-



Fig. 1. Low energy ion beam sputtering system.



Fig. 2. Two inch diameter ion beam source just after replacement of neutralizing filament.

5. Silicon and Aluminum Nitride Films

The current work on dielectric films is concentrated on the deposition of aluminum nitride films for encapsulation and MIS purposes, and on the surface electronic properties of GaAs which has been overlaid by either Si_3N_4 or ALN. Mr. Sung Pak from Korea has been doing the sputtering, Mr. Joe Bowden the photoluminescence studies, and Dr. Hülya Birey, a visitor from Turkey, the electrical and optical measurements.

The aluminum nitride deposition closely parallels that of the silicon nitride described above. A high purity aluminum target replaces the silicon wafer. Initial results show that high resistivity (> $3 \times 10^{13} \ \Omega$ -cm) layers the order of 500 Å can be relatively easily deposited. We have not yet subjected these films to high temperature annealing cycles. The surface electronic properties are also just beginning. We have seen through capacitance-voltage measurements that the Fermi level can be moved somewhat when either a Si₃N₄ or ALN dielectric layer is used. At this point we have not made frequency dispersion, temperature dependence, or surface conductivity measurements.

6. Surface Damage

A second project being pursued at present by Mr. Helmut Schmidt and Mr. Phil Jensen is a study of GaAs surface damage resulting from ion beam bombardment. The motivation for this project is to understand the effects of pre-deposition sputter cleaning on the surface electronic properties. To date we have measured the current-voltage and capacitance-voltage curves of Schottky barriers made on sputtered surfaces of GaAs. They show a progressive decrease in barrier height as the argon ion beam energy is increased. For practical purposes, the barrier disappears with a bombardment energy around 200 eV. Other measurements currently underway include

-6-

measurement of damage depth using a calibrated chemical etch, photoluminescence evaluation of the bombarded samples, and surface conductivity studies of the damage layer.

Appendix A

SILICON NITRIDE LAYERS ON GALLIUM ARSENIDE BY LOW ENERGY ION BEAM SPUTTERING

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ABSTRACT

Silicon nitride layers are formed on gallium arsenide for encapsulation purposes. The process utilizes a 500 eV neutralized ion beam containing argon for sputtering and nitrogen for reactive deposition, directed at a pure silicon target. It is found that with proper surface preparation layers having mechanical stability to above 900°C can be formed. Photoluminescence shows that no radiative transitions are introduced in the deposition process, but that annealing inevitably leads to diffusion of silicon into the GaAs. Auger studies reveal significant oxygen impurity in the Si_3N_4 , particularly near the interface. Index of refraction was found to be a sensitive, non-destructive test of encapsulant quality.

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-8-

Appendix B

ELECTRONIC PROFILE OF n-InAs ON SEMI-INSULATING GaAs

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ABSTRACT

The electron density and mobility of VPE grown 15 µm n-type indium arsenide epilayers have been determined as a function of distance from the gallium arsenide substrate. Both epilayer surfaces show significant increases in density and decreases in mobility from the bulk values $(10^{15}-10^{16}\text{cm}^{-3} \text{ and } 10^5\text{cm}^2/\text{V-sec}$ at 77°K). The interfacial, or back, surface is apparently dominated by defects to a depth of about 3 µm. The density and mobility profiles are roughly exponential; integrated values are $1.6 \times 10^{13}\text{cm}^{-2}$ and $2 \times 10^3\text{cm}^2/\text{V-sec}$. The front surface, highly dependent on applied gate bias, has a density range in accumulation from 0 to $5 \times 10^{12}\text{cm}^{-2}$ and mobility from 2.5 × 10^4 to $3 \times 10^3\text{cm}^2/\text{V-sec}$. The parameters for both surfaces are essentially temperature independent below 80° K. The front surface effective mass increases with electron density from its band edge value of 0.0215 m_a to nearly 0.06 m_a.

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