### Indiana University of Pennsylvania

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1011 South Drive
Idiana, Pennsylvania 15705-1087

724-357-3184
Fax: 724-357-2332
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January 5, 1999

Mr. David L. Purdy RD #2 Box 121A Marion Center, PA 15759-9003

Dear Mr. Purdy:

I hope you, Peg and the rest of your family had a lovely holiday season filled with moments that will someday be fond memories.

I am happy to provide you with the enclosed report prepared by Dr. Dennis Whitson, professor of physics, detailing the use and impact of the gift you and Mrs. Purdy made to Indiana University of Pennsylvania in 1995.

I hope this report will demonstrate the impact your generosity has had in strengthening the learning environment and research capabilities of our students and faculty. Thanks to you, today's students are learning the skills needed to become tomorrow's scientists and researchers.

If you have any questions concerning the enclosed report, I am certain Dr. Whitson would be happy to discuss them with you. Also, please do not hesitate to call upon me if I can be of further assistance.

Once again, I thank you for your continuing friendship and interest in the University and for your leadership efforts with the Foundation for IUP.

Wilte best wishes to you for the New Year.

Sincerely,

cc:

Shari Trinkley

Director, Major & Planned Giving

Dr. Joan M. Fisher, Vice President for Institutional Advancement

Ms. Barbara A. Ender, Executive Director, Foundation for IUP

Dr. Dennis W. Whitson, Professor, Department of Physics

### Development of a Schottky Barrier Infra-Red Detector in Conjunction with Biocontrol Technology

Submitted by:

Dr. Dennis Whitson, Professor of Physics, Physics Department,

Indiana University of Pennsylvania (IUP)

Other Personnel:

Dr. Kathy Meehan Mr. Layi Fapohunda Mr. David Ramsey Mr. John Ball

Background

This is a joint project with Biocontrol Technology, Inc. of Indiana, Pennsylvania which was funded by a generous gift of Biocontrol Technology stock from David and Margaret Purdy. The stock was sold for \$87,062 and the money was used to purchase and refurbish an used sputtering machine. An accounting of how the money was spent is given in the spread sheets that accompany this report.

Biocontrol personnel have developed a non-invasive blood glucose monitor for use by diabetics. The technique used to predict the blood glucose level is based on the analysis of the reflected and re-emitted spectrum of the skin obtained by illuminating the patient's arm with infrared radiation. A key component in the system is the infrared photodetector. Biocontrol Technology is interested in working with the Physics Department at IUP to evaluate possible alternatives to the photodetector currently used in its monitor. We are attempting to decrease the component cost and to increase the detector stability.

The research effort at IUP is based on Schottky barrier diodes on silicon wafers. The wavelength of the infrared radiation that can be detected is determined by the metal used for the formation of the Schottky barrier. No one has developed this type of photodetector for use over the wavelength range required for Biocontrol's application. The goal for this project is to detect electromagnetic radiation over much of the infrared spectral range cheaply and efficiently. As silicon is used in most semiconductor components and Schottky barriers are commonly used in the fabrication of these components, the photodetector technology developed at IUP can be readily transferred to large-scale manufacturing. The sputtering machine will play a crucial role in this research since it is extremely versatile and can be used to deposit many different materials on many different substrates.

**Refurbishing the Sputtering Machine** 

The sputtering machine was purchased as an used machine and it had been sitting in a warehouse for a few years before we purchased it. Because of this, many parts had deteriorated and many parts were missing. The following is a partial list of the repairs and purchases that had to be made in order to make the sputtering machine operational. It should be kept in mind that most of these repairs and purchases could not be done in parallel since we had to fix one problem before we would find out about the next problem.

- 1. The backing pump that came with the unit had water and other contaminates in the oil and the price of sending it back to the manufacturer to be cleaned and refurbished wasn't much less than the purchase of a new pump and the future reliability of the repaired unit could not be warranted. Therefore we purchased a new pump.
- 2. The cryogenic absorbing array for the cryogenic pump had to be replaced.

- 3. The vacuum and gas valves are actuated by air pressure and the vacuum chamber cover is lifted by a piston that is actuated by air pressure. We had to purchase an air compressor and then we had to replace all of the air lines in the unit since they had deteriorated and were leaking.
- 4. We were missing many of the manuals for the machine and we had to find out who had these manuals and purchase them.
- 5. In order to hook up the water chiller we had to purchase parts for it.
- 6. We had to purchase and replace some vacuum gauges.
- 7. In the planetary system, there are ceramic sleeves that isolate the sample holder from electrical ground. Only a few of these ceramic sleeves came with the system. We had to have new sleeves custom produced and when we received the sleeves we found out that the inner diameter was too small and had to be reamed out. A method had to be developed in order to enlarge the inner diameter without breaking the fragile ceramic sleeves.
- 8. Some of the bearings in the planetary system were bad and had to be replaced. The problem here is that they had to be oil free since they are in a high vacuum during the deposition process.
- 9. Air lines, gas (Argon and Nitrogen) lines, and regulators had to be installed.
- 10. The RF power supply had to be sent back to the manufacturer in order to be fixed. This caused a few months delay.
- 11. The RF wave guide was missing and the drawings that we had were not very clear about its shape and size. Using a cut and try method we were able to produce one that worked.
- 12. The quartz lamps that are used to heat the vacuum chamber were not operable so we had to find someone (Yeagle Technology) that could construct a new lamp for us.
- 13. The sputtering machine only came with one sputter gun, but with three positions for sputter guns. All three guns were needed to implement the photodetector as originally conceived, thus we needed to purchase two more sputter guns. As with the quartz lamps, these can not be purchased on the open market so we had to find someone (Yeagle Technology again) that would construct them for us.
- 14. With the three sputter guns in place, it was obvious that the shutters used to regulate the deposition of the target materials were not working correctly. We had to redesign the shutters and the pistons used to rotate the shutters in and out of place.
- 15. All the water hoses used for cooling the sputter guns had to be put in.
- 15. The motor that drives the planetary system is outside of the vacuum chamber (it would contaminate the vacuum chamber if it was inside). The Ferrofludics feedthrough that translates the motion into the vacuum chamber had to be replaced.

**Fabrication of Schottky Barrier Diodes** 

In order to have a Schottky Barrier Diode one must have an Ohmic contact and a barrier (or rectifying) contact. The ohmic contact is usually made by depositing Al on the silicon and the barrier contact is made by depositing a metal such as Ni, Pt, Ti, Co, or W on the silicon. In order to know the thickness of Ni deposited on the silicon we had to calibrate the crystal monitors for Ni and we will have to calibrate for each metal.

We fabricated a number of Schottky barrier diodes, some of which produced good I-V (current-voltage) characteristics. Some of the better results are shown in the accompanying graphs. However, many of them would break down after a short time and would no longer rectify the signal. There are two possible reasons for this problem: (1) dust and dirt being incorporated in the device and (2) samples being exposed to oxygen during the annealing process. The first problem should be alleviated with our new clean room which is about ready to go on line. The second problem would be alleviated by the purchase of an annealing oven (approximately \$5000).

A series of diodes were fabricated with different annealing temperatures using Ni as the barrier metal (see the attachment "Independent Study Report by Layi Fapohunda"). The barrier height and therefore the range of sensitivity to the infrared radiation should depend on the annealing temperature. The approximate barrier height can be found from the I-V curve. Unfortunately the results were inconclusive. It is felt that the problems mentioned above along with a problem with the shadow mask caused these results. The shadow mask is attached to the wafer and then deposition of one of the contacts is made. The mask is moved over and the second contact is deposited. The problem is that there is no way to accurately locate the second placement of the shadow mask with respect to the first placement. We are in the process of designing a new shadow mask where the dots will be a chosen distance apart.

A series of diodes were sputter etched before deposition to see if the etching would inprove the I-V response. The conclusion was that all the materials with high etch power showed poor contact, i.e. the I-V response was poor.

Photo-response of the Schottky Barrier Diodes

In order to measure the photo-response of the Schottky Barrier Diodes we purchased the following equipment: (a) Monochrometer, (b) Computer, (c) Wavelength Drive, and (d) Infrared source. This equipment was tuned up and calibrated using Ge detectors (see attachment). However, no response was discernible using the equipment to measure the Schottky Barrier diodes. It is felt that a lock-in detector and light chopper are needed to measure this response. The light chopper has been purchased and money (other than Biotechnology Institute money) has been found to purchase the lock-in detector. It will be ordered shortly.

### **Down Time**

The sputtering machine has been out of commission since May of 1998 because of the construction of a class 1000 clean room in Room 2 of Weyandt Hall. The money for this project came from the National Science Foundation (NSF) funding a grant proposal submitted by Dr. W. Larry Freeman and Dr. Dennis Whitson. IUP matched the funds from the NSF. It is felt that the probability of success in the fabrication of the Schottky barrier diodes will be greatly increased with the addition of this clean room. We should be able to move into the new facility the week of January 11th, 1999.

### **Future Work**

Besides the items mentioned above (new clean room, new shadow mask, new lock-in detector, and new annealing oven) there are some other initiatives that will be explored. (1) Using a "finger" geometry for the contacts will increase the current flow of the diodes and make them more sensitive to the infrared radiation. In order to do this we need a mask aligner. We have an used one which was donated to us from the University of Pittsburgh and we are going to see if we can make it operational. (2) Using anti-reflective coatings on the diodes. We will probably have to go to an outside source for this coating.

One other piece of equipment that may need to be replaced is the water chiller (\$7000). We will again attempt to fix the current water chiller, but it has been repaired about four times and it continues to be unreliable.

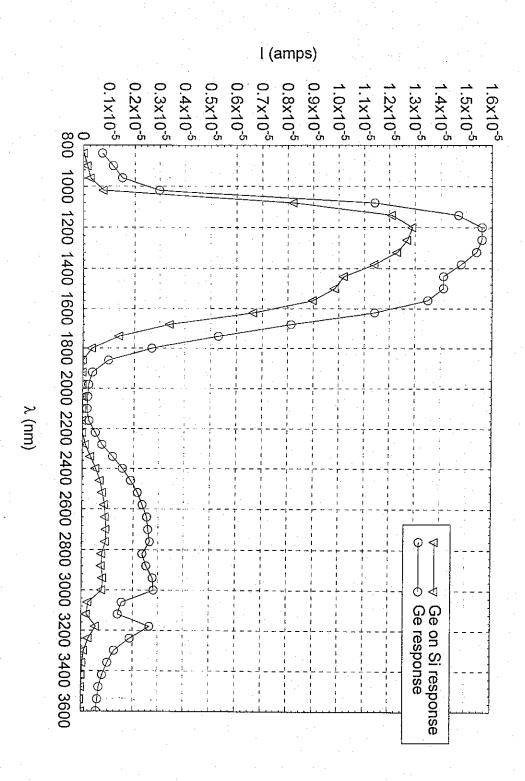
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х	Commission for sale of stock	\$1,411.14	5/31/95	
Х	Foundation Fee	\$2,325.00	6/30/95	·
Х	Leybold Vacuum Products, Inc.	\$5,404.20	7/11/95	7/20/95
	Bosch Sputtering Unit	\$14,217.00	6/24/95	7/6/95
	Layi's Salary Transfered to 521216	\$1,587.02	7/14/95	X
$\overline{}$	Ramsey's Salary transferred to 521216	\$3,512.95	7/14/95	Х
	Cryogenic Absorbing Array	\$463.27	7/14/95	8/22/95
$\overline{}$	Air Compressor (CT) / Farm & Country	\$356.12	7/14/95	7/14/95
	Petty Cash-Dave Ramsey-Supplies	\$18.20	7/18/95	Х
	Innotec / Manual for Sputtering Machine	\$229.76	7/25/95	8/17/95
-	Petty Cash - Dave Ramsey-Air Compres.	\$12.50	7/25/95	Х
	Lesker Co. / Instrument Manual MMPC	\$85.00	7/21/95	7/31/95
X	Shank Spec. / Water Chiller Parts	\$196.20	7/31/95	7/31/95
X	McMaster-Carr / Air Comp. Supplies	\$278.60	7/21/95	7/31/95
X	AEC / Pressure Gauge / Manual	\$88.00	7/31/95	9/15/95
X	Leybold Inficon / Manual IC6000	\$67.99	8/2/95	8/10/95
X	U.S. Tech Ceramics / Ceramic Sleeves	\$700.00	9/2/95	9/22/95
X	Technotrade Inter / Vacuum gauge	\$425.00	9/5/95	9/21/95
Х	Allied Electronics / Ceramic Sleeve	\$27.50	9/8/95	9/21/95
Х	CTI-Cryogenics / Manifold & Hose	\$513.00	9/11/95	10/2/95
Х	Leybold Vacuum / trap, flange	\$680.80	9/18/95	10/13/95
X	Bearings, Inc. / Bearings for planetary Sys.	\$785.71	9/19/95	9/28/95
X	Erie Bearings Col.	\$33.13	9/28/95	10/5/95
X	McMaster Carr / PVC Vacuum Tube	\$141.26	9/26/95	10/5/95
X	McMaster Carr / Air hose parts	\$255.67	9/27/95	10/5/95
X	McMaster Carr / lapping material	\$57.25	9/28/95	10/8/95
X	VWR / Gas regulators	\$535.77	10/3/95	10/12/95
X	McMaster Carr / Fitings Gas Manifold	\$219.16	10/10/95	10/19/95
	McMaster Carr / Tubing Cutter	\$36.28	10/10/95	10/19/95
	McMaster Carr / Garolite Sheet	\$11.80	10/12/95	10/19/95
	C & J Sales / Gas Regulator (Dave Ramsey)	\$40.04	10/13/95	10/19/95
	Allied Seals / O rings	\$152.84	10/13/95	10/19/95
	McMaster Carr / Nylon Tubing	\$27.52	10/16/95	10/19/95
-	Consultant's Fee / John Ball	\$240.00	10/17/95	10/19/95
-	McMaster Carr / Compressor Air Regulator	\$45.51	10/20/95	11/2/95
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-	Teledyne / Vacuum Gauges	\$83.62	10/25/95	11/2/95
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\$486.97	\$486.97	\$182.61	\$42.78	\$243.48	\$1,897.00	\$1,585.00	\$486.97	\$486.97	\$2,252.00	\$486.97	\$225.22	\$198.00	\$14.40	\$220.00	\$298.95	\$173.10	\$363.20	\$118.80	\$55.30	\$57.40	\$226.54	\$205.00	\$525.00	\$791.00	\$25.00	\$58.90	\$740.00	\$50.00	\$25.90	\$1,587.00	\$1,925.00	\$11,000.00
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### Investigation of Electrical and Optical Properties of Sputter Deposited Ni-Si and Ni Silicide-Si contacts

Independent Study Report
Presented by

Layi Fapohunda

December 16, 1997.

## PROJECT OBJECTIVES

- Commissioning of Sputtering System
- Development of Operating techniques
- Sputter/Etch Optimization
- ➤ Deposition Rate and Properties as a Function of DC/RF Power and Gas Pressure
- > Etch Rate as a Function of Power and Gas Pressure
- Thickness Monitor Calibration
- Deposition and Formation of Ohmic Contacts
- Deposition and Formation of Ni-Si and Ni Silicide-Silicon Contacts
- Silicide Formation (200, 400, 750°C)
- Electrical Characterization (I-V)
- Optical Characterization

### MOTIVATION

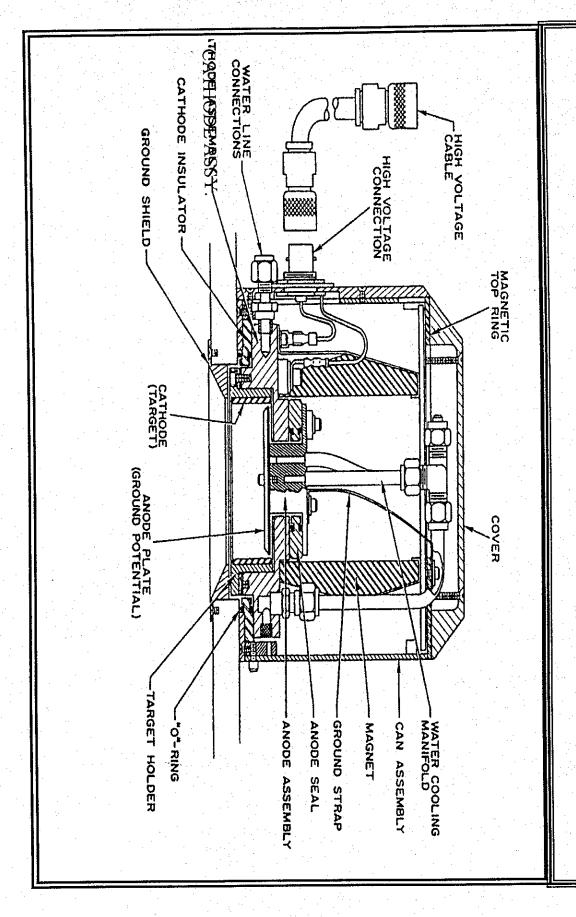
- Possible achievement of cut-off wavelengths corresponding to nearand mid-infrared region of the EM spectrum
- 1.3-1.55 µm range needed for optic fiber transmission
- Pt-Si, Pt-Au Schottky diodes have been demonstrated as PDs for this range[M.
- Cut-off wavelength is ~0.8µm for GaAs, ~1.1µm for Silicon
- constituents in human tissue. Use of an array of different silicides Potential high-speed optical detection of concentration of biological will give the sensitivity over the entire wavelength range of interest 1.1-2.5 µm)
- Biocontrol Technology, Inc. is developing non-invasive blood glucose was initiated by a former Biocontrol Technology, Inc. Engineer monitors using sensors operating in the same wavelength range. This project
- Capability for large scale integration and application
- Military (Laser range finding, near infrared image processing)
- > near-infrared spectroscopy (water vapor, CO<sub>2</sub>, Hydrocarbon gas detection)
- read-out electronics on same chip on same chip Use of highly advanced S1 technology will allow integration of

### MECHANICAL PARTS

- The Roughing Pump: Rotary type
- Cryogenic type The High Vacuum Pump and Compressor Unit:
- The Cooling Water unit: water chiller and pump
- The Chamber Top Works: electrical and mechanical control links
- The Vacuum Valves: Roughing, Hi-Vac, Foreline, Orifice plate
- Other Valves: Gas control, Gas isolation, Chamber cover
- operation The Sputter Gun: 3 Magnetron type for RF or DC power

### Chamber Gas Valve Argon Tank Matching Network Chamber Cover/Topwork Vent **T** Planetary System HiVac Valve Orifice Plate System Schematic **¹**Valve Roughing Roughing Pump Regen. Valve Cryopump Compressor Cryopump Helium Supply/ Return lines

## SPUTTERGUN DETAILS



### Distribution of Sputtered Material ANODE -

## INSTRUMENTATION

- Vacuum Control System
- Ionization and TC gauges
- Manual Controller
- Flow switch
- DC Power Supply
- RF Controller And Impedance Matching Network
- Gas Flow And Pressure Controller
   Capacitive pressure gauge
- Thickness Monitor
- Cover Hoist Control
- Temperature Controller

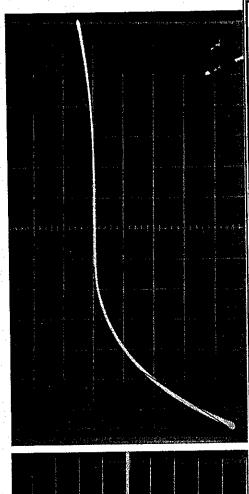
## CONTACT FORMATION

- Substrate cleaning (etch) Substrate doping level: 10<sup>17</sup>
- Ohmic Contact Deposition and Annealing
- Masking
- Sputtering A1 at  $\sim 75^{\circ}C$  chamber temperature (final thickness between 500 and 1000Å)
- Annealing at 450°C for 30mins
- Ni Schottky diode contact Deposition
- Masking
- Sputtering Ni at ~75°C chamber temperature (final thickness between 300 and 1000Å)
- As-deposited IV Characterization
- Silicide formation (Annealing)
- Annealing at 200 or 400°C to form Ni<sub>2</sub>Si and NiSi respectively
- For NiSi<sub>2</sub> formed at 750°C, the Silicide is formed before the Ohmic contact is deposited and annealed

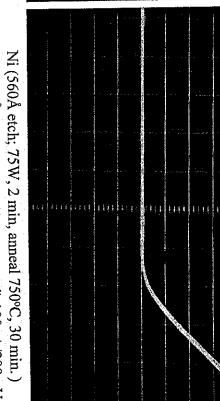
### I-V Characterization

- Curve Tracer
- Gives approximate I-V curve for Schottky diode
- The supply voltage can be varied for desired range
- · Current and Voltage per division adjustments
- Polaroid picture of screen taken for record
- Variable Voltage Source/MPLI DAQ System
- Voltage Source: Keithley 236 with range up to -10 to 10VDC Resolution of 0.01VDC used
- DAQ Unit: data plotted directly on computer screen and data stored in table
- Raw data are supply voltage and voltage drop across 10kΩ resistor
- Data Analysis
- MS Easyplot used for arithmetic operation and data plot
- Determination of Leakage Current and Dark Current
- Determination of Turn-on and Break-down Voltages

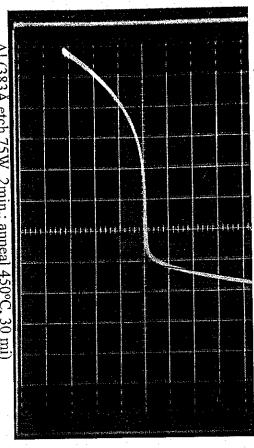
### Curve Tracer Images



Al (485Å, etch 75W, 2min.; anneal 450°C, 30 mi) Ni (610Å, etch; 75W, 2min; no anneal (200µA/200mV)

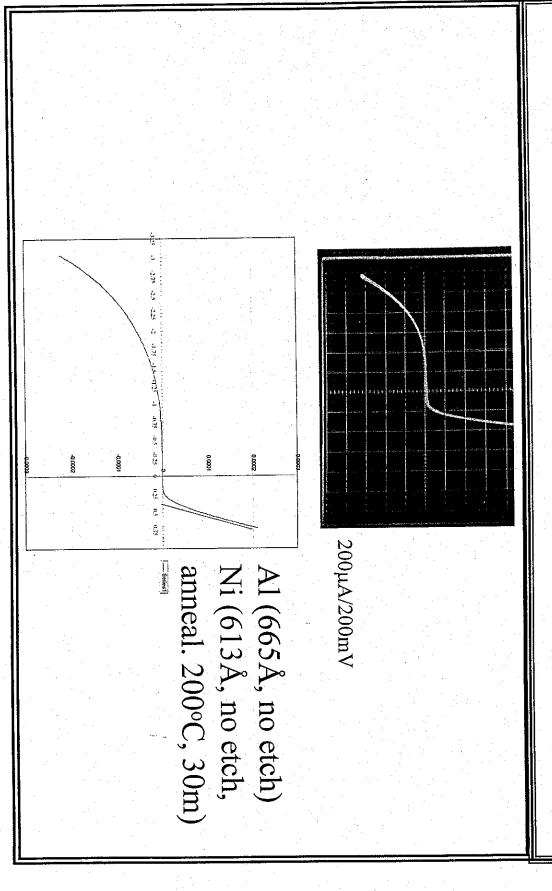


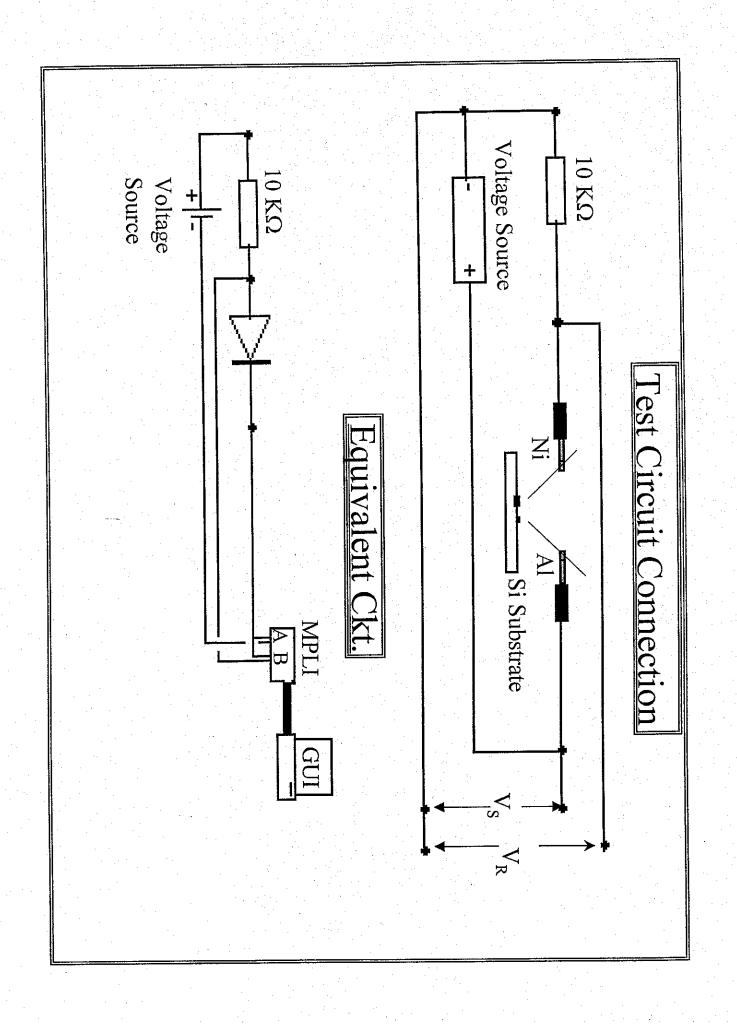
Al (450Å, etch 75W, 2 min, no anneal) 100μA/200mV



Ni (318Å, etch; 75W, 2min; no anneal (200µA/200mV) Al (383Å,etch 75W, 2min.; anneal 450°C, 30 mi)

# OBSERVATION AND RESULTS





## Optical Characterization

- Construction and testing of optical testbed using Ge PL
- Alignment of light source for optimum intensity
- Measurement of light Intensity wrt wavelength
- Measurement of response in "filtered white light" at various bias voltages (BV's)
- Measurement of response as a function of wavelength at various BV's
- Measurement of the "Dark Current" wrt BV's
- (response Dark current) Determination of Signal-to-noise ratio

### Voltage Source 10 KΩ OPTICAL TEST CCIRCUIT CONNECTION Light Source Optic Fiber Bunch Filter Monochrometer Opaque Box Si Substrate \_Test Platform

# **OBSERVATION AND RESULTS**

- MSM were fabricated on n-type Si(111) carrier concentration  $\sim 10^{17}$
- No specific attention was paid to the finger spacing
- The area of contact deposited were 7.85x10<sup>5</sup>µm<sup>2</sup>
- Variation in the dark-current densities from as-deposited Ni/Si to Ni<sub>2</sub>Si/Si Schottky barriers observed from  $\sim 6 \times 10^{-3}$  to  $\sim 10^{-3}$
- All materials with high etch power showed poor contact
- Optical characterization of diodes was not successful due to lack of high intensity light source (already ordered)
- Use of chopped light and lock-in amplifier did not help