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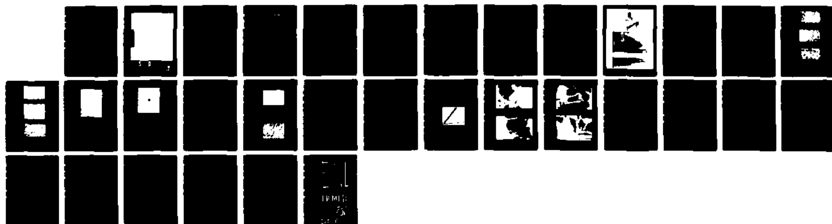

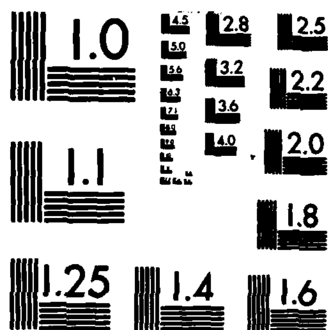

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EPITAXIAL (100) GaAs THIN FILMS ON SAPPHIRE FOR SURFACE ACOUSTIC WAVE/ELECTRONIC DEVICES

Submitted to:
AFOSR
Directorate of Electronic and
Material Sciences
Bolling Air Force Base, WA 20332

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20. ABSTRACT *(Continue on reverse side if necessary and identify by block number)*

In the past year it has been demonstrated that undoped <111> single crystal gallium arsenide could be grown on <01$\bar{1}$2> sapphire using the metalorganic chemical vapor deposition (MO-CVD) growth technique.

An interesting and unexpected result from this work was that the GaAs films grown had a <111> orientation instead of theproposed <100> orientation.

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TABLE OF CONTENTS

Section		Page
1	INTRODUCTION	1-1
2	MO-CVD GROWTH SYSTEMS	2-1
3	EXPERIMENTAL	3-1
	3.1 Substrates	3-1
	3.2 Deposition Studies	3-1
	3.2.1 Deposition Temperature	3-1
	3.2.2 Deposition Rate	3-7
	3.2.3 As/Ga Ratio	3-9
	3.2.4 Effects of Deposition Temperature on Orientation	3-9
4	MEASUREMENTS	4-1
	4.1 X-Ray Diffraction	4-1
	4.2 Mobility	4-1
	4.3 TEM	4-1
	4.4 SIMS	4-5
	4.5 Polaron C-V	4-5
5	SUMMARY	5-1
6	REFERENCES	6-1
7	ASSOCIATED PERSONNEL	7-1

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LIST OF ILLUSTRATIONS

Figure		Page
1	MO-CVD Reactor With IBM-PC Control System	2-2
2	Schematic of MO-CVD Growth Chamber	2-3
3	Nomarski Micrographs of 6 Micron Polycrystalline GaAs Films Grown on $\langle 01\bar{1}2 \rangle$ Al_2O_3	3-3
4	Nomarski Micrographs of 6 Micron Highly Oriented (111) GaAs Films Grown on $\langle 01\bar{1}2 \rangle$ Al_2O_3	3-4
5	Nomarski Micrograph of 6 Micron (111) Single Crystal GaAs Film on $\langle 01\bar{1}2 \rangle$ Al_2O_3 Grown at 725°C	3-5
6	Laue Photograph of $\langle 01\bar{1}2 \rangle$ Al_2O_3	3-6
7	Nomarski Micrograph of 6-Micron Polycrystalline GaAs Film on Al_2O_3 Grown with Non-Optimum Conditions.	3-8
8	Nomarski Micrograph of 6-Micron Single Crystal GaAs Film Grown on Al_2O_3 With Optimum Conditions.	3-8
9	GaAs on Sapphire - Resistivity Measurement	4-2
10	TEM Micrographs of $\langle 111 \rangle$ GaAs Films on $\langle 0112 \rangle$ Al_2O_3	4-3
11	TEM Micrographs of Stacking Faults in 6-Micron GaAs Film Grown on $\langle 01\bar{1}2 \rangle$ Al_2O_3	4-4
12	SIMS Profile of a 6-Micron GaAs Film Grown on $\langle 01\bar{1}2 \rangle$ Al_2O_3. .	4-6

LIST OF TABLES

Table		Page
1	Growth Conditions to Deterine Optimum Deposition Temperature . . .	3-2
2	Two Temperature Deposition Process	3-2
3	Growth Conditions to Determine Optimum Deposition Rate.	3-7
4	Growth Conditions to Determine Effect of As/Ga Ratio on Film Quality	3-9

SECTION 1
INTRODUCTION

This report covers the past years work on a program sponsored by U.S. Department of Defense to demonstrate that high-quality $\langle 100 \rangle$ GaAs/$(01\bar{1}2)$] sapphire (Al_2O_3) epitaxy could be achieved.

GaAs, with the $\langle 100 \rangle$ surface orientation, is preferred both for monolithic surface acoustic wave (SAW) and electronic device fabrication. The successful deposition of $\langle 100 \rangle$ GaAs/$\langle 01\bar{1}2 \rangle$ Al_2O_3 is of great value because it would allow for the integration of SAW and electronic devices on a common chip.

Metalorganic chemical vapor deposition (MO-CVD) was used for all depositions during this work. Sapphire with an orientation of $\langle 01\bar{1}2 \rangle$ was chosen for the substrate, since its four-fold symmetry was expected to initiate $\langle 100 \rangle$ epitaxial growth. The quality of the GaAs films were characterized as a function of deposition temperature, As/Ga ratio and growth rate, in an attempt to optimize the deposition process.

The result that the films grown are of a $\langle 111 \rangle$ orientation, leads to the conclusion that the $\langle 01\bar{1}2 \rangle$ orientation of the Al_2O_3 does not appear to have a significant effect on determining the orientation of the films. This in turn leads to the belief that epitaxial GaAs films could be deposited on other types of substrates, such as quartz.

SECTION 2
MO-CVD GROWTH SYSTEMS

All GaAs depositions were performed in our second MO-CVD system which is also being used in a continuing program, funded by the Solar Energy Research Institute to fabricate GaAs/GaAlAs heteroepitaxial structures.

The MO-CVD reactor, shown in Figure 1, features a microprocessor control system and a rotating, vertical-geometry reaction chamber with a large growth area.

The MO-CVD growth chamber is shown schematically in Figure 2. The pyramid-shaped susceptor has a growth area of 100 cm^2 and can hold five 2" wafers per batch. A rotating thermocouple inside the RF-heated susceptor provides for feedback temperature control. The water-cooled fused-quartz vessel is sealed to the water-cooled stainless steel baseplate by means of a double O-ring arrangement. The rotating fused-quartz shaft is sealed to the baseplate by means of a custom-designed ferrofluid feedthrough.

The entire MO-CVD process sequence is controlled by a Westinghouse PC-900 programmable controller in conjunction with an IBM-PC resulting in a sophisticated computer control system. This system allows for maximum ease of operation, reproducibility, documentation capabilities, and continual monitoring of all safety conditions for operator safety.

The gas-delivery system consists of face-seal fittings and bellows valves butt-welded to stainless steel tubing. A palladium-alloy purifier is used in the hydrogen line and all flows are controlled by electronic mass flow controllers. A filtered-air cabinet is provided in the wafer-loading area and organometallic sources are maintained in temperature-stabilized cooling baths.

This MO-CVD reactor is designed to grow GaAs and GaAlAs over the entire alloy range. Maximum doping levels are mid-10^{18} range for N-type and about 10^{19} for p-type GaAs. Growth rates can be varied from 0.01 to 2 μm/min and the growth temperature is typically between 650°C and 800°C.

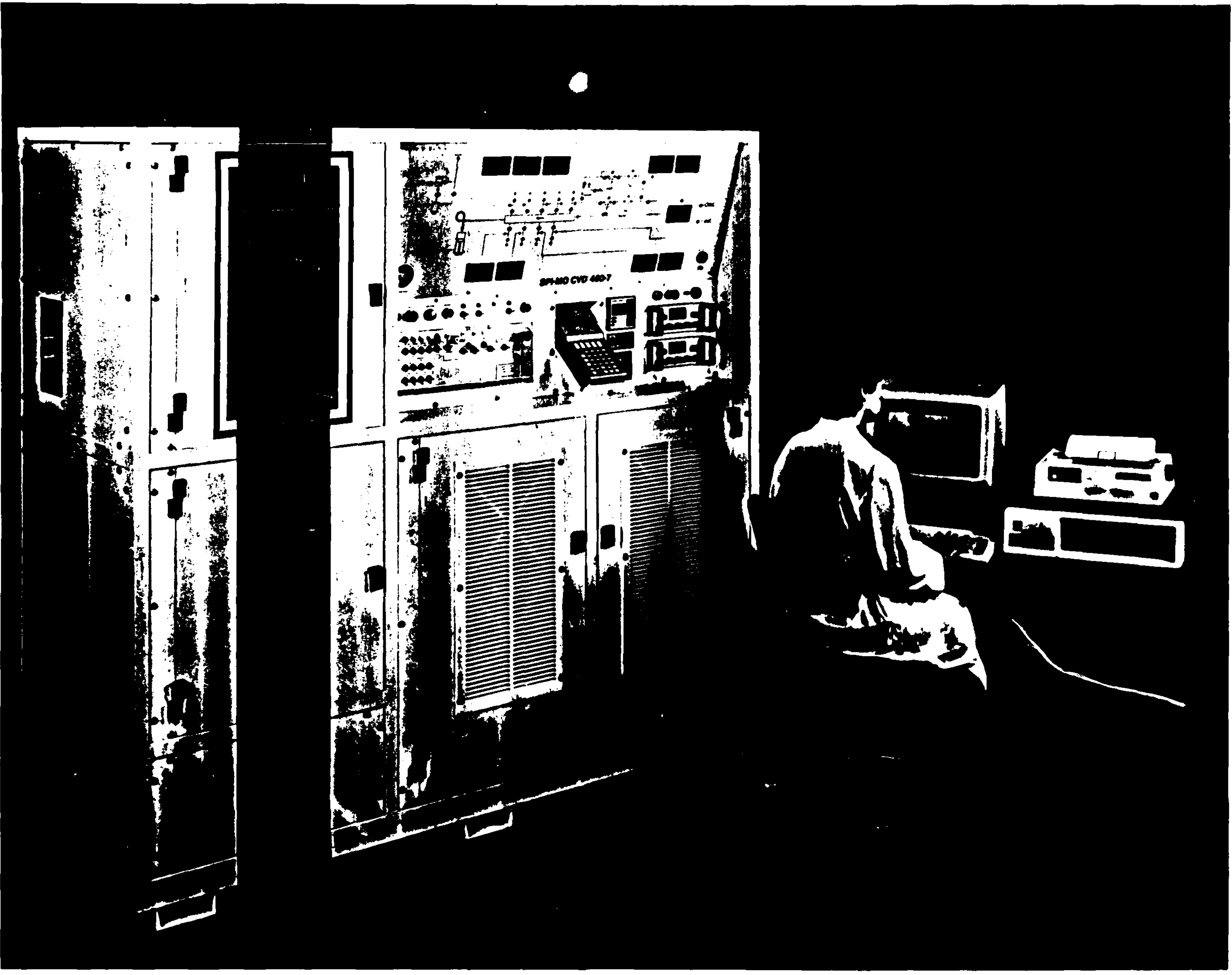


FIGURE 1. MO-CVD REACTOR WITH IBM-PC CONTROL SYSTEM.

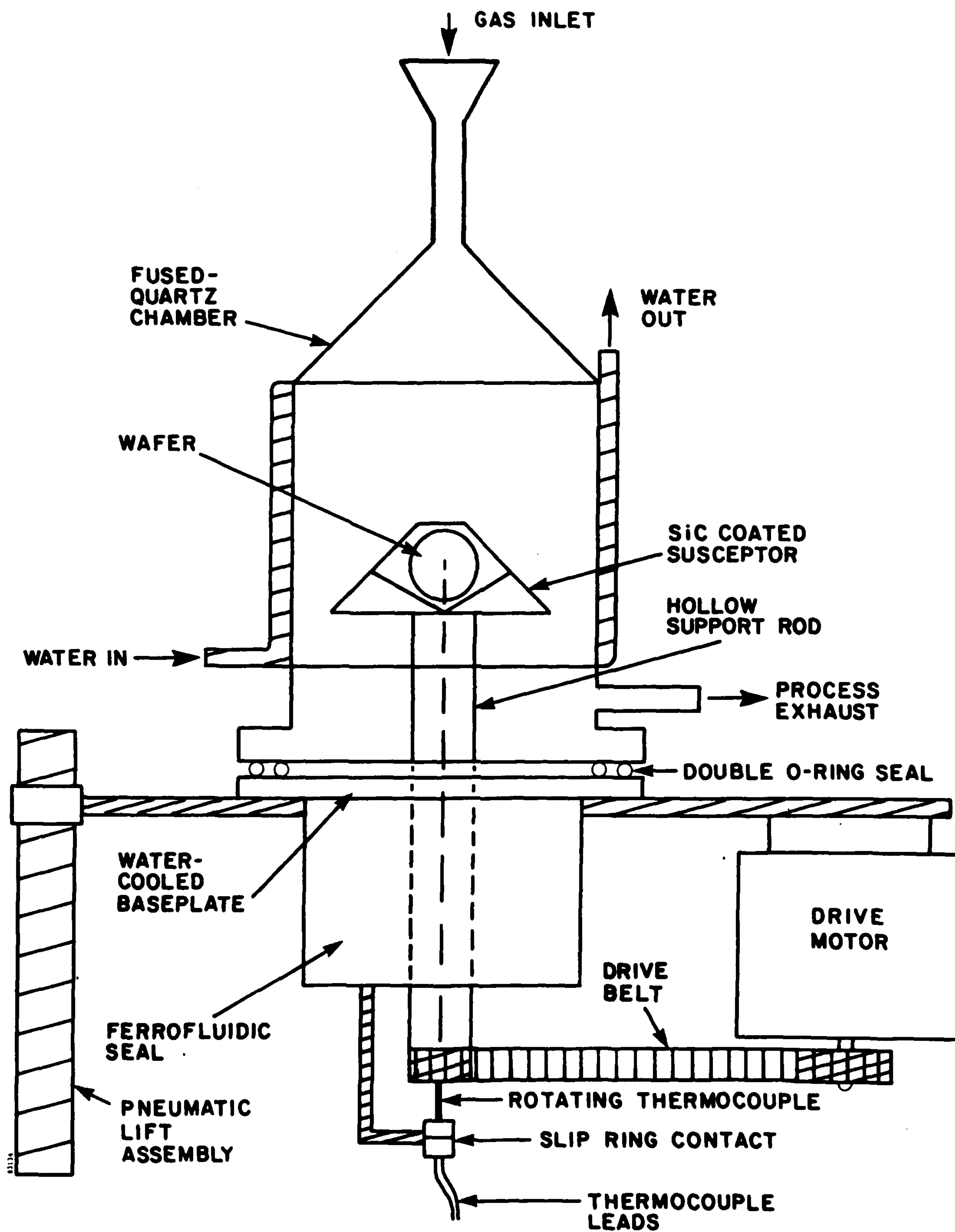


FIGURE 2. SCHEMATIC OF MO-CVD GROWTH CHAMBER.

SECTION 3
EXPERIMENTAL

3.1 SUBSTRATES

The sapphire (Al_2O_3) substrates used for this work were supplied by Saphikon.[2] They were all <01$\bar{1}$2> Al_2O_3 cut in 1" x 1" squares with the front side polished to EPI quality and the back side polished to an optical finish. Before loading, the substrates were cleaned in acetone and methanol followed by a DI rinse. The substrates were then cleaned in $NH_4OH:H_2O_2:H_2O$,2:1:10 for 30 seconds. Each sample was dipped in HF and rinsed just prior to loading. A small piece of N^+GaAs was also loaded each run for evaluation.

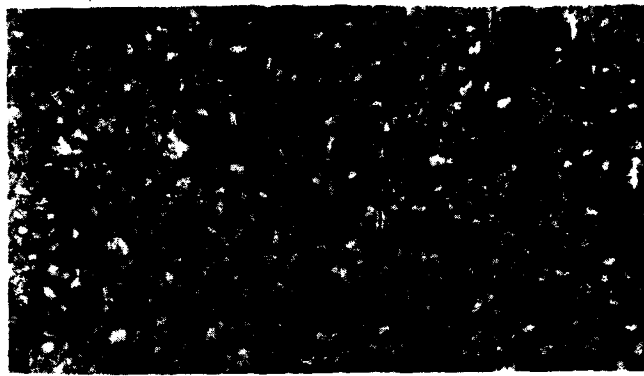
3.2 DEPOSITION STUDIES

Experiments were designed to determine the best conditions for the deposition of GaAs on <01$\bar{1}$2> Al_2O_3. The growth parameters studied were deposition temperature, As/Ga ratio and deposition rate. The deposited layers were evaluated and characterized in terms of surface morphology, electronic quality and structural perfection.

3.2.1 Deposition Temperature

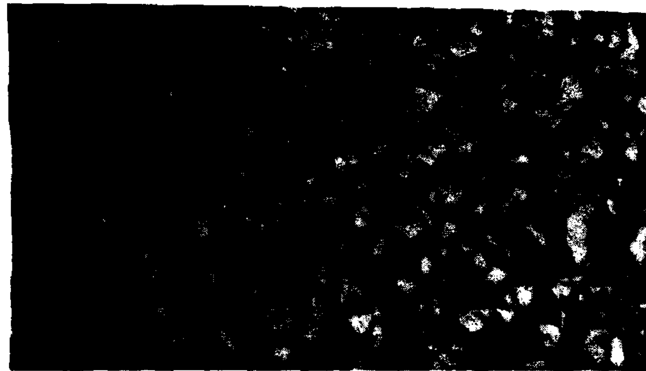
The first growth experiments were done to determine the optimum deposition temperature. A series of runs were made at a fixed growth rate and As/Ga ratio while the temperature was varied for each run. A 6-micron thick undoped layer was deposited at each temperature. The growth conditions are shown in Table 1.

Each sample was evaluated by eye, Nomarski phase-contrast microscopy and by a Sloan Dektak surface profiler. The samples grown at 600°C, 620°C, 650°C all have a dull mat-gray appearance to the eye and look polycrystalline when viewed by the Nomarski microscope (Figure 3). The samples grown at 675°C, 700°C, 725°C, and 750°C all have a smooth, reflective surface to the eye. The samples grown at 675°C, 700°C, and 750°C when viewed by microscopy, reveal a large grain texture (Figure 4). The surface of the sample grown at 725°C has a very definite crystalline appearance as can be seen by the triangular morphology in Figure 5.

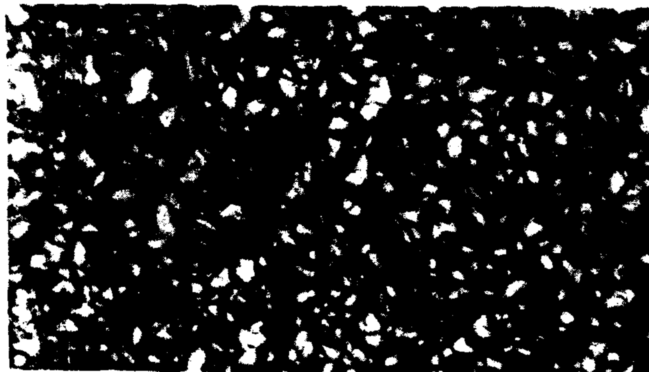


10μm

(a) 600°C

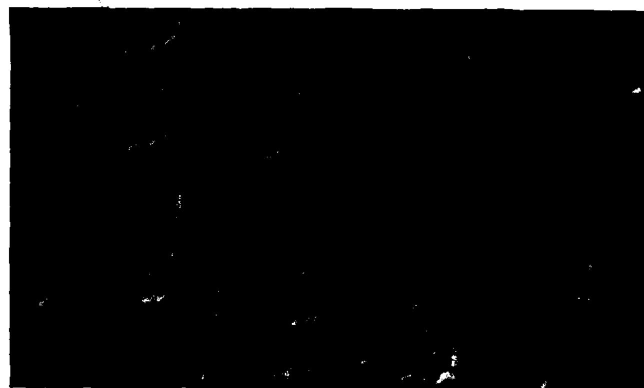


(b) 620°C



(c) 650°C

FIGURE 3. NOMARSKI MICROGRAPHS OF 6 MICRON POLYCRYSTALLINE GaAs FILMS GROWN ON $\langle 01\bar{1}2 \rangle$ Al_2O_3.



(a) 675°C

(b) 700°C

(c) 750°C

FIGURE 4. NOMARSKI MICROGRAPHS OF 6 MICRON HIGHLY ORIENTED (111) GaAs FILMS GROWN ON $\langle 01\bar{1}2 \rangle$ Al_2O_3.

FIGURE 5. NOMARSKI MICROGRAPH OF 6 MICRON (111) SINGLE CRYSTAL GaAs FILM ON ⟨01$\bar{1}$2⟩ Al_2O_3 GROWN AT 725°C.

The sample grown using the two step process is mat-gray and appeared polycrystalline w

Selected sa nt to Manlabs[3] for x-ray diffraction analy aAs layers are oriented in the (111) directi mples grown at 650°, 675°, 700°, 725° all sh (333) reflection indicating a highly oriented

The sample indicating this sample is somewhat polycr

The sample lanlabs, revealed a slightly sharper (111) pe this probably indicates less strain in the cry

The Al_2O_3 substrate was also examined by x-ray diffraction and a laue photograph (Figure 6) which verified it was ⟨01$\bar{1}$2⟩ Al_2O_3 as expected.

FIGURE 6. LAUE PHOTOGRAPH OF ⟨01$\bar{1}$2⟩ Al_2O_3.

After examining the data from Manlabs, comparing photographs from the microscope, examining the samples by eye and comparing Dektak measurements, it was concluded that the sample grown at 725° gave the best crystal. A highly oriented <111> GaAs layer with an average surface roughness of .5 microns.

3.2.2 Deposition Rate

A second series of growth experiments were done to determine how the deposition rate effected the films. 725° was the temperature chosen for this series of experiments because it yielded the best crystal in the first series. The As/Ga ratio was kept constant. Table 3 shows the growth conditions.

TABLE 3. GROWTH CONDITIONS TO DETERMINE OPTIMUM DEPOSITION RATE

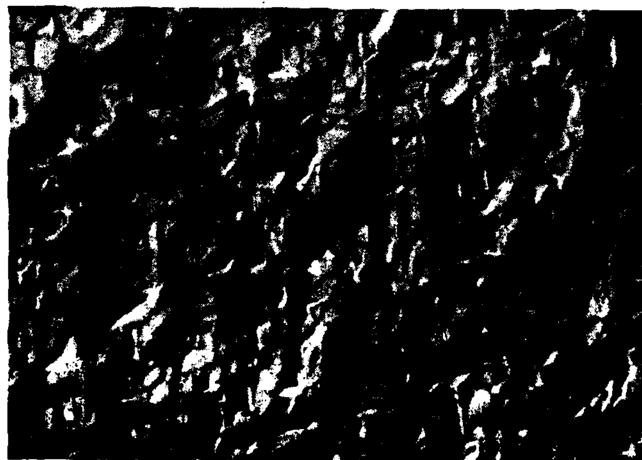
Run #	Deposition Rate	As/Ga Ratio	Deposition Temperature	Layer Thickness
1	5.5Å/sec	10:1	725°C	6 µm
2	22Å/sec	10:1	725°C	6 µm
3	44Å/sec	10:1	725°C	6 µm

Samples were again examined and evaluated by eye and by microscopy. All samples other than the one grown at 22Å/sec appeared polycrystalline when viewed by microscopy (Figure 7) and had the same mat-gray appearance by eye. The sample grown at 22Å/sec again appeared to be cyrstalline (Figure 8) and have a bright reflective surface as before.

The above experiments led to the conclusion that the GaAs film quality is very depended on the deposition rate. The optimum growth conditions after completing the first two series of runs was deposition temperature 725°C, AsGa ratio 10:1 and deposition rate of 22Å/sec.

10 μm

FIGURE 7. NOMARSKI MICROGRAPH OF 6-MICRON POLYCRYSTALLINE GaAs FILM ON Al_2O_3 GROWN WITH NON-OPTIMUM CONDITIONS.



10 μm

FIGURE 8. NOMARSKI MICROGRAPH OF 6-MICRON SINGLE CRYSTAL GaAs FILM GROWN ON Al_2O_3 WITH OPTIMUM CONDITIONS.

3.2.3 As/Ga Ratio

A third series of runs were done to determine how the As/Ga ratio would effect the film quality.

Two runs were made at the optimum temperature of 725° and growth rate of 22Å/sec. The growth conditions are shown in Table 4.

TABLE 4. GROWTH CONDITIONS TO DETERMINE EFFECT OF As/Ga RATIO ON FILM QUALITY

Run #	Deposition Rate	As/Ga Ratio	Deposition Temperature	Layer Thickness
1	22 Å/sec.	20:1	725°C	6 μm
2	22 Å/sec.	5:1	725°C	6 μm

Samples grown at both the above As/Ga ratios are mat-gray and looked polycrystalline under the microscope.

3.2.4 Effects of Deposition Temperature on Orientation

One run was made to determine if a high deposition temperature would possibly enhance <100> growth due to the high energy level of the GaAs atoms. The run was made at 825°C with optimum growth conditions (deposition rate=22Å/sec, As/Ga ratio=10:1). This sample also looks mat-gray and polycrystalline by microscopy.

SECTION 4
MEASUREMENTS

4.1 X-RAY DIFFRACTION

Selected samples were again sent to Manlabs for x-ray diffraction. Along with the GaAs/(01T2) samples, one GaAs/0001 Al_2O_3 sample was also sent. This sample was grown during one of the runs with optimum growth conditions and was used as a comparison. $\langle 0001 \rangle$ Al_2O_3 is known to initiate $\langle 111 \rangle$ GaAs growth from previous work done at Spire which was funded by industry and has been reported elsewhere.[1] Data from Manlabs reinforced the first findings that the sample grown at the optimum settings of 725°, AsGa ratio 10:1 and deposition rate of 22 Å second is the best crystal. They concluded that this sample is a single crystal layer with a $\langle 111 \rangle$ orientation. It was also found that there is very little difference if any between the layer grown on $\langle 0001 \rangle$ Al_2O_3 and $\langle 01\bar{1}2 \rangle$ Al_2O_3 both layers are $\langle 111 \rangle$ single crystal GaAs.

The best samples were then chosen for closer examination. Electronic qualities were attempted to be measured by Hall effect measurement. Defect populations were examined by transmission electron microscopy (TEM). The purity and autodoping were investigated by secondary ion mass spectroscopy (SIMS). A measurement of the doping level was attempted by Polaron C-V profilometry.

One sample was used for all the above measurements. The sample used was grown at the optimum conditions, 725°C, As/Ga ratio=10:1 and growth rate of 22A/sec. It was sectioned as needed for each measurement.

4.2 MOBILITY

A mobility measurement was attempted by Hall effect. This proved to be unsuccessful due to the fact that the resistivity (ρ) of the layer is too high (Figure 9) for our Hall setup, which is sensitivity limited by its small magnet.

4.3 TEM

A section was sent to ARACOR[4] for examination by TEM. ARACOR verified that the crystal is a (111) crystal, however they did detect some evidence of polycrystallinity.

Sample: Van der Pauw Hall configuration ICM X ICM

Room light only

$$\frac{V}{I} = \frac{10V}{10nA} = 10^9$$

$$RSH = 4.53\frac{V}{I} = 4.53 \times 10^9$$

$$= R_{sh} \cdot t = 2.27 \times 10^6$$

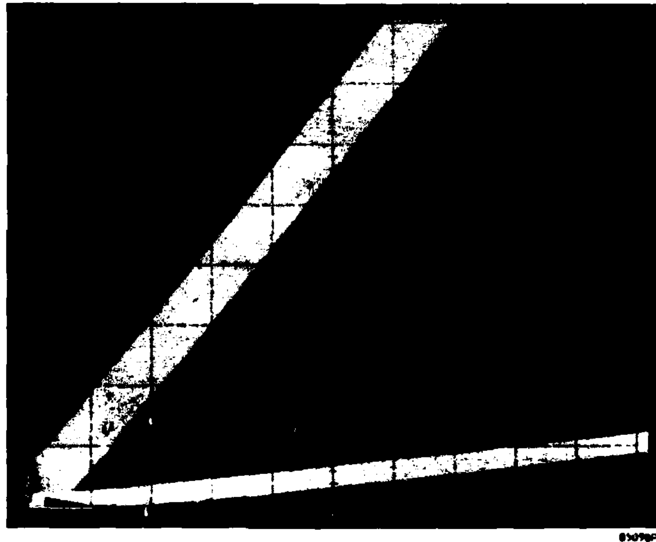


FIGURE 9. GaAs ON SAPPHIRE - RESISTIVITY MEASUREMENT. Top Illuminated, Bottom Dark.

Figure 10 A and B shows a grain which is of a different orientation then the surrounding <111> region. As can be seen, the grain is rotated with respect to the matrix. This is also confirmed by the extra non-symetric spot in the laue photograph (Figure 10A). Aracor was not able to determine the orientation of this grain. The sample also has a high density of stacking faults around $10^7 cm^{-2}$ calculated from the micrographs (Figure 11 A and B).

FIGURE 10-A.

FIGURE 10-B. TEM MICROGRAPHS OF <111> GaAs FILM ON <01$\bar{1}$2> Al_2O_3.

FIGURE 11-A.

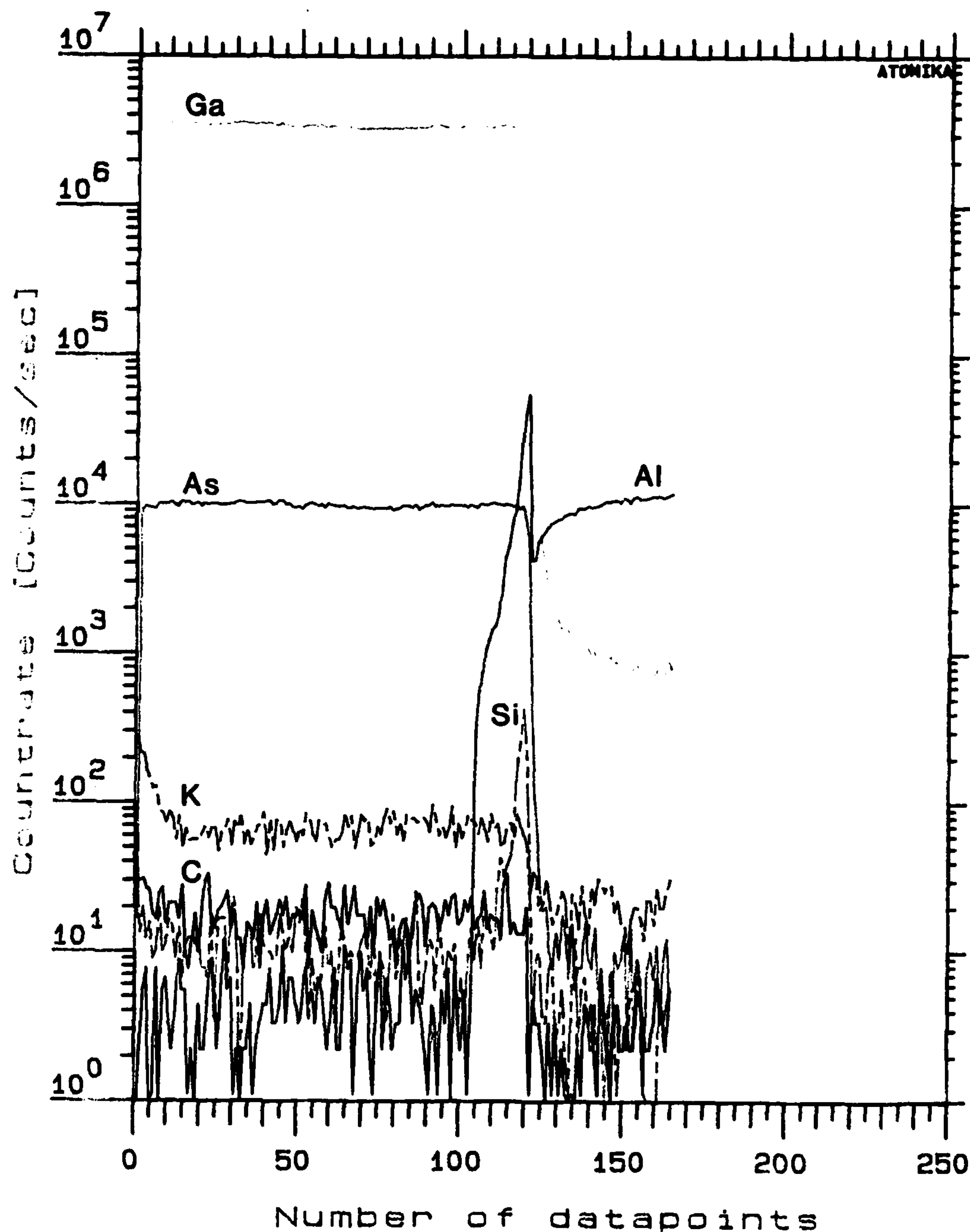
FIGURE 11-B. TEM MICROGRAPHS OF STACKING FAULTS IN 6-MICRON GaAs FILM GROWN ON <01$\bar{1}$2> Al_2O_3.

4.4 SIMS

Another section was sent to Northern Analytical Lab[5] for examination by SIMS. A depth-profile was taken through the GaAs layer into the Al_2O_3 substrate. The profile (Figure 12) shows the plots for As, Ga, Al, C, SI and K. As can be seen there is an Al spike at the GaAs/Al_2O_3 interface which quickly disappears early into the GaAs layer. Northern Analytical believes that the Al spike in the GaAs layer is not actually there, but was just an artifact of the measurement due to a charge build up at the surface of the Al_2O_3. The Al in the first portion of the GaAs film is believed to be real and quickly dissipates as mentioned above.

4.5 POLARON C-V

An attempt was made to measure the carrier concentration of the best film using a Polaron profile plotter. The carrier concentration is calculated from the capacitance-voltage data taken from a Schottky barrier formed between the GaAs film and an electrolyte solution. Omic contact must be made either to the back of the substrate or to the deposited film in order for the Polaron to operate. Due to the insulating nature of the Al_20_3 substrate and the highly resistive grown layer, omic contact could not be achieved so the carrier concentration could not be measured.



Primary Ions	:	Oxygen
Ion Energy	:	12 [kev]
Beam Current	:	500 W/CC [nA]
Scan Width	:	0.400 [mm]
Scan Speed	:	2 [s/Frame]
Scan Gate	:	30 [%]

Label	Mass	Energy	Cycles
AS	75.0	-5.0	5
AL	43.0	-5.0	5
GA	71.0	-5.0	2
C	12.0	-5.0	5
SI	28.0	-5.0	5
K	39.0	-5.0	5

FIGURE 12. SIMS PROFILE OF A 6-MICRON GaAs FILM GROWN ON <01$\bar{1}$2> Al_2O_3.

SECTION 5
SUMMARY

The conclusions, observations and results of the past year are as follows:

1. A reproducible process for depositing $\langle 111 \rangle$ single crystal GaAs on $\langle 01\bar{1}2 \rangle$ Al_2O_3 has been developed using the MO-CVD technique. The reason for the films being of $\langle 111 \rangle$ orientation instead of the proposed $\langle 100 \rangle$ orientation must still be investigated.

2. The $\langle 01\bar{1}2 \rangle$ orientation of the Al_2O_3 substrate did not appear to have a significant effect on determining the $\langle 111 \rangle$ orientation of the GaAs films.

3. The best films grown are highly reflective to the eye and have a surface roughness of approximately .4 microns.

4. Deposition temperature, GaAs ratio and deposition rate, all affected the quality of the films.

5. The defect density of the films is approximately $10^7 cm^{-2}$. More work is needed to improve the films.

6. The SIMS profile showed very little autodoping or contamination in the GaAs films attributed to the Al_2O_3 substrate.

SECTION 6

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2) Saphikon, 51 Powers Street, Milford, NH 03055

3) Manlabs, 21 Erie Street, Cambridge, MA

4) ARACOR, 1223 East Arques Avenue, Sunnyvale, CA 94086

5) Northern Analytical Lab, Inc., 3 Northern Blvd., B2, Amherst, NH 03031

SECTION 7

ASSOCIATED PERSONNEL

ROBERT G. WOLFSON

EDUCATION: B.S., Metallurgy, M.I.T.
M.S. and Ph.D., Materials Science, Northwestern University

PROFESSIONAL EXPERIENCE:

Present — Spire Corporation. Vice President/Research. Dr. Wolfson directs R&D on materials and processes for photovoltaics, solid-state micro-electronics, and transducer/detector technology. He is also responsible for managing Spire's research programs from the U.S. Departments of Defense and Energy and from private industry. Current activities center upon the elemental and compound semiconductors: silicon (both crystalline and amorphous), germanium, and the III-V and II-VI compounds; there are, in addition, major efforts on other materials, such as amorphous beryllium and cubic (adamantine) boron nitride. The chief applications for this work are in the areas of thin-film solar cells, discrete devices and integrated circuits, microwave communications, infrared imaging and optics, fiberoptics, and protective coatings for wear and chemical resistance.

Prior to 1979 — Before joining Spire, Dr. Wolfson was Manager, Advanced Materials Equipment for the Varian/Lexington Vacuum Division and earlier was Associate Director, Materials Technology Center, General Instrument Corporation. For six years, he served as Associate Professor of Engineering at Dartmouth College; still earlier, he was Project Leader, Epitaxy Research at P.R. Mallory Company and Manager, Materials R&D at Sylvania Semiconductor Division, where he began his career.

PUBLICATIONS:

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EDUCATION: A.S. and B.S., Electronic Engineering Technology, Wentworth Institute of Technology, 1978

PROFESSIONAL EXPERIENCE:

1978-Present Spire Corporation. Research Scientist. Responsible for: (1) MO-CVD of GaAs/GaAlAs for various research programs, (2) Lab supervision. Previously, as Process Engineer, designed software control for MO-CVD reactor product line. Wired, programmed, operated and maintained first MO-CVD reactor. Other experience with photolithographic processes for solar cells.

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EDUCATION: M.S., Electrical Engineering, Rutgers University, 1976
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PROFESSIONAL EXPERIENCE:

1981-Present Spire Corporation. Manager, Epitaxial R&D. Responsible for: (1) design and development of metalorganic chemical vapor deposition (MO-CVD) systems product line, (2) establishment and operation of MO-CVD GaAs/GaAlAs epitaxial growth service, and (3) GaAs/GaAlAs deposition for various research programs concerned with high-efficiency, thin-film solar cells for flat-plate and concentrator applications.

1976-1981 IBM Thomas J. Watson Research Center. Associate Engineer. Responsible for the chemical-vapor-deposition portion of the GaAs-GaAlAs research effort on solar cells and MESFETS. Designed and built an organometallic-CVD growth system. Successfully grew high-quality GaAs and GaAlAs layers, and produced first vapor-grown GaAs-GaAlAs solar cell and GaAs MESFET at IBM. Fabricated and tested Schottky barrier solar cells on Si and on single- and polycrystalline GaAs films. Developed recrystallization process which increased grain size of poly-GaAs films by three orders of magnitude. Achieved large-grained GaAs films on Si and SiO_2 by graphoepitaxy.

1974-1976 Rutgers University. Research Fellow, Electrical Engineering Department. Designed, fabricated and tested Schottky barrier (MIS) solar cells on Si. Discovered means of achieving reproducibly high open-circuit voltages by control of fabrication parameters.

1972-1974 Rutgers University. Research Assistant, Physics Department. Performed research on homogeneous nucleation in a fluid mixture.

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1970-1971 Boston College. Research Assistant, Physics Department. Studied thermal annealing of radiation-damaged Si solar cells.

PUBLICATIONS:

"Growth of Epitaxial GaAs Films on Silicon Substrates," S. M. Vernon, S. Shanfield, and R. G. Wolfson, (submitted).

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PROFESSIONAL EXPERIENCE

1984-present Spire Corporation. Research Technician. Responsible for: (1) Operation and maintenance of MO-CVD reactor, (2) Design of GaAs/GaAlAs growth conditions for research programs.

1980-1984 MA/COM Corporation. Diffusion Technician. Responsible for Lab equipment, deposition and diffusion of silicon devices.

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