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SCHOTTKY BARRIER SURFACE SCIENCE
FINAL REPORT

Prepared For

Office Of Naval Research
Department Of The Navy
Arlington, VA 22217

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Report 7855

July 1985

AEROJET ELECTROSYSTEMS COMPANY

AZUSA, CALIFORNIA

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A DIVISION OF AEROJET GENERAL ⊖

SCHOTTKY BARRIER SURFACE SCIENCE

Final Report

Report Number 7855

July 1985

Contract Number   N00014-83-C-0680

By

Dr. Patricia M. George

Prepared for

Office of Naval Research
Department of the Navy
Arlington, VA   22217

Prepared by

Aerojet ElectroSystems Company
1100 West Hollyvale Street
Azusa, California 91702

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ABSTRACT

Thermal desorption mass spectrometry (TDMS) and X-ray photoelectron spectroscopy (XPS) are utilized to study GaAs(100) surfaces prepared by MBE and chemical etching. XPS results on the chemically etched GaAs samples revealed As present in GaAs and As^{+3} in the form of As_2O_3. TDMS of both the chemically etched and MBE prepared GaAs showed several impurities such as H_2, H_2O, CO_2 and CO. Also, the surface reactions of water and ammonia with atomically clean GaAs surfaces are studied with XPS and TDMS. These results help identify possible contaminants that may interfere with GaAs device performance.

Key Words: GaAs(100)
                     Thermal Desorption Mass Spectrometry
                     X-ray Photoelectron Spectroscopy
                     Surface Science
                     GaAs Processing
                     Surface Reactions

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1.   INTRODUCTION

The goal of the investigation supported by ONR Contract #N00014-83-C-0680 was to initiate a study of reactions on clean GaAs surfaces and at GaAs metal interfaces. Major objectives of our extended program are to establish the structural features, energetics and fundamental mechanisms associated with adsorbing selected molecules at various stages of Schottky barrier formation. Results of research into the chemical, physical and electrical properties with adsorbing specific molecules are essential for the development of techniques to construct reproducible barrier heights and ohmic contacts.

Initial experiments have been accomplished using a multi-probe, ultra-high vacuum system. The first stage of the research involved studies of small molecules on GaAs. The molecules were chosen with one of two criteria in mind. The first was that the molecules provide experimental information on the relationship between atomic geometry, electronic structure and chemical reactivity of each GaAs surface. It is anticipated that precise chemical probes can be used to detect and identify different electronic sites present at the surface. In this study a well-defined sequence of gaseous Lewis bases, ammonia, methylamine, dimethylamine and trimethylamine, was chosen. The second criteria was that the molecule(s) be important in processing, passivation or as contaminants on GaAs. For this investigation the reaction of water with the different surfaces of GaAs was chosen. During the course of these studies it was necessary to characterize the material obtained from various sources. This has resulted in a third investigation in which thermal desorption methods are developed to compare materials grown and/or processed under different conditions.

This report summarizes the preliminary data obtained from the three investigations mentioned above. Also detailed are problems and approaches which did not prove to be viable and an outline of future experiments which would extend the studies.

2.   EXPERIMENTAL

The experiments were performed in a bakeable mu-metal and stainless steel ultra-high vacuum system specifically designed for the above investigations. Schematic representations of the instrument, built by V. G. Scientific, are given in Figure 1 and Figure 2. It comprises two analytical chambers (I and II) separated by a preparation chamber. Each chamber can be isolated, thereby allowing pressures up to an atmosphere to be maintained in the preparation chamber with no adverse effects on analytic capabilities. All three chambers are fitted with liquid nitrogen-trapped diffusion pumps and each is capable of reaching a base pressure of $<2 \times 10^{-11}$ Torr. In addition, they are all equipped with temperature controlled manipulators that can be cooled to liquid nitrogen temperatures. Samples are introduced via an additional chamber, equipped with a fast-entry lock which opens into a dry box. Thus substrates can be chemically etched and characterized without exposure to air. Once inside the preparation chamber, samples are transported by a specimen transfer system based on a rack and pinion drive. Samples may then be removed to each manipulator as required.

The preparation chamber houses a mass spectrometer (0 - 300amu) for identification of gas phase species and thermal desorption experiments as well as an evaporator and quartz crystal monitor for thin film measurements. In addition, the chamber is equipped with MgF laser ports and a molecular beam gas doser.

Chamber I consists of an X-ray photoelectron spectrometer (XPS) equipped with a 150° spherical sector analyzer and with both an Al monochromated X-ray source and a dual anode (Al/Mg) source, a Secondary Ion Mass Spectrometer (SIMS) with a mass range from 0-800 amu, low energy Ion Scattering Spectrometry (ISS), Scanning Electron and Auger Microscopy (SEM and SAM) with imaging to 2000 Å. Qualitative angle-resolved XPS measurements can be performed by rotating the sample. Thermal desorption mass spectrometry is also performed in this chamber using the MM-1212s quadrupole mass analyzer. Chamber II comprises a rear-view Low Energy Election Diffraction gun and screen, a Low Energy Electron Loss Spectrometer with 10 meV resolution and angle-resolved UPS.
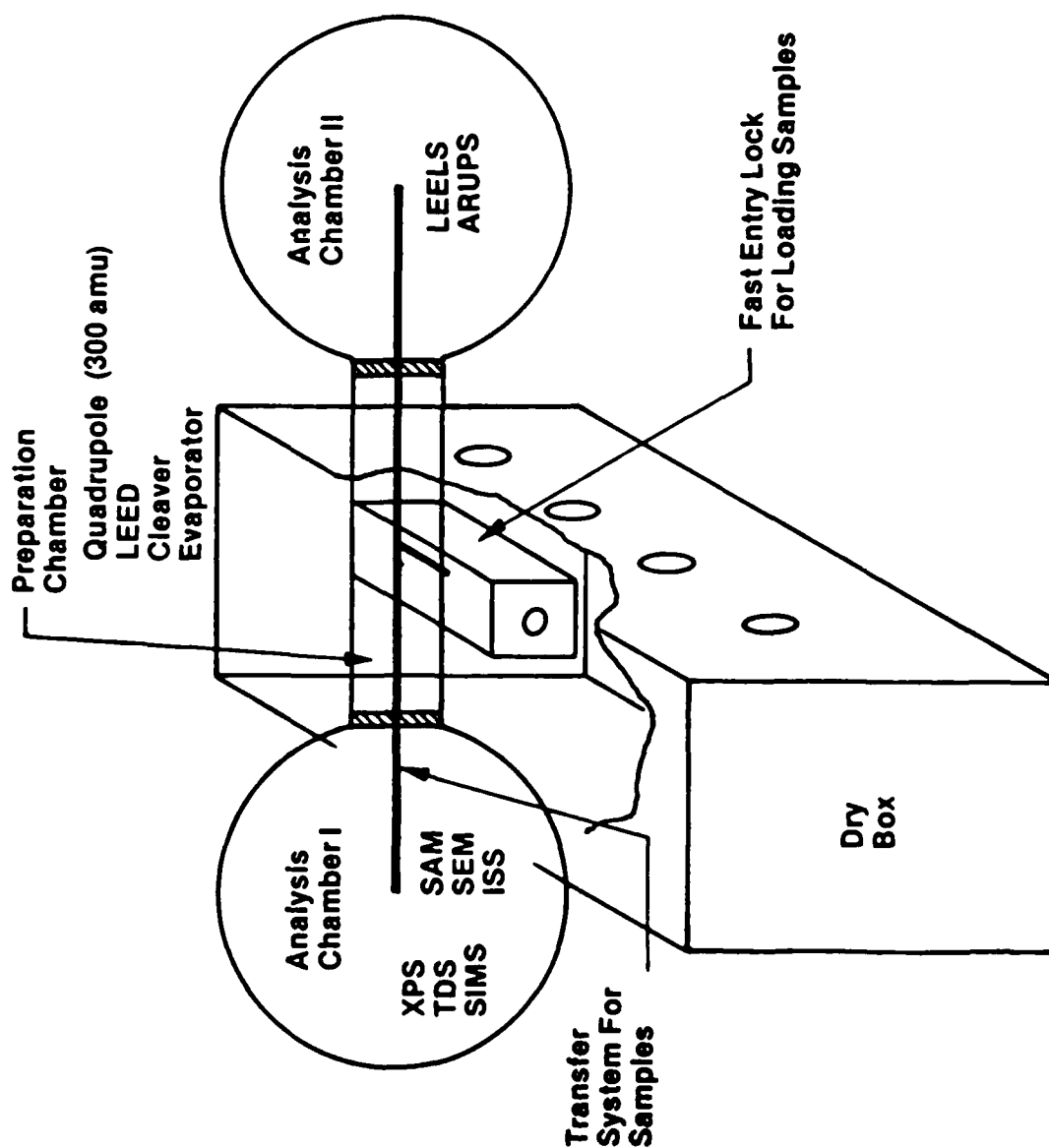
FIGURE 1. Spatial arrangement of surface science instrument

-3-

583-2047

X-Ray Monochromator

150mm Mean Radius Spherical Sector Analyser

2000Å Electron Gun
Scanning Ion Gun For Depth Profiling/SIMS
Specimen Tranfer Rotary Drive

Quadrupole
300 amu RGA

UPS Source

LEELS

Evaporator

Static Spot Ion Gun
Cleaning & Etching

LEED

Quadrupole
800 amu

Twin Anode
X-Ray Source

Fast
Entry Lock

High Precision
Manipulator With
Heating And Cooling
Titanium Sublimation
Pump

High
Precision
Translator
XYZ Shift
& Rotation
Heating
& Cooling

High
Precision
Translator
XYZ Shift
& Rotation
With Heating
And Cooling

Analysis Chamber I

XPS
TDS
SIMS
SAM
SEM
ISS

Preparation

LEED
Evaporator
Cleaver

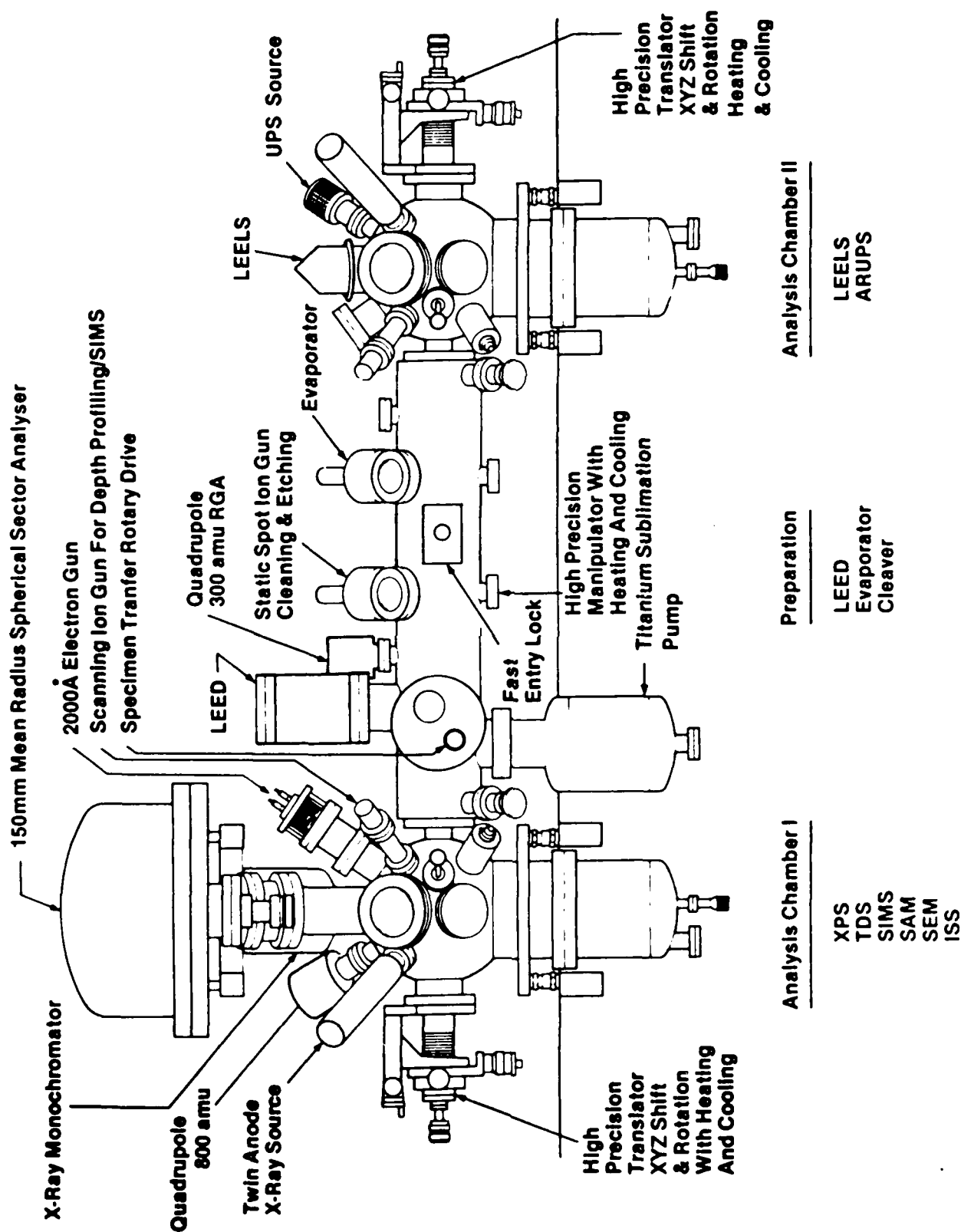Analysis Chamber II

LEELS
ARUPS

FIGURE 2.   Schematic representation of surface science instrument

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The initial GaAs studies were performed on cut ($\pm$ $\frac{1}{2}^{O}$) polished GaAs(100) purchased from Sumitomo Electric Co. This was cleaned and etched using a procedure which has been successfully used to clean substrates for MBE growth. The procedure consists of boiling the GaAs(100) in trichloreothane, rinsing in acetone and isopropyl alcohol, etching with a 1:3:135 hydrogen peroxide, ammonium hydroxide, water solution followed by a deionized water rinse. XPS was used to determine the surface composition. Thermal desorption mass spectrometry was used to monitor the evolution of oxides as we as Ga and As. Initially, these were the only peaks monitored but subsequent mass spectra taken while heating the samples revealed the need to monitor peaks at mass 28, 44, 18 and 2. XPS was again used to determine the surface composition after oxide removal. These experiments were repeated for MBE grown Si-doped GaAs(100) with and without a 1um buffer layer between the substrate and epitaxally grown film. Both MBE grown substrates were arsenic-capped before transfer from thee MBE system to the analysis system[1]. Substrates were mounted with indium on nickel stubs and cooled to 180K. Control experiments were performed to ensure no contribution to the thermal desorption data was coming from the mounting stubs and surrounding manipulator. LEED patterns were recorded for each sample. The substrates used for experiments were 3000Å n-type (Si-doped) MBE grown GaAs(100) with no buffer layer. These were cleaned by annealing cycles to 820K to remove oxides, as evidenced by XPS spectra. $\sim 10^3$L of H_2O was admitted via a molecular beam doser while the substrate was held at 180K. Substrates used for the ammonia/amine experiments were polished, chemically-etched GaAs(100) with the oxide layer removed thermally.

3.   METHODS OF ANALYSIS: THERMAL DESORPTION MASS SPECTROMETRY AS A
        PROBE OF IMPURITIES IN GaAs

Thermal Desorption Mass Spectrometry (TDMS) has been used previously to characterize GaAs[2] as well as the reaction of oxygen on GaAs[3]. In addition, mass spectra have been taken of both gallium[4] and arsenic oxides[5]. No mention has been made, however, of impurities observed in the mass spectra. Ranke et al[3] monitored AsO^+, As^+, Ga^+, O^+, Ga^+ and O_2^+ when desorbing 0.1 Torr-s oxygen from ion bombarded, annealed GaAs(100) and observed loss of AsO^+

and O_2^+ at 340K, Ga_2O^+ and Ga^+ at ~780K before As^+ and Ga^+ started evaporating from the crystal at ~820K. It is not clear whether they monitored other masses.

Our specific study was instigated as a result of several attempts to understand Thermal Desorption reactions on polished, chemically etched GaAs(100) surfaces, which had been annealed to remove the passivating oxide. Reports in the literature[6] indicate that heating chemically-etched GaAs, to ~530°C removes the passivating oxide layer formed during the final water rinse of the chemical etch process. LEED, Auger and XPS data have shown that surfaces prepared in this way yielded gallium rich surfaces. In an effort to both reproducibly remove oxides and monitor the surface for arsenic loss thermal desorption spectra were taken during oxide removal. This led to the observation that additional species were being desorbed and preliminary investigations have concentrated on characterizing the species which evolved from GaAs grown under varying conditions.

A limited number of experiments have examined the reaction of water of GaAs[7, 8]. Büchel and Lüth[7] used UPS to study water on GaAs(110). They observe two molecularly adsorbed phases at 300K while at lower temperatures (180K) only the condensed H_2O is found. UPS, XPS and SIMS were used to examine water on ion bombarded and annealed GaAs by Webb et al[8]. Contrary to the findings on GaAs(110) they inferred a dissociative mechanism preceeded by molecular adsorption of H_2O. The formation of Ga-OH bonds were postulated as a result of these experiments.

4.   RESULTS
4.1   Polished, chemically-etched GaAs(100)

Initial XPS results (Figure 3) show that, after etching, the n-type GaAs surfaces comprises As present in GaAs and As^{+3} in the form of As_2O_3. No evidence was seen for higher arsenic oxides. Broad shoulders on the Ga 3d peak indicates the presence of oxides or hydroxides. These data were obtained under UHV conditions on as-prepared samples, cooled to 180K[9]. Figure 4 shows the same As and Ga 3d core levels after heating the sample in situ to ~670K. Difference spectra indicate that, on heating, loss of As_2O_3 occurs with a concommitant
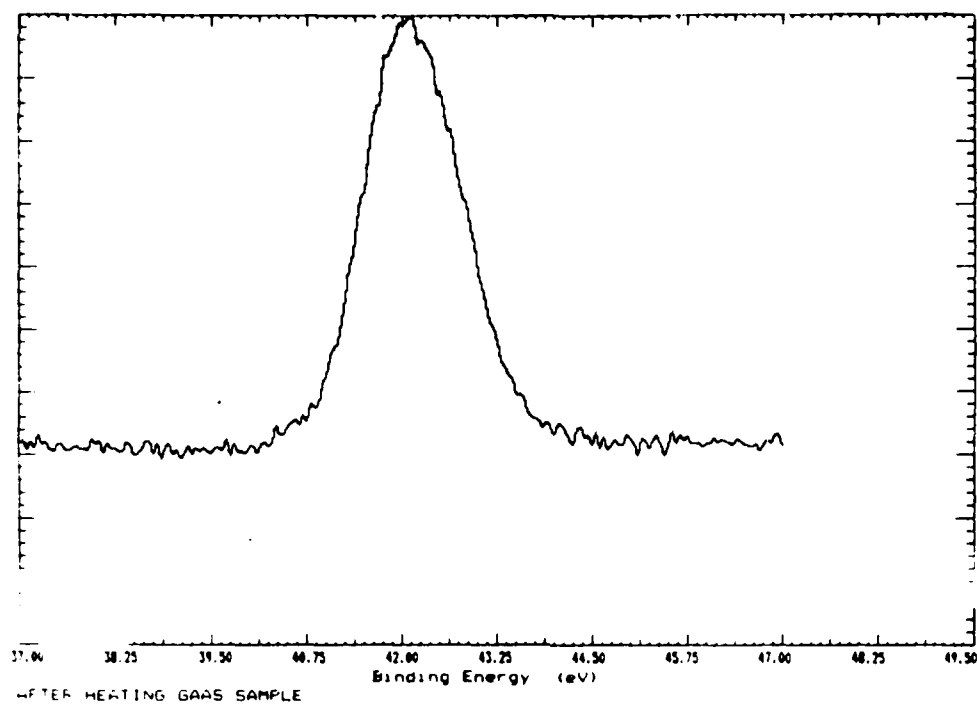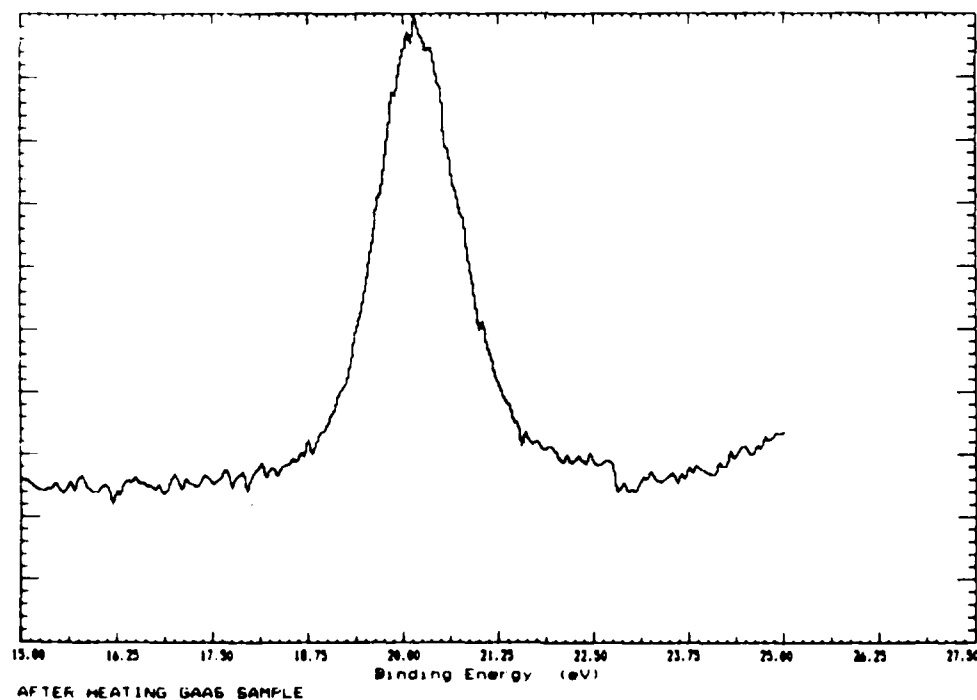
-6-

a.

BEFORE HEATING SAMPLE

b.

BEFORE HEATING SAMPLE
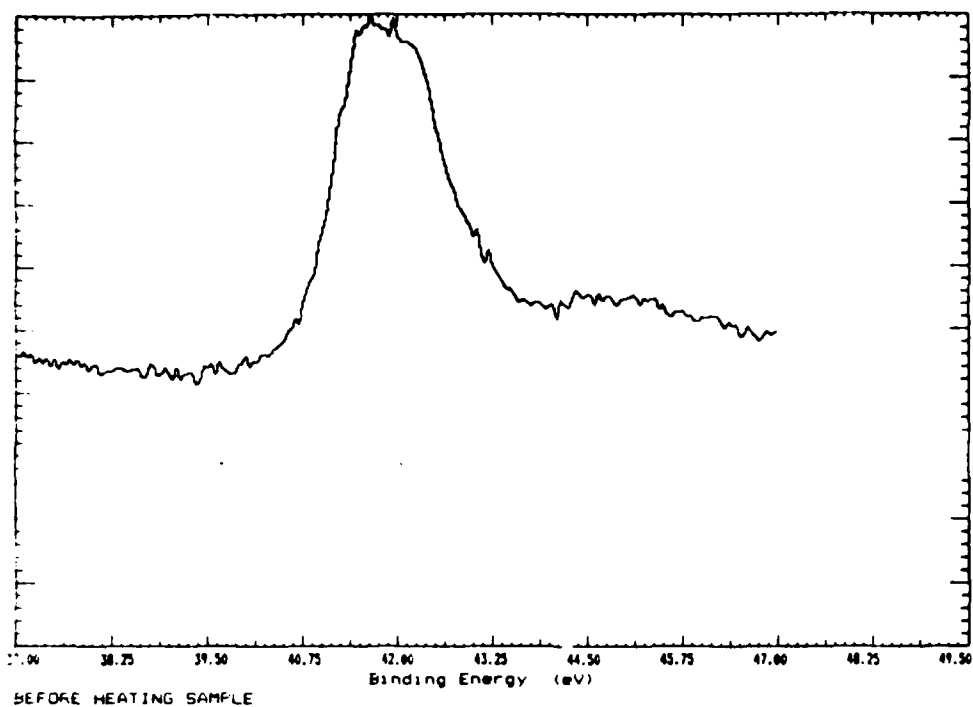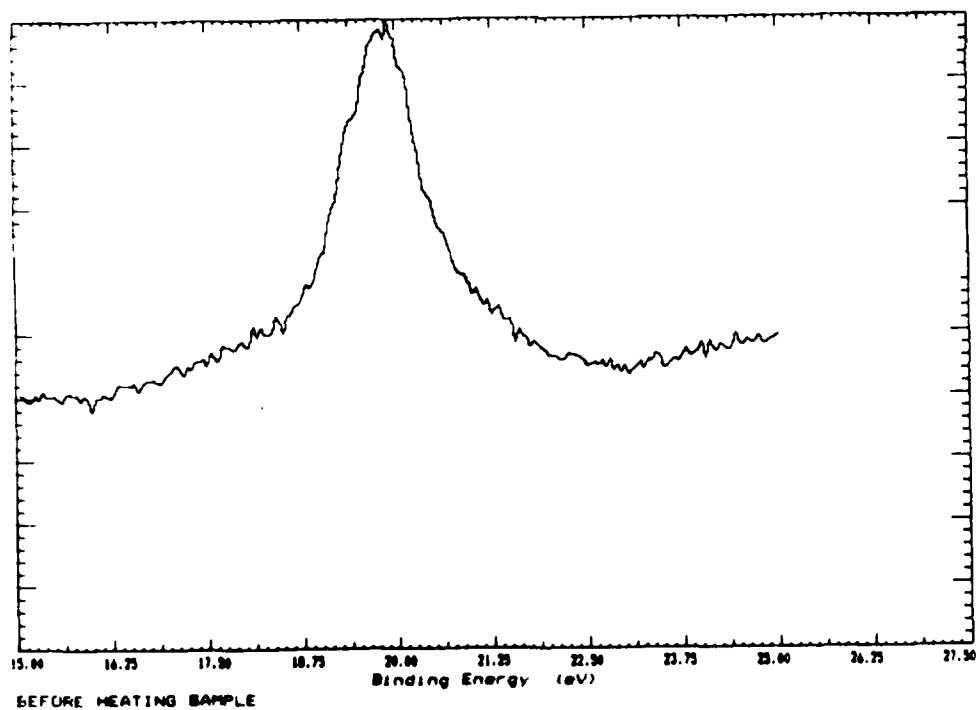
FIGURE 3.   a) Arsenic 3d region   b) Gallium 3d region of polished
            etched GaAs(100) at 180K

Binding Energy (eV)

AFTER HEATING GAAS SAMPLE

a.

Binding Energy (eV)

AFTER HEATING GAAS SAMPLE

b.

FIGURE 4.   a) Arsenic 3d region b) Gallium 3d region of polished,
            etched GaAs(100) at 180K, after heating sample to 670K.

loss of Ga_2O_3 (or $Ga(OH)_3$), leaving a surface which has a Ga:As ratio of
1.2. This compares with a value of approximately 1.0 obtained prior to
heating the oxidized substrate. Both the broadened oxygen 1s peak and the
carbon Auger peak, evident in the oxidized sample, were reduced to almost
zero on heating.

Thermal desorption mass spectrometry was also used to characterize
the annealing process, in part to identify the temperature at which oxide
removal was complete prior to bulk GaAs evaporation, evidenced by As_2^+ or
As_4^+ peaks in the mass spectrum. Initially masses 16, 32, 69, 75 and
91 amu were monitored. These masses correspond to O^+, O_2^+, Ga^+, As^+, and AsO^+
and were selected because they represent decomposition products of several
different oxides[2-5]. Subsequent data agreed with the original selection,
but an additional thermal desorption peak at mass 16 was observed, which did not
appear to correspond to either arsenic or gallium oxide (Figure 5). A mass
spectrum taken while heating the sample showed that H_2, H_2O, CO, CO_2 were
additional desorption products. Figure 5 also illustrates the relationship
between desorption of O_2^+, GaO^+ and AsO^+, indicating that along with O^+, O_2^+
is an ionization product of both As_2O_3 and Ga_2O_3. As^+ is also seen to arise
from both AsO^+, Point B, and, at higher temperatures, from As_4, Point C,
formed on evaporation of the GaAs lattice. Figure 6 illustrates the low
temperature desorption products (<660K). No evaporated As is removed from the
GaAs lattice and once again, excess O^+ is observed at lower temperatures. On
cooling the sample to 180K and reheating it is observed (Figure 7) that not all
of the oxides were removed during the first anneal and small quantities of
additional oxides were desorbed during the second thermal treatment. In addi-
tion, it is clear that bulk GaAs has been decomposed with evolution of arsenic
clusters. This was ascertained in subsequent experiments by monitoring As_4^+,
As_2^+ and As^+. It was also found that experimental conditions such as heating
rate, can be adjusted so that oxygen/oxide desorption occurs prior to bulk
arsenic loss, Point A in Figure 8.

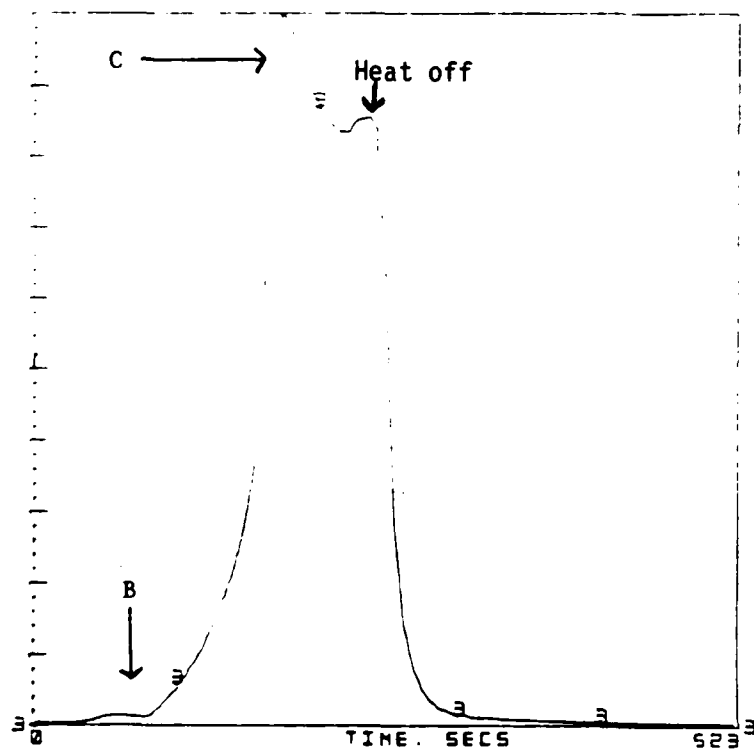4.2   Arsenic-capped MBE grown GaAs(100) - no buffer layer
        Similar results were obtained for arsenic-capped MBE grown GaAs(100)
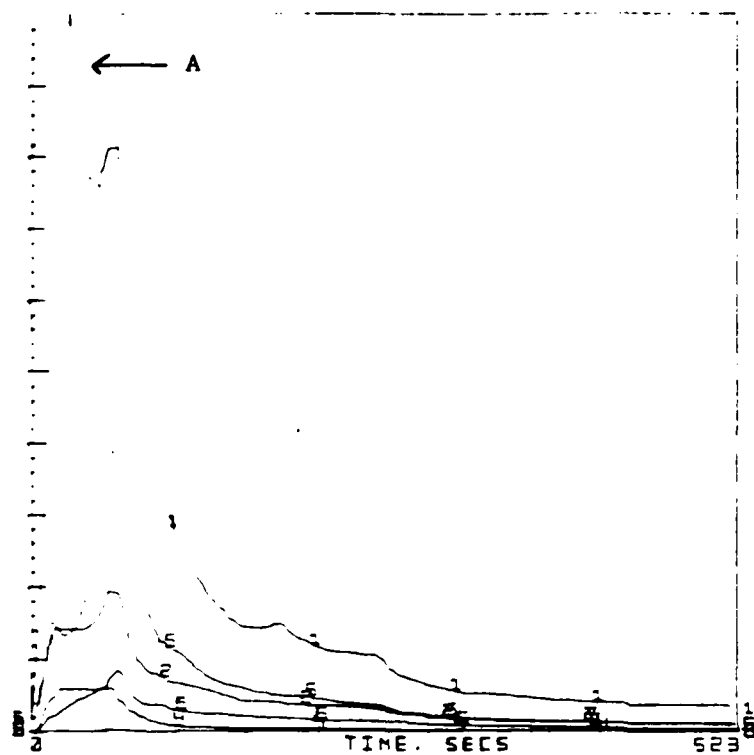
FIGURE 5. Thermal Desorption Mass Spectra of polished
chemically etched GaAs(100). Temperature
was raised from 180K to 820K, at approximately 2K/sec.

-10-
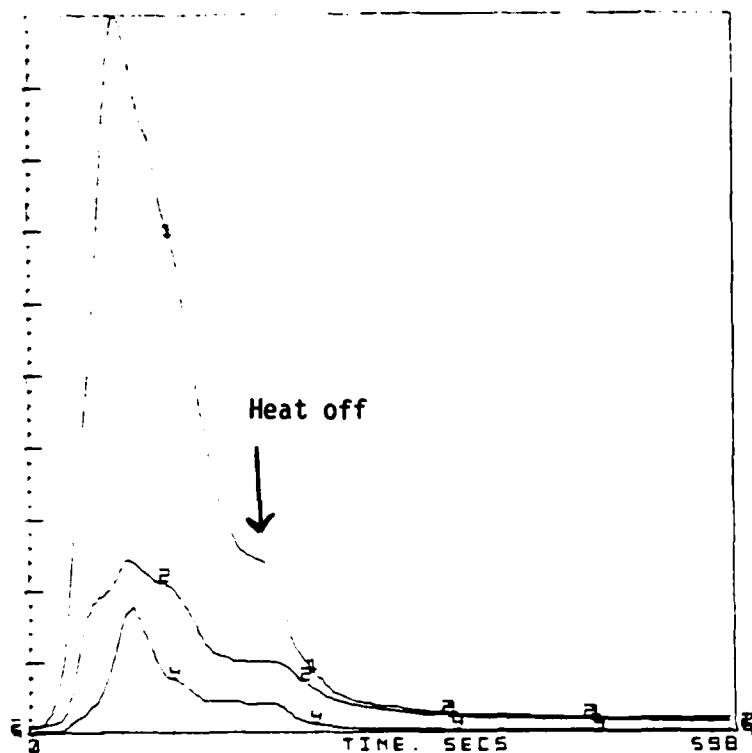
MAX HEIGHT 6658
NUM   MASS   TRGT INT SENS
   NAME   BIAS TIME FAC

1 O   16.0   0.0 1.00
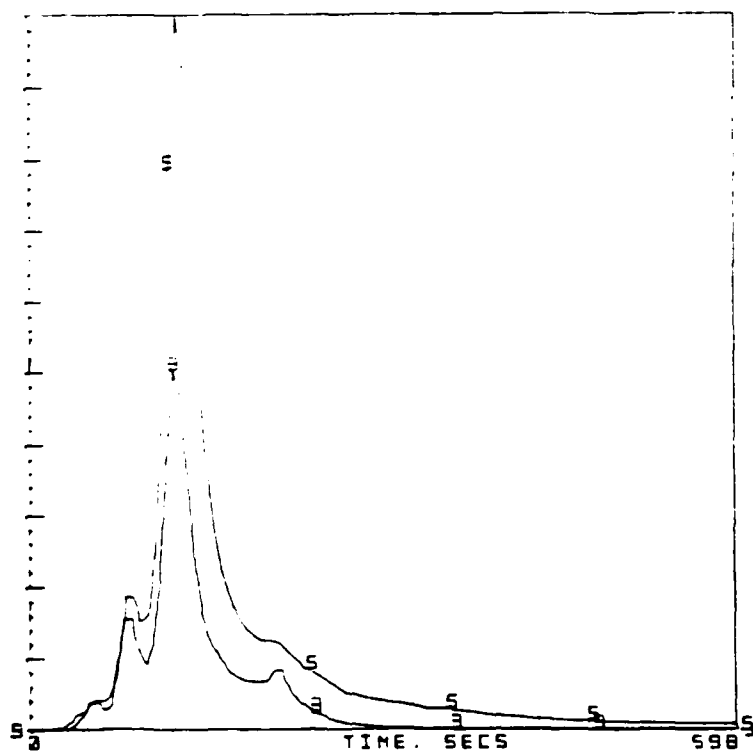2 GA   69.0   0.0 1.00
4 O₂   32.0   0.0 1.00

Heat off

a.

TIME. SECS

MAX HEIGHT 92994
NUM   MASS   TRGT INT SENS
   NAME   BIAS TIME FAC

3 AS   75.0   0.0 1.00
5 ASO 91.0   0.0 1.00

b.

TIME. SECS

FIGURE 6.   Low temperature (< 660K) Thermal Desorption
            Mass Spectra of polished, chemically-etched GaAs(100).
            Heating rate is ∿ 2K/sec.

Heat off
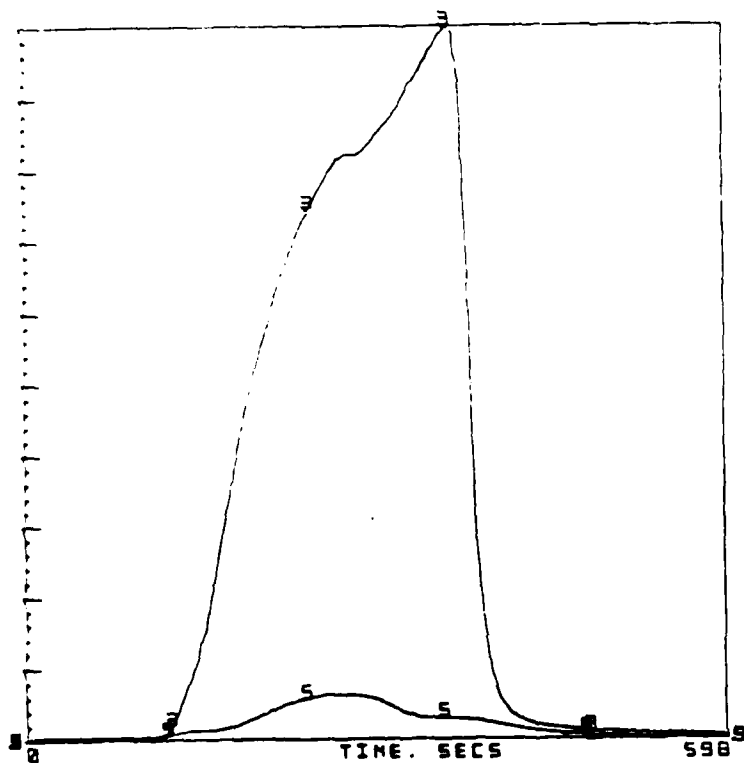
FIGURE 7. Thermal Desorption Mass Spectra of polished chemically-
etched GaAs(100). Second heating from 180-820K after
heating previously to 660K (Figure 6).
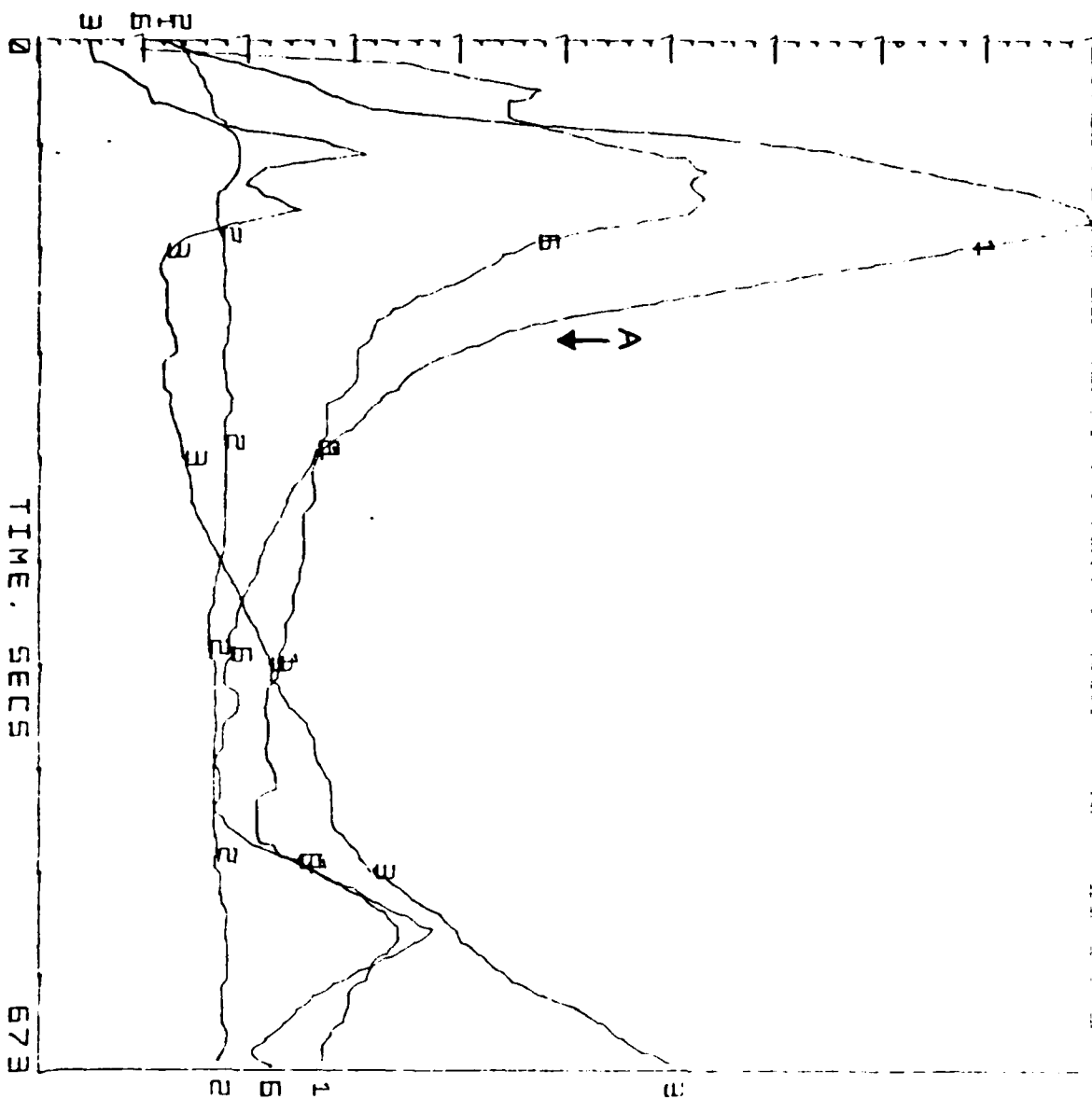
TIME. SECS   673

FIGURE 8. Thermal Desorption
Mass Spectrum. Temperature
was raised from 180K to
~730K, at ~1K/sec.

MAX HEIGHT 2255

NUM	NAME	MASS	TRGT	INT BIAS	TIME	SENS FAC
1	0	20	10.0	0.0	0.0	1.00
2	02	42	2.0	0.0	1.00	1.00
3	85	75.0	0.0	0.0	1.00	1.00
5	C0	22.0	0.0	0.0	1.00	1.00

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although the details of desorption are different. As expected XPS survey spectra showed considerably less oxide formation, arsenic oxide alone being observed, with very little carbon impurity present. Narrow, detailed scans have not been performed, as yet. Thermal desorption experiments, however, indicate that there are significant impurities in the material, most notably, H_2, H_2O, CO_2 and CO. Repeated heating cycles show additional loss of these impurities. In this case it appears that the Ga^+ peak tracks the second H_2O^+ peak, indicating the possible presence of $Ga(OH)_3$. Figure 9 illustrates this feature along with the observation that arsenic oxide does not appear to desorb as $As_2O_3^+$, but rather must form decomposition products. As_4^+ and As_2^+, however, are seen as decomposition products of the GaAs lattice, the latter probably formed by ionization of As_4. This was observed when the sample was heated to 670K prior to heating to 820K. Figure 9 also illustrates two of the three peaks associated with water desorption. Mass 18 peaks were observed at ⁓ 240K, ⁓ 270K and ⁓ 490K although the 240K peak was primarily evident in the first low temperature desorption (not shown). The shoulder observed at the high temperature side of 490K peak, which shifts to higher temperatures with increasing heating rate appears to correlate with Ga desorption from the GaAs lattice.

4.3   Arsenic-capped MBE grown GaAs(100) (with buffer layer)

Since there was no buffer layer on the previous sample it was considered a possibility that the CO, CO_2 and H_2O were due to diffusion from the substrate. To test this, 3000Å of n-type (Si) was grown by MBE on top of a 1μm buffer layer and evaluated. This material is of the quality used to make high mobility HEMPT structures and is grown at 870K. It showed considerably lower quantities of impurities than the previous two samples, however, H_2, H_2O, O, CO and CO_2 were still present. Again, thermal desorption spectra were not identical with those obtained previously. Initial heating to approximately 730K results in a Ga:As ratio of 1.6, while heating to 820K completely removes all traces of oxygen leaving the Ga:As ratio at 1.2. Further heating cycles do not appear to alter this ratio. Again, desorption of CO, CO_2, H_2O, and H_2 occurs over repeated heating. Figure 10 illustrates some of the differences observed. Water, still appears as three distinct peaks but now it appears as if the H_2O, CO and CO_2 peaks all desorb simultaneously. Ga^+ is also seen to desorb, correlating with the low temperature water peak. In addition, $As_2O_3^+$ is observed to correlate with H_2^+ desorption while As_4^+ desorption is
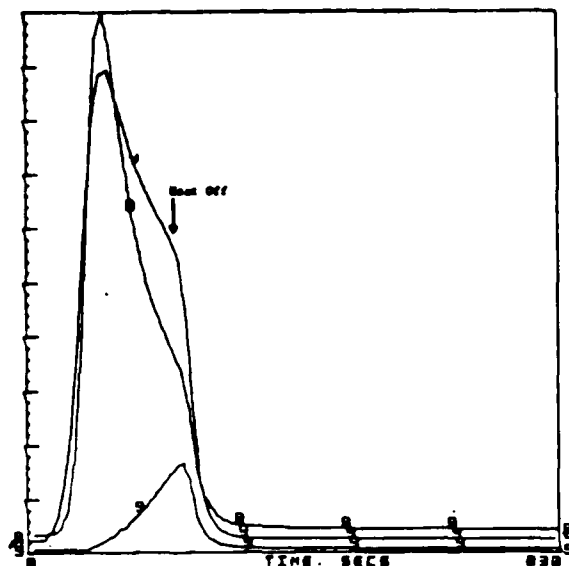
-14-
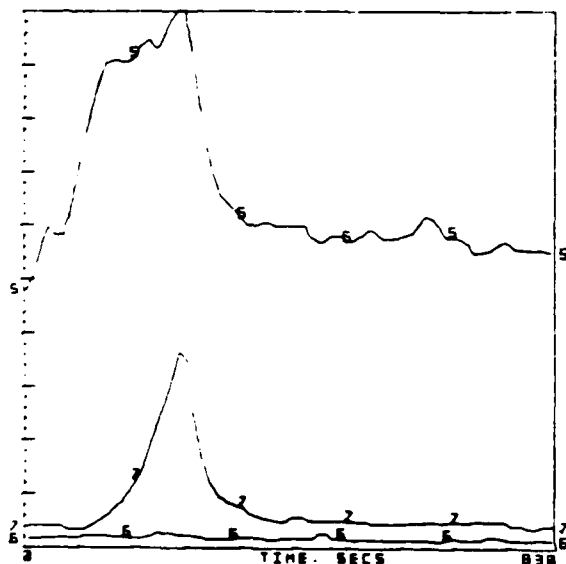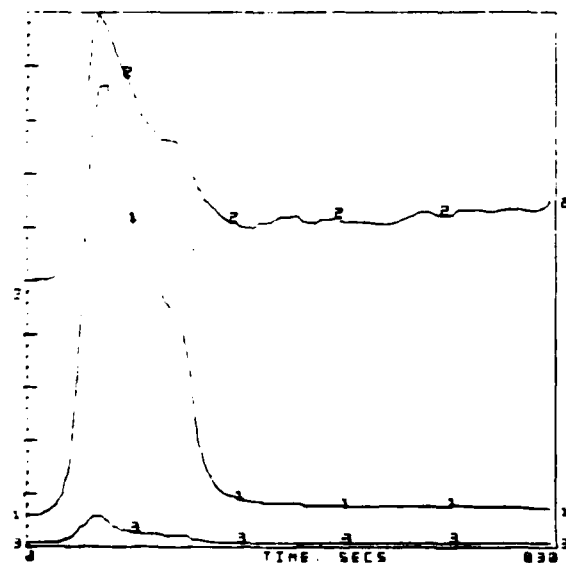
FIGURE 9. Thermal Desorption Mass Spectra of Arsenic-capped MBE grown GaAs(100), 3000Å n-type (Si) GaAs on top of polished chemicall etched substrate material. Temperature was raised from 180K at $\sim$ 2K/sec.
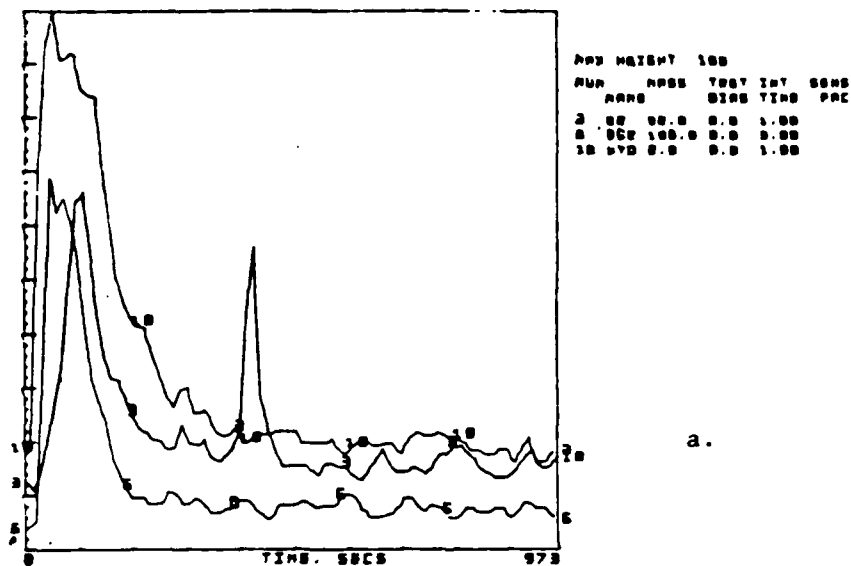
a.
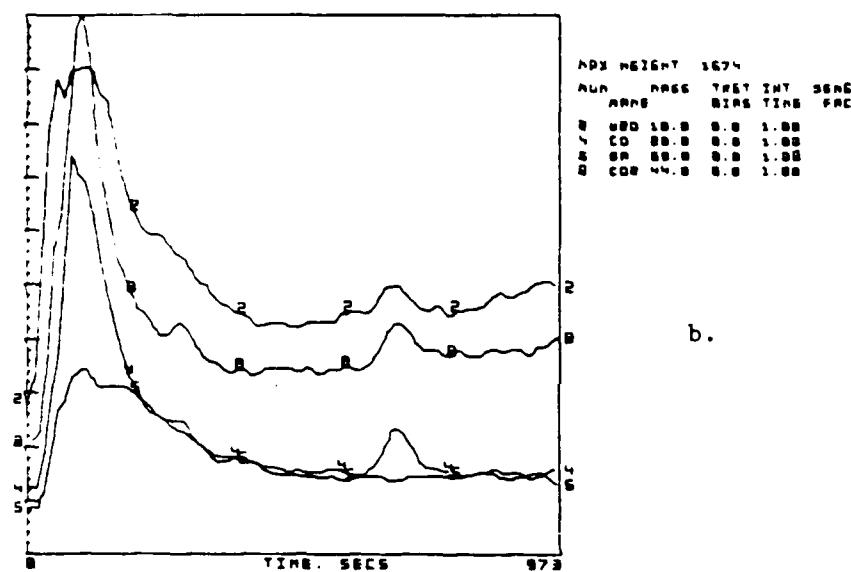
b.
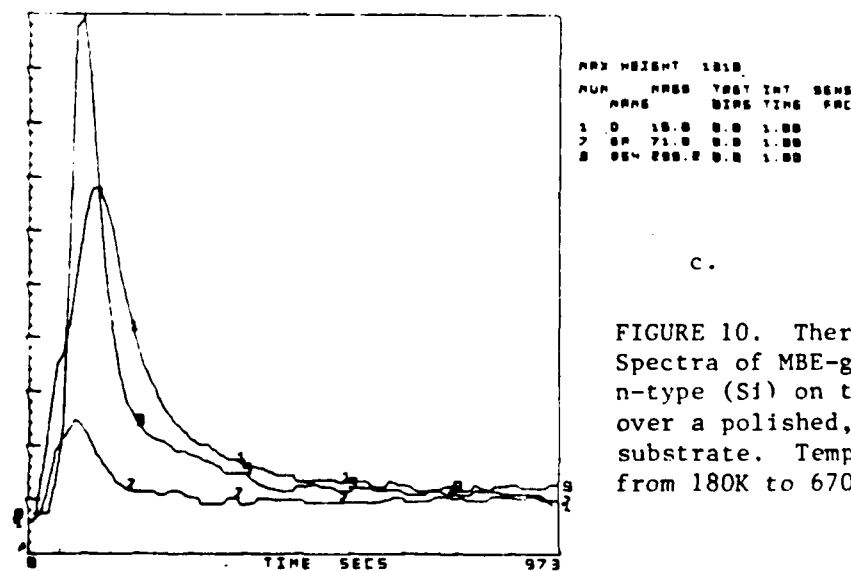
c.

FIGURE 10. Thermal Desorption Mass Spectra of MBE-grown GaAs(100), 3000Å n-type (Si) on top of 1 μm buffer layer over a polished, chemically-etched substrate. Temperature was raised from 180K to 670K.

associated with the O_2^+ peak. As_2 and As_4 from the GaAs lattice are not
seen to desorb at these temperatures. The four-fold LEED pattern is shown
for the MBE grown material after oxide removal in Figure 11.

4.4   Reaction of Water with GaAs(100)

In order to better characterize the passivation process on GaAs(100)
the reaction of water from 180K - 820K was studied. While the doser substrate
was held at 180K, approximately 10^3L of H_2O was admitted via a molecular beam. A
survey XPS scan was taken from heating the sample to 570K to obtain a thermal
desorption profile. An oxygen 1s peak was observed at a binding energy
corresponding to hydroxide species. However, no detailed narrow scans were
taken at the time. These are planned for future experiments. In addition,
the initial four-fold LEED pattern was removed upon addition of the water and no
new pattern emerged. Figure 12 shows the major peaks in the desorption experiment.
Although the heating rate is non-linear it is evident that only two peaks desorb
below 570K, the first at ~270K, the second at ~480K. As expected the O^+
peak tracks the H_2O^+ peak. Most of the residual H_2O, H_2, CO and CO_2 had
been removed by successive annealing cycles and no gallium or arsenic was seen
to desorb in any form. A similar experiment showed H_2^+ desorption also tracked
the H_2O^+. The H_2^+ was not ionization product. XPS spectra taken from thermal
desorption indicated that oxygen present on the surface was probably an oxide.
Again no high resolution spectra have been taken. The Ga:As ratio now
appeared to have increased to 1.6.

4.5   Reaction of Ammonia, Monomethylamine and Trimethylamine with GaAs(100)

XPS data show that ammonia, monomethylamine and trimethylamine all
absorb on the Ga-rich, polished, etched GaAs(100) surface. However, there
were continuing problems with the temperature at which we could maintain the
manipulator. At 190K these species have a vapor pressure of 35 torr, 8 torr
and 1 torr respectively. Thus, absorption at this temperature is difficult.
Modifications to the manipulator are currently being made which will allow
experiments to be performed routinely below 150K.

-17-
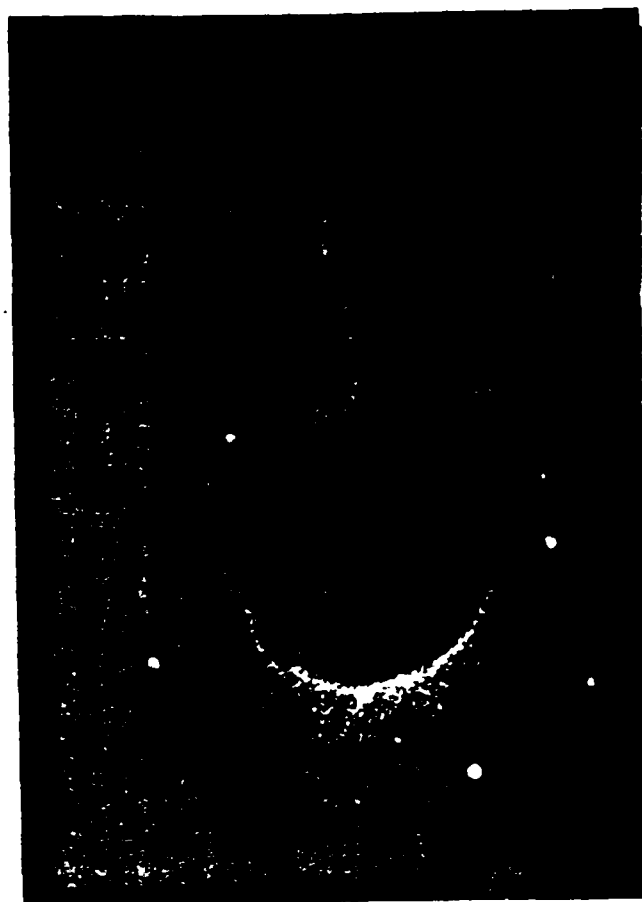
FIGURE 11. Four-fold Low Energy Electron Diffraction pattern
of thermally-treated chemically-etched GaAs(100).
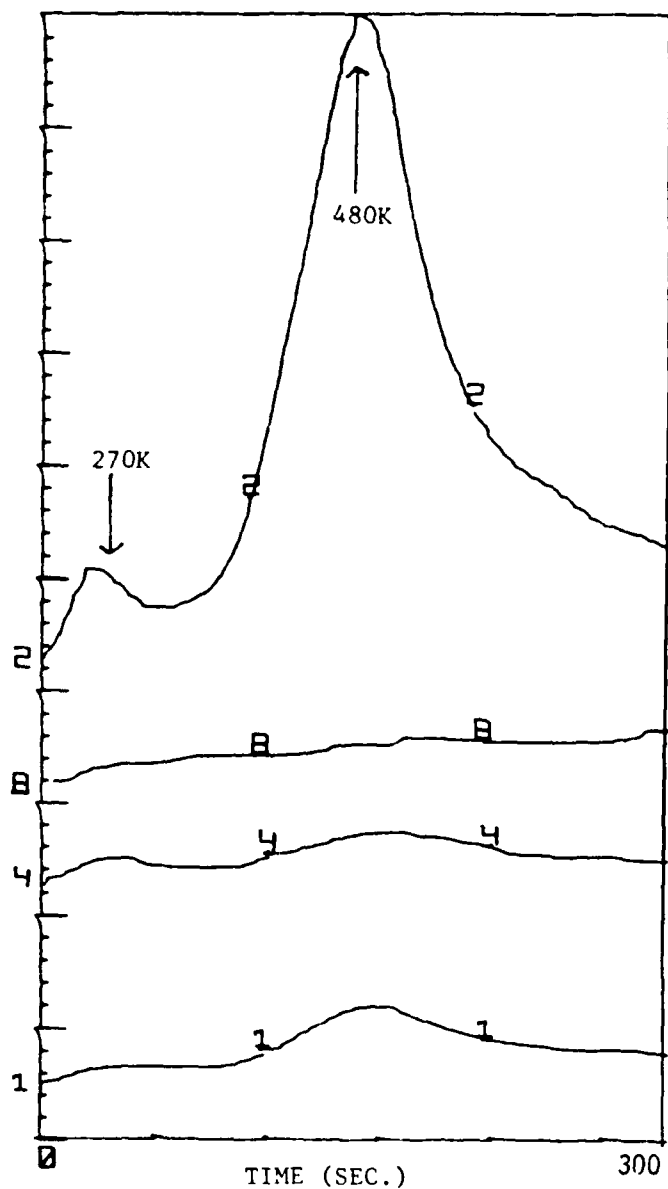
MAX HEIGHT 7510

NUM	MASS NAME	TRGT BIAS	INT TIME	SENS FAC
1	O 16.0	0.0	1.00	
2	H2O 18.0	0.0	1.00	
4	CO 28.0	0.0	1.00	
8	CO2 44.0	0.0	1.00	

FIGURE 12. Thermal Desorption Mass Spectrum of water on clean GaAs(100).

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5.   DISCUSSION

The chemistry occurring in all three samples of GaAs(100) is more complex than was anticipated. The oxidation of the polished, etched GaAs(100) appears to be quite different from the surface formed by the more traditional acid etch.[6,9] As^{+5} was not observed at 180K or after heating, while the surface appears to comprise GaAs, and As_2O_3 but no elemental As. Hydroxide formation was not clearly observed, although based on subsequent data, this could be monitored by thermal desorption experiments. A monochromatic X-ray source can be used for XPS spectra in order to verify that no elemental arsenic is present on the surface. None was observed in the thermal desorption data (Figure 5b) until the GaAs lattice evaporated, the peak at lower temperatures corresponding to an ionization product of arsenic oxide. Our results support observations that an NH_4OH final etch after an anodic NH_4OH etch eliminates excess As and produces a nearly stoichiometric surface.[10] In our case, the NH_4OH/H_2O_2 etch was the only etch and anodic oxidation was unnecessary.

The effect of the thermally desorbed CO, CO_2, H_2O and H_2 depends on the source of contamination. It is likely that the use of acetone as a degreaser (or isopropyl alcohol as a rinse) may be responsible for these products since the CO_2, CO, H_2O and H_2 desorption peaks seem to be correlated with each other. Two possibilities are that the acetone is thermally decomposing and as the temperature increases the products diffuse through the GaAs and desorb, or the acetone diffuses and decomposes at the surface. We propose to investigate these reactions further by comparing the reactions of acetone on the GaAs(100) surface with those on clean, cleaved GaAs(110). It may also be instructive to compare another degreaser, such as hexane, with acetone.

The observations could also expain why the MBE material grown without a buffer layer exhibit CO, CO_2, H_2O and H_2 desorption, in a qualitatively similar fashion. Repeated heating would allow diffusion to occur through the 3000Å n-type (GaAs) epitaxial layer. When a 1μm buffer layer is present impurity

diffusion is sufficiently blocked such that a ⌣90% reduction in peak intensities is observed. It may be true that this buffer layer completely blocks diffusion from the substrate. However, in order to clean indium from the back-side of the wafer, MBE-grown material is removed from the sample with wax which is subsequently dissolved in acetone. So the source of contamination is difficult to determine at present. Further experiments are in progress where removal of the MBE grown wafers are accomplished without the use of wax and acetone.

Although the results of water absorption of GaAs(100) are by no means definitive, it appears likely that H_2O adsorbs both associatively and dissociatively on GaAs(100). This is evidenced by the presence of both H_2O^+ and excess H_2^+ in the desorption spectra, as well as oxide formation on the surface during heating. It appears that the 270K peak is multilayer water desorption while the peak at ⌣480K represents a chemisorbed species. The 240K peak observed in TDMS during oxide removal may have been formed as a result of Ga sites reacting with other species. As mentioned earlier, literature reports of water reactions on GaAs(100) are sparse and none have been examined for a similar surface with a similar preparation. However, both associative[7] and dissociative mechanisms[8] have been postulated for surfaces prepared by cleaving and argon ion bombardment and annealing, respectively, the latter being GaAs(100) while the former represents low temperature absorption on GaAs(110).

To understand the ammonia/amine adsorption on GaAs(100), it is useful to examine XPS studies of these species in the gas phase, as solids and on surfaces. The binding energy of the gas phase ammonia 1s nitrogen peak is 405.43 eV while that of solid NH_3 (powder) is 398.8 eV[11]. This value is reduced to 399.0 eV in solid KCN and 397.9 in BN[12]. It thus seems likely that monomethylamine and trimethylamine have dissociatively adsorbed while ammonia has remained associatively adsorbed.

Nitrogen 1s Binding Energies (in ev)

NH_3	401.0		
$(CH_3) NH_2$	399.9	402.2	
$(CH_3)_3 N$	398.3	401.0	403.5

TABLE 1

6. SUMMARY

We have initiated investigations in three areas of study. These are (1) the use of Thermal Desorption Mass Spectrometry (TDMS) to study contamination or impurity reactions of GaAs, (2) the reaction of water on GaAs together with characterization of the resulting surface and (3) study of specific acid and base sequences as chemical probes for electronic sites in compound semiconductors. Results, to date, indicate that all three investigations will lead to valuable information in the area of GaAs processing. TDMS has already allowed us to identify contaminants which would interfere with device performance. Carbon is known to be a very shallow acceptor and a p-type dopant while oxygen is deep level trap. By identifying and removing causes of such contamination, device performance will be improved.

Future work could include studies of both acetone, isopropyl alcohol and hexane on GaAs in an attempt to identify chemically adsorbed species that could cause contamination. These studies, in conjunction with the completion of the experiments on water adsorption on GaAs will provide a useful basis for understanding cleaning processes in III-V compound semiconductors. In addition, experiments involving the sequence of Lewis bases may lead to schemes whereby the surfaces can be passivated in a reproducible fashion. By depositing metals on top of the passivated surface, precise control of Schottky Barrier heights could be achieved. Completeted manuscripts of this work will be submitted so that they may be attached to this final report.

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