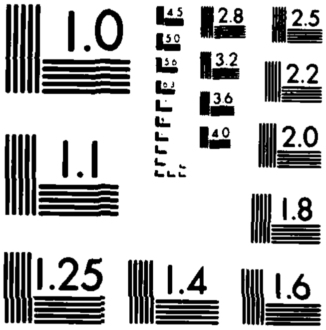
AD-A148 777 MATERIALS-PROCESS INTERACTIONS IN TERNARY ALLOY SEMICONDUCTORS(U) HUGHES RESEARCH LABS MALIBU CA K V VAIDYANATHAN ET AL. SEP 83 N00019-82-C-0066 1/1

UNCLASSIFIED F/G 20/12 NL

END

FILMED

DTIC



MICROCOPY RESOLUTION TEST CHART
NATIONAL BUREAU OF STANDARDS-1963-A

MATERIALS–PROCESS INTERACTIONS IN TERNARY ALLOY SEMICONDUCTORS

K. V. Vaidyanathan and J.F. Wager III

Hughes Research Laboratories
3011 Malibu Canyon Road
Malibu, CA 90265

April 1984

N00019–82–C–0066
Quarterly Report 5
1 May 1983 through 31 July 1983

Sponsored by
DEPARTMENT OF THE NAVY
Naval Air Systems Command
Washington, DC 20361

UNCLASSIFIED
SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)

REPORT DOCUMENTATION PAGE

READ INSTRUCTIONS
BEFORE COMPLETING FORM

1. REPORT NUMBER

2. GOVT ACCESSION NO. AD-A145 777

3. RECIPIENT'S CATALOG NUMBER

4. TITLE (and Subtitle)
MATERIALS-PROCESS INTERACTIONS IN TERNARY
ALLOY SEMICONDUCTORS

5. TYPE OF REPORT & PERIOD COVERED
Quarterly Report 5
1 May 1983 - 31 July 1983

6. PERFORMING ORG. REPORT NUMBER

7. AUTHOR(s)
K.V. Vaidyanathan and J.F. Wager III

8. CONTRACT OR GRANT NUMBER(s)
N00019-82-C-0066

9. PERFORMING ORGANIZATION NAME AND ADDRESS
Hughes Research Laboratories
3011 Malibu Canyon Road
Malibu, CA 90265

10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS

11. CONTROLLING OFFICE NAME AND ADDRESS
Department of the Navy
Naval Air Systems Command
Washington, DC 20361

12. REPORT DATE
September 1983

13. NUMBER OF PAGES
15

14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)

15. SECURITY CLASS. (of this report)
Unclassified

15a. DECLASSIFICATION/DOWNGRADING SCHEDULE

16. DISTRIBUTION STATEMENT (of this Report)

APPROVED FOR PUBLIC RELEASE
DISTRIBUTION UNLIMITED

17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)

18. SUPPLEMENTARY NOTES

19. KEY WORDS (Continue on reverse side if necessary and identify by block number)
Properties of InGaAs, surface studies on InGaAs, x-ray photoelectron spectroscopy on InGaAs, Electron energy loss spectroscopy on InGaAs.

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)
The report describes the properties of InGaAs and the results of detailed x-ray photo-electron and electron energy loss (XPS, ELS) spectroscopies on $In_{0.53}Ga_{0.47}As$ samples. The results show that in this alloy system, the native oxide consists of As_2O_3 and oxides involving In. Oxides involving Ga with other elements are also present.

SECTION 1

INTRODUCTION

Recent improvements in optical fiber technology have resulted in the availability of fibers exhibiting low loss and low dispersion in the 1.2 to 1.6 μm range. These fibers are also radiation resistant and are expected to play a major role in long baseline, high speed, secure communication systems. The availability of reliable semiconductor light sources and detectors capable of operating in this wavelength range is crucial to the development of such communication system.

Experimental results suggest that photodiodes fabricated in $In_{0.53}Ga_{0.47}As$ (grown lattice-matched to InP substrates) can act as detectors for the desired wavelength range. For such systems applications, it is desirable to integrate the detectors with circuits capable of performing signal processing functions. These circuits can either be fabricated in InGaAs epitaxial layers or in InP substrates. To accomplish such a level of integration, a mature process technology in InGaAs must be developed. The device technology in this alloy system is in a truly primitive state. The primary objective of this program is to develop a reliable and mature process technology in InGaAs, paying particular attention to ion implantation doping of InGaAs and studying the dielectric-sesmiconductor interface so that InGaAs MISFET device and photodetector technology can be established.

SECTION 2

CHARACTERIZATION OF InGaAs SURFACES

Development of a mature InGaAs MISFET device technology depends critically on our understanding of the semiconductor-to-dielectric interface. As is the case for most other III-V semiconductor systems, InGaAs MISFETs have been fabricated using deposited dielectrics. In this instance, the interface consists of the deposited dielectric as well as any native oxide present on the semiconductor surface. In this section we describe the results of experiments performed to elucidate the nature of the native oxide present on InGaAs surfaces.

A. DESCRIPTION OF EXPERIMENTAL TECHNIQUES

In this experimental investigation, two spectroscopic techniques were used to study the chemical nature of the InGaAs surface: x-ray photoelectron spectroscopy (XPS) and electron energy loss spectroscopy (ELS). The experiments were performed using a VG Scientific ESCALAB Mark II spectrometer.

B. X-RAY PHOTOEMISSION SPECTROSCOPY (XPS)

In this process an incident x-ray beam ionizes core electrons. Some of the ionized photoelectrons escape from the sample into vacuum and can be detected and energy analyzed. Knowing the incident photoenergy ($h\nu$) and the kinetic energy (K.E.) of the electrons emitted, one can calculate the binding energy of the electrons, BE, which is given by

$$BE = h\nu - K.E. \quad , \qquad (1)$$

where K.E. is the kinetic energy of the emitted electron. In our experiments, X-rays from a Mg target ($K\alpha$) with an energy of 1253.6 eV were used. In a typical XPS spectrum the number of electrons, N(E), is displayed as a function of the binding energy. Thus, a peak in XPS spectrum is characterized by a certain binding energy and represents the electrons emitted from a certain atomic orbital.

Changes in the local chemical bonding (for example, indium bonded to oxygen versus In metal) may result in a chemical shift of the XPS peak. The observed chemical shifts can then be related to the nature of chemical bonding present in the analyzing volume. Electrostatic charging of the sample can create problems in determining the exact chemical shift. In the present work we have circumvented this potential problem by referencing all binding energies with respect to the omnipresent carbon peak at 284.6 eV. A major advantage of XPS stems from the fact that the photoelectron escape depth is less than ~ 100 Å and therefore can be used to non-destructively analyze native oxide/semiconductor and deposited oxide/semiconductor interfaces. In Figure 1 we schematically show the experimental arrangements as well as a stylized XPS spectrum.

C. ELECTRON ENERGY LOSS SPECTROSCOPY (ELS)

In this technique, a beam of low energy electons, typically < 1 keV, impinges on the sample surface. These primary electrons can undergo either elastic or inelastic scattering. Electrons scattered elastically change direction but do not suffer energy loss. This process is used in low energy electron diffraction (LEED). The inelastically scattered electrons suffer energy loss. This loss may either be due to excitation of surface or bulk plasmons or interband transitions. Again, some of the primary electrons escape from the sample and can be energy analyzed.

ELS measurements can be performed in two modes. in the first mode, the second derivative of the number of electrons with respect to energy ($- d^2N(E)/dE^2$) is plotted as a function

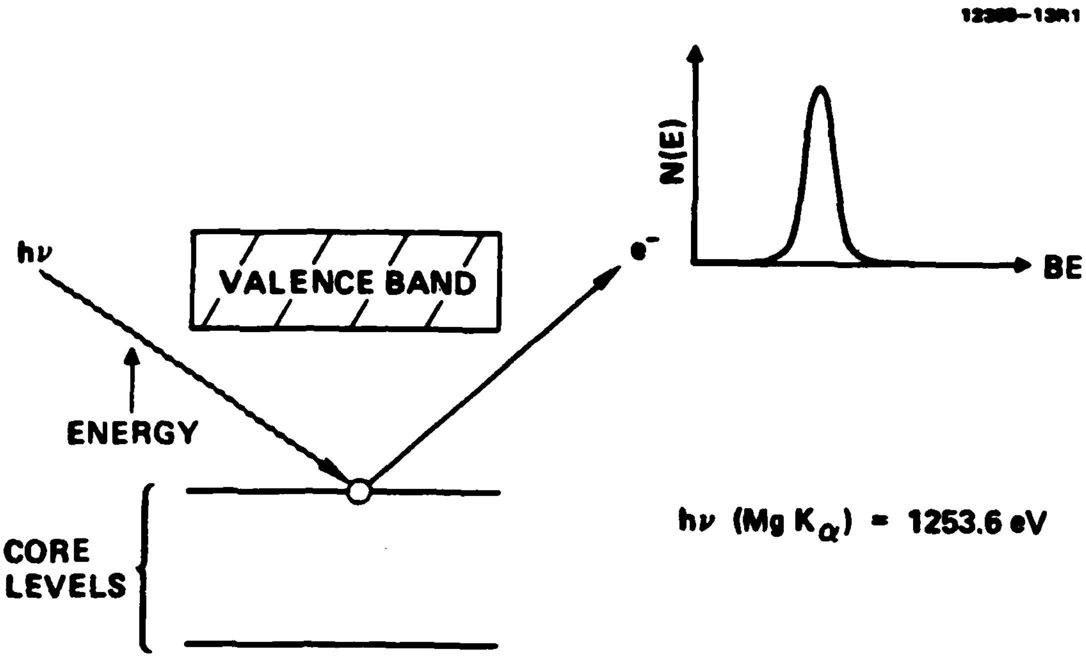


Figure 1. A schematic illustrating the XPS technique.

of the energy loss, E_L. This technique is used when studying plasmons and valence-to-conduction band interband transitions. In the second mode, a very low energy primary beam (~10 eV) is used. A high resolution ELS spectrometer is required for these studies. Resolution of the elastic peak of FWLIM of ~ 10 meV is required in such measurements. This analysis is typically used to study lattice vibrations.

The experiments described in this section were performed in the following manner. A standard Auger electron gun was used with a prmary electron energy of 300 eV and a beam current of 10 nA. Data were collected directly in the N(E) mode using standard retarding potential and pulse-counting electronics. The resolution of the system was 0.55 eV FWHM. Very low beam currents can be used in this process, thereby avoiding the problem of electron beam damage to the analyzing surface. Because the spectrum is collected in the N(E) mode which is similar to surface sensitive optical absorption measurements, any absorption edges in the spectrum can be used to estimate bandgaps of surface compounds present and also to provide useful information regarding the trap structure of surface compounds. Finally, the collected N(E) spectrum can be double differentiated using a computer if needed. A schematic of the ELS process is presented in Figure 2.

D. EXPERIMENTAL RESULTS

InGaAs epitaxial layers grown by LPE and lattice matched to InP substrates were first subjected to a thorough cleaning process. The n-type layers were nominally undoped, with a carrier concentration of ~ 5×10^{-15} cm-^{3}. These layers were then etched for ~ 1 min in a solution of 1:2:400 parts by volume of $NH_4OH:H_2O_2:H_2O$, for 30 sec. in a solution of 1:15 parts by volume of $NH_4OH:H_2O$, and rinsed in a 4:1 mixture of methanol and acetone and blown dry in nitrogen. After etching, the samples were exposed to amient air for ~ 1 hour before loading into the VG ESCALAB Mark II system for analysis.

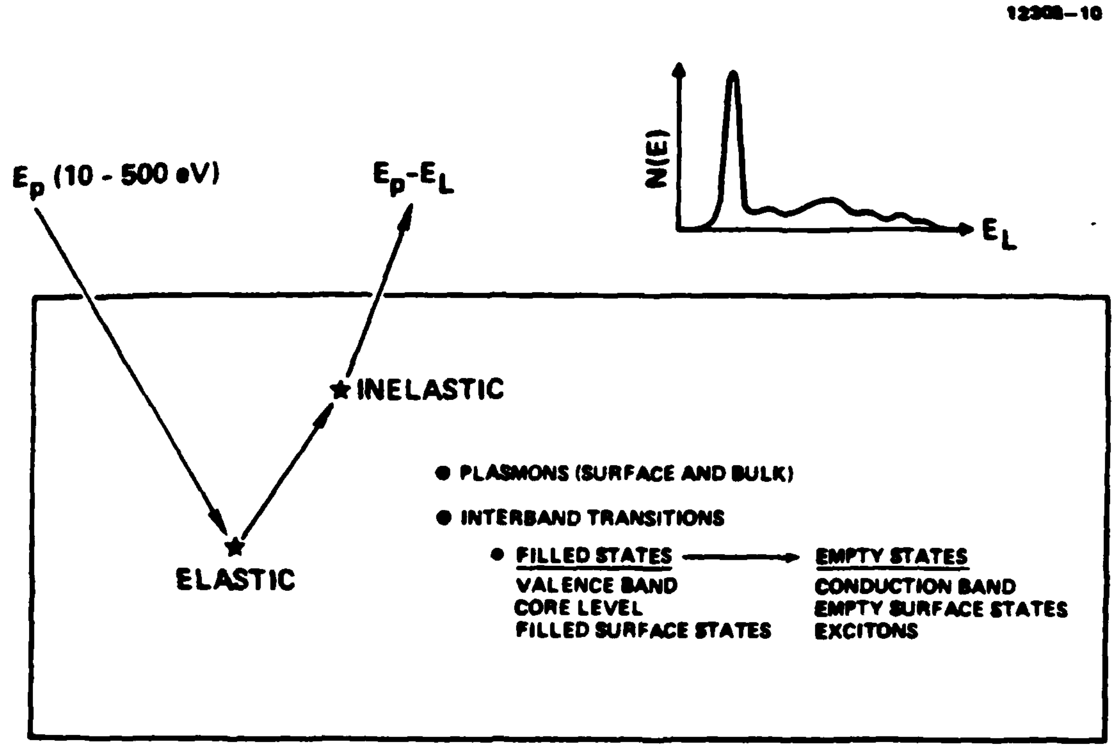


Figure 2. A schematic illustrating the ELS technique.

XPS studies were performed on these etched surfaces using the procedures described earlier. Carbon and oxygen were the only detectable contaminants on the surface.

In Figure 3 we illustrate the XPS spectrum of the As 3-d states. The spectrum consists of two well-defined peaks. The As substrate peak is located at a binding energy of 40.4 eV, while the second weaker peak is located at 43.9 eV, with an energy separation of 3.5 eV between the two peaks. The position and shape of the major As peak is consistent with bonded As. No metallic As is observed. The weaker peak is related to oxidized As; its binding energy is consistent with that of As_2O_3.

In Figure 4 we show the In $3d_{5/2}$ XPS spectrum. The spectrum again shows clear evidence of two peaks, at 444.2 eV and 445.0 eV. The 44.2 eV peak is related to the InGaAs substrate, while the 445.0 eV peak is related to the presence of oxidized indium. The observed binding energy of the oxidized In peak is too high for In_2O_3. Clark et al.[1] report a binding energy of 444.4 eV for In_2O_3, while Bertrand[2] claims it to be 444.3 eV. Our observed binding energy of 445.0 indicates that the dominant oxide in this case is not In_2O_3. Potential candidates include $InAsO_4$, InO-OH, In $(OH)_3$, etc. More work is required to resolve this issue.

The XPS spectrum obtained from Ga 3d/In4d levels is quite complex and is rather difficult to interpret. It clearly showed the presence of oxidized In; however, it was not clear from this spectrum whether gallium oxide was present or not. From heat-of-formation considerations, one would expect to find oxidized gallium.

We therefore decided to perform detailed XPS measurement on Ga $2p_{3/2}$ level. Spectra were obtained with two different take-off angles, 0° and 50°, respectively. In Figure 5, we show the 0° spectrum. A substrate peak at 117.8 eV and an oxide peak at 1118.2 eV can be seen. The oxide peak is more clearly evident in the 50° take-off spectrum (which is more surface sensitive),

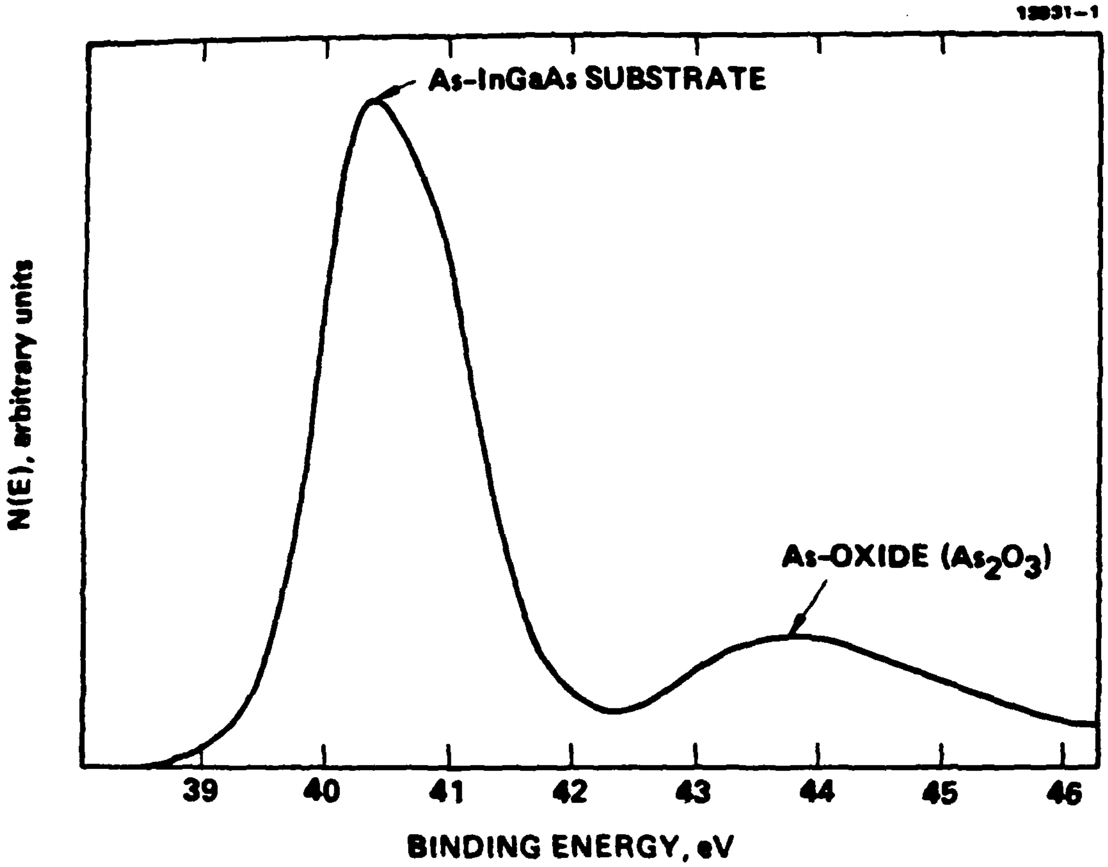


Figure 3. XPS As 3d spectrum from air-oxidized InGaAs.

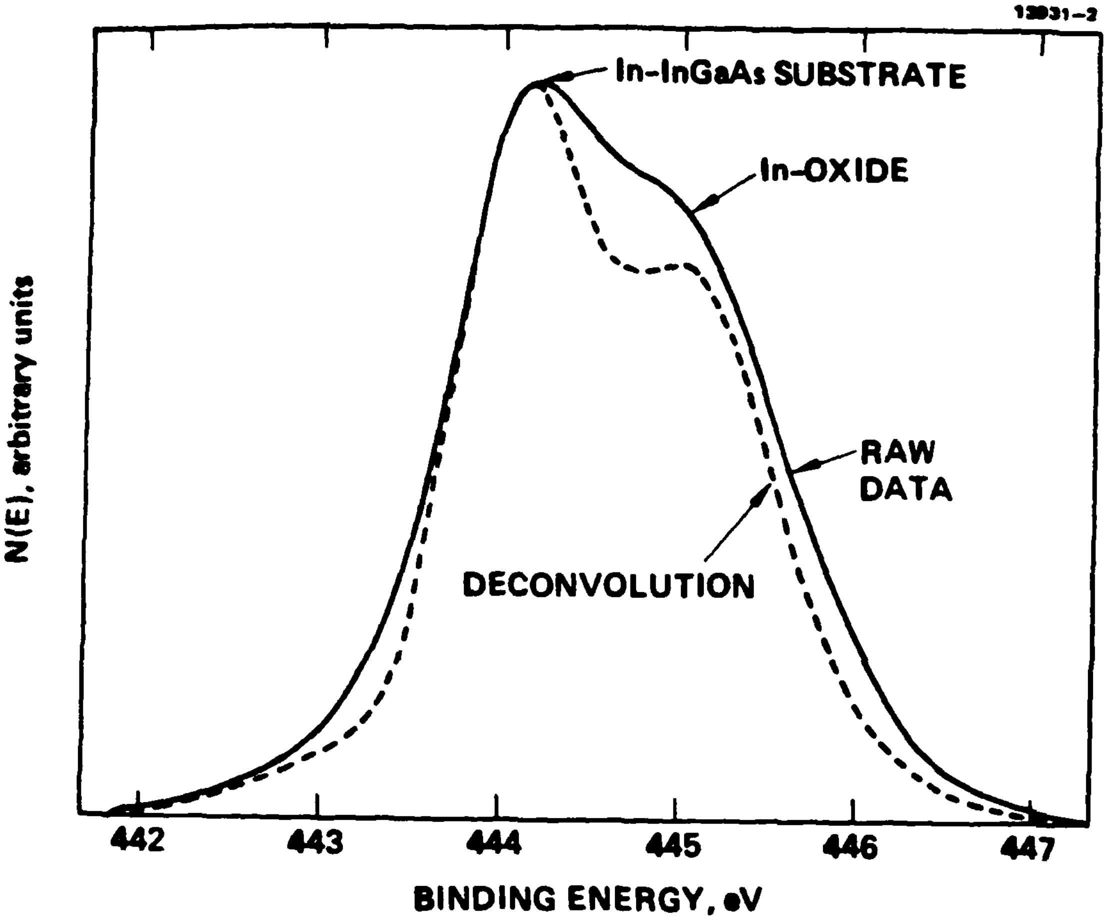


Figure 4. XPS in $3d_{5/2}$ spectrum from air-oxidized InGaAs. The deconvolution spectrum has been corrected for system broadening.

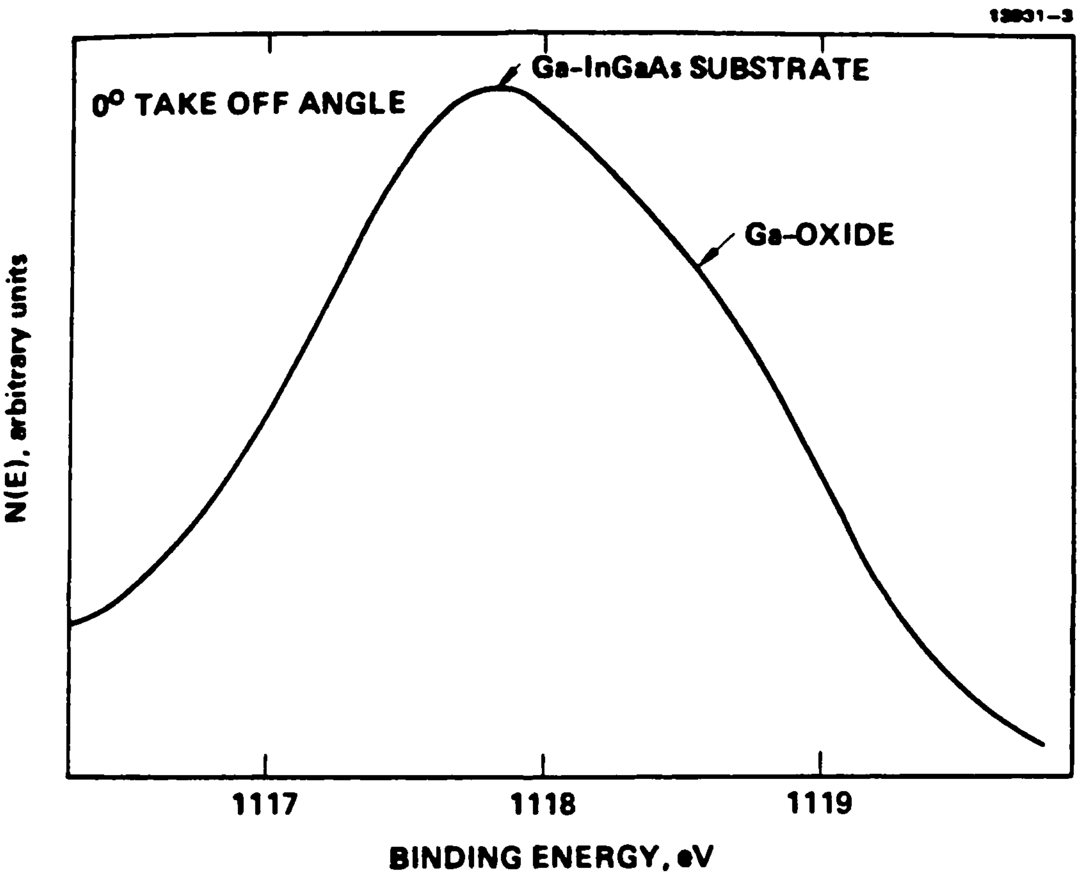


Figure 5. XPS Ga $2p_{3/2}$ spectrum from air-oxidized InGaAs taken at a take-off angle of 0°.

as seen in Figure 6. Clearly, more detailed work is required before we can unambigiously determine the exact oxidation state of gallium.

We estimate the thickness of the native oxide to be ~ 6 Å from the As 3d data, to be ~ 15 Å from In data, and ~ 18 Å from Ga data. We can also estimate the "near surface" composition of the $In_{0.53}Ga_{0.47}As$ layers, and is presented in Table 1. From these data, it is clear that while the measured In concentration is close to the bulk composition, the surface is gallium-rich and As deficient. This near surface composition may be extremely sensitive to the nature of surface treatment that the InGaAs layer undergoes prior to analysis.

The electron energy loss spectroscopy (ELS) spectrum is illustrated in Figure 7. The primary electron energy used was 303.5 eV. The ELS spectrum shows an absorption edge at 2·1eV and an interband transition at 6.4 eV. We have measured the ELS spectra of etched GaAs and oxidized In surfaces. Comparison of these data suggests that the ELS spectrum on an etched InGaAs surface is dominated by the presence of In oxide, similar to In_2O_3. Again, more careful analysis is required in order to understand the condition of chemically etched $In_{0.53}Ga_{0.47}As$ surfaces.

Table 1. Measured Composition of the Near Surface Region of $In_{0.53}Ga_{0.47}As$ Layers as Compared with Theoretical Composition

Line	Measured Atomic %	Theo. Atomic %
In $3d_{5/2}$	25	26.5
In 4d	24	26.5
Ga 3d	34	23.5
As 3d	41	50

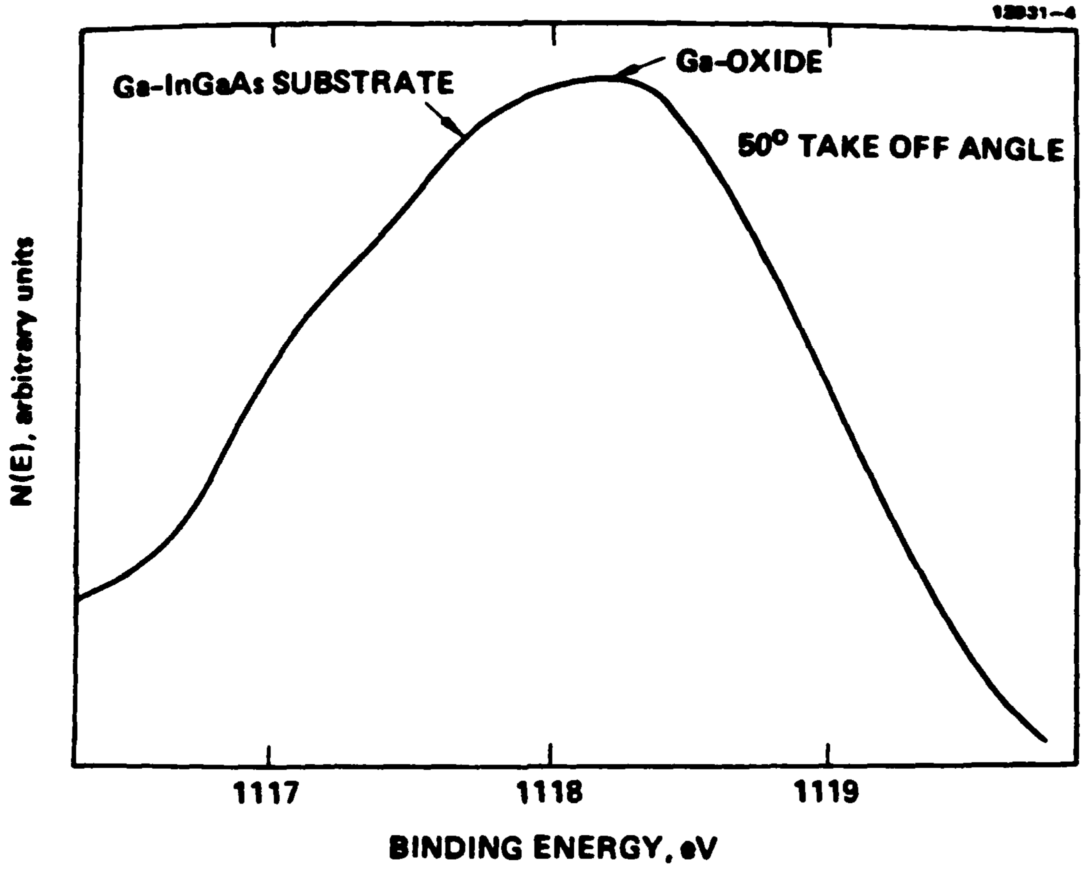


Figure 6. XPS Ga $2p_{3/2}$ spectrum from air-oxidized InGaAs taken at a take-off angle of 50°. This condition is more surface-sensitive.

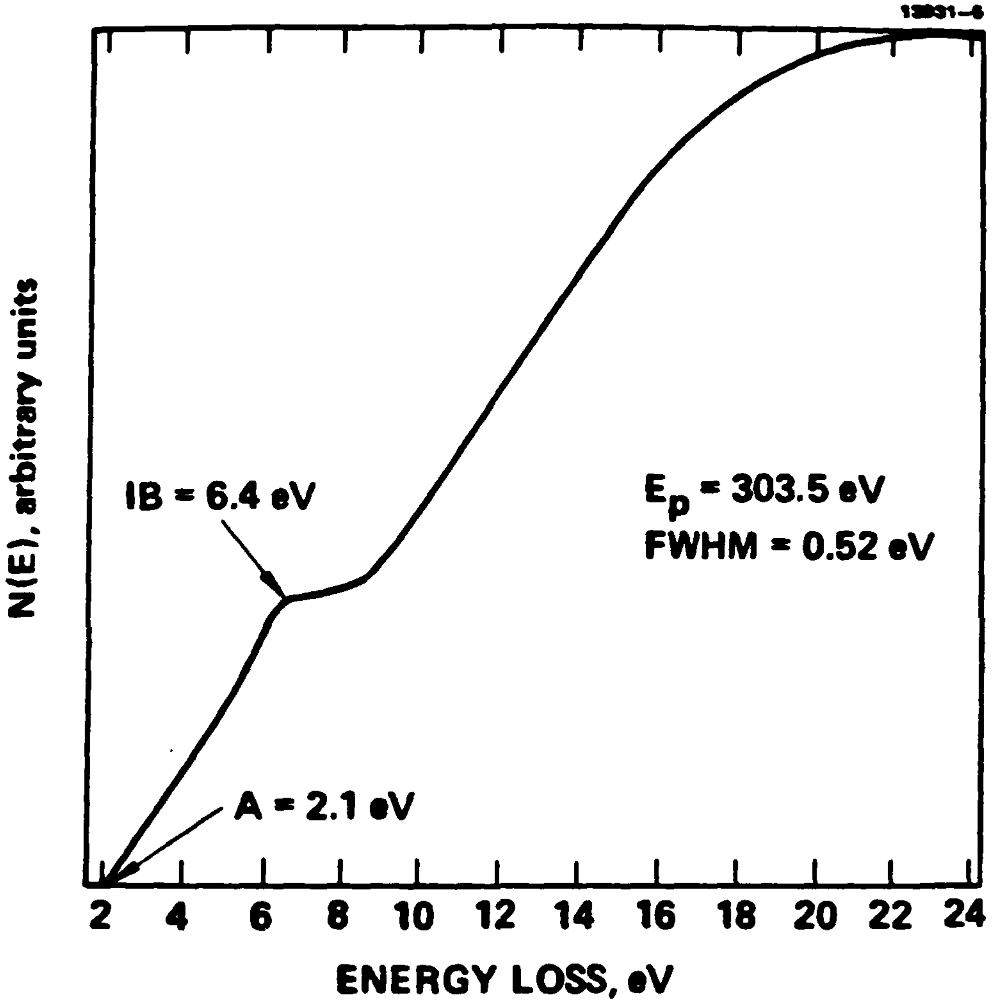


Figure 7. ELS spectrum from air-oxidized InGaAs.

SECTION 3

SUMMARY

From detailed analysis of the x-ray photoelectron spectroscopy (XPS) and electron energy loss spectroscopy (ELS) studies on $In_{0.53}Ga_{0.47}As$ epitaxial layers, we conclude that (1) the native oxide in the samples analyzed is ~ 15 Å thick; (2) the oxidation state of As is consistent with As_2O_3, while the chemical identities of In and Ga oxides are not clearly defined; (3) the near surface region of $In_{0.53}Ga_{0.47}As$ is Ga rich and As deficient; (4) the ELS spectrum on $In_{0.53}Ga_{0.47}As$ layers is dominated by the oxidized indium.

During the next quarter we will perform more detailed experiments to characterize the semiconductor-dielectric interface of MIS structure on $In_{0.53}Ga_{0.47}As$ and correlate them with capacitance measurements on such MIS capacitors.

REFERENCES

1. D.T. Clark, T. Fox, G.G. Roberts, and R.W. Sykes, Thin Solid Films 70, 261 (1980).

2. P.A. Bertrand, J. Vac, Sci. Tech. 18, 28 (1981).