

## **XPS STUDY OF pHEMT STRUCTURE CLEANED USING HCl, HF AND BOE ETCHANTS**

Hesly Afida Hashim<sup>a</sup>, Asban Dolah<sup>a</sup>, Mohamad Deraman<sup>b</sup>, Hariyadi Soetedjo<sup>a</sup>,  
Mohamed Razman Yahya<sup>a</sup> and Abdul Fatah Awang Mat<sup>a</sup>

<sup>a</sup>*MENT, TM Research & Development Sdn. Bhd., Idea Tower I & II,  
UPM-MTDC Lebuh Silikon, 43400 Serdang, Selangor.*

<sup>b</sup>*School of Applied Physics, Faculty of Science and Technology,  
Universiti Kebangsaan Malaysia, 43600 Bangi, Selangor.*

### **ABSTRACT**

Unetched and HCl, HF, BOE solutions (acid to water = 1:10 and 1:20) etched surface of GaAs top layer of GaAs based pHEMT samples were analyzed by XPS technique. It was found that the unetched sample consists of about 9-10% oxide (Ga-oxides and/or As-oxides) species whereas other etched samples consist of 5-8% oxide content. The optimum effect of removing oxide layer was shown by HCl: H<sub>2</sub>O (1:10) treatment, where about 40% of oxides content reduction was achieved. At this concentration the XPS spectra of this sample also shows that (a) oxide contribution to O 1s peaks only come from As elements, (b) the treated surface is an As-rich surface. This is consistent with the decreasing value of Ga/As ratio before and after HCl treatment.

### **INTRODUCTION**

X-Ray Photoelectron Spectroscopy (XPS) is one of the important techniques for the study of semiconductor materials because it can measure both the composition and the electronic structure of materials, which are present on the sample surface. The surface sensitivity of XPS, which is typically 40-100 Å, enables the technique to measure oxidation states and oxide layer thickness on III-V compounds and other semiconductor materials. The XPS spectra can be obtained by allowing massive amounts of incident photons on the sample to produce a large number of photoelectrons. The difference between the kinetic energy and the initial energy of the photons is related to the binding energy of the materials and the work function of the analyzer. Comparing the spectra of photoelectrons intensity versus binding energy of an unknown sample to those of other known standards is a routine step for the analysis of materials at the surface of the semiconductor materials.

GaAs is perhaps one of the most popular compound semiconductor materials and observation by XPS technique found that oxides on the surface of GaAs exhibit various types of chemicals and structural configurations [1, 2, 3, 4]. The present work reports the XPS surface characterization of chemically etched and unetched pHEMT structure samples. These samples were made of several epitaxial layers, i.e.: GaAs as top layer, AlGaAs as supply, InGaAs as channel, AlGaAs and GaAs as the buffer layers and

finally GaAs as the substrate.

The XPS analysis was carried out to obtain the spectra covering peaks within the binding energy ranges from 0 to 1200 eV and C KLL peak was used as a reference line for the charging effect. The XPS spectra were recorded before and after HCl, HF and BOE wet etching. The emphasis of this study was on the variation of Ga/As and Ga- and As-oxides contents at the surface of the samples against the chemical treatment conditions.

## EXPERIMENTS

The top layer of pHEMT samples was etched with HCl, HF and BOE solutions, which were prepared having the concentration ratio of acid to water at 1:10 and 1:20. The etching time was selected for 60 s for each samples and successfully rinsed for 30 s in deionized water and blow-dried by N<sub>2</sub> gas. The XPS analysis was performed on these samples using a PHI Model 5600 Multi Techniques equipped with X-Rays emitted by an anti-cathode of Al at energy ( $K_{\alpha} = 1486.6$  eV). The analysis chamber was maintained at ultra high vacuum (pressure,  $p = 3 \times 10^{-9}$  Torr) and the samples position were oriented at  $\theta = 70^{\circ}$  in respect to the analyzer.

## RESULTS AND DISCUSSIONS

The recorded XPS wide scan spectra of the investigated samples were found to consist of the following XPS peaks namely Ga 2p<sup>3/2</sup>, Ga 3p, As 3s, As 3p, C KLL, C 1s, As 3d, O KLL and O 1s. The value of specific binding energy for those elements and their compounds can be found elsewhere [5] and these values were used for the interpretation of our XPS data. Figure 1 shows a typical XPS spectra from one of the sample-investigated, sample B1 (HCl: H<sub>2</sub>O= 1:10). Our study only focuses on the analysis of the O 1s, As 3d and Ga 2p<sup>3/2</sup> peaks. The mass concentrations of the species estimated from these three peaks are shown as a histogram in Figure 2. The patterns of this histogram clearly show that the surface stoichiometry of the samples vary with the treatment conditions, i.e. acid concentration, types of acid used and cleaning time.

Further analysis of this histogram shows the ratios of O1s-peaks between the treated samples (B to D) and untreated sample (A) in Table 1. These ratios clearly indicate that HCl and BOE can remove a significant amount of oxide on the samples surface. A similar significant change of oxide contents were observed on the GaAs samples etched with HCl solution (36 %) and 10 s rinsed with water [4], and on the Ti/GaAs interface produced after chemically etched (HCl: H<sub>2</sub>O =1:1 for 30 s) and rinsed (water 60 s) [2]. A contrary behavior is however exhibited by the ratio for the C1 and C2 samples, where HF etched causes the oxide content to increase by 7 to 8 %. The ratios for the samples B seem to indicate the existence of optimum etching time (1 min); 10 min could be much longer than necessary.

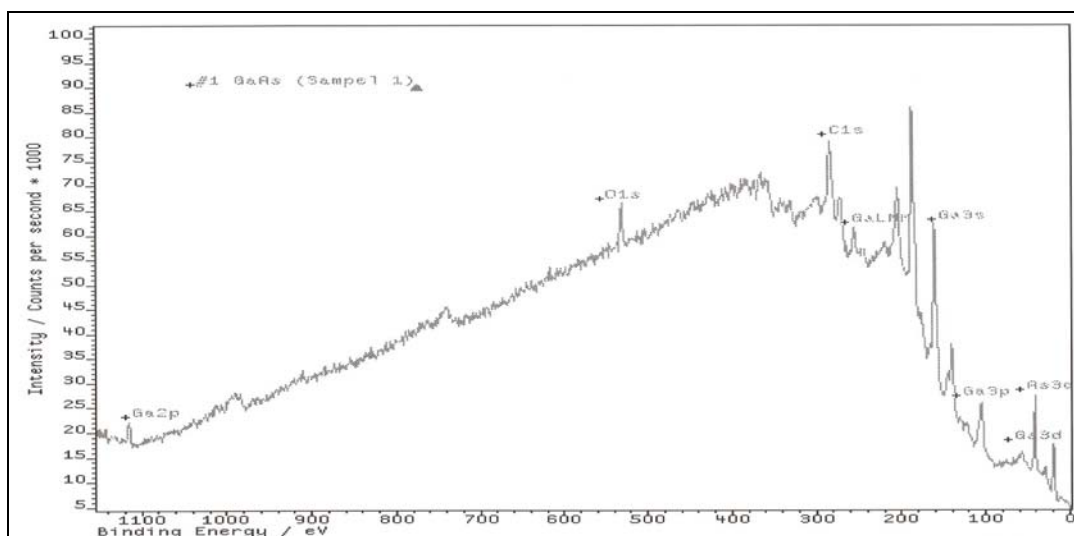


Figure 1: XPS Wide Scan Spectra of Sample B1 (HCl: H<sub>2</sub>O=1:10)

Table 1: Ratio of Elemental and Oxides Contents

Sample	Treatment Condition	$[O\ 1s]_{\text{sample}} / [O\ 1s]_A$	$[Ga\ 2p^{3/2}] / [As\ 3d]$
A	Untreated	1.00	1.51
B1	HCl: H <sub>2</sub> O=1:10, 1 min	0.565	1.28
B2	HCl: H <sub>2</sub> O= 1:10, 10 min	0.693	0.70
B3	HCl: H <sub>2</sub> O= 1:20, 1 min	0.715	1.11
C1	HF: H <sub>2</sub> O= 1:10, 1 min	1.080	0.72
C2	HF: H <sub>2</sub> O= 1:20, 1 min	1.070	0.61
D1	BOE: H <sub>2</sub> O= 1:10, 1 min	0.698	1.26
D2	BOE: H <sub>2</sub> O= 1:20, 1 min	0.891	0.50

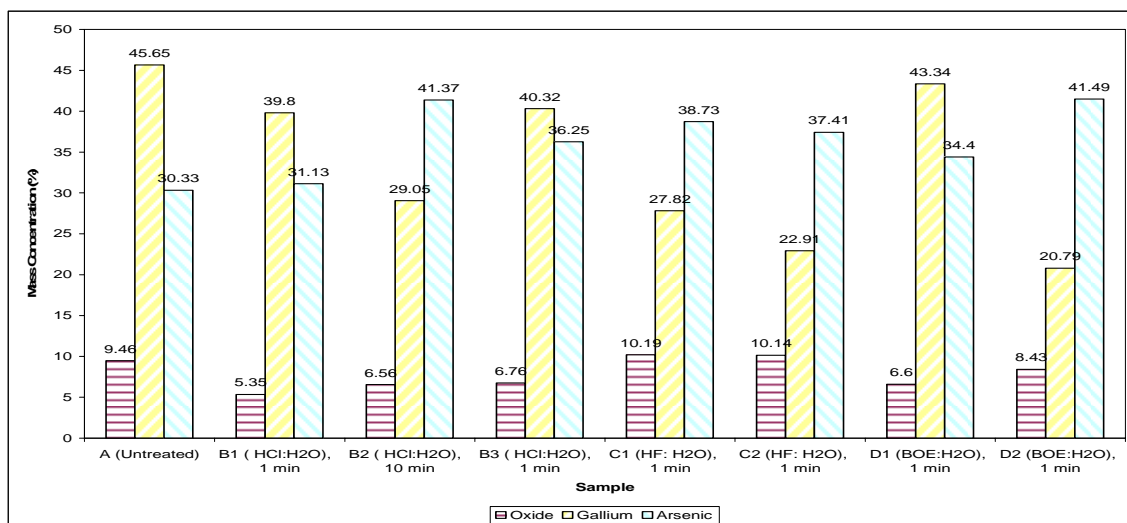
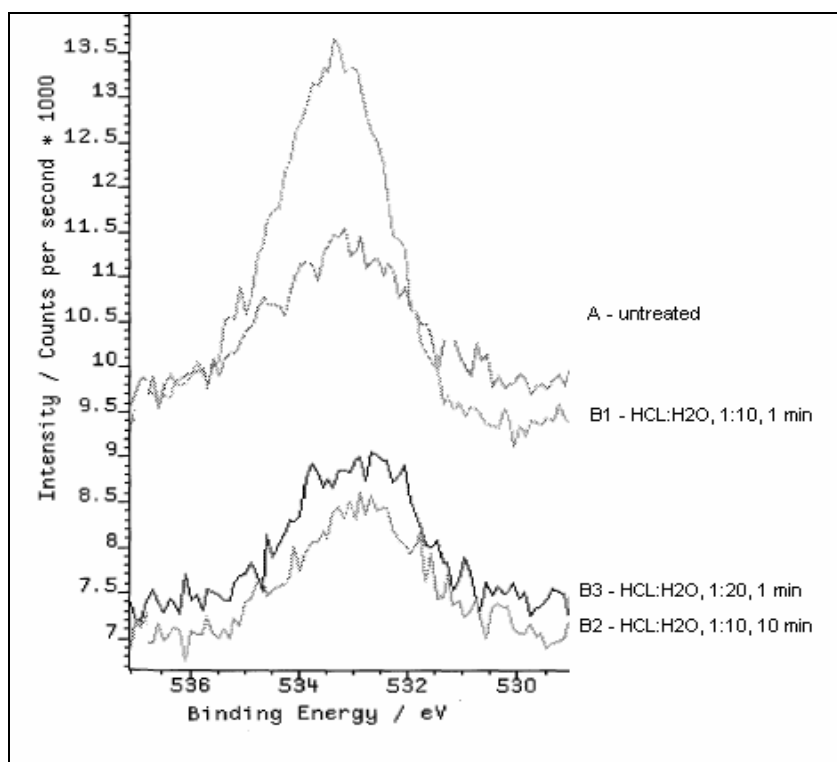
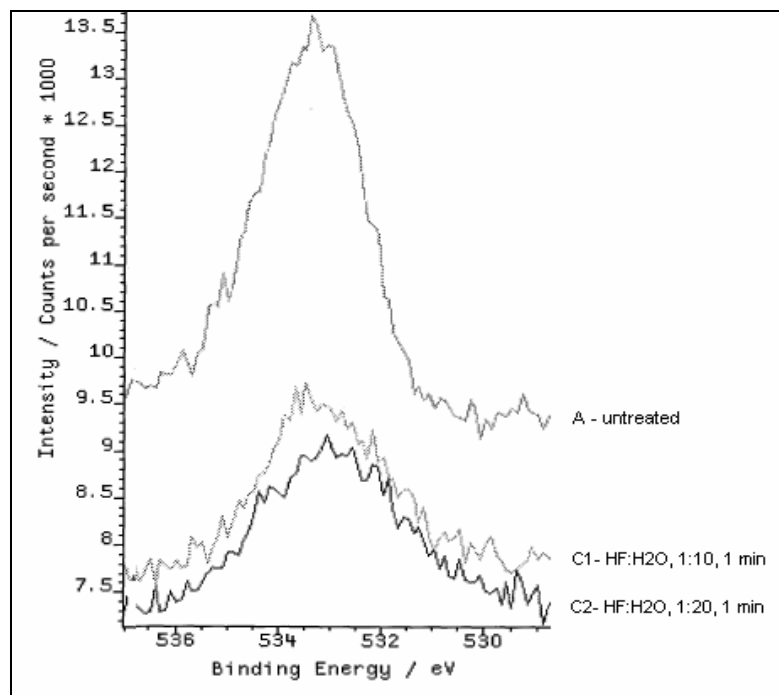


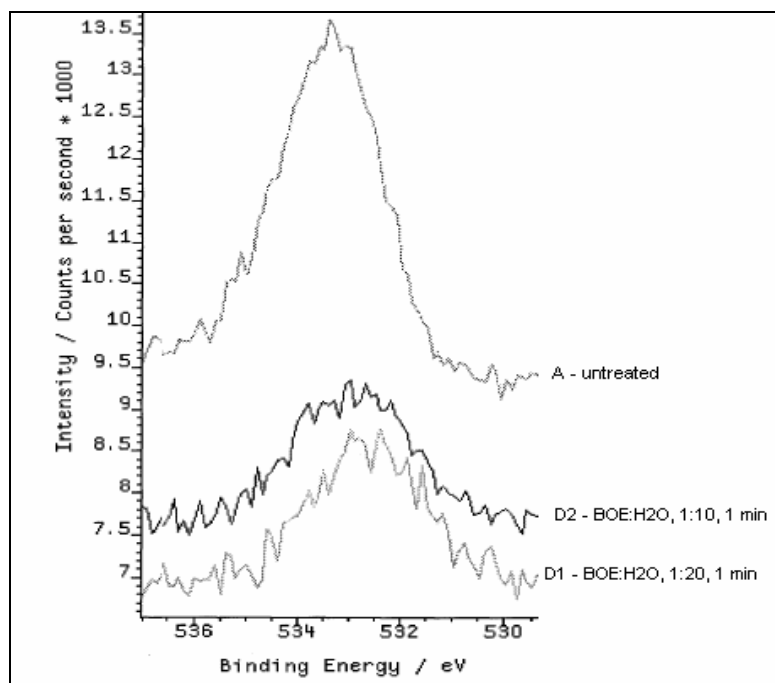
Figure 2: The Histogram of Mass Concentration (%) of Chemical Species



(a): XPS Spectra of O 1s from HCl etching



(b): XPS Spectra of O 1s from HF etching



(c): XPS Spectra of O 1s from BOE etching

Figure 3: XPS Spectra of O1s fro the untreated and treated samples.

Figure 3 is the XPS spectra O1s line for the untreated and treated samples, which show the variation of peak shape and intensity representing the presence/absence and concentration quantity of the gallium oxide and arsenic oxides on the samples surface. Detailed analysis of these peaks found that some of these curves are made up of both or one of the  $\text{As}_2\text{O}_3/\text{As}_2\text{O}_5$  peak and  $\text{Ga}_2\text{O}_3$  peak;  $\text{Ga}_2\text{O}_3$  peak (A and D1); both peaks (B2 and C2) and  $\text{As}_2\text{O}_3/\text{As}_2\text{O}_5$  peaks (B1, B2, C1 and D2). As for the sample A, the presence of only  $\text{Ga}_2\text{O}_3$  peak is attributed to the well known behavior that Ga is very less stable than the As in term of the tendency to form oxide. This is in contrary to the results of Song et. al (1994), which consist of a mixture of both  $\text{Ga}_2\text{O}_3$  and  $\text{As}_2\text{O}_3$  species. As for the other samples, the overall trend of the data clearly indicates that due to certain treatment conditions, the disappearances of  $\text{Ga}_2\text{O}_3$  species on the sample surface that followed by the formation of  $\text{As}_2\text{O}_3/\text{As}_2\text{O}_5$  species. The function of HCl/ $\text{H}_2\text{O}$  etching in producing the GaAs surface As-rich can be explained by the following etching mechanism [4]. When the GaAs surface is immersed in the HCl solution, the thick over layer of oxides reacts with HCl and most of the oxides dissolve into the solution, leaving a mixture of gallium chlorides and oxichloride on the surface. When these etched samples are sequently dipped in water for 30 seconds, the surface species (gallium chlorides and oxichlorides) dissolves leaving an As-rich surface. The data in Table 1 (column 4), i.e. the ratio of  $[\text{Ga } 2p^{3/2}]/[\text{As } 3d]$  which is in decreasing trend for the treated samples are consistent with the fact that after treatment the surface of the samples tend to become As-rich surface.

## CONCLUSIONS

A significant reduction of about 40% of oxide layer at GaAs top surface can be achieved by acid HCl and BOE treatment. The effect of HF treatment in removing the oxide layer is not significant. The ratio of 1:20 for BOE:  $\text{H}_2\text{O}$  is too diluted to give a significant reaction to oxide layer. This study suggests that the treatment with (HCl: $\text{H}_2\text{O}$  of 1:10, 60 s immersion; and water, 30 s rinse) gives the most effective reduction of oxide layer from our substrate samples, where it can reduce the surface oxide content from about 9.5% to about 5.4%.

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