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IN-SITU STUDY OF GaAs OXIDATION

M.E. Hall
(M.S. Thesis)

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IN-SITU STUDY OF GaAs OXIDATION

by

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INTRODUCTION

One of the newer semiconductor materials is gallium arsenide (GaAs). The electronics industry has high hopes for GaAs since:¹ 1) its energy band structure allows electrons to move through it five times faster than Si; 2) its switching speed is ten times faster than Si; 3) GaAs has lower power dissipation; 4) it can withstand up to 10^8 rads of radiation; and 5) it has a maximum operating temperature of up to 300°C . The electronics industry currently uses it in LED's, microwave amplifiers, and solid state lenses; they would like to expand its use to solar cells, and integrated circuits.

There are problems with GaAs, however. For example, no one has developed a way to grow a good insulating oxide on GaAs. The electronics industry defines a good oxide as one which: 1) has high insulating properties; 2) is chemically inert; 3) has no lattice mismatch between the substrate and the oxide; 4) is chemically homogeneous in all directions; and 5) is cheap and easy to produce. Current GaAs devices use deposited layers of SiN and/or SiO_2 for their passivating layer. The deposited layers can cause coupling and leakage problems in device manufacture.

This study uses a transmission electron microscope (TEM) equipped with an environmental cell (E-cell) to study the growth of thermally produced native oxides on GaAs. Since an E-cell is used, the actual growth of the oxide through time can be observed.

Crystallographic and compositional changes can quantitatively be studied using an E-cell.² In-situ tests can also provide qualitative data on the specimen's microstructure.

In-situ TEM experiments can provide a means to study fundamental mechanisms of gas-solid reactions. However, there are some general guidelines that must be followed for accurate results. The phenomena being studied must occur on a scale less than the foil thickness and at a number of random sites. Oxidation and reduction reactions do not seem to be affected by the specimen's thickness.³ The electron beam should not be left in any one area for extended periods. The electron beam can inhibit or initiate reactions, and also cause radiation damage to the specimen.

There are some disadvantages to using an E-cell. First, a thorough defect analysis cannot be done since the specimen tilt is limited by the E-cell design. Second, resolution is reduced due to the presence of an atmosphere.

Research has shown that GaAs oxidizes similarly to a binary alloy.⁴ It differs from oxidation in a binary alloy in that: 1) the excess solubility of As in Ga_2O_3 is limited; 2) Ga and As have low self diffusion rates; and 3) there is a high binding energy between Ga and As. The oxidation growth follows a parabolic rate law in the temperature range of 400°C to 450°C .^{5,6} The rate equation is:

$$d^2 = A_p \exp\left(-\frac{\Delta E_p}{kT}\right)t$$

where d \equiv thickness of the oxide layer,
 A_p \equiv constant,
 ΔE_p \equiv activation energy dependent on the atmosphere,
 k \equiv Boltzmann constant,
 T \equiv temperature, and
 t = time of exposure to atmosphere.

Wilmsen has found the parabolic rate constant for GaAs oxidation to be:⁷

$$K_{AB} = K_A \exp(\Delta F_{AB}/4RT) \quad ;$$

where K_{AB} \equiv rate constant for formation of Ga_2O_3 ,
 K_A \equiv constant, and
 ΔF_{AB} \equiv free energy of formation of GaAs.

A linear rate law applies for the temperature range of 480°C to 530°C.^{8,9} The linear rate equation is:

$$d_L = A_L \exp\left(-\frac{\Delta E_L}{kT}\right) \quad ;$$

where d_L \equiv thickness of the oxide layer,
 A_L \equiv constant, and
 ΔE_L \equiv activation energy dependent on the atmosphere.

No information was found in the literature on why the formation laws are reversed.

The only published TEM study done on the oxides of GaAs was done in the mid-1970's by Bull and Sealy.¹⁰ They performed an ex-situ study on the oxide morphology. Pure GaAs with a (110) face was oxidized in dry oxygen between 450°C and 750°C for .5 to 6 hours. An unidentified amorphous oxide was produced at temperatures below 500°C. An amorphous oxide layer approximately 300 Å thick was grown at 450°C. At temperatures above 500°C, crystalline β -Ga₂O₃ was produced. The orientation of the monoclinic β -Ga₂O₃ was: $\langle 002 \rangle_{\beta\text{-Ga}_2\text{O}_3} || \langle 002 \rangle_{\text{GaAs}}$ and $\langle 10\bar{2} \rangle_{\beta\text{-Ga}_2\text{O}_3} || \langle 1\bar{1}1 \rangle_{\text{GaAs}}$.

Others have studied GaAs and its oxides using AES, SIMS, and XPS. Grunthaner, et al.¹¹ found that the stability of GaAs oxides were dependent on the temperature. They found that thermal oxidation of GaAs produced: Ga₂O₃, As₂O₃, GaAsO₂, GaAsO₃, and GaAsO₄. The Ga in the substrate was found to maintain a +3 charge. Kazmerski, et al.¹² found that dry oxygen at atmospheric pressure and 200°C produced a polycrystalline layer of Ga₂O₃. Wet oxidation at 26°C produced a layer of Ga₂O₃ and As₂O₅. The As₂O₅ concentration varied throughout the oxide layer. Schwartz, et al.¹³ examined the oxidation of GaAs with air, As₂O₃/O₂ mixtures, and O₂. They found that As₂O₃/O₂ gas mixtures produced an amorphous oxide at temperatures below 500°C; above 600°C, a crystalline oxide was produced. Air and oxygen were found to produce a layer of Ga₂O₃ on the substrate. Schwartz and his group also proved that As₂O₃ and As₂O₅ volatilized when dry oxygen was the oxidizer. Watanabe, et al.¹⁴ thermally oxidized GaAs with dry air between 500°C and 720°C. They found that Ga was preferentially

oxidized over As. An enriched As zone was formed in the substrate near the interface. Their results also show that Ga_2O_3 was the only solid oxide produced in thermal oxidation. The oxidation of GaAs with H_2O was studied by Webb and Lichtensteiger.¹⁵ XPS data shows that Ga-OH compounds can form in the first stages of oxidation.

The purpose of these experiments is to observe native oxide formation on GaAs. Since it is done in-situ, the oxides produced will not be affected by oxygen or water vapor in the air. Nor, will the oxides' morphology be altered by specimen preparation techniques. Arsenic oxides are water soluble; Bull and Sealy note this and use it as a possible explanation for why arsenic oxides are not detected in their study. Also, an in-situ study allows any intermediate phases and/or compounds to be detected.

EXPERIMENTAL

All microscopy was done on a Hitachi 650 kV transmission electron microscope equipped with a Gatan environmental cell (E-cell) and hot stage. Operating voltages were either 650 kV or 500 kV. The microscope's smallest intermediate aperture is .5 microns. Without the E-cell, the microscope has a resolution of 5\AA . The resolution of the microscope with the E-cell in depends on the gas' density, the gas pressure, and flow rate.

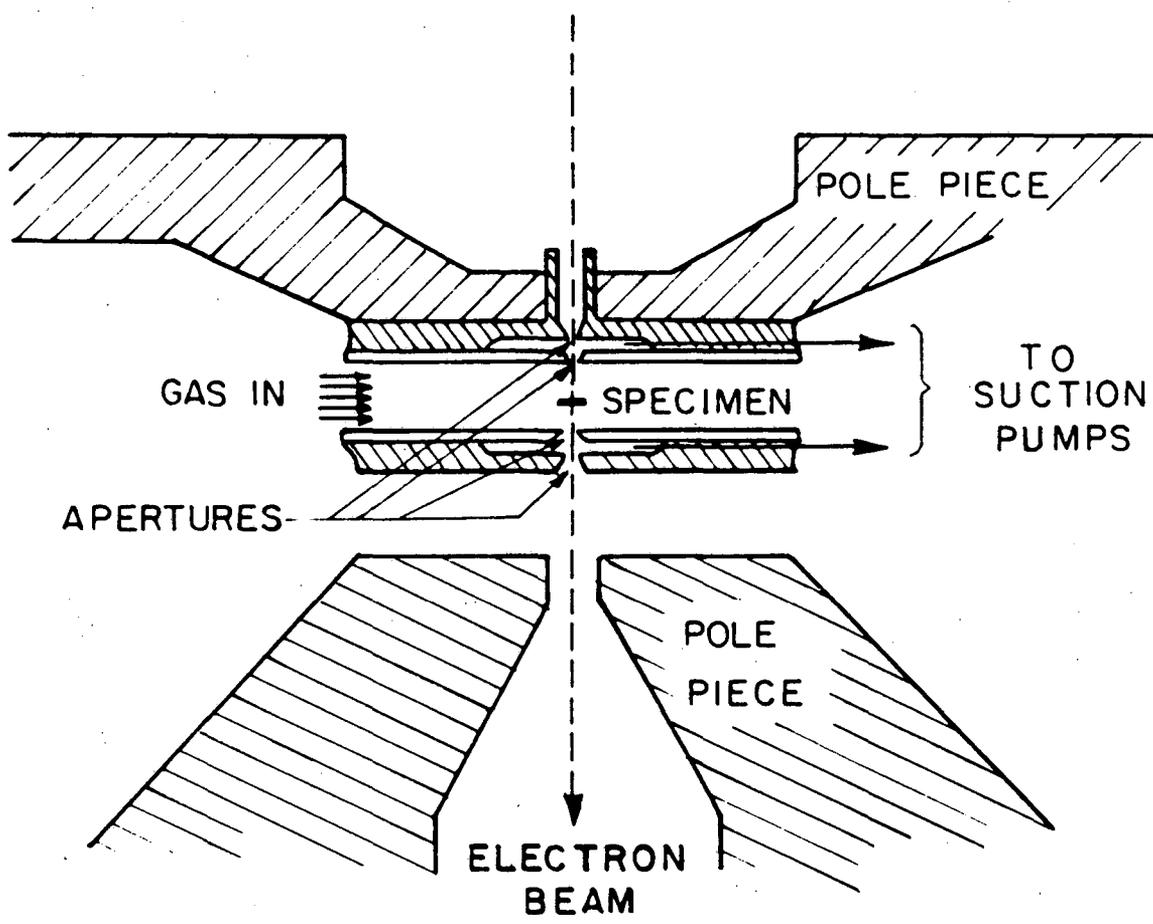
Figure 1 is a schematic diagram of the side loading Gatan E-cell used in these experiments. It sits between the pole piece of the microscope and allows the specimen to be exposed to an atmosphere up to 100 torr in pressure. The body of the E-cell is 41 mm in diameter and 12 mm deep. The gas reservoir can hold approximately 50 ml gas. Gas pressure in the E-cell is regulated by a needle valve. To prevent filament damage, all experiments were done with 50 torr of pressure or less. The gas flow rate ranged from 25 cm/min to 48 cm/min.

The gas is removed from the E-cell by differential pumping. The apertures of the E-cell are 100 μm in diameter.

A side loading E-cell allows the specimen to be tilted about one axis. A top loading E-cell would not allow the specimen to be tilted. Side loading E-cells are susceptible to vibration and thermal drift perpendicular to the beam.¹⁶

The GaAs specimens came from a single GaAs wafer supplied by Hewlett-Packard. The wafer came from an ingot grown by the Czochralski method. The concentration of the Si, acting as an n-type

FIGURE 1
Cross Section of E-cell in Microscope



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dopant, was $5 \times 10^{17} \text{ cm}^{-3}$. The wafer was oriented close to a (100) face that was 4° toward the (110). The specimens were cut from the wafer, mounted in wax and thinned by polishing them on a series of emery papers. The specimen's surface was brought to a shiny finish by using .6 and .4 micron diamond paste. By using three acetone baths, the specimens were removed from the wax. To further thin and clean the specimen, it was soaked in a bath of 1% Br-methanol for 15 minutes. The specimens were then mounted on 3 mm grids and ion milled. Ion milling was done for 7 hrs. at $6\frac{1}{2}$ kv at an angle of 20° to 30° . To prevent contamination, the milled specimens were loaded into the microscope as rapidly as possible.

The chemical reactivity of the specimen is increased due to ion milling. No studies were done to see if all the polishing damage was removed from the specimen's surface.

The oxygen atmosphere was produced from a tank of pure oxygen. The oxygen was not dried. Mass spectrograph data indicates that the oxygen atmosphere contained 2% water vapor. The atmospheres containing water vapor were created by passing pure Ar through a bubbler filled with distilled, de-ionized water at 21°C .

RESULTS AND DISCUSSION

Listed in Table 1 are the average conditions under which GaAs was oxidized. Temperature was the main variable; the oxygen pressure was kept as constant as possible.

Figure 3 shows the results of oxidizing GaAs at 200°C and 50 torr of O₂ for 5 hours. Small particles, 20 nm in size, are visible at the specimen's edges. Due to the size of the particles and their low density, they do not generate any features in the diffraction pattern. The dots occupying the [200] position in Figure 3b are double diffraction dots. They disappear when the specimen is tilted a few degrees.

No visible change was observed when GaAs was oxidized at 280°C and 40 torr of O₂. The specimen was oxidized for 2½ hours. Figure 4 shows the specimen before and after oxidation.

An amorphous layer was grown on the GaAs specimen oxidized at 320°C and 44 torr of O₂ for 1½ hours. There were no visible changes to the specimen's surface. As seen in Figure 5, the changes show up in the diffraction pattern of the material. A dark field image taken from the 2nd band shows that the layer covers the surface. The average d-spacing for the amorphous bands is: $d_1 = 2.00 \text{ \AA}$ and $d_2 = 1.46 \text{ \AA}$. There is not enough data to positively identify the oxide.

Polycrystalline $\beta\text{-Ga}_2\text{O}_3$ is produced at 450°C. This process is shown in Figure 6. Figure 6a is the starting material and its diffraction pattern. After 15 minutes of exposure to O₂ at 450°C, the specimen has an irregular surface. The diffraction pattern contains

TABLE 1
EXPERIMENTAL CONDITIONS

<u>Temperature</u>	<u>Oxidizing Agent</u>	<u>Average Pressure</u>	<u>Total Exposure Time</u>	<u>Product</u>
(°C)		(Torr)	(hours)	
200	O ₂	50	5.00	No reaction
280	O ₂	40	2.25	No reaction
320	O ₂	44	1.50	Amorphous oxide
450	O ₂	46	2.00	β-Ga ₂ O ₃
550	O ₂	47	2.00	γ-Ga ₂ O ₃
450	H ₂ O	46	2.00	γ or β-Ga ₂ O ₃

Arsenic and its oxides are volatile above 312°C. A plot of vapor pressure versus temperature is in Figure 2.

FIGURE TWO

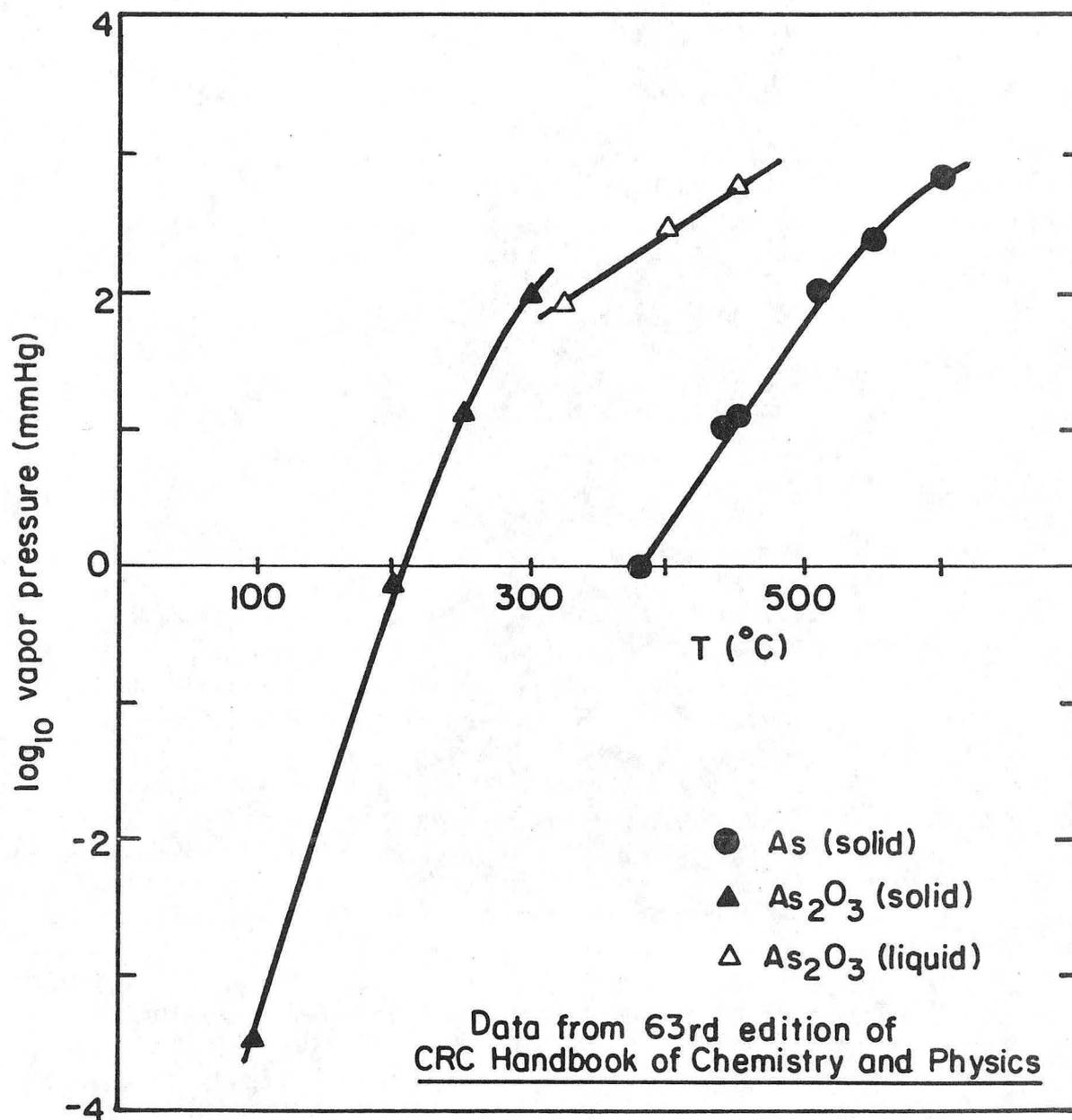
 \log_{10} Vapor Pressure versus Temperature for As and As_2O_3 

FIGURE 3

Oxidation of GaAs at 200°C

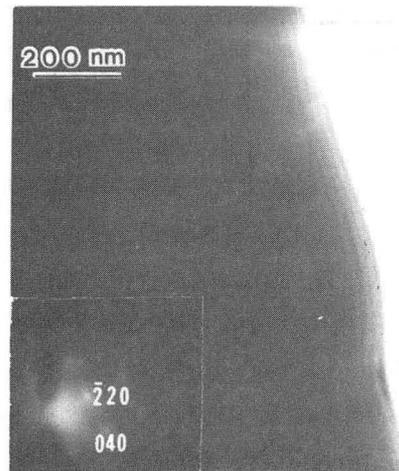


Figure 3a

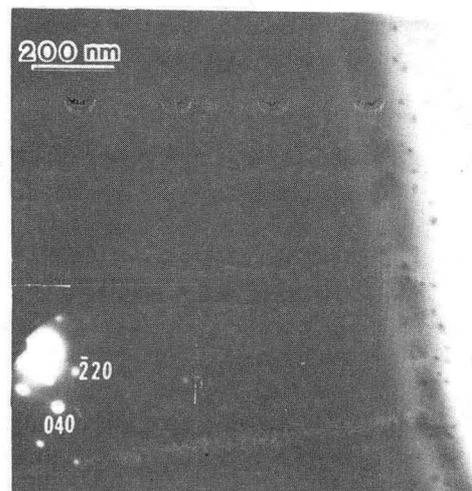


Figure 3b

In this figure are the results of oxidizing GaAs at 200°C and 50 torr O_2 . The starting material and its diffraction pattern are shown in Figure 3a. The diffraction pattern is characteristic of the [100] orientation in diamond cubic materials. The specimen, after 5 hours of oxidation is in Figure 3b. The extra spots in the [200] position of the diffraction pattern arise from double diffraction

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FIGURE 4
Oxidation of GaAs at 280°C



Figure 4a

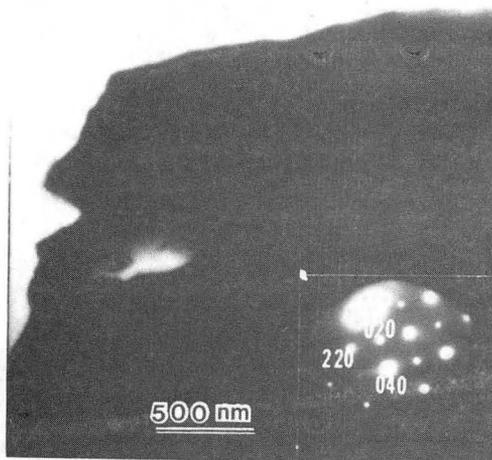


Figure 4b

Figure 4a is the GaAs before oxidation. Figure 4b is the same area after 2½ hours at 280°C and 40 torr O₂. The specimen is much darker, but analysis of the diffraction pattern yielded no new compounds. The dots in the [200] position of the diffraction pattern were caused by double diffraction.

XBB 854-3402

FIGURE 5
Oxidation at 320°C

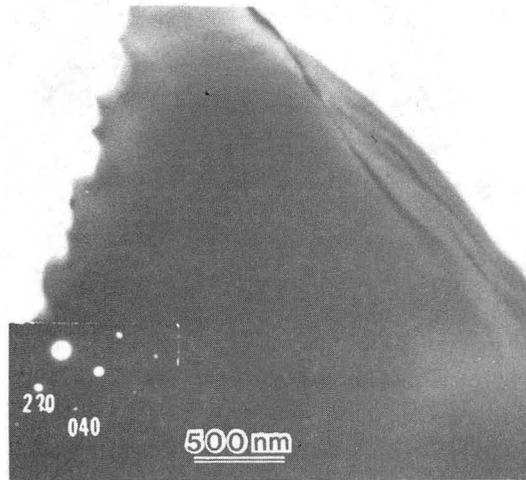


Figure 5a

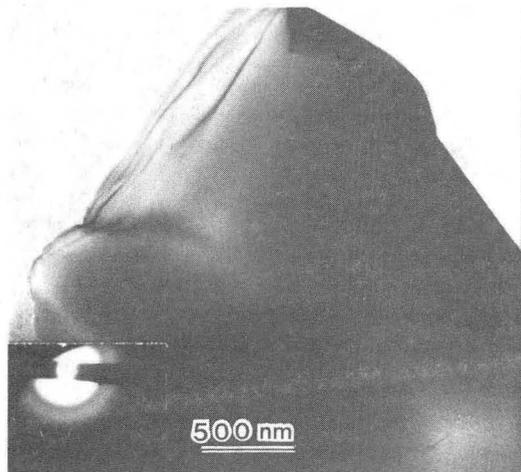


Figure 5b

The starting material is shown in Figure 5a. After oxidizing for 1 hour at 320°C and 44 torr O_2 , an amorphous film has formed. This is shown in Figure 5b. Figure 5c is the dark field from the 2nd band. Figure 5d is the specimen after 1½ hours. The thickness of the oxide layer could not be determined.

XBB 854-3403

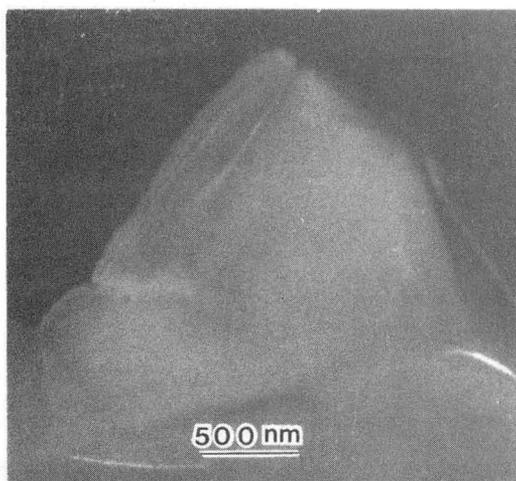


Figure 5c

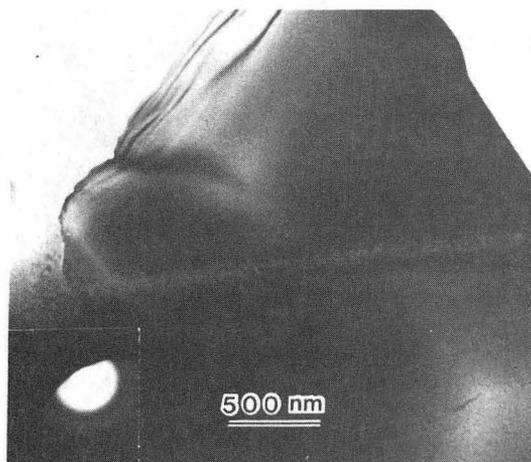


Figure 5d

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FIGURE 6
Oxidation at 450°C

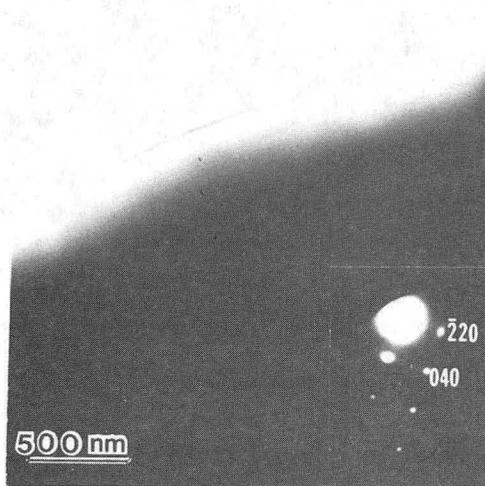


Figure 6a

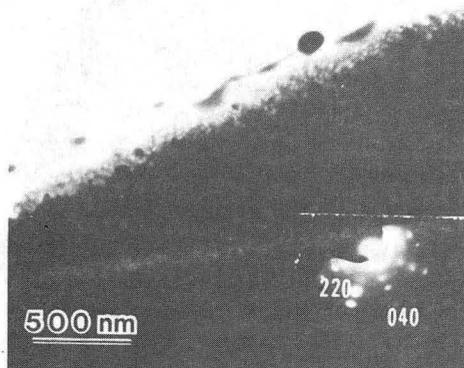


Figure 6b

Figure 6a is the starting GaAs area. After 15 minutes of exposure to 450°C and 46 torr O_2 , a polycrystalline oxide layer is formed. This is shown in Figure 6b. Figure 6c shows the material after $1\frac{1}{4}$ hours.

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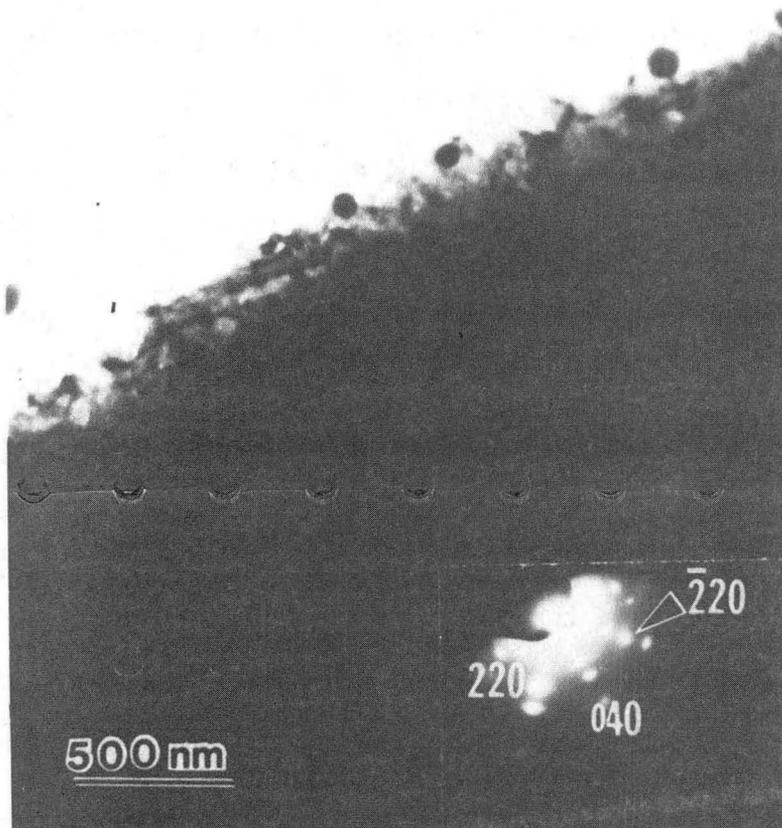


Figure 6c

XBB 854-3406

both rings and extra dots (Figure 6b). Measurements for the diffraction pattern are contained in Table 2. The data best corresponds to β -Ga₂O₃. The oxide is polycrystalline since none of the spots and their angles correspond to each other.

As the reaction proceeds, the oxide layer becomes more polycrystalline and thicker. Figure 6c shows the area after being oxidized for 1½ hours. The specimen's surface is much more irregular and the diffraction pattern is fogged.

Dark field studies indicate that the oxide grows from the edge to the inside. Figure 7a shows an oxidized region and its diffraction pattern. Figure 7b is a dark field photograph from a GaAs matrix spot; a dark field photograph from an oxide spot is in Figure 7c. The oxide is at the specimen's edge, while the GaAs is further in. More evidence for this is shown in the series of photographs in Figure 8.

Polycrystalline γ -Ga₂O₃ was produced at 550°C and 44 torr of O₂. Figure 9 shows the starting material and its structure after 8 minutes. The d-spacings for the oxide are listed in Table 3; they correspond very closely to γ -Ga₂O₃. Also listed in Table 3 are the d-spacings for the area pictured in Figure 10. Figure 10 is another region at higher magnification containing polycrystalline γ -Ga₂O₃.

GaAs was also oxidized with water. Distilled water at 21.5°C was used; argon was the carrier gas. The total pressure into the E-cell was 46 torr. As shown in Figure 11, a polycrystalline oxide was produced. It formed in the same amount of time as when oxygen was used. d-spacings (Table 4) obtained from the diffraction patterns

TABLE 2
Interplanar Spacings for Figure 6b

<u>d, Å</u>	<u>ASTMS Powder Pattern Index Values for β-Ga₂O₃, Å</u>
2.82	2.816
2.11	2.103
1.37	1.353

FIGURE 7
Oxide Growth Characteristics

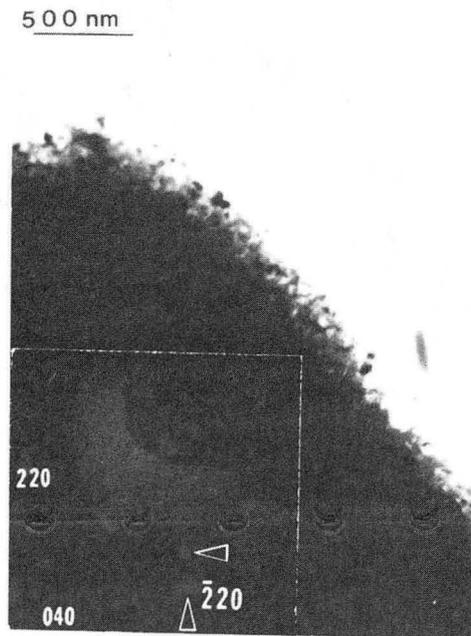


Figure 7a

The bright field image and its diffraction pattern is shown in Figure 7a. The dark field image from a GaAs spot is shown in Figure 7b. The β -Ga₂O₃ is the bright areas of Figure 7c; that dark field image was generated from a Ga₂O₃ spot. The diffraction spots used are indicated by arrows.

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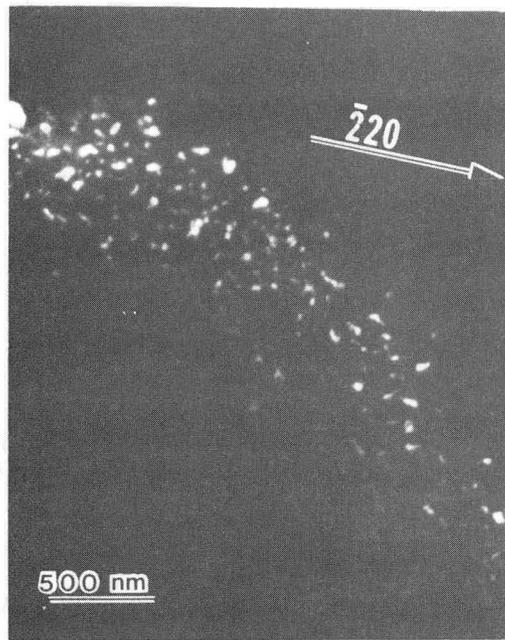


Figure 7b

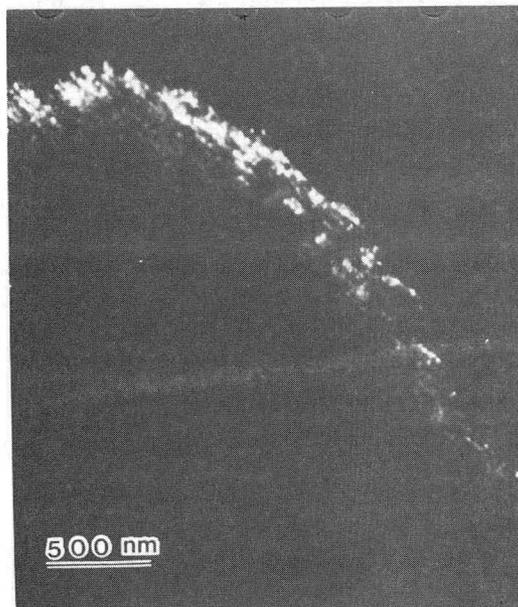


Figure 7c

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FIGURE 8
Oxide Growth Characteristics

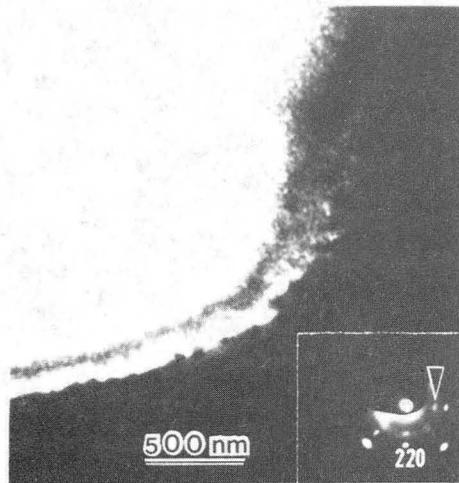


Figure 8a

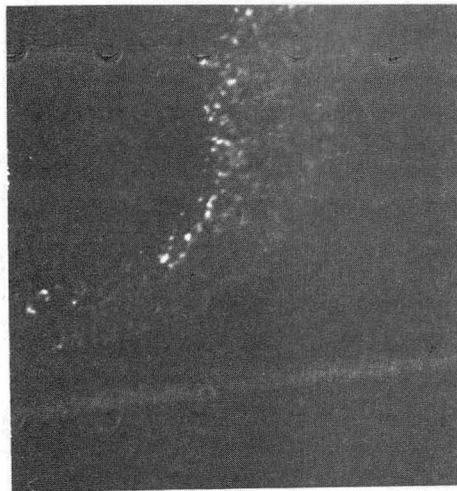


Figure 8b

Figure 8a shows an area of GaAs that has been oxidized for $1\frac{1}{4}$ hours at 450°C and 46 torr. The dark field image in Figure 8b is from a $\beta\text{-Ga}_2\text{O}_3$ spot indicated by the arrow.

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FIGURE 9

Oxidation at 550°C

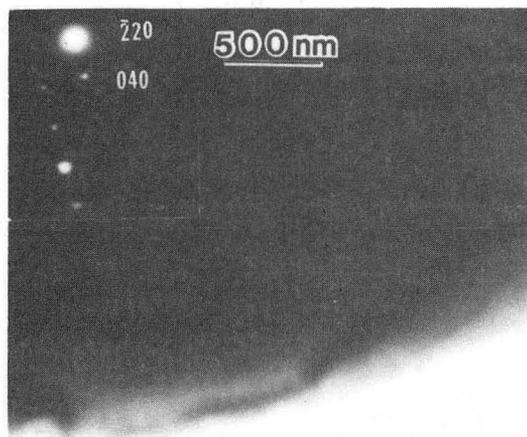


Figure 9a

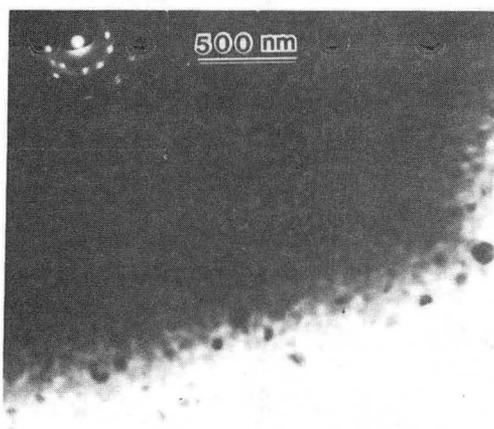


Figure 9b

Figure 9a is the starting region of GaAs and its diffraction pattern at 20,000X. After being exposed to 44 torr O₂ at 550°C, a layer of γ -Ga₂O₃ forms on its surface.

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TABLE 3

Interplanar Spacings for 550°C Specimens

Spacings for Figure 9 (Å)	Spacings for Figure 10 (Å)	ASTMS Powder Pattern Index Values for γ -Ga ₂ O ₃ (Å)
	2.96	2.90
2.51	2.44	2.48
2.07	2.00	2.05
		1.68
		1.58
1.41	1.46	1.45
		1.25

FIGURE 10
Oxidation at 550°C



Figure 10 is a photo of an area of GaAs oxidized for 10 minutes at 550°C and 50 torr O₂.

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FIGURE 11
Oxidation at 450° with H₂O

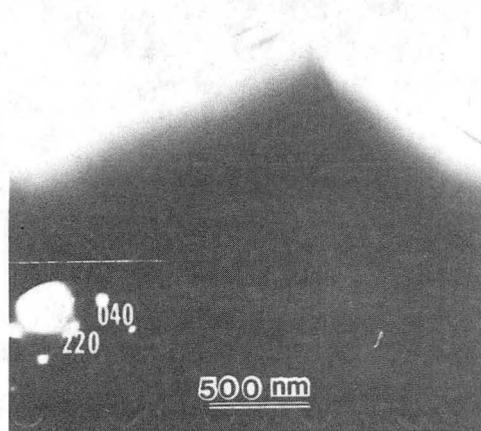


Figure 11a

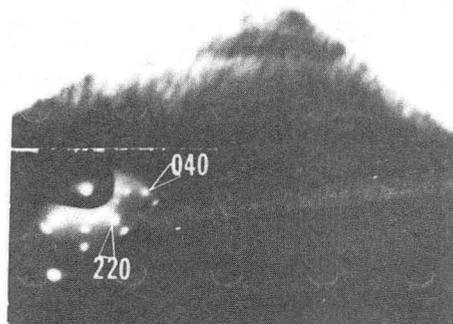


Figure 11b

Figure 11a is the specimen at start. After 1 hour of exposure to argon saturated with water, a polycrystalline oxide is produced. This oxide is either β or γ -Ga₂O₃.

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TABLE 4
Interplanar Spacings for Figure 11

<u>d, Å</u>
2.005
1.413
1.230

indicate that the oxide is either γ - or β -Ga₂O₃. This oxide also grows from the edge of the specimen to the interior.

This in-situ TEM oxidation study disagrees with the findings of Bull and Sealy. In this study, at temperatures of 450°C and above, polycrystalline Ga₂O₃ was formed. Bull and Sealy's ex-situ TEM study found that single crystal β -Ga₂O₃ formed at temperatures above 500°C and was oriented: $\langle 002 \rangle_{\beta\text{-Ga}_2\text{O}_3} \parallel \langle 002 \rangle_{\text{GaAs}}$ and $\langle 10\bar{2} \rangle_{\beta\text{-Ga}_2\text{O}_3} \parallel \langle 1\bar{1}1 \rangle_{\text{GaAs}}$. Their study also found amorphous films growing on GaAs at temperatures below 450°C. An amorphous film was produced in this study at 320°C.

One hypothesis for the formation of a polycrystalline oxide is the loss of arsenic. The gallium oxide does not form a continuous crystal in this model; pores and grain boundaries are in it to allow the arsenic oxides to escape. Arsenic and its oxides are volatile at temperatures above 312°C. Also, Navratil, et al.¹⁷ found that the surface of GaAs roughened when heated.

Two experiments were performed to test this hypothesis. Amorphous layers were grown on two GaAs specimens at 320°C. One was heated to 450°C in a vacuum; the other was heated to 450°C in 44 torr O₂. In both experiments a polycrystalline oxide did not form. The amorphous layer remained unchanged after several hours.

Neither of the two experiments support the hypothesis. Ideally, the arsenic (and/or its oxides) should volatilize at 450°C and form a polycrystalline layer of Ga₂O₃.

The only phase diagram for the Ga-As-O system is valid at temperatures below 2780C.¹⁸ At 2780C and below, β -Ga₂O₃ is the stable oxide. γ -Ga₂O₃ could very easily be a higher temperature phase; Bull and Sealy examined their specimens at room temperature.

Part of the disagreement could also be due to the two microscope techniques. Samples used in this study were highly reactive due to their thinness and having been ion-milled. A lower oxygen pressure was also used in this study. Bull and Sealy do not go into detail on how clean their sample was before they oxidized it. Their samples could have contained a thick coating of native Ga₂O₃ before it was oxidized in their furnace.

No traces of As, As₂O₃, or As₂O₅ are found in any of the polycrystalline diffraction patterns. These compounds and/or GaAsO₄ could be present in the amorphous layer. The electron diffraction pattern (Figure 5b) does not allow one to determine if these compounds are present.

Dark field photographs show the oxide forming first at the specimen's edges. This is due to the nature of the specimen. The edges are the thinnest part of the specimen and are ideally the most reactive.

Band and thickness contours were evident in the samples, but there were no visible dislocations. Two-beam conditions could not be obtained since the specimen holder could only tilt $\pm 20^\circ$ in one direction.

CONCLUSIONS

At temperatures above 450°C, polycrystalline Ga₂O₃ is produced when GaAs is oxidized with O₂ or H₂O. Thermal oxidation at temperatures above 450°C is not recommended for GaAs. Ga₂O₃ has poor insulating properties; plus a polycrystalline oxide does not have uniform electrical properties.

An amorphous oxide layer was formed on GaAs at 320°C using pure O₂. Since the oxide could not be identified, further research should be done on it. The amorphous oxide's growth characteristics, electrical properties, and chemical composition should be investigated. This oxide could be suitable to the electronics industry.

Other experiments that are worthwhile conducting are:

- 1) See if there is a catalyst that promotes the oxidation of GaAs. Trichloroethylene has been shown to promote the steam oxidation of Si.
- 2) Do an in-situ study of oxidizing GaAs with As₂O₃ or As₂O₅.

FOOTNOTES

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