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**Reactive ion etching of gallium arsenide and aluminum gallium
arsenide using boron trichloride and chlorine**

Hendricks, Douglas Ray, M.S.

The University of Arizona, 1987

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REACTIVE ION ETCHING OF GALLIUM ARSENIDE
AND ALUMINUM GALLIUM ARSENIDE
USING BORON TRICHLORIDE AND CHLORINE

by

Douglas Ray Hendricks

A Thesis Submitted to the Faculty of the
COMMITTEE ON OPTICAL SCIENCES (GRADUATE)
In Partial Fulfillment of the Requirements
For the Degree of
MASTER OF SCIENCE
In the Graduate College
THE UNIVERSITY OF ARIZONA

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SIGNED: Douglas R. Henderson

APPROVAL BY THESIS DIRECTOR

This thesis has been approved on the date shown below:

Hyatt M. Gibbs
H. M. GIBBS
Professor of Optical Sciences

3-25-87
Date

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ABSTRACT

A reactive ion etch process is described that will etch both gallium arsenide and aluminum gallium arsenide. Gas atmospheres containing boron trichloride and chlorine in various concentrations and under different conditions were tested. Smoothly etched features with vertical walls were obtained when the ratio of chlorine to boron trichloride was less than 0.4 and the chamber pressure was less than 70 millitorr. Changes in radio frequency power level were found not to affect the etch as long as the power level was maintained above 0.24 W/cm^2 .

For reasons of process stability, an etch process using pure boron trichloride at 40 millitorr with an rf power of 0.41 W/cm^2 was selected as optimum. Waveguide devices were successfully fabricated using this process.

CHAPTER 1

INTRODUCTION AND THEORY

1.1 General Introduction

Etching is a vital step in the fabrication of almost every microelectronic or optoelectronic device. By removing material from certain areas of a semiconductor (or other material) and leaving material in other areas, it is possible to create structures that can act as waveguides, transistors, lasers, or many other useful devices.

This paper describes a reactive ion etching (RIE) process that was developed to etch gallium arsenide and/or aluminum gallium arsenide. The motivation for this project was the desire to fabricate waveguides and isolated Fabry-Perot etalon devices in GaAs, AlGaAs or in material that was grown with alternating thin layers of GaAs and AlGaAs (multiple quantum-well structures). A reactive ion etch process using Freon-12 (CCl_2F_2) had previously been successful in etching GaAs but was ineffective on AlGaAs so a new process had to be developed.

1.2 The Lithographic Process

Before launching into a description of the etch process, it will be helpful to briefly outline the entire

microlithographic process (of which etching is just one step).

The starting point is a flat GaAs wafer illustrated in Figure 1-A. After layers of epitaxial AlGaAs and/or GaAs are grown on top of it (Figure 1-B), the sample is coated with photoresist (Figure 1-C). A glass plate with the desired pattern drawn on it is then placed in contact with the wafer. Figure 1-D shows ultra-violet light shining through the glass plate onto the sample. Since portions of the plate are dark, some areas of the photoresist will not get exposed to the light. After the plate is removed and the wafer is rinsed in developer, the photoresist that was exposed to light will be gone but the photoresist that was protected from light by the opaque pattern in the glass plate will still be on the wafer (Figure 1-E). A bake step is required to harden the photoresist and then the wafer may be exposed to the etchant. The etchant will remove GaAs and AlGaAs everywhere except for the areas protected by photoresist (Figure 1-F). Figure 1-G shows the finished product after the remaining photoresist has been stripped off.

Clearly, success of the etch process is dependent on success of all preceding steps. Photoresist troubles such as lack of adhesion, apparent changes in sensitivity, the presence of dust and other contaminants and failure to withstand attack by the etchant are very common problems.

Many other similar difficulties must all be controlled before acceptable etching can be obtained, but this paper will not mention them further. The following discussion will assume that all lithographic steps prior to etching have resulted in an ideal photoresist mask sitting on top of the substrate to be etched.

1.3 Reactive Ion Etching

The etchant could be a liquid, gas, plasma, ion beam, or even a laser beam, as long as it removes the desired material without significantly affecting the photoresist. Plasmas have proven to be useful etchants for fabrication of semiconductor devices that have very small structures (less than ten or twenty micrometers).

Reactive ion etching uses a plasma as the etchant with equipment arranged as shown in Figure 2. This figure is a simplified drawing, but it shows all the key elements of the system. A vacuum pump keeps the reaction chamber at a pressure usually less than 100 mtorr while a gas supply system replenishes the gas as it is pumped away. A radio frequency power supply excites the plasma between the electrodes, and a matching network serves to promote efficient coupling of energy from the power supply into the plasma. The plasma exists in the area between the electrodes and consists of three general types of particles: neutral gas atoms, positively ionized gas atoms, and electrons.

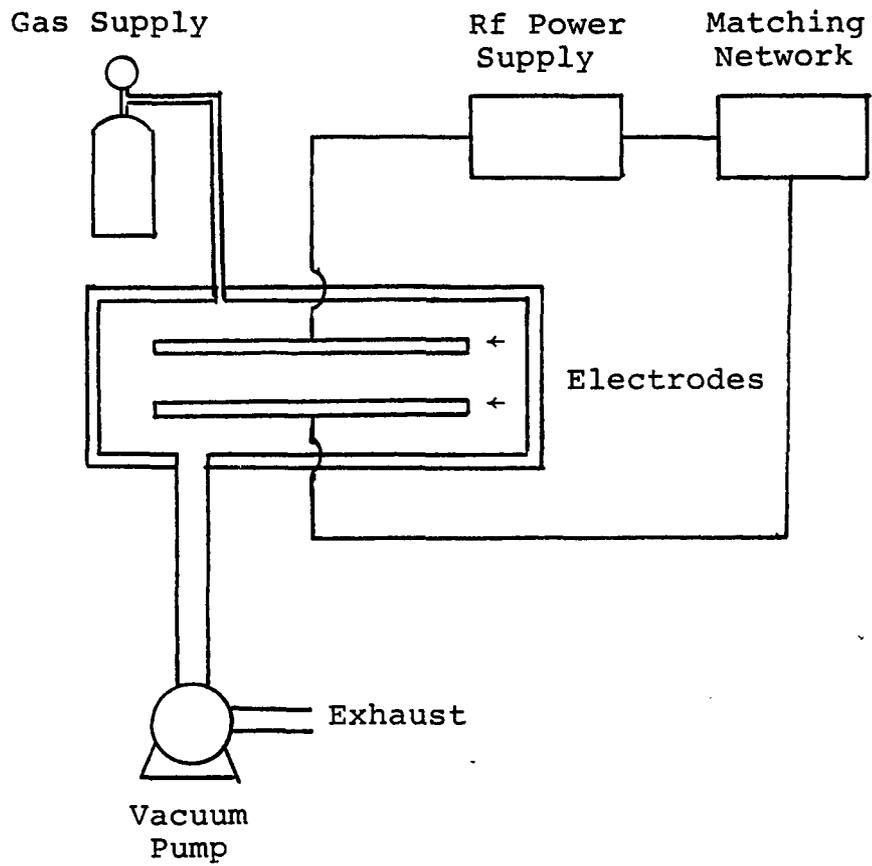


Figure 2. Simplified RIE Setup.

When an alternating electric field is applied between the electrodes, electrons and ions will be accelerated so that both types of particles strike the wafer sitting on the bottom electrode. The electric field does an equal amount of work on the electrons and the ions but because an electron's mass is much less than that of an ion, the electron's velocity will be much much greater. A high-velocity electron colliding with a neutral gas atom can do any of three things. It could knock an electron free from the atom, creating another electron-ion pair and thus replenishing the plasma which would otherwise be depleted of ions. It could excite the neutral atom to a higher electronic state which later relaxes, giving off a photon and creating the glow that is observed between the electrodes, or the electron could just bounce off the atom with no important effect other than a change of direction.

One consequence of the electron's significantly greater velocity is that initially many more electrons than ions will strike the bottom electrode and it will build up a net negative charge. This negative charge will act to repel electrons approaching from the plasma so that after a short time steady state is reached where equal numbers of electrons and ions strike the electrode. The electrode's net negative charge also causes electrons to avoid the region of the plasma directly near it. Since collision of electrons

with neutral gas atoms is the cause of the plasma's glow, and since there are fewer electrons in the space near the electrode, a relatively dark space can be observed there.

The etch mechanism is partially chemical and partially physical. Gas molecules that are ionized in the plasma are much more reactive than un-ionized molecules. They strike the wafer's surface and chemically combine with the wafer material to form reaction products. If these products are volatile, they will leave the wafer's surface and expose underlying layers, allowing etching to proceed. Because the ions are accelerated by the electric field in the plasma, they strike the wafer with a significant momentum and the physical aspect of the etch mechanism becomes apparent. When ions hit the wafer's surface, the energy of collision can result in enhanced chemical reactivity or possibly even knock the molecule out of the wafer as in sputter etching.

Because the ions are travelling essentially vertically when they strike the wafer, it is possible to etch material without undercutting the photoresist. The straight walled structures that result are one reason why plasma etching is a very common process for semiconductor device fabrication involving small feature sizes. Other reasons are that plasma etching can be more easily automated than liquid etch processes and it is conducted in a closed

chamber, protecting the operator from the hazards of working with chemically reactive liquids.

1.4 The Chemistry

Prior to the work described in this thesis, Freon-12 (CCl_2F_2) had been used as an etchant in the RIE chamber in combination with various other gases (He, Ar, H_2). Acceptable results had been obtained in etching GaAs but it was not possible to etch AlGaAs with that process. Also, even with GaAs, run-to-run variations in etch depth were quite troublesome, and unexplained surface roughness appeared from time to time.

The gases used for this thesis, boron trichloride and chlorine, were chosen because they were reported to etch both GaAs and AlGaAs very well. Other researchers (Tamura and Kurihara, 1984) had shown excellent results with these gases in an RIE setup. They reported etch rates of 0.4 microns/min for $\text{Al}_{(.45)}\text{Ga}_{(.55)}\text{As}$ and 0.6 microns/min for GaAs under the conditions that they considered optimum. Their data suggested that it was possible to obtain equal etch rates by sacrificing etching speed, but they did not report pursuing that goal.

CHAPTER 2

DETAILS OF RIE EQUIPMENT

2.1 Gas System

The equipment setup that was used in this project is shown in Figure 3. This picture includes all the details omitted in Figure 2 for simplicity's sake.

The gas system includes a bank of four mass flow controllers (MFCs) that can accurately deliver small amounts of the etch gases to the intake manifold. BCl_3 and Cl_2 were plumbed in for this project; CCl_2F_2 and H_2 were already connected. A master controller that is not shown in the figure monitors the gas flow through the MFCs and automatically makes any necessary adjustments to maintain the set flow rates. Another gas, O_2 , can be used to remove photoresist after the etch if desired. It comes into the manifold through a manually operated rotameter. The last gas in the system, N_2 , is needed to vent the chamber after a process is finished. It is admitted to the manifold through a valve operated automatically by the master controller.

All gases pass through the intake manifold, enter the reaction chamber through holes in the top electrode, and are exhausted from the chamber through the space around the

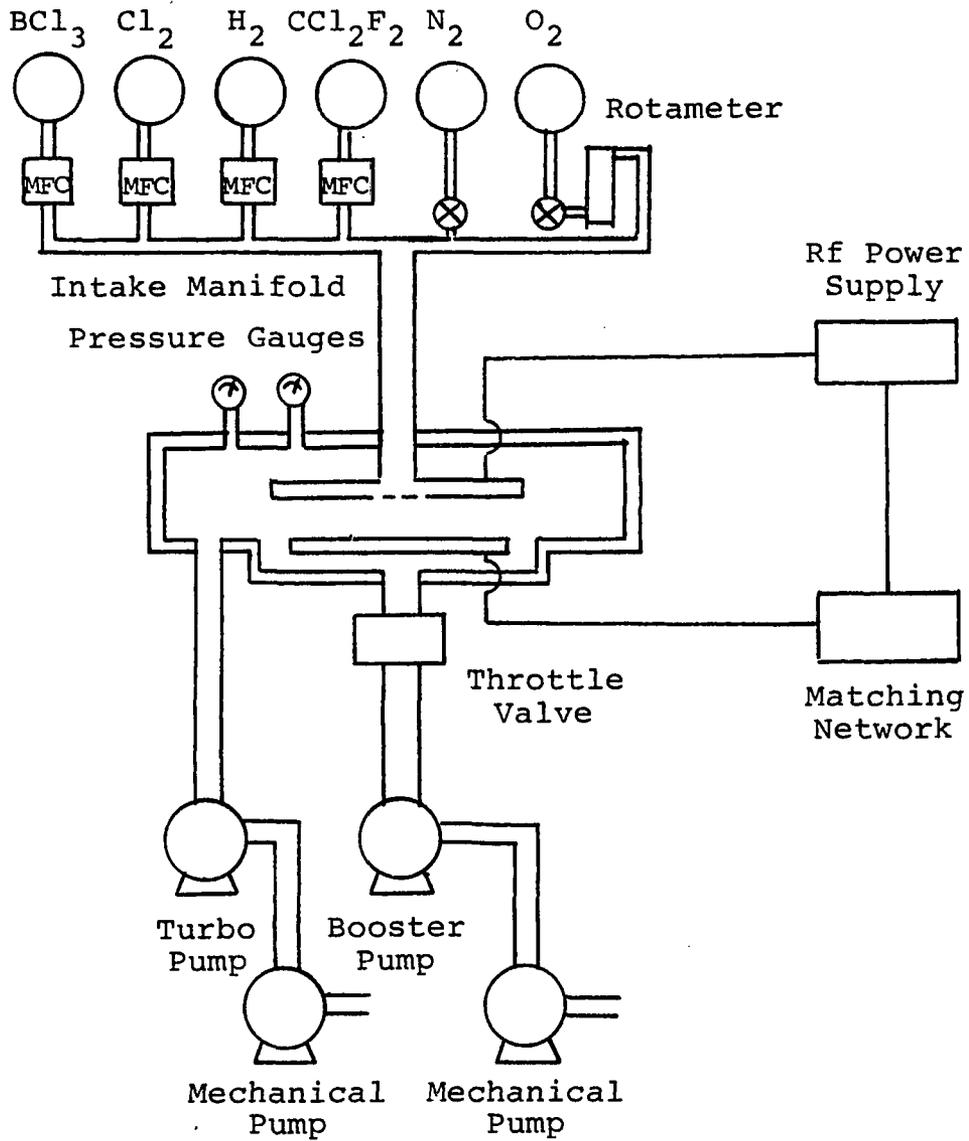


Figure 3. Detailed RIE Setup.

lower electrode. A throttle valve on the vacuum line below the chamber is adjusted by the master controller to maintain the set pressure in the chamber. The vacuum pump arrangement used while etching consists of a booster pump backed up by a mechanical pump and is capable of bringing the chamber down to a pressure of approximately 10 mtorr.

A separate turbo pump can also be used just before each etching run begins. This pump brings the chamber to a lower pressure than the regular process pumps are capable of reaching but cannot be used during the etch process. By using the turbo pump for a few minutes just before the etch gases come in, the chamber can be cleaned of residual contaminants that might have condensed on the walls while the chamber was open to atmosphere.

Two gauges are used to determine the chamber pressure. A thermocouple gauge tube (Hastings DV6) is reliable when air or nitrogen is in the chamber but not when other gases are present. It is used as a reference for the second gauge, an MKS Baratron, which is more reliable when the gas composition varies during a run, but drifts slightly from day to day. The pressures reported in this thesis are the readings recorded from the Baratron gauge during processing, adjusted downward by the difference between the Baratron and the thermocouple gauge when air or nitrogen was in the chamber.

Because the two etch gases used for this project, Cl_2 and BCl_3 , are both corrosive and toxic, safety regulations required installation of a gas leak detector that is not shown in any of the figures. It monitors room air and sets off an alarm if either of the etch gases is detected.

2.2 Electrical System

The alternating electric field needed to excite the plasma is provided by a radio frequency generator operating at 13.56 MHz, the frequency allotted by international communications authorities at which energy can be radiated without interfering with communications. The generator supplies energy to the two electrodes. The bottom one is 23 cm in diameter and the top one is 30 cm in diameter. They are separated by 2 cm.

A matching network monitors reflected power and automatically makes adjustments to maximize the power dissipated in the chamber. It operates in a manner analogous to a DC circuit in which a DC power supply's internal resistance is varied to maximize the power dissipated. This DC dissipation is largest when the external resistance matches the internal resistance of the power supply. For the AC discharge in an etcher, the impedance is partially resistive and partially capacitive so the matching network adjusts the complex impedance in doing its job.

CHAPTER 3

PHOTOLITHOGRAPHY PROCESS

Because the etch process is so strongly affected by the photoresist process, mention must be made about the photolithography details before starting into a description of the etch details.

Before applying photoresist, all samples are scribed and cleaved into pieces that measure between one and two centimeters on a side. Whole wafers are not used because the samples with the multiple quantum-well structures are too precious to process as complete wafers. In order for the test runs to simulate the runs using multiple quantum-well samples, the test samples also have to be cleaved into the same size pieces.

A de-hydration bake is performed at 90°C for 30 minutes and then the photoresist (Kodak 820, 27 centi-stoke) is applied by an eyedropper on the stationary sample. After a three to five second spread time, the sample is spun at 5000 rpm for 20 seconds. Pre-exposure bake is 90°C for 30 minutes.

Exposure is done with the sample underneath and in contact with the photomask. Intensity of the exposure light

at the wafer plane during this study was 7 mW/cm^2 at 436 nanometers and 28 mW/cm^2 at 405 nanometers. Experiments had shown that no significant difference in resist profile resulted when exposure time was varied from four to eight seconds, so six seconds was chosen as the exposure time.

Development is done in a beaker for 30 seconds using KTI 932 developer mixed 1 to 1 with DI water, followed by a rinse in DI water for at least 30 seconds. Pre-etch bake (hard bake) is for 20 minutes at 115°C .

Scanning electron microscope (SEM) pictures of the photoresist as it appears after the hard bake are shown in Figure 4. The pattern in both pictures was made using the waveguide mask. Figure 4-A is a top view tilted slightly so the side walls can be seen. The ripples on the side walls occur because of the standing wave that results when the incident exposure light interferes with the light that is reflected from the wafer's surface. This effect is commonly observed and does not degrade the etch results. Figure 4-B is an end view of the resist where the wafer has been cleaved. The side walls are between 10 and 20 degrees away from vertical.

After the pre-etch lithography steps are complete, the samples are cut into even smaller pieces, placed on top of a two-inch silicon wafer, and laid on the bottom electrode. Etching can now begin.

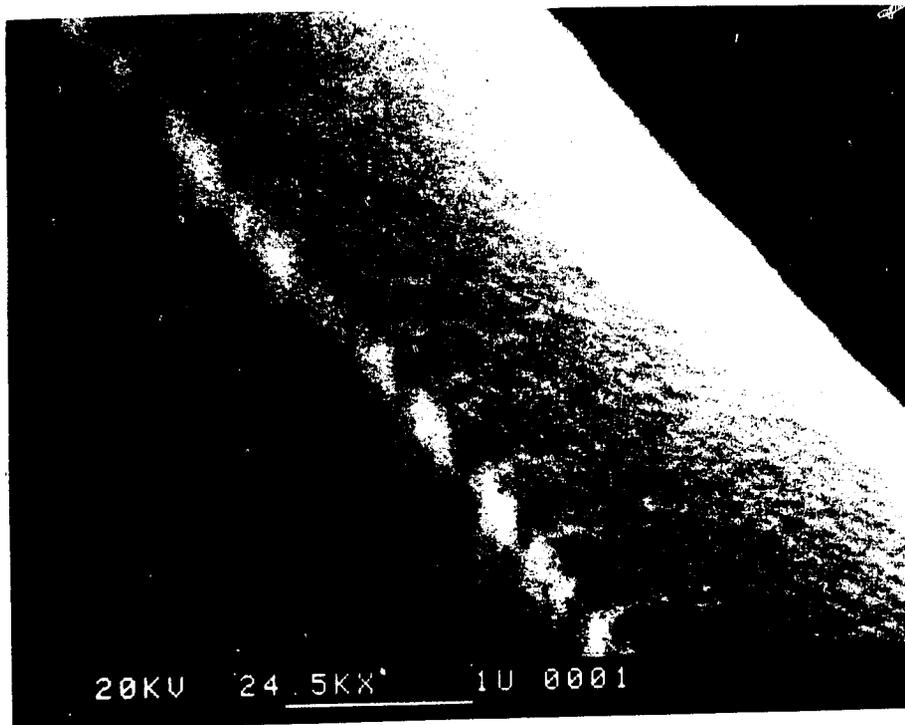


Fig. 4-A. Photoresist Ridge. -- Top view.

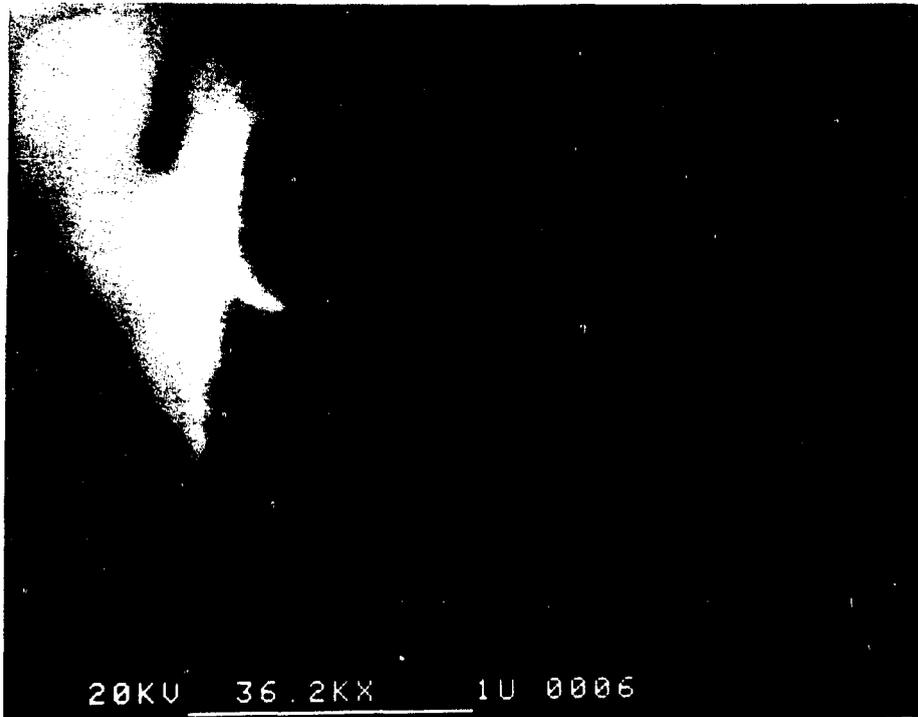


Fig. 4-B. Photoresist Ridge. -- End view.

CHAPTER 4

EXPERIMENTAL RESULTS

For the experiments described in this chapter, etch rates of AlGaAs were measured using samples made at TRW with a single epitaxial layer of AlGaAs grown by Metallo-Organic Chemical Vapor Deposition (MOCVD) on a GaAs wafer. The ratio of aluminum to arsenic was 0.3 and the ratio of gallium to arsenic was 0.7. Etch rates of GaAs were measured on pure GaAs samples that had no epitaxial growth. In all cases, etch rates were obtained by measuring the etch depth with a Dektak profilometer or by SEM observation and then dividing the etch depth by the etch time.

4.1 Gas Ratio

The first runs were designed to find a good gas flow ratio. Rf power was held constant at 170 W (0.41 W/cm^2). This value was chosen in order to reproduce the setup of Tamura and Kurihara for comparison of results. Although they used AlGaAs with a ratio of Al to As of 0.45 and a ratio of Ga to As of 0.55, a similar etch rate was expected.

The pressure controller was set to keep a constant pressure of 40 mtorr for all runs. Figure 5 shows the

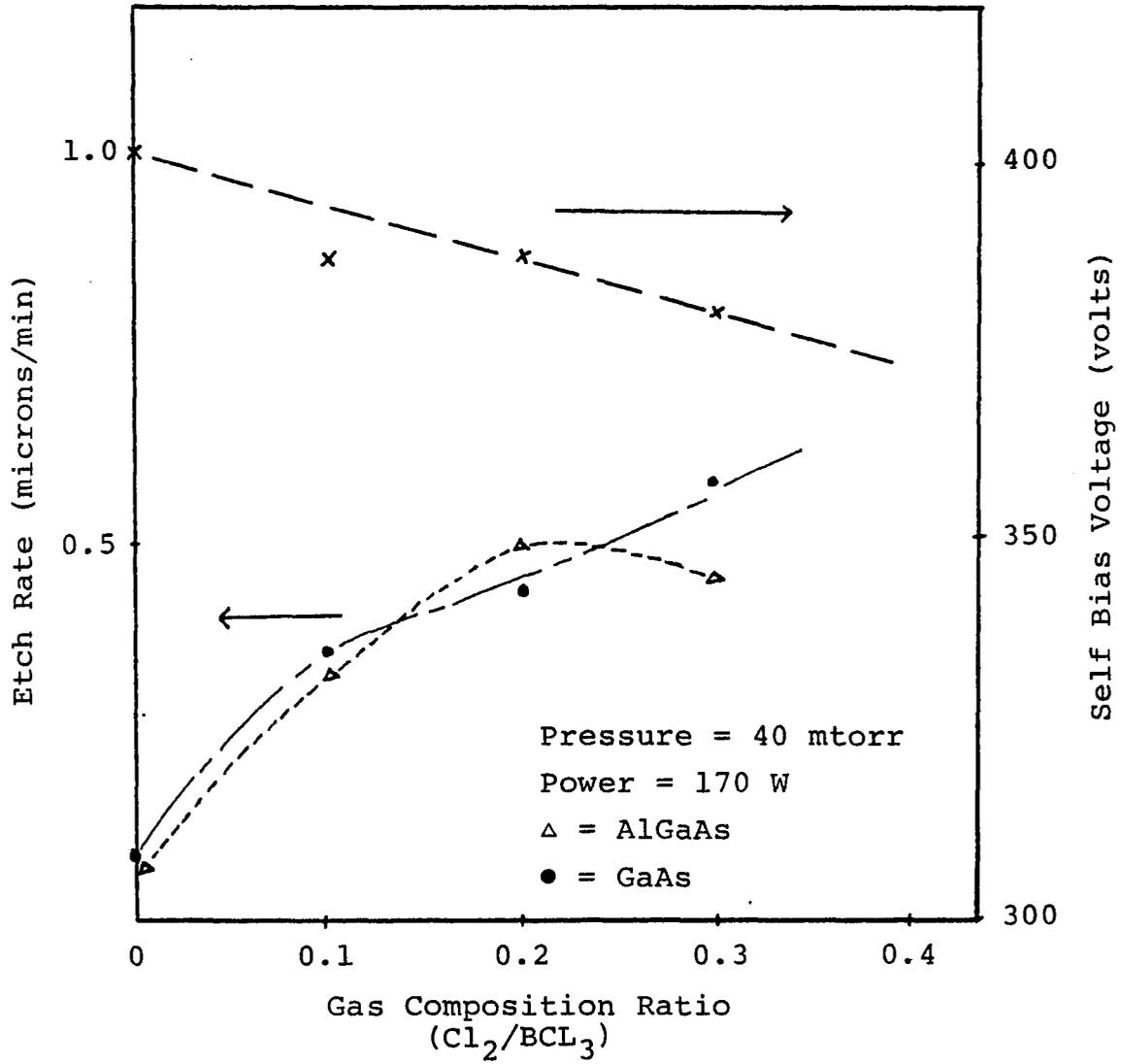


Figure 5. Etch Rate and Self Bias Voltage as a Function of Gas Composition Ratio

results with etch rate shown on a left vertical axis and self bias voltage shown on the right vertical axis. The Cl_2 flow rate divided by the BCl_3 flow rate is shown on the horizontal axis of Figure 5 as the Gas Composition Ratio. Although the figure only shows up to a ratio of 0.3, the ratio was actually varied from 0 (pure BCl_3) to infinity (pure Cl_2). For gas ratios higher than 0.3, the etch quality was so bad that the etch depth could not be measured. Figure 6 shows a typical result that was etched at a ratio of 1. At gas ratios higher than 8, etching did not occur. At gas ratios of 0.3 and lower, relatively smoothly etched walls were obtained. Figure 7 shows a typical result that was etched at a gas ratio of 0.2.

When the gas composition ratio was zero (pure BCl_3 at a flow rate of 25 SCCM) the etch rates of GaAs and AlGaAs were identical. The rate was 0.08 microns/min which is only slightly lower than the rate obtained by Tamura and Kurihara (0.12).

At a gas composition ratio of 0.1 (2.5 SCCM of Cl_2 , 25 SCCM of BCl_3) the etch rates were higher for both GaAs and AlGaAs and the two etch rates were still within 10% of each other. This general trend agrees with Tamura and Kurahara's results although they reported slightly lower etch rates (about 0.2 microns/min for both GaAs and AlGaAs instead of 0.35).

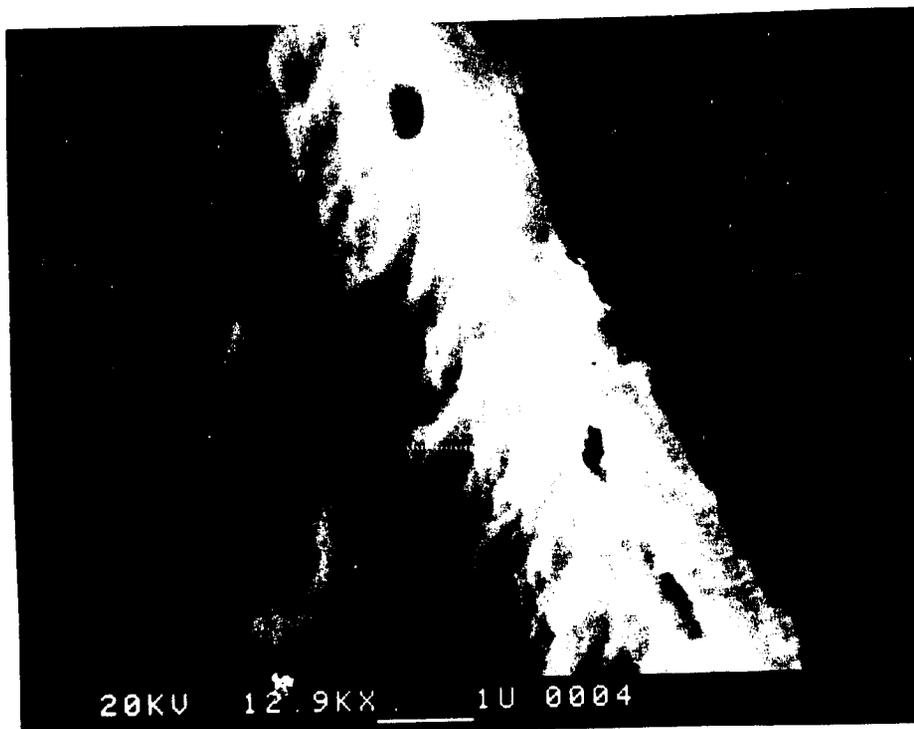


Fig. 6. GaAs Ridge Etched with $\text{Cl}_2/\text{BCl}_3 = 1$.



Fig. 7. GaAs Ridge Etched with $\text{Cl}_2/\text{BCl}_3 = 0.2$.

At a gas ratio of 0.2 (Cl_2 flow = 5 SCCM, BCl_3 flow = 25 SCCM) the etch rates were still within 10% of each other (0.45 microns/min for GaAs and 0.5 for AlGaAs). Here the results are different from Tamura and Kurihara's. They show the GaAs etch rate increasing much more than AlGaAs under these conditions (0.6 microns/min for GaAs and 0.4 microns/min for AlGaAs).

When the ratio was increased to 0.3, the etch rate of GaAs went up to 0.6 microns/min but the etch rate of AlGaAs stayed down at 0.45. These results agree exactly with Tamura and Kurihara.

The general agreement between current results and those obtained by previous researchers was encouraging because it showed that the goal of etching AlGaAs was obtainable. But before that goal could be reached, the process had to be more fully characterized.

4.2 RF Power

The relationship between power density and etch rate of GaAs was explored using a fixed gas ratio of 0.2 (5 SCCM of Cl_2 , 25 SCCM of BCl_3). All runs were done at a chamber pressure of 40 mtorr.

The results are shown in Figure 8. There appears to be a threshold at about 50 W (0.12 W/cm^2) below which no etching occurs. Below 80 W (0.19 W/cm^2) the etched surface was rough while above that value the etch was acceptably

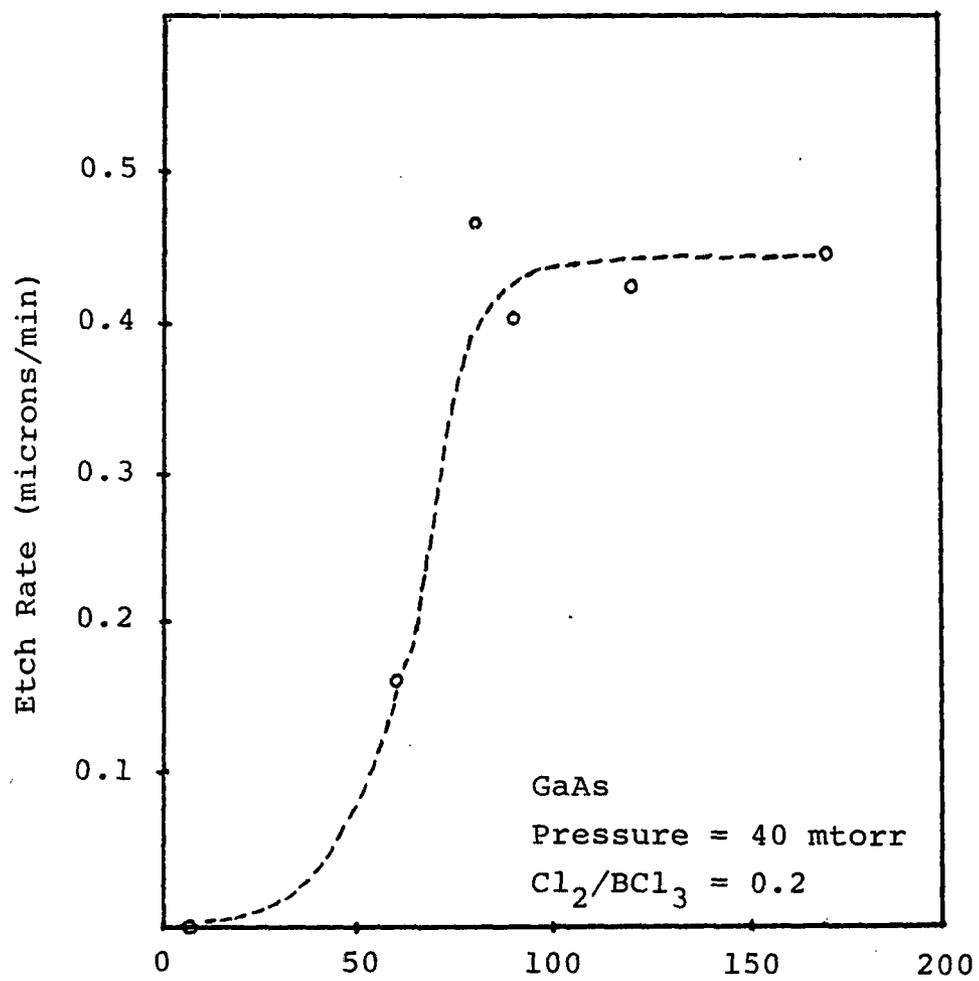


Figure 8. Etch Rate of GaAs as a Function of Rf Power.

smooth. Above 100 W (0.24 W/cm^2) the etch rate does not appear to be a function of power density.

Since the power level of 170 W that was used in Section 4.1 is well into the stable region of the curve, that value was chosen to use for the rest of the runs studied in this project.

4.3 Etch Depth vs. Time

One question that was investigated was whether or not there was a period right after the etch started when the etch rate was very slow. This phenomenon is not uncommon in other etch processes and often occurs because of the presence of an oxide layer that must be etched through before the plasma can reach the substrate. If this initialization period were present, it would have to be taken into account when characterizing the process.

Figure 9 shows the etch depth as a function of etch time. The two sets of data reflect runs that were made on two different days. The fact that one day's runs all showed a higher etch rate than the other day's runs raises a question of repeatability that will be addressed in Section 4.5. However, even though the two lines have different slopes, both of them extrapolate back to zero etch depth for zero time. This important result suggests that there is not an initial slow period in the etch.

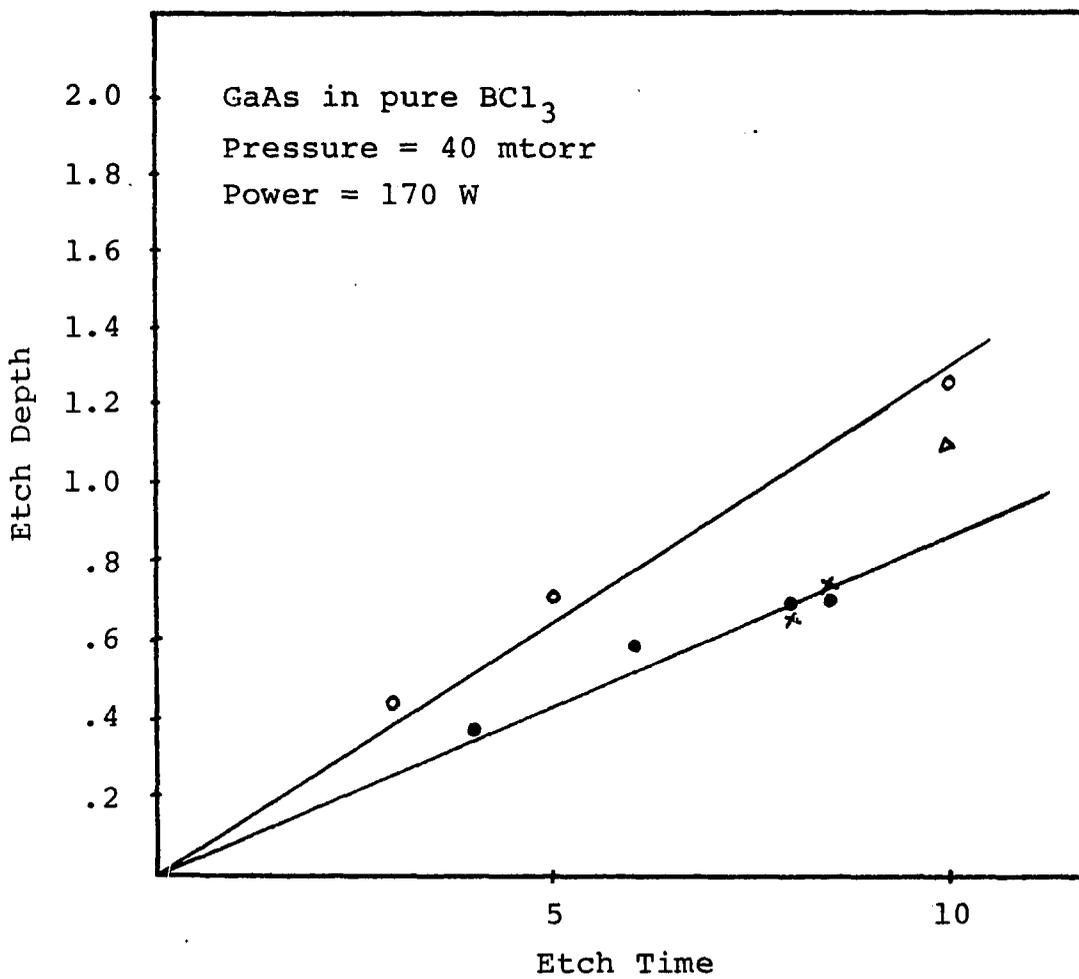


Figure 9. Etch Depth as a Function of Etch Time.

4.4 Etch Rate vs. Pressure

In order to determine the effect of pressure, samples were etched at 70 and 120 mtorr with a gas ratio of 0.2 ($\text{Cl}_2 = 5$ SCCM, $\text{BCl}_3 = 25$ SCCM). The samples came out so poorly that reliable etch depth measurements could not be taken. SEM pictures of the results are provided in Figures 10 and 11.

The first figure (10) shows that the waveguide etched at 70 mtorr is badly eroded on the sides, and the surface surrounding the guide is very rough. The next figure (11) shows the same surface roughness on the sample etched at 120 mtorr and even worse erosion on the side of the guide than before.

For an interesting comparison, turn back to Figure 7 which showed the run etched at the same gas ratio but at a lower pressure. That guide had vertical edges and a smooth surrounding surface. These results suggest that pressure variations can be devastating for the process using a gas ratio of 0.2.

The effect of pressure was also studied when the etch was done using only BCl_3 in the chamber. This time reliable etch rate measurements could be taken and the waveguides made at the higher pressures were not eroded. Figure 12 shows a sample etched at 160 mtorr with pure BCl_3 .

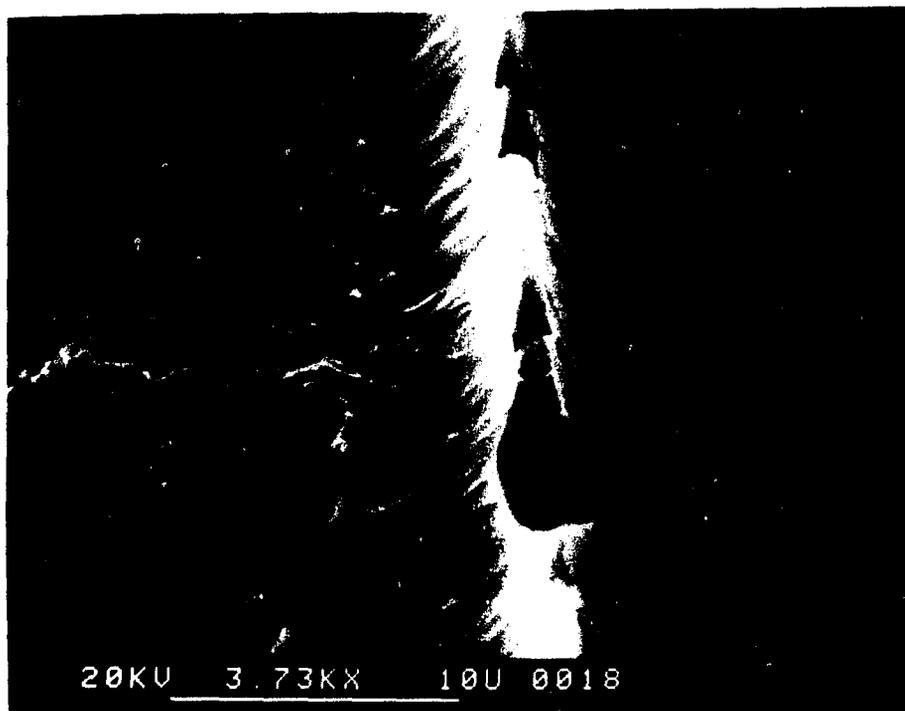


Fig. 10. Ridge Etched at 70 mtorr with $\text{Cl}_2/\text{BCl}_3 = 0.2$.

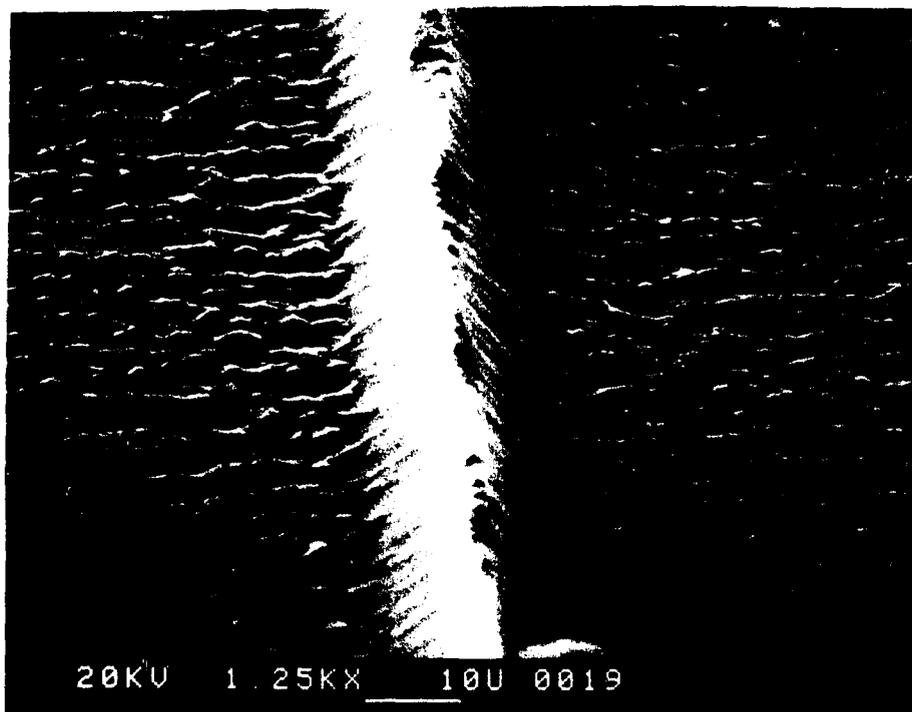
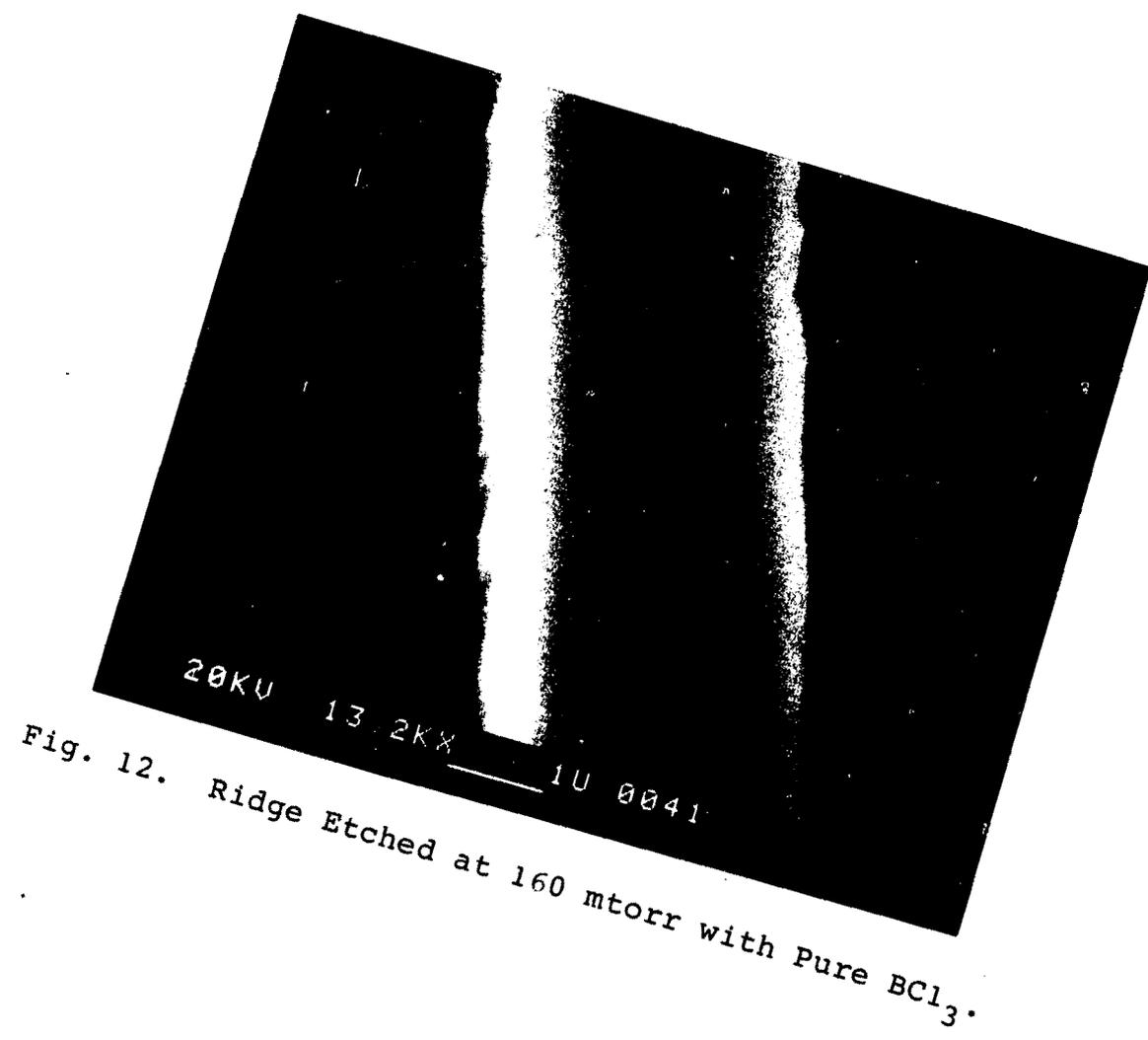


Fig. 11. Ridge Etched at 120 mtorr with $\text{Cl}_2/\text{BCl}_3 = 0.2$.



The relationship between chamber pressure and etch rate in pure BCl_3 is shown in Figure 13. The general trend is for increased etch rate as pressure increases. Surface roughness was not a problem at higher pressures. Even at 160 mtorr the surface was acceptably smooth.

4.5 Repeatability

Back in Figure 10 of Section 4.2 it was seen that runs done on different days gave different etch rates. Unfortunately it was not uncommon during this study to see differences in etch rates from day to day and even from run to run on the same day although they usually differed by less than 20%.

Figure 14 shows four pairs of runs etched in pure BCl_3 at different times. Each pair consisted of a GaAs sample and an AlGaAs sample etched side by side in the chamber. The etch rates of GaAs varied from a low of 0.8 microns/min to a high of 0.13. For one run the etch rates of GaAs and AlGaAs were exactly the same and for another run they differed by 20%.

Two main theories were postulated to explain these differences. One theory blamed the pressure variations caused by the drift in the Baratron. Since the master controller used the Baratron as a reference when controlling chamber pressure during a run, an incorrect Baratron reading would affect the chamber pressure. A problem with this

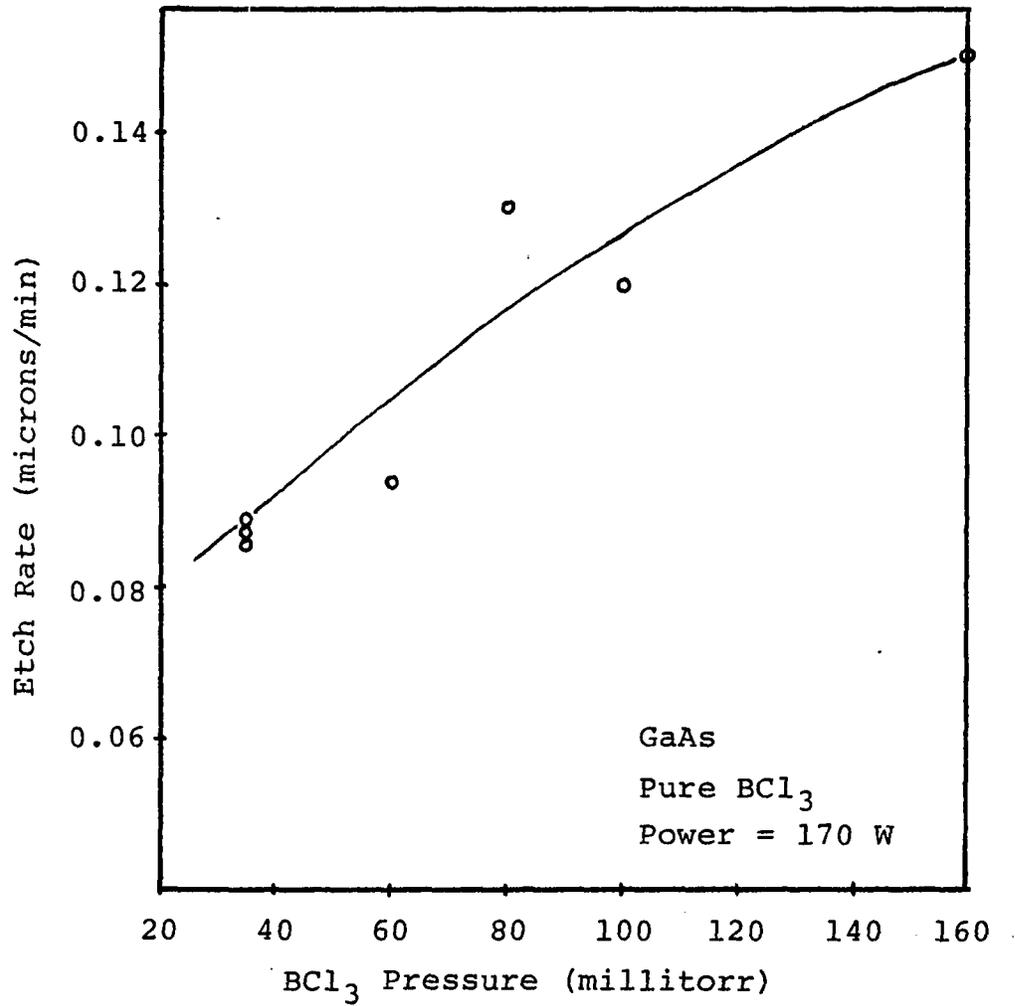


Figure 13. Etch Rate as a Function of Pressure in Pure BCl₃.

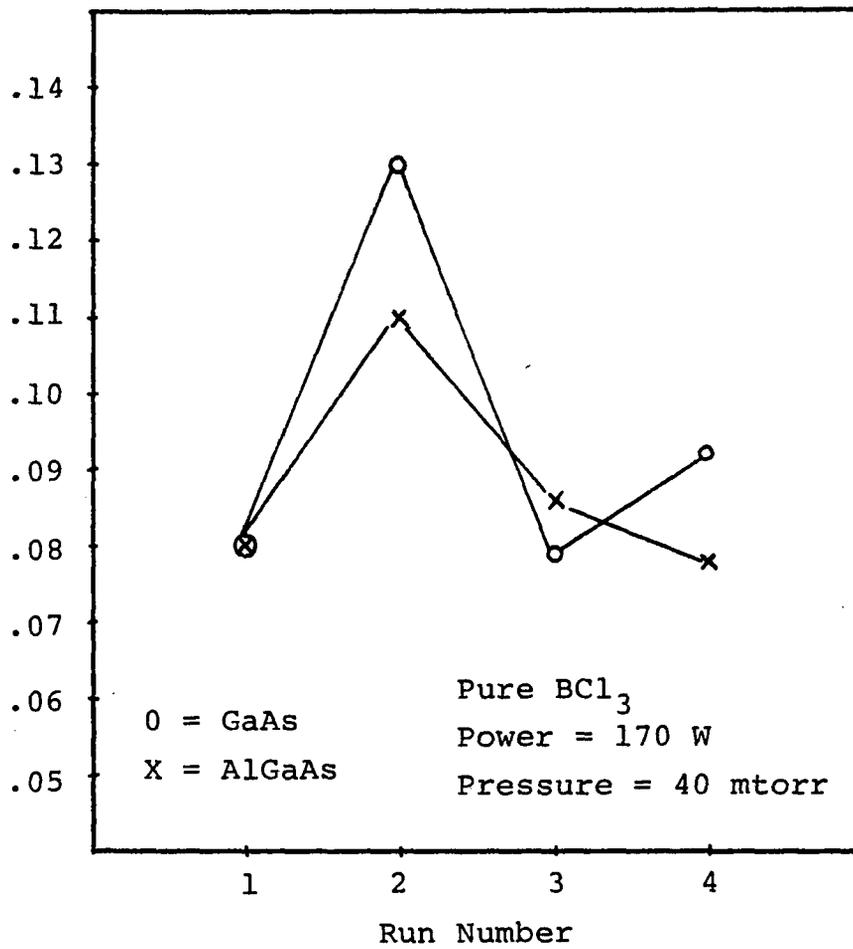


Figure 14. Etch Rate of Four Different Runs Etched in Pure BCl_3 .

theory is that the Baratron always drifted in the upward direction by a small amount each day. This would result in a lower chamber pressure and (according to Figure 13) a continuously falling etch rate, but the actual etch rate fluctuations were both up and down. The purchase of an ion gauge is currently being investigated to see if it would help solve the repeatability problem.

A second theory to explain the etch rate variations blamed the technique used when sticking the sample to the wafer with vacuum grease. Occasionally the sample would be blown off the wafer by the rush of air that occurred when the chamber was initially pumped down before etch. To keep the sample in place, a dab of vacuum grease was put between the sample and the wafer. The theory to explain the variations held that sometimes the sample didn't contact the wafer while other times it did. If the sample was held completely away from the wafer, it could either heat up or charge up more than otherwise, which would affect the etch rate.

To test the theory, several runs were done using grease under one sample and no grease under another one and etching the two samples side by side in the chamber. The samples without the grease consistently showed almost twice the etch rate of the samples with the grease. This large difference indicates that careless use of grease could

possibly account for the variation in etch rate. After discovering this, greater care was taken to ensure that the samples were always completely isolated from the wafer with the grease in between. Although this gives a slower etch, it should be more repeatable. As of this writing, not enough runs have been done to decide whether or not this technique solves the problem.

CHAPTER 5

DEVICE FABRICATION

For reasons that will be discussed in Chapter 6, the best process was chosen to be RIE in a pure BCl_3 atmosphere at 40 mtorr and 170 W of rf power. These conditions were used to fabricate waveguides in samples with a one micron thick AlGaAs layer on top of a stack of alternating GaAs and AlGaAs multiple quantum-wells 97-Angstroms thick. The etch process was purposely stopped before the multiple quantum-well layers were reached, so the ridge that resulted was in the top AlGaAs layer only. These waveguides were shown to successfully guide light, and work is in process to observe optical bistability in them as this thesis is being written.

Other plans for future work include using the etch process developed here to fabricate directional couplers, arrays of Fabry-Perot etalons, and quantum-dot structures in AlGaAs and/or GaAs.

CHAPTER 6

CONCLUSIONS

Based on the results of this study, the recommended RIE process for GaAs and AlGaAs uses pure BCl_3 at a pressure less than 150 mtorr and an rf power between 125 and 175 watts. The etch rate for both GaAs and AlGaAs at 40 mtorr and 170 W is about 0.1 micron/min although variations of as much as 20% have been observed. Structures with vertical walls and smooth surfaces can be easily fabricated.

This etch process enjoys the luxury that the tolerance on the pressure and power level need not be very tight. Increased pressure increases the etch rate in pure BCl_3 but does not degrade the etch quality, whereas a process using a combination of BCl_3 and Cl_2 shows significant degradation of etch quality when the pressure rises to only 70 mtorr. Power level is not a major concern because it would have to drop to half of the recommended levels before etch rate or etch quality would suffer.

The presence of Cl_2 at low pressures does not hurt the etch quality as long as the ratio of Cl_2 to BCl_3 is less than 0.4. However, the Cl_2 does not really help either. The higher etch rate obtained by adding Cl_2 is not a true

advantage because the etch rate in pure BCl_3 is quite acceptable, and in fact the slower etch rate allows more precise control over etch depth.

REFERENCE

Tamura, H. and Kurihara, H., Japanese Journal of Applied Physics, Vol. 23, No. 9, pp. L1731-L733 (Sept. 1984).