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COVER PAGE

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Plasma Etching of CVD Tungsten using ECR Discharges

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Abstract

An electron cyclotron resonance (ECR) plasma was used to etch patterned CVD (chemical vapor deposition) tungsten from single crystal silicon wafers, thermally grown silicon oxide, and boron-phosphorous doped glass. Etch rates of 2700 Å/min and photoresist selectivity of 2.3 were observed at substrate temperature below 323 K in a $P_{forward}$ = 1000 W, 1 mtorr SF_6/O_2 (20%) plasma with -20 V applied bias on the substrate. A two-level factorial experiment was designed to determine the optimum process condition with adjustable parameters of pressure, microwave power, RF bias, and oxygen addition. Etch rate and selectivity increase with $P_{forward}$ = 600 to 1000 W, decrease with pressure = 1 to 3 mtorr, and increase with RF bias = 0 to -40 V. For oxygen addition, etch rate and selectivity variations were more complicated. Etch rate and selectivity increased with increasing oxygen addition at a constant pressure p = 1 mtorr, whereas etch rate and selectivity decreased with oxygen addition at p = 2 mtorr. Preliminary etch data showed that good etch profiles with no undercut and good photoresist selectivity can be obtained at -20 V RF bias. At -60 V bias, although a higher etch rate was obtained, the surface becomes pitted and the photoresist selectivity begins to deteriorate, consistent with the increase in ion bombardment energy across the sheath.

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I. INTRODUCTION

Refractory metals have long been investigated for use as metal interconnects, plugs in contact holes, and deep vias. Tungsten, specifically, has attracted major attention in the past decade because of its high resistance to electromigration and hillock formation, ability to withstand high temperature processing, and good step coverage over high aspect ratio trenches through chemical vapor deposition.[4,11-13] As device dimensions decrease, process conditions become more demanding in order to provide good etch profiles, and at the same time, maintain good etch rate and selectivity over the underlying substrate and overlying photoresist pattern. Studies on tungsten etching in a fluorinated plasma have been done using an RIE (reactive ion etch) source[4,6,11-13]; however, issues of radiation damage and degree of undercut motivate the search for an alternative source.

The unique features of ECR (electron cyclotron resonance) such as low pressure, low ion energy, and independent control of RF bias and input power, will allow better control of anisotropy, etch rate, and selectivity, and can also reduce radiation damage from the plasma. Studies have been performed to characterize ECR systems and their etch characteristics for metal silicides and aluminum-silicon-copper alloys[8,9]; no data is available, however, for tungsten etching.

In this study, tungsten etch profiles, etch rate, and selectivity will be examined in fluorine-containing plasmas generated by an electron cyclotron resonance discharge. The gases used are CF_4 and SF_6, and their admixtures with oxygen. The effects of power, pressure, RF bias, and oxygen concentration on the etch rate of CVD (chemical vapor deposited) tungsten and its selectivity over photoresist are also investigated.

II. EXPERIMENTAL

The ECR system used in this study has been described elsewhere.[10] The reactor configuration is shown in Figure 1. Two and three inch wafer pieces were clamped onto the wafer holder via three metal fingers. The samples were supplied by Jeff Bukhman of the Motorola Advanced Technology Division, and are composed of 1 μm CVD tungsten film on top of boron-phosphorous doped glass, patterned by 1.2 μm photoresist. The wafer holder was maintained at a temperature below 323 K by circulating cooling water through the holder assembly, as monitored by a thermocouple.

A two-level factorial experiment was designed to study the effects of power, pressure, oxygen concentration, and RF bias on the etch rate of tungsten and the selectivity over photoresist. In each case, all variables were kept constant except for the one of interest. The different levels are : $P_{forward} = 600 - 1000$ W ($\Delta = 200$), pressure = 1 - 3 mtorr ($\Delta = 1$), bias[2] = 0 - -40 V ($\Delta = -20$), and percent oxygen = 0 - 20% ($\Delta = 10$).

Before each run, the chamber was conditioned with an argon plasma for 5 minutes. Each sample was etched for 3 minutes to eliminate the subjectiveness in determining a clear time for the tungsten film; the 3 minute etch did not clear the film. Since tungsten is an opaque film, the thickness cannot be determined using the Nanospec thin film measurement system; therefore, an Alphastep 200 profilometer was used. The step height was measured before and after resist strip to determine the thickness of photoresist and tungsten etched. The measured film thickness was divided by the etch time to yield an etch rate. The selectivity of tungsten over photoresist was determined by taking the ratio of the thickness of tungsten removed to that of the photoresist.

A Langmuir probe was used to determine the ion density (n_i) and electron temperature (T_e) as a function of the parameters investigated. Scanning Electron Microscopy (SEM) was used to analyze the etch profile of the tungsten structures.

III. RESULTS AND DISCUSSIONS

A. Characterization of CF_4 discharge

The ECR magnet current was set at 260 A and a Langmuir probe was used to determine the plasma parameters via the method of Laframboise.[7] The ion density measured 1 cm in front of the wafer was linear with power from 400 to 1400 W as shown in Figure 2. Ion density and electron temperature variations with pressure were also measured (Figure 3); ion density increases from 1 to 2 mtorr, goes through a maximum at 2 mtorr, and decreases with increasing pressure. The reasoning

[2]Bias reported in this paper is the measurement of a DC offset from an applied RF bias

behind the trend is probably two fold: 1). At fixed power, the initial increase in n_i is due to the decrease in electron temperature T_e as pressure is increased, because $P_{forward} \sim n_i T_e$; 2). Diffusional losses to the chamber walls are enhanced at higher pressure, which lead to the subsequent decrease in plasma density. Electron temperature was observed to decrease slowly with increasing pressure, which is a result of the decrease in the required ionization rate constant for particle balance at higher pressures. These variations of n_i and T_e versus power and pressure are consistent with results from other ECR systems.[2]

Radial ion density profiles were also measured to obtain uniformity information. As can be seen from the top graph in Figure 4, a drop in ion density was observed at the edge of the wafer holder (diameter = 10.5"), possibly due to the change in boundary conditions and plasma absorption by the wafer holder; a parabolic profile also develops in front of the wafer holder with $n_{i,max}$ at the center. To verify that the wafer holder perturbs the density profile, data was obtained in the absence of the wafer holder. As shown in the bottom graph of the same figure, the density profile was fairly uniform with the exception of the radial position =-4 inches. This might be due to plasma asymmetry or perturbation of the plasma by the probe body.

B. CF_4/O_2 Etch Results

Mixtures of CF_4 with different concentrations of oxygen (10 - 20%) were used to etch tungsten samples. No etching was observed when the wafer holder was not biased; varying the percent oxygen, etch time, power, and wafer holder position yielded no significant improvement. When bias was applied to give a DC potential of -60 V, an etch rate of 420 A/min (see Table 1) was observed in a CF_4/10% O_2 mixture. Increasing the DC potential to -120 V improves the etch rate by approximately 80% but the resist selectivity was extremely poor. Since an increase in oxygen concentration was known to improve the tungsten etch rate[1,6,12], the DC potential was held constant while increasing the oxygen percentage from 10 to 20. Again, the etch rate was improved but the photoresist selectivity was less than 0.5. The decrease in resist selectivity with an increase in DC bias and percent oxygen is obvious for reasons that have long been understood. The increase in ion bombardment energy due to the higher potential drop across the sheath enhances the etch rate of photoresist by sputtering, and the increase in oxygen concentration further depletes the resist thickness by chemically reacting with the polymer substrate. (Oxygen plasmas have long been used in resist stripping.)

With the thickness of the tungsten layer being 1 μm, an etch time of 20 - 25 minutes will be required if CF_4/O_2 is used, and the tungsten to photoresist selectivity is not in an acceptable range. This is neither efficient nor practical from a process standpoint; therefore, an alternative gas mixture must be used. Along with CF_4, SF_6 and SF_6/O_2 chemistry have also known to etch tungsten[4-6,11,13] on much faster time scales.

Table 1. Tungsten Etch in CF_4/O_2 Plasma

■ CF_4/10% O_2

RF Bias	Etch Rate (A/min)	Selectivity (W:Photoresist)
-60 V	420	------
-120 V	760	0.37

■ CF_4/O_2, -60 V bias

% O2	Etch Rate (A/min)	Selectivity (W:Photoresist)
10	420	------

20	525	0.44

C. SF_6/O_2 Etch Results

Pure SF_6 was observed to etch the 1 μm tungsten film with no complications (clear time was less than 10 minutes). A factorial experiment was designed to study the effects of power, pressure, RF bias, and oxygen concentration on etch rate and photoresist selectivity. (Figure 5) A study of etch depth versus time was first conducted to confirm the linear dependence of etch rate on time, which was proven as can be seen in Figure 6. Data points were collected at 1 minute increments and fitted by linear regression.

From Figures 7 - 9, etch rate and selectivity increase with increasing power, decreasing RF bias and pressure. For all the variables studied in the factorial experiment, none appears to affect the photoresist etch rate significantly. Selectivity trends seem to follow the tungsten etch rate. This particular finding is contrary to what is observed in an RIE system, where selectivity decreases with increasing power, pressure, and bias[4,11].

Etch rate improvements with increase in power and RF bias have been observed previously.[4,11] For the power factorial, etch rate enhancement can be explained as follows: plasma density is increased because $P_{forward} \sim n_i T_e$ and T_e is relatively independent of power. Similarly, the dissociated fluorine atom density goes up. Increases in n_i and n_F both enhance the tungsten etch rate. This is consistent with the fluorine etch mechanism, which states that product formation is dependent on the degree of halogenation of the surface. The amount of fluorine available is particularly important for a tungsten substrate, since the main volatile product formed is WF_6.[5] With increase in RF bias, the etch rate can be raised because of the corresponding increase in potential drop across the sheath. As the potential drop goes up, the ion bombardment energy also increases, thus enhancing the etch rate.

The decrease in resist selectivity and tungsten etch rate at higher pressure might be explained by the variation of n_i, T_e and n_F with pressure. A decrease in ion density and electron temperature with increasing pressure has been observed in other ECR works.[2,3,10] This is consistent with the observations in the current study. A decrease in temperature can also lead to a drop in fluorine atom density, since the dissociation rate is a sensitive increasing function of temperature. Although one may expect that $n_F \sim p$, the preceeding effects, as well as the enhanced diffusion and recombination loss of fluorine atoms at higher pressures, may more than compensate for the tendency of n_F to increase due to an increase in feedstock gas density. A decrease in n_i and n_F at high pressures will lead to a decrease in tungsten etch rate.

Figures 10a and 10b show the etch rate and selectivity dependence on oxygen addition for different pressure ranges. The trends are complicated; both etch rate and selectivity increase with increasing oxygen concentration when p = 1 mtorr, and both decrease when p = 2 mtorr. Both trends have been observed in the literature[4,12, 13]; however, the reactor configurations and process conditions were different for each trend. A drastic change in etch rate and selectivity behavior when different pressure ranges are studied has not previously been observed in the same system. Two mechanisms may be considered: 1) addition of oxygen frees fluorine atoms in the main gas (SF_6), which increases the fluorine atom density, 2) oxygen and fluorine are competitively adsorbed onto the tungsten surface, such that an increase in oxygen concentration in the plasma will oxidize the surface and inhibit adsorption of fluorine atoms. The behavior of the etch rate for the two different pressures suggests that the dominating mechanism must change when switching from 1 to 2 mtorr. In other words, at low pressure, the generation of fluorine atoms might dominate, and the etch rate should increase with increasing oxygen concentration. However, when the pressure is increased, the oxygen concentration also increases, making the surface "oxide-like", and thereby preventing fluorine atoms from adsorbing onto the surface, so that the etch rate goes down. Although the exact mechanism is unknown, the above reasoning has been used to explain why the tungsten etch rate decreases monotonically with increasing oxygen concentration in an RIE system.[6] A simple kinetic model will be developed to provide a quantitative view of the above mechanisms. The model will include both gas phase and surface reactions of SF_6, O_2, and the tungsten substrate. Surface adsorption mechanism will initially be described by the use of the Langmuir-Hinshelwood isotherm.

SEM photographs of the tungsten structure are presented in Figure 11. A preliminary study was conducted by varying the bias and obtaining the etch profile. Biases ranging from 0 to -60 V were applied, with -20 V being the best case, and -60 V the worst. At high negative DC potential, the substrate surface becomes pitted. The optimum point was obtained from the factorial experiment. As can be seen, -20 V bias is sufficient to provide good etch profiles.

D. Future Work

Plasma parameters such as ion density and electron temperature will need to be obtained under conditions identical to the factorial experiment to provide better understanding of the experimental data. Consistency of experimental results will also need to be verified. Diagnostic techniques such as Langmuir probes and optical emission spectroscopy along with actinometry will be used to obtain ion density and fluorine atom concentration under various process conditions.

The photoresist behavior will require a separate set of experiments in which blanket films of photoresist over silicon are etched using the identical conditions for patterned tungsten etch and for a pure oxygen plasma. If the same trends are observed, further studies of these electronegative plasmas will be needed. However, if a difference is observed, then the interactions between the tungsten and the photoresist during etch might be responsible for the results obtained from the factorial experiment.

IV. CONCLUSIONS

Patterned tungsten over BPSG was etched in an ECR reactor following the design of a factorial experiment. Effects of power, pressure, percent oxygen, and RF bias on etch rate and selectivity were investigated. Etch rate and photoresist selectivity were found to increase with increasing power, decreasing pressure, and increasing RF bias. Effect of oxygen addition depends on the pressure range used. At low pressure (1 mtorr), both etch rate and selectivity increase with increasing O_2, however, the opposite trend was observed when the pressure was doubled. SEM analysis of the etch profile showed nearly vertical sidewalls and a smooth surface at -20 V RF bias.

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FIGURE CAPTIONS

Fig. 1: Reactor schematic.

Fig. 2: Plot of n_i versus microwave power with p = 1 mtorr.

Fig. 3: Plot of n_i and T_e versus pressure. Microwave input power was set at 600 W with probe tip located 1 cm in front of the wafer holder.

Fig. 4: Plot of experimental radial n_i profiles with and without the wafer holder at pressure = 1 mtorr, $P_{forward}$ = 600 W.

Fig. 5: Design for two-level factorial experiment.

Fig. 6: Plot of etch depth versus time. SF_6 gas at 1 mtorr and $P_{forward}$ = 800 W.

Fig. 7: a) Plot of tungsten etch rate and photoresist selectivity versus microwave power. b) Tungsten and photoresist etch rate. Total pressure = 1 mtorr; gas mixture = SF_6/10%O_2 with wafer holder grounded.

Fig. 8: a) Plot of tungsten etch rate and photoresist selectivity versus RF bias. b) Tungsten and photoresist etch rate. Pressure = 1 mtorr (SF_6/10% O_2); $P_{forward}$= 800 W.

Fig. 9: a) Plot of tungsten etch rate and photoresist selectivity versus pressure. b) Tungsten and photoresist etch rate. $P_{forward}$ = 800 W with waferholder grounded.

Fig. 10: a) Plot of etch rate and photoresistselectivity versus percent oxygen at different pressures. b) Tungsten and photoresist etch rate. $P_{forward}$ for both cases was 800 W with wafer holder grounded.

Fig. 11: SEM photograph of tungsten structure at various RF bias.

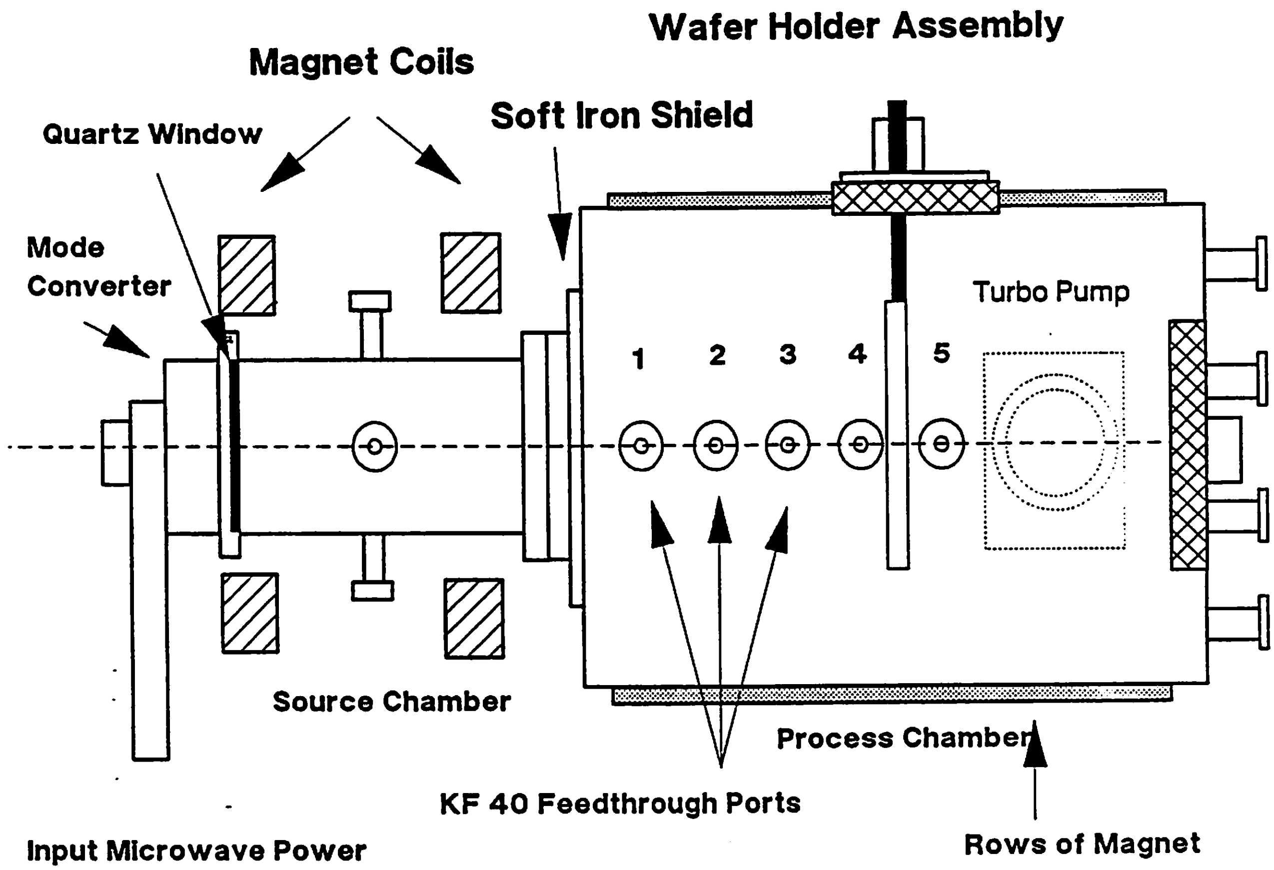
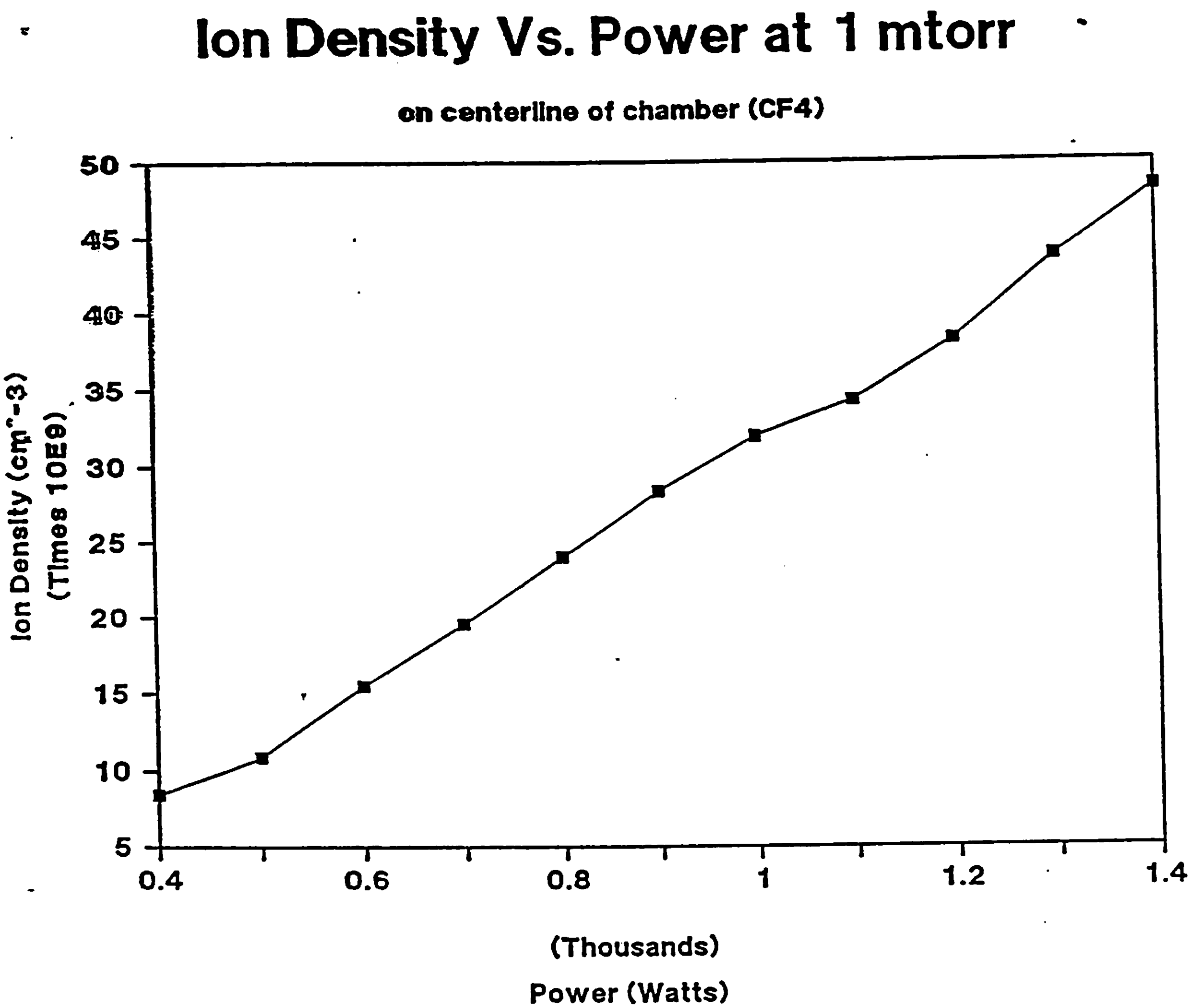

Wafer Holder Assembly
Magnet Coils
Soft Iron Shield
Quartz Window
Mode
Converter
Turbo Pump
1
2
3
4
5
Source Chamber
Process Chamber
KF 40 Feedthrough Ports
Input Microwave Power
Rows of Magnet

Figure 1



Ion Density Vs. Power at 1 mtorr
on centerline of chamber (CF4)
Ion Density (cm^-3)
(Times 10E9)
50
45
40
35
30
25
20
15
10
5
0.4
0.6
0.8
1
1.2
1.4
(Thousands)
Power (Watts)

Figure 2

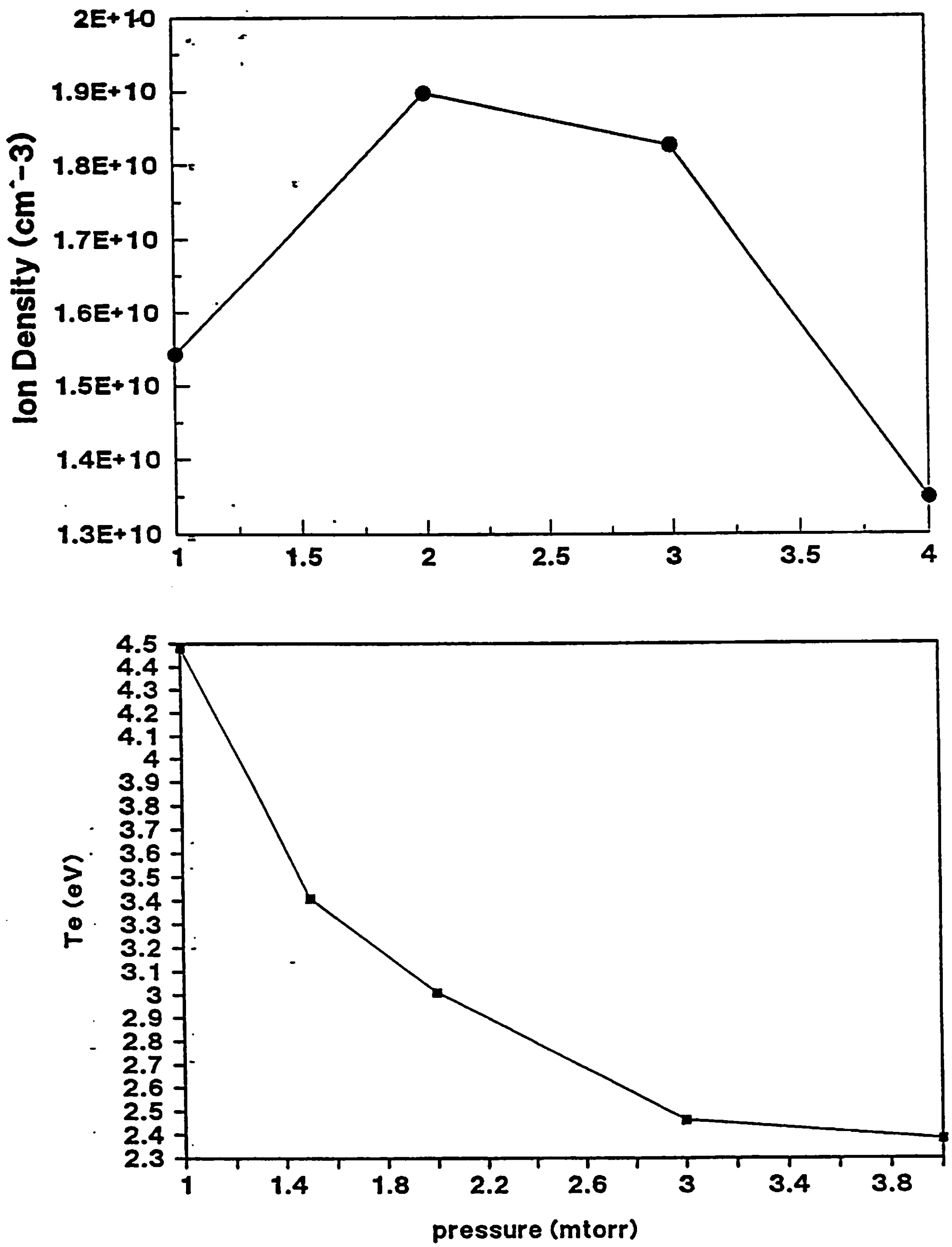

Ion Density (cm^-3)
2E+10
1.9E+10
1.8E+10
1.7E+10
1.6E+10
1.5E+10
1.4E+10
1.3E+10
1
1.5
2
2.5
3
3.5
4
Te (eV)
4.5
4.4
4.3
4.2
4.1
4
3.9
3.8
3.7
3.6
3.5
3.4
3.3
3.2
3.1
3
2.9
2.8
2.7
2.6
2.5
2.4
2.3
1
1.4
1.8
2.2
2.6
3
3.4
3.8
pressure (mtorr)

Figure 3

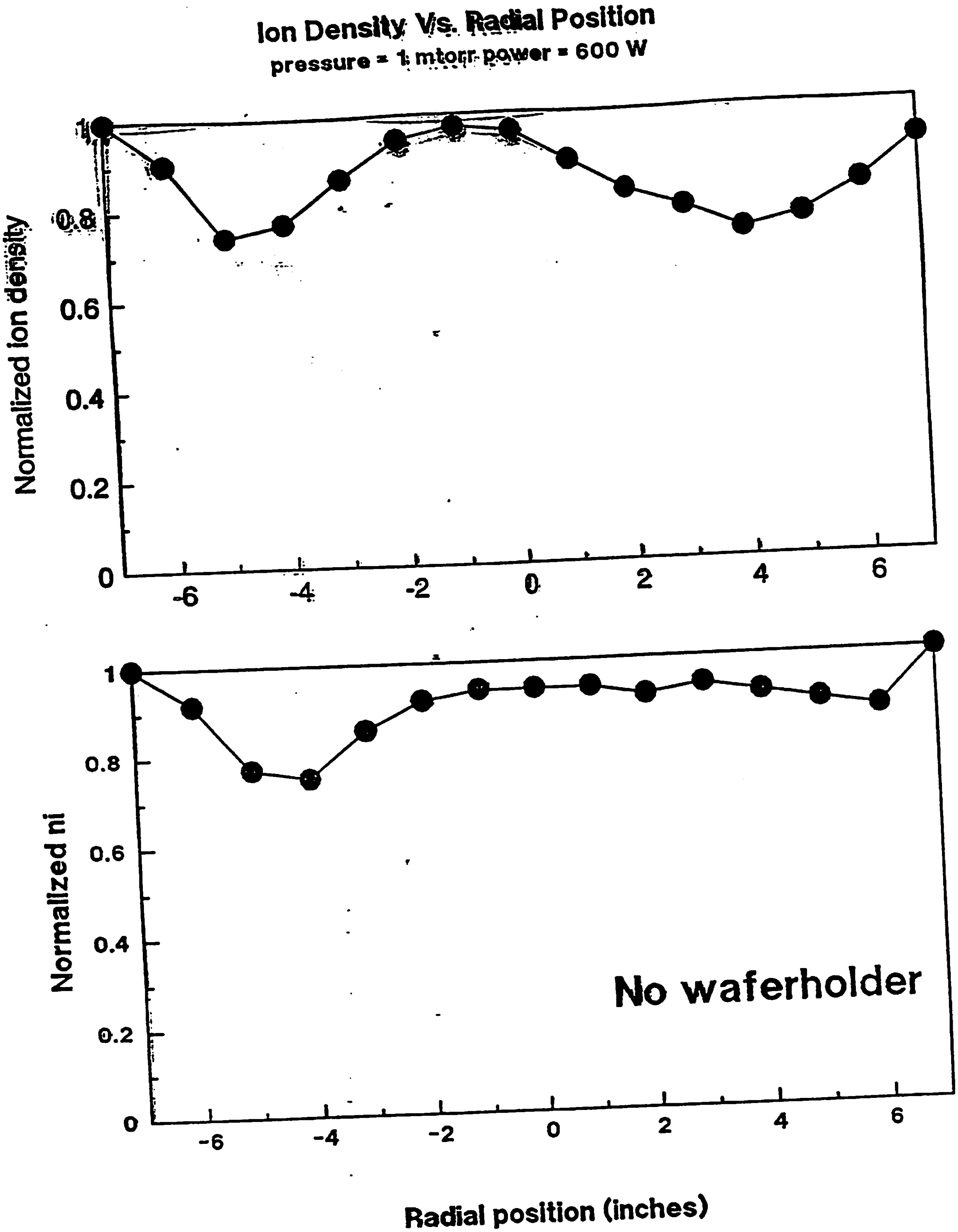
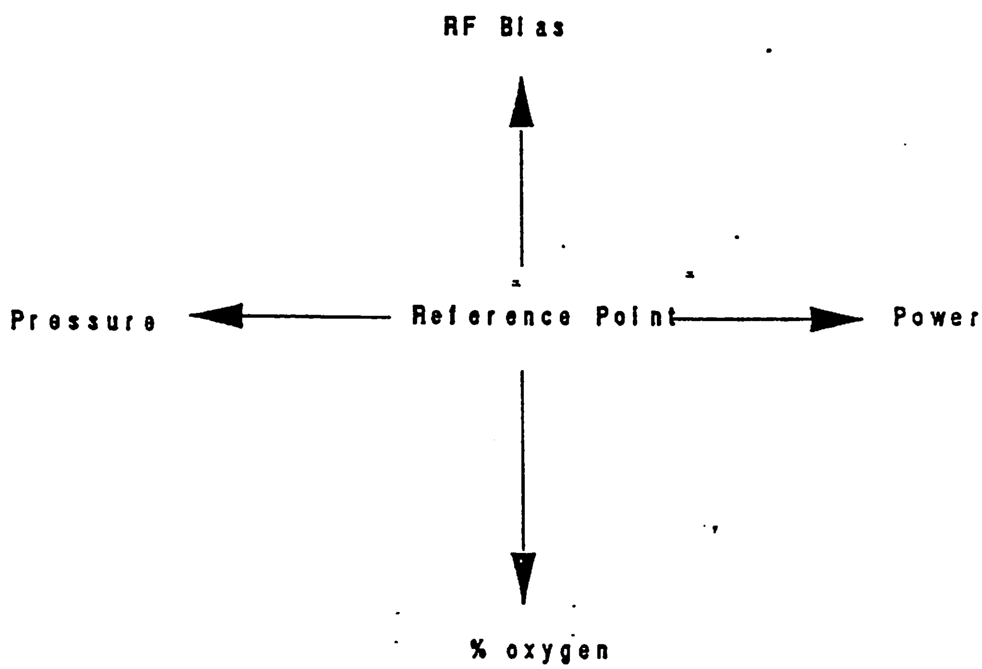

Ion Density Vs. Radial Position
pressure = 1 mtorr power = 600 W
Normalized ion density
0
0.2
0.4
0.6
0.8
1
-6
-4
-2
0
2
4
6
Normalized ni
0
0.2
0.4
0.6
0.8
1
No waferholder
-6
-4
-2
0
2
4
6
Radial position (inches)

Figure 4

Tungsten Etch Results in SF_6/O_2 Plasma

■ Factorial experiment



■ Reference point (variation)

Power = 800 W (± 200)

10% oxygen (± 10)

Pressure = 2 mtorr (± 1)

No bias (delta = -20)

Figure 5

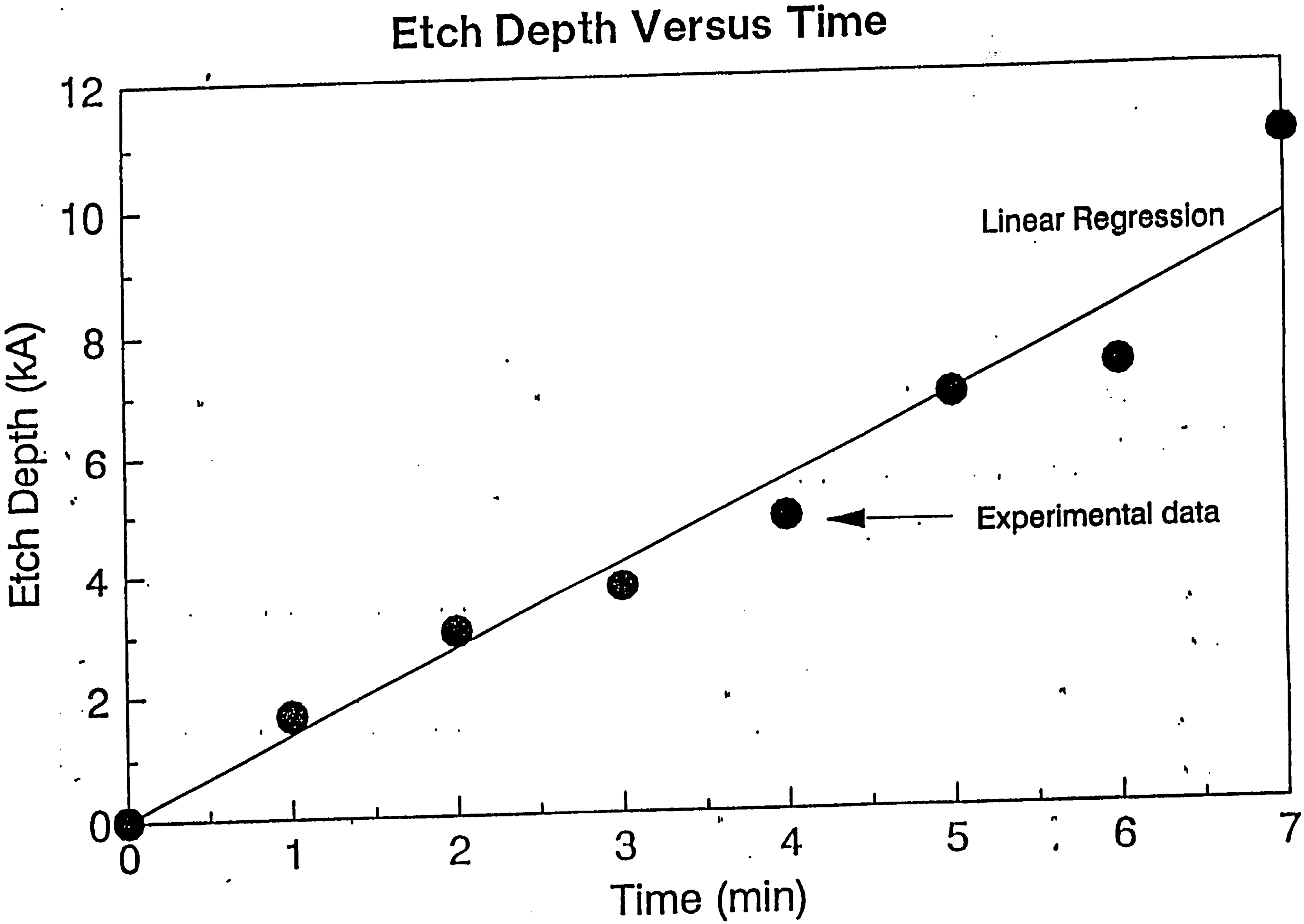

Etch Depth Versus Time
Linear Regression
Experimental data
Etch Depth (kA)
Time (min)
0
2
4
6
8
10
12
0
1
2
3
4
5
6
7

Figure 6

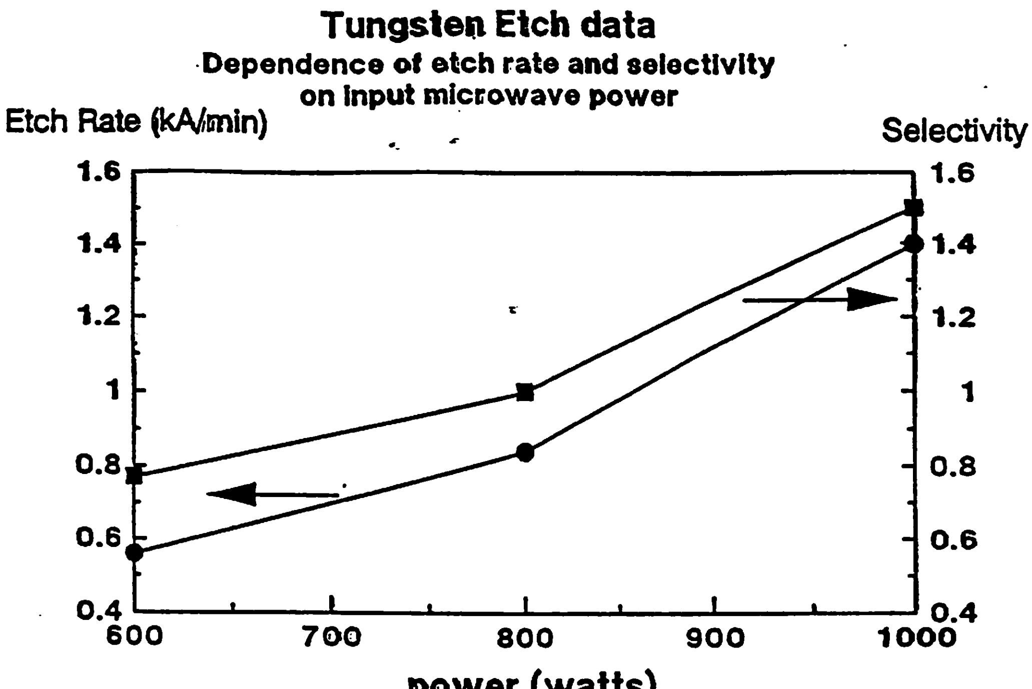


Figure 7a

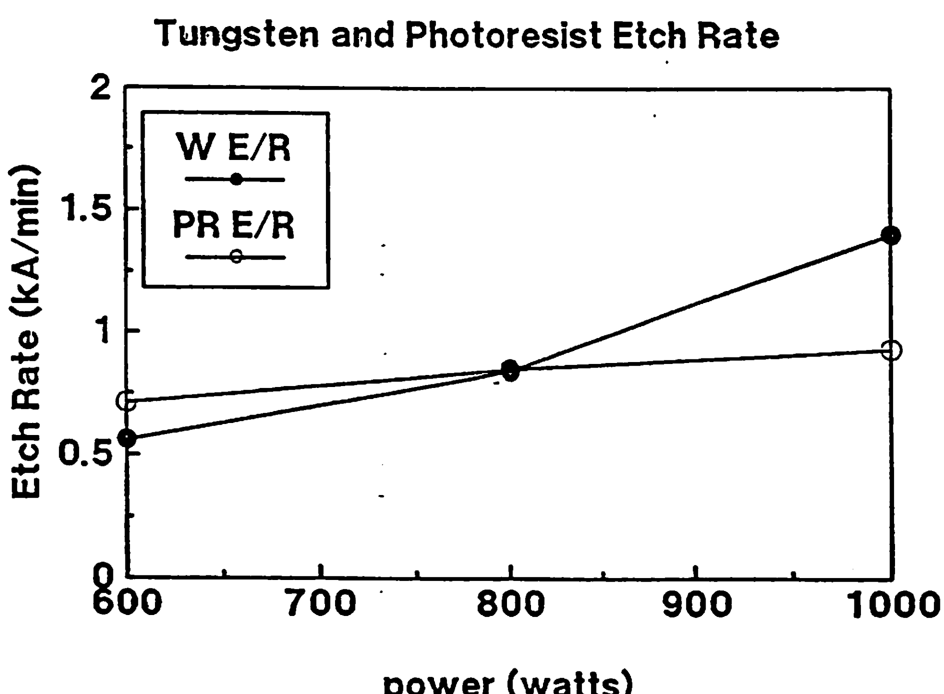


Figure 7b

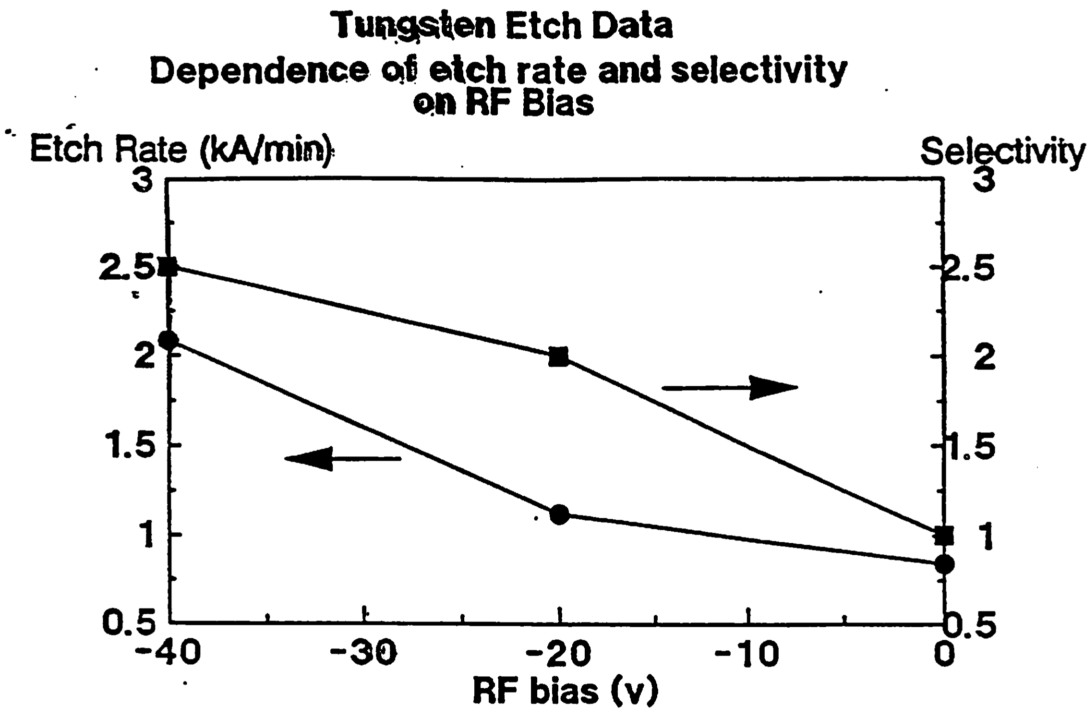


Figure 8a

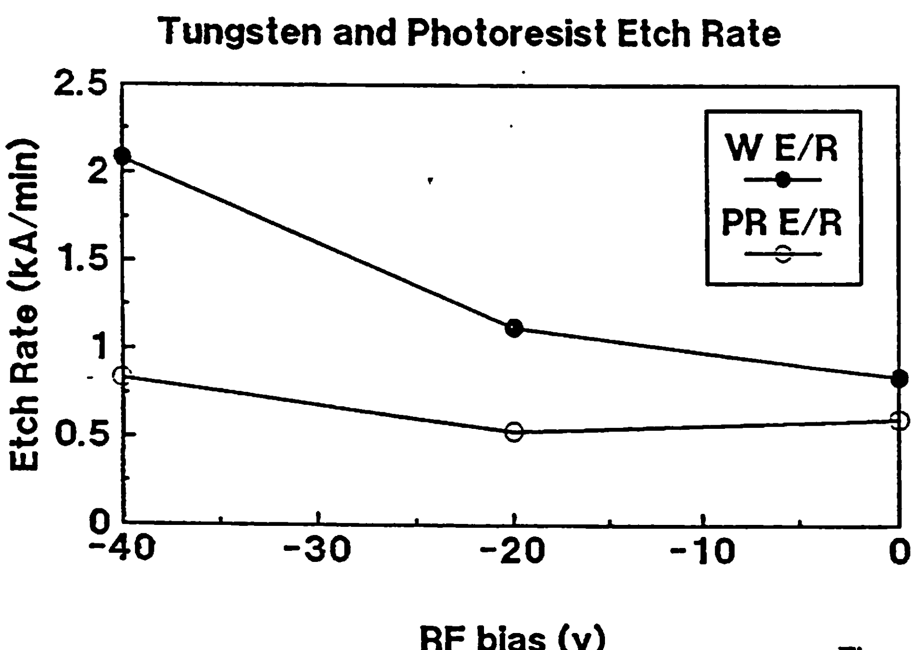


Figure 8b

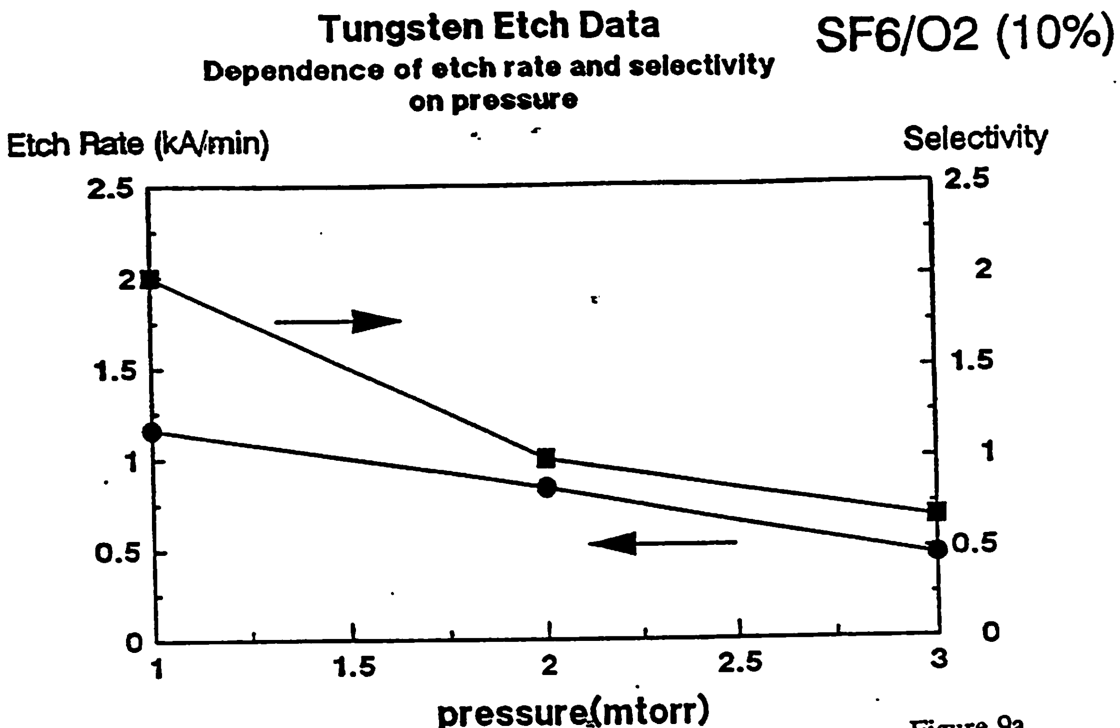


Figure 9a

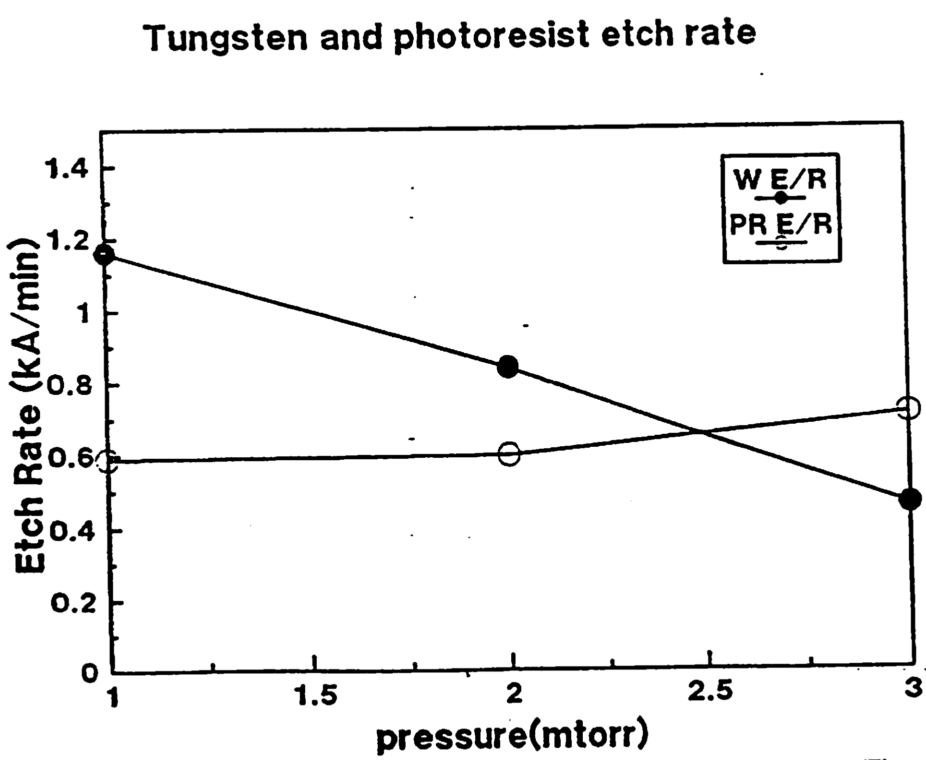
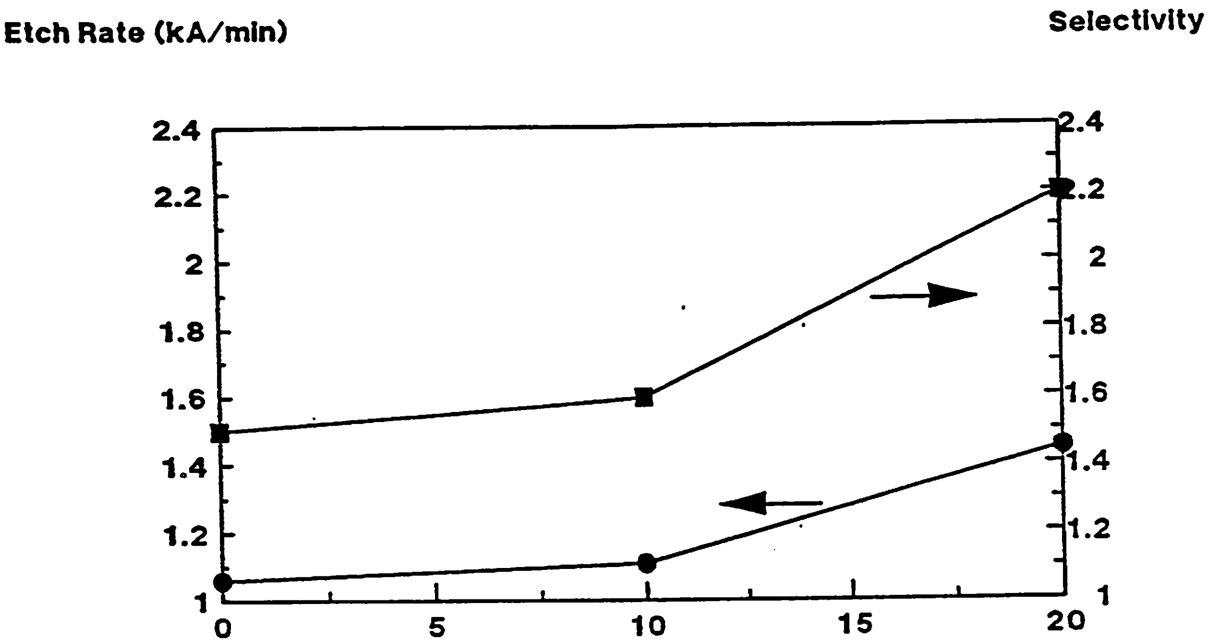


Figure 9b

Tungsten Etch Data

Dependence of etch rate and selectivity on oxygen concentration (pressure = 1 mtorr)



Pressure = 2 mtorr

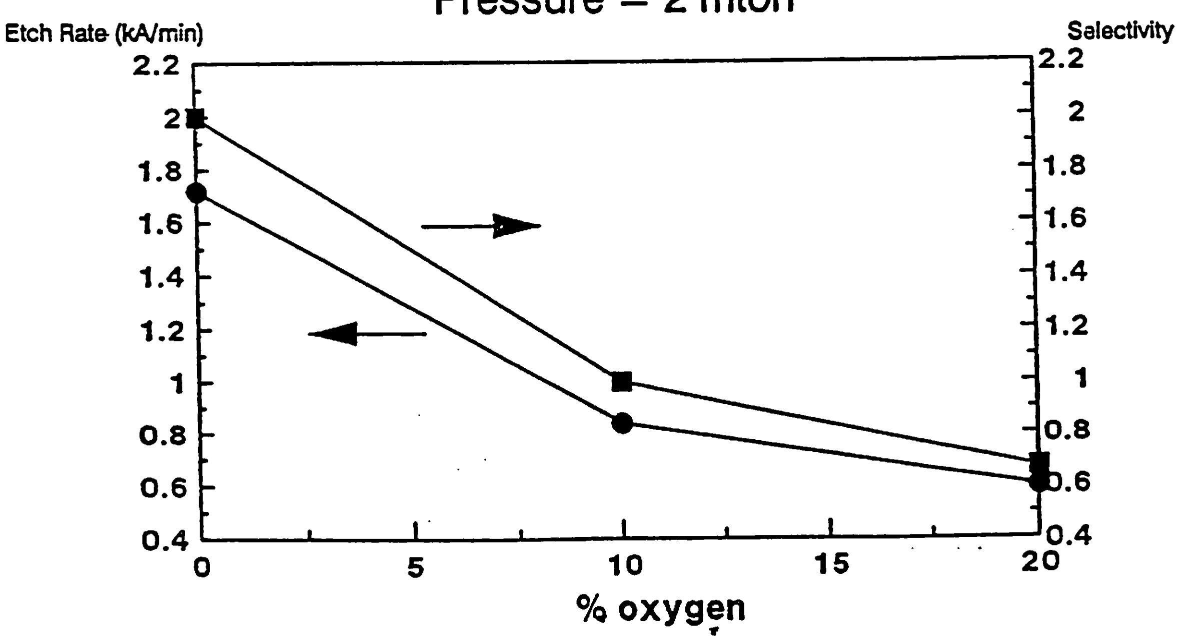
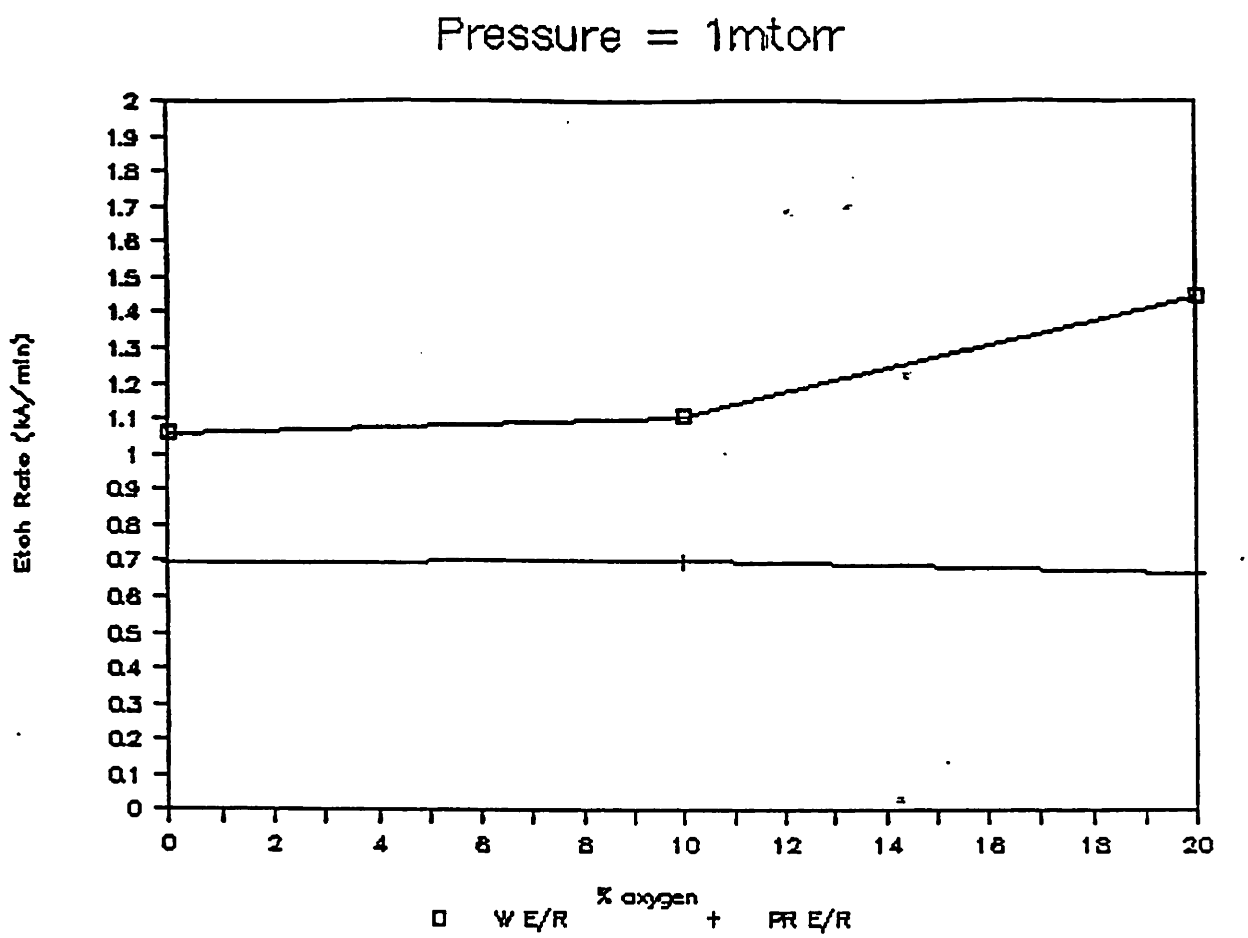


Figure 10a


Pressure = 1mtorr
Etch Rate (kA/min)
% oxygen
W E/R
PR E/R

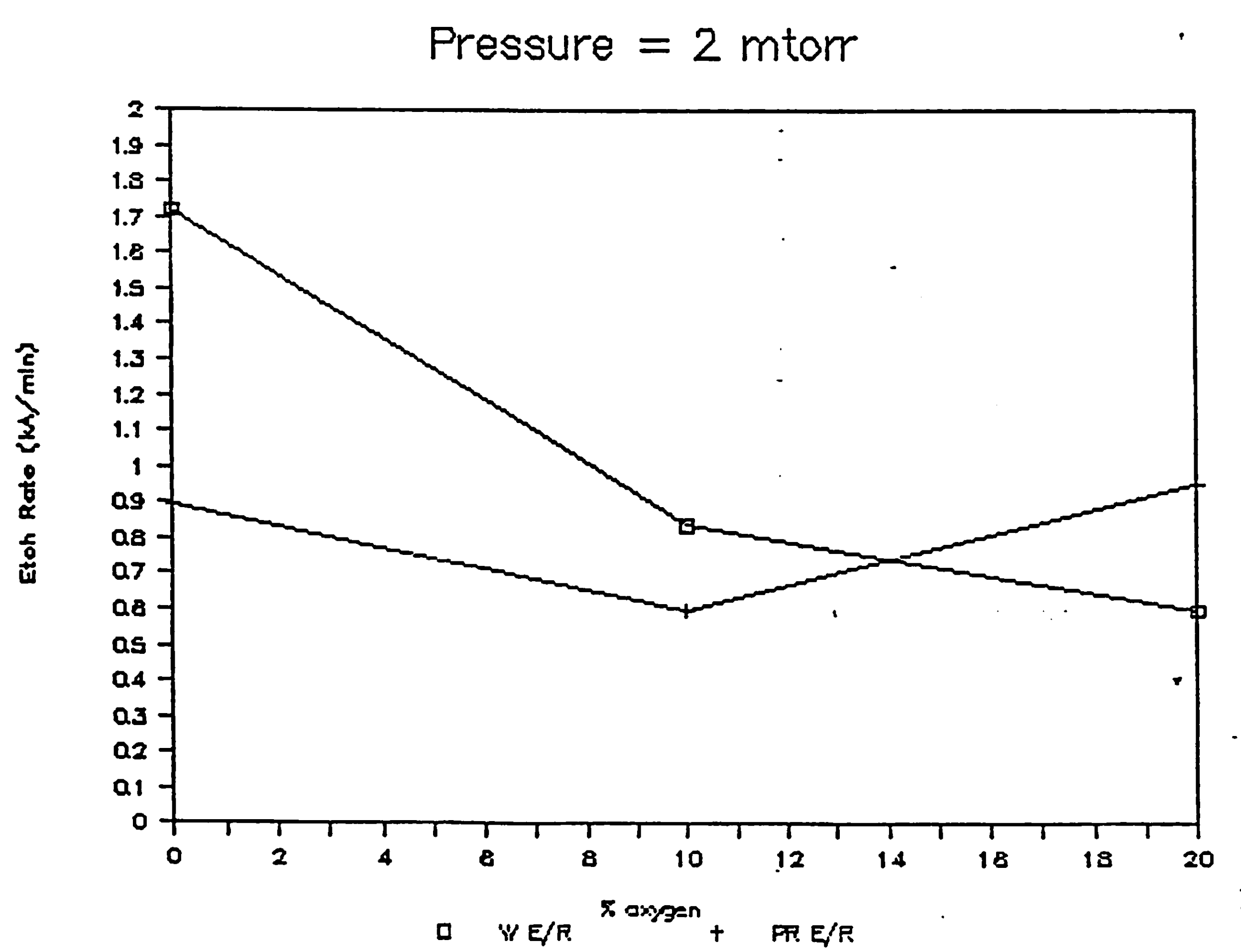
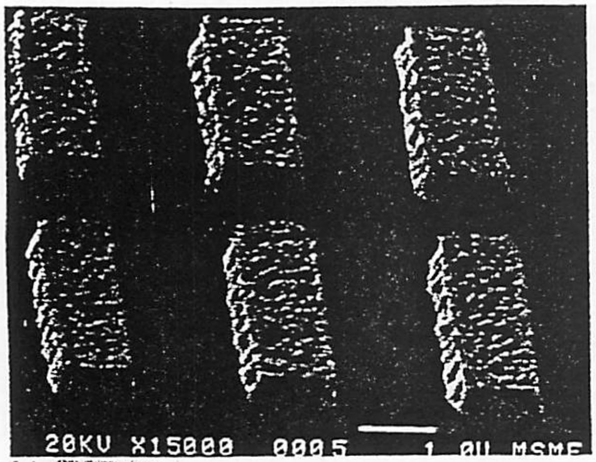
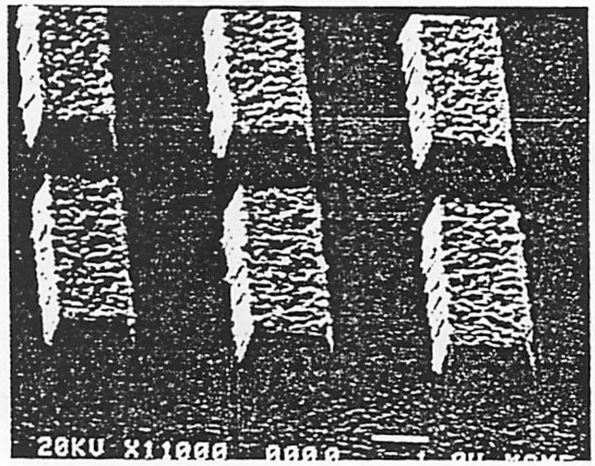

Pressure = 2 mtorr
Etch Rate (kA/min)
% oxygen
W E/R
PR E/R

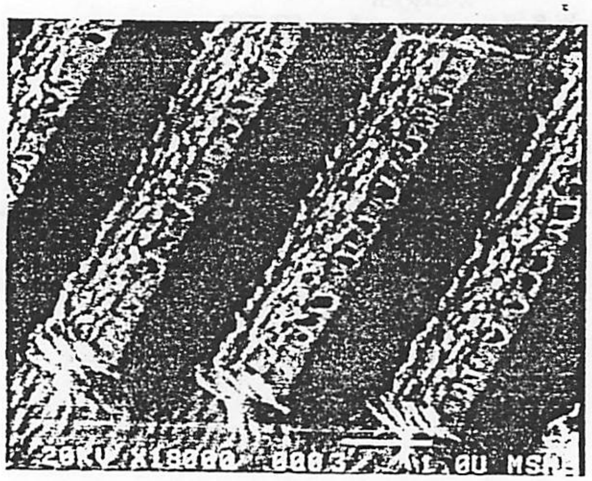
Figure 10b



wafer bias = -10 V



wafer bias = -20 V



wafer bias = -60 V

SF6/O2 plasma

Figure 11