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REACTIVE ION ETCHING OF POLYIMIDE FILMS
USING A RADIO FREQUENCY DISCHARGE

JAMES G. FAGAN

SEPTEMBER, 1987

THESIS
SUBMITTED IN PARTIAL FULFILLMENT OF THE
REQUIREMENTS FOR THE DEGREE OF MASTER OF SCIENCE

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REACTIVE ION ETCHING OF POLYIMIDE FILM
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A very special thanks goes to a secretary whom has been gifted with extreme patience, as demonstrated by her skill in typing this thesis without physically harming me due to all my revisions, modifications and misspellings. Thank you Betty! We'll do lunch!
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1.0. INTRODUCTION

The use of Radio Frequency (RF) plasmas containing oxygen \( \text{O}_2 \) for etching organic films has been investigated previously \([1-6]\). When etching of organic films is conducted on the powered electrode of a RF plasma reactor it is possible for the ions to interact with the internal and external electrical fields of the discharge and cause ion bombardment of the film's surface. This effect leads to etching which has been termed reactive sputter etching, RSE \([7]\), or reactive ion etching, RIE \([8]\), due to the combined effects of ion bombardment and chemical reactivity of the various plasma species \([9,10]\). The RF system used for the work reported herein was designed to allow for such reactive ion etching.

The plasma etching of polyimide (PI) films is of interest in the fabrication of VLSI and other integrated circuits due to its potential for use as an insulating layer and planarization material with high temperature applications \([2,6,11]\). The use of oxygen plasmas in conjunction with dopant gases to enhance production of atomic oxygen, such as tetrafluoromethane \( \text{CF}_4 \) and sulfur hexafluoride \( \text{SF}_6 \), have been investigated in PI etching \([1,2,12]\) as well as plasmas containing pure oxygen \([4,13,14]\). The behavior of PI during RIE in the presence of an oxygen plasma was seen to relate to the emission
ratio intensity of [O atomic] / [O$_2$$^+$ ion] within the
discharge, as determined by emission spectroscopy. The lower
the O atom/O$_2^+$ ion emission intensity ratio was shown to
correlate to a lowering of the resultant etch rate and degree
of directionality in etching [13, 14]. The work of Egito et
al [1] and Matuszak [12] have shown that whether ions are
present or not during etching of PI the etch rate is
dependent upon the O to F atomic ratio ([O]/[F]) within an
O$_2$-CF$_4$-Ar discharge. The etch rate was maximized at an
optimum [O]/[F] ratio near the PI film determined by in situ
emission spectrographic analysis of the plasma.

The mechanism for plasma etching of PI is complex. It,
as above, has been associated with the presence of atomic
oxygen and fluorine, if present in the proper concentration
[15]. Additionally, the effects of the ionic and neutral
species within a RF plasma are seen to have influence on the
rate and type (degree of anisotropy) of resultant etching
[10,13]. This is exemplified by the decrease in PI etch rate
without the presence of ion bombardment [1]. This is not
unexpected since ion bombardment of polymeric films, such as
PI, is thought to facilitate the generation of reactive
surface sites which thereby enhances the etch rate [1,16-18].

The positive ions within a RF discharge play a major
role in the ion bombardment in a RIE configuration. The
negative ions within the RF discharge are not expected to
play a direct role in the etching process since the plasma potential is usually most positive with respect to all surfaces in contact with the plasma [19]. Therefore negative ions are not able to reach the surfaces unless they are imparted with a substantial amount of kinetic energy [20]. The energy of positive ions in the plasma are effected by both the pressure of the discharge and the presence of induced or applied electric fields.

The difference in the low mobility of ions as compared to that of electrons in response to the alternating electric field of a RF discharge induces self biasing of the target electrode, also referred to as the DC offset voltage (|Vdc|) [21]. This self biasing can be effected by the geometric asymmetry of the RF reactor electrode design as shown by Coburn [20] and Koenig [22]. The more asymmetric a system is with respect to the target electrode, the greater the |Vdc| becomes. This imparts greater energy to ions transversing the target sheath. Increasing the voltage asymmetry is also seen to increase the amount of residence time for positive ion bombardment the biasing electrode will experience in a given RF cycle [21]. This asymmetry effect is often used in RIE system design [8,21,23].

The bias voltage within a RF plasma will also be influenced by the discharge pressure. The bias voltage, |Vdc|, is seen to decrease with increasing pressure above
0.01 torr at a constant input power as noted for etching Si and SiO$_2$ in RIE systems [23,24]. The decrease can be explained by the increased number of collisions and recombinations an ion will incur while moving to the target at higher pressures, thereby reducing the ion density [13,21,25]. Fortuno [26] has shown the target sheath voltage in RIE of SiO$_2$ to decrease with increasing pressure. The target sheath voltage was reported as the average of the resultant of the plasma and dc offset potentials. The drop in sheath potential with pressure can also be explained by the increase in the number of collisions within the discharge and sheath at higher pressures, thereby reducing the average ion energy [13].

Both the $|V_{dc}|$ and peak to peak RF voltage ($V_{pp}$) as well as the input power are often used to characterize RF sputtering processes [27,28]. Recently Horwitz [23] reviewed such parameters as they apply to RIE and showed that the relative ion bombardment energy ratio, as defined by $|V_{dc}/V_{pp}|$, trends in a manner similar to that of $V_{dc}$ with pressure for RF sputtering/sputter-etching at a constant power. This ratio indicated the relative amount of ion bombardment energies which will strike the target as compared to the chamber within a RIE system. The highest ratio being 0.5 for completely asymmetric systems. Determining the relation between ion bombardment energies at the two electrodes within a RIE system in this way
results in a simple method of analysis as compared to the methods employing Langmuir probes and retarding-field analyzers [23]. This results in a simplified means for gaining qualitative information regarding the role of ion bombardment in a RIE process.

The etch rate of PI can also be effected by vessel geometry and linear gas flow velocity, (which is affected by the flow rate, pressure and vessel geometry, for a given plasma etching process. A systems geometry plays an important role in the voltage symmetry of a RF system as well in the power densities within the discharge [28, 29]. Vukanovic et al [30] has examined the effect of linear gas flow velocity (Vr) when etching PI with the absence of ions and has found at increasing linear dependence of etch rate on Vr from approximately 300 to 700 cm/min at 0.3 torr.

The activation energy for etching photoresist has been shown to depend upon gas composition [12]. The activation energy (Ea) in plasmas containing pure oxygen, 0.048% C2F6 in O2 and 0.81% C2F6 in O2 without the presence of ion bombardment were reported at 0.5, 0.3 and 0.13 eV/mole respectfully [31]. Other investigators have reported the Ea for etching polyimide to be 0.14 ± 0.03 eV [30] for O2 - CF4 - Ar containing plasmas in a downstream etching system. In addition Battey [3] has reported the Ea for an organic photoresist in a pure oxygen RF plasma to be 0.28 eV/mole.
In summary, previous work has shown the etch rate of PI and other organic films to be influenced by the presence of ions [1,17] and pressure [2,5,11,14,17] for RIE. Other investigations have shown that the etch rate of Si and SiO₂ to vary in accordance with the various plasma voltages: Vpp, Vdc, and the target sheath voltages as well as pressure [23,24,26]. Additionally, factors such as linear gas flow velocity, geometry and discharge gas composition are seen to influence PI etching [1,12,30].

The intention of this work is to investigate the influence of ions (ion bombardment) on the etch rate of PI in a RF discharge as pressure is varied. In order to examine the effect of ions on PI etching the resultant plasma potentials of Vdc and Vpp shall be monitored as pressure is varied. This will allow a characterization of the ion energy, relative discharge voltage asymmetry and relative ion bombardment energies between the target and chamber electrodes of the discharge. Such a characterization enables the evaluation of the various factors impacting ions in a RF discharge and their effect on PI etching. The influence of linear gas flow velocity and activation energy for etching of PI in the presence of ions will also be examined.
2.0. EXPERIMENTAL

2.1. Radio Frequency Etching System

The radio frequency (RF) system used for polyimide (PI) etching experiments consisted of a Plasma Therm HFS 3000E three kilowatt RF generator, Perkin Elmer Randex Model 3140 impedance matching network, reaction chamber, electrode assembly and vacuum system to be detailed in the following experimental section. The etching system is shown schematically in Figure 1.

The plasma generator was manually tuned at 13.56 MHz in accordance with the Plasma Therm operating procedures [32] using a 50 ohm resistive load, HeathKit Canteena. The generator's power cable was attached to the power receiver bulk head on the matching network powering the RF chamber's electrode. The impedance matching network allowed tuning of the RF discharge by a variable inductor and variable capacitor as shown in Figure 2. The matching network provided the capability of matching the generator's output impedance by combining the RF discharge load with the variable matching network load (21). Impedances were matched by adjusting both the variable inductor and capacitor on the matching network and the tuning and load controls on the RF generator. Tuning adjustments were made by maximizing incident power at 100 watts while minimizing
Figure 1
Schematic of the Radio Frequency etching apparatus.
Captions For Figure 1

CH  Chart Recorder (Varian G-1100)
DP  Diffusion Pump (NRC Type 184)
EL  Power Electrode and Shield
F   Mass Flow Controllers (Tylan FC260)
FV  Foreline Valve
HV  High Vacuum Valve
La  Laser (Metrologic ML 840 He/Ne 3.0mW)
LD  Laser Detector (Metrologic 45-540 Digital Power Meter)
MN  Matching Network (Perkin-Elmer 3140)
MP  Mechanical Pump (Balzers DUO 35)
OSC Oscilloscope (Tektronix Type 561B)
PM  Capacitance Manometer (MKS Baratron 310, 1 Torr Head) with a 170-27D Digital Readout
PS  Radio Frequency Power Supply (Plasma Therm HFS 3000 E)
RV  Roughing Valve
S   Substrate
TC  Thermocouple Gauge Tube
    (TC1) Roughing Line Pressure
    (TC2) Foreline Pressure
    (TC3) Chamber Pressure
T   Fluoroptic Thermometer
W   Window for Laser Etch Rate Monitoring
MS  Molecular Sieve Trap
Figure 2

Schematic of the Radio Frequency matching network.
reflected power to ≤5 watts as monitored on the incident and reflected power meters of the RF generator.

The reaction chamber used in the RF etching experiments of PI films was assembled to gain high linear gas flow rates. The chamber was constructed with two stainless steel collars, of the dimensions 20" I.D. by 10" wide, each containing two 8" diameter access ports and two 22" diameter aluminum cover plates as shown in Figure 3. To achieve high linear gas flow velocities a 7" long by 3" I.D. Pyrex conical cylinder was extended downward from the chamber cover plate inside of which the electrode assembly was inserted from the chamber base plate. The electrode assembly extended to within 3 7/8" of the chamber cover plate. The reaction chamber configuration is detailed in Figure 3.

Gas was fed to the chamber through a port in the cover plate above the electrode as in Figure 3. Pressure was also monitored during discharge experiments above the electrode through an additional port in chamber cover plate. The chamber's cover plate was raised and lowered by the use of a mechanical hoist (see Figure 4) to access the chamber and electrode between experiments. The chamber cover plate, base plate and access ports vacuum seals were made using Vitron o-rings.

The electrode assembly shown consisted of three parts: electrode shield, electrode/conductor, water
Figure 3
Radio Frequency electrode configuration.
Figure 4

Photograph of Ar discharge in RF system. Substrate raised into glass cylinder that is attached to the top of the chamber.
Figure 5
Vacuum system for RF etching apparatus.
cooling system. First is the electrode shielding that was constructed of aluminum. The electrode shield was built in two sections allowing ease in assembly and flexibility in future electrode design changes. The lower section of this shield was bolted to the base plate while the upper shield section threaded on to the lower. Each section was machined to a 2 1/4" O.D. with an inner dimension conforming to the electrode shape with a 1/8" gap between the two. This gap size between the shielding and electrode avoided the generation of discharges outside of the upper electrode surface and the potential for shorts.

The electrode consisted of a machined brass electrode, 2 1/4" diameter x 1" width, connected to a 3/8" copper conductor by a brass Swagelock fitting. The copper conductor functioned as both power feed and part of a water cooling system for the electrode. The conductor was encased by a steel tube with porcelainized interior capped at the ends with a teflon machined part providing locations for Vitron o-ring vacuum seals. The conductor assembly was fed into the chamber through a Cajon fitting (Ultra Torr B-16 UT6) that provided a mechanical lock and vacuum seal for the electrode vacuum feed through, in addition to a series of o-ring seals (1" I.D. x 1/8") within the base plate.

The conductor was electrically connected in the matching network by a 3/4" O.D. x 3/8" I.D. x 2" long brass
coupling placed on the copper conductor and fitted into a brass power feed receptacle. The coupling and power feed receptacle were mechanically attached by the use of brass set screws.

The electrode and copper conductor also had the capability to function as a closed loop water cooling system in conjunction with a refrigerated water bath. A 1/8" stainless steel tube was inserted into the 3/8" copper conductor allowing both a water feed and return for the electrode. The electrode water cooling system is schematically shown in Figure 6. This capability was however not used in the experiments discussed in this paper.

2.2 Vacuum System

The vacuum system was connected to the RF reaction chamber mentioned above by the use of a 8" I.D. flanged throttle valve to one of the chamber’s upper collar access ports. This system is shown schematically in Figure 1 as well as pictorially in Figures 4 and 5.

The vacuum pumping system consisted of a NRC Type 184 8" water cooled oil diffusion pump and Sergent-Walsh Model 1397 mechanical pump. Due to the use of oxygen in the experiments the mechanical pump was refitted with new seals, gaskets, o-rings and filled with a perfluorinated
Figure 6

Schematic of RF electrode water cooling design.
(Fomblin) pump fluid. The perfluorinated fluid was used in order to avoid the detrimental polymerization reactions which may occur to hydrocarbon based pumps oils when exposed to oxygen [12]. A molecular sieve trap was placed on the roughing line before the mechanical pump, as shown in Figure 1. This trap was used in order to avoid contamination from backstreaming vapors out of the mechanical pump into the vacuum system.

The vacuum system's air actuated valves (roughing, foreline, and high vacuum) were controlled by the use of a Davis and Wilder valve sequence controller, Model 4035. The throttle valve mentioned above was manually operated when varying pumping speeds were required during the experiments. A liquid nitrogen (LN₂) cold baffle was placed on the diffusion pump stack as in Figure 1. The LN₂ cold baffle fill level was controlled by a Model 100L Cryotrol controller. Stainless steel flange type fittings and piping along with Vitron o-ring seals and gaskets were used for this vacuum system.

2.3 Pressure Measurement

The reaction chamber pressure was measured by the use of a Type 310-BHS-1 Torr MKS Baratron capacitance manometer pressure sensor in conjunction with a MKS Baratron 170-M-27D series digital readout for pressures between
$10^{-4}$ and 1.0 Torr. For pressures of 1.0 to 10 Torr a MKS Baratron LD1 gauge was used with a MKS Baratron Type 222-BA-00010AB capacitance manometer sensor. The Baratron 310-BHS-1 Torr sensor was maintained at 45°C by internal heaters with a DC proportional controller to minimize the effect of ambient air temperature changes. The capacitance manometer sensors were connected to the upper part of the reaction chamber, as indicated in Figure 3, by the use of 1/4" stainless steel Swagelock fitting threaded into reaction chamber top plate.

Foreline and roughing line pressures between 1 and $10^{-3}$ Torr were monitored with Kurk J. Lesker (KJL) thermal couple gauges and sensors of the type KJL 1518 and KJL Model 200 respectfully. A Varian NRC 524-2 cold cathode ionization gauge connected to a NRC-855-A gauge controller was used to measure pressures of $10^{-9}$ to $5 \times 10^{-3}$ Torr. This gauge was placed between the high vacuum valve and the liquid nitrogen cold baffle of the diffusion pump. A vacuum seal for this gauge was attained by the use of o-ring compression adaptor.

2.4 Temperature Measurement

Substrate temperature was monitored during etching experiments by a fluoroptic temperature measurement system. This system consisted of a Luxtron Model 1000A/A
microprocessor-control instrument and a Luxtron Type LIG optic fiber probe. The use of this probe allowed a range of temperatures from \(-65^\circ C\) to \(240^\circ C\) to be measured with an accuracy of \(\pm 1^\circ C\).

The optic fiber probe was fed into the reaction chamber through a Swagelock vacuum seal fitting which was threaded into the cover plate of the chamber’s lower collar access port. The probe was the inserted through the electrode shielding and the electrode itself until contact was made with the bottom of the PI coated substrate as shown in Figure 3. The probe was held in contact during experiments by adjusting and tightening the probe's vacuum feed through.

Fluoroptic thermometry is based upon the light-emitting properties of a rare earth photoluminescent phosphor which varies in a reproducible manner with changing temperature [33]. In our application this phosphor sensor was placed at the end of the optic fiber probe. This probe was then placed in contact with the substrate to measure its temperature. Due to the low thermal mass and thermal conductivity of the probe, as compared to other thermal couple type probes, such contact temperature measurements are not seen to be adversely effected [34]. Additionally, the fluoroptic probe was constructed with no metallic or conductive components electrically isolating the probe from the surrounding
environment. This allows temperature measurement within a RF discharge environment without adverse effects from RF radiation and field effects.

2.5 Gas Flow Rate

The gas flow rate of Ar, CF$_4$, and O$_2$ gases were controlled by Tylan FC-260 mass flow controllers. A Starret needle valve flow controller was used for O$_2$ gas flow rates greater than 50 sccm. A Tylan RO-20A flow control readout was used for control and monitoring flow rates of 1 to 550 sccm and 10 to 500 sccm for Ar and CF$_4$ gases respectfully. At the lower flow rates of 1 to 10 sccm for Ar and 1 to 50 sccm for CF$_4$ and O$_2$ gases, a RO-1-400 Tylan flow controller/readout was used.

The gas composition used for experimentation was a 75% O$_2$, 20% CF$_4$ and 5% Ar gas mixture. Total gas flow rate was varied between experiments while maintaining the above gas composition. Information regarding the gas flow rates used are detailed in the experimental conditions section (2.8) of this paper. The flow rates of the gases were calibrated using a constant volume technique [12].
2.6 Laser Interferometry

The etch rate of PI was measured with the use of a laser interference method. Measurements were taken by directing a helium-neon laser beam, of a 632.8 nm wavelength, downward onto the PI coated substrate surface at a near normal angle of incidence (Figure 1). The incident and reflected beams passed through a 1" port machined into the chamber cover plate which was covered by a 1 1/4" diameter by 1/8" polished Pyrex window. Vacuum seal was attained by placement of a 1 1/8" diameter by 1/8" Vitron o-ring between the chamber cover plate and the Pyrex window. The laser used for such etch rate measurements was a Metrologic ML 840 He/Ne 3.0 mW laser. A Metrologic photometer was used for monitoring the reflected beam from the substrate during etching. This photometer contained a photodiode connected to an ammeter from which a signal was transmitted to a strip chart recorder for etch rate measurement with time.

The use of interferometry is based upon the variations induced upon the reflected laser beam intensity due to constructive and destructive optical interference produced between the reflected beams from the top and bottom surfaces of the PI film as etching progressed. Using the equation:

\[ d = \frac{\lambda}{2n} \]  

(1)
the amount between occurrence of consecutive maxima (minima), \( d \), can be related to the laser wavelength, \( \lambda = 632.8 \text{ nm} \) and the refractive index, \( n \), of the PI film \( (n = 1.76 \text{ for PI}) \). Therefore, when monitoring the signal intensity, as shown in Figure 7, of the reflected laser beam with time during an etching experiment the distance between adjacent maxima (minima) can be related to film thickness changes using equation 1. This yields a measure of etch rate (units of \( \AA/\text{minute} \)).

2.7 Measurement of Plasma Voltages

The mean plasma potential, \( V_p \), and self bias potential, \( |V_{dc}| \), were measured using a Tektronix Type DM-44-475 Digital Oscilloscope. Potentials were measured between the electrode power feed within the system's matching network and ground. This was accomplished by attaching an electrical lead from the electrode power feed within the matching network to a BNC bulkhead connect exiting the matching network. A coaxial cable, (RG-59/U Type), was connected to the BNC bulkhead and the oscilloscope allowing monitoring of the voltage signal.

The voltage signal from the electrode was attenuated at a 100:1 ratio before entering the oscilloscope. A voltage divider circuit consisting of 10K ohm and 1 M ohm resistors, as shown in Figure 8, was employed to accomplish this attenuation. The attenuation of the voltage signal then
Figure 7

Chart recording of laser interference pattern during RF plasma etching of polyimide.
$R_1 = 1\text{M ohm}$

$R_2 = 10\text{K ohm}$

$V_{IN} = V_{OUT} \cdot \left(\frac{R_1 + R_2}{R_2}\right)$

Figure 8

Voltage Divider Circuit
allowed the final signal to be monitored within the voltage range of the oscilloscope. Resistances of the voltage divider circuit were measured between each experiment to correct the attenuation factor for any component resistance changes.

The voltage signal was monitored with AC and DC coupling modes on the oscilloscope during an experiment to facilitate measurement of the plasma potentials, |Vdc| and Vpp. The oscilloscope was adjusted to allow suitable measurement of both peak to peak potential (Vpp) and |Vdc| at the RF discharge frequency of 13.6 MHz. The |Vdc| was measured from the peak potential difference between the AC and DC coupled signals as received from RF discharge by the oscilloscope. The difference in peak potentials was assumed to equal that of the potential difference between ground and the dc offset target potential V_T, where V ground was assumed to equal zero, as shown in Figure 9. Therefore, the following relationship was used in determining Vdc:

\[
V_{dc} = V_{\text{peak (AC)}} - V_{\text{peak (DC)}} = V_{\text{ground (0)}} - |V_{\text{target}}| \quad (2)
\]

The mean plasma potential (V_p) was calculated from the voltage signal's Vpp and |Vdc| using the equation (3):

\[
V_p = \frac{V_{pp}}{4} - \frac{|V_{dc}}{2} \quad (3)
\]
2V_p \sim \frac{(V_{rf})_{pp} - V_{dc}}{2}

V_p \text{ - Plasma Potential}
V_{dc} \text{ - Self Bias Voltage on cathode electrode}
(V_{rf})_{pp} \text{ - Peak to Peak RF voltage applied to cathode}

Figure 9
Approximate RF cathode and plasma waveforms.
In deriving both $V_{pp}$ and $|V_{dc}|$ the measured oscilloscope potentials were multiplied by the appropriate attenuation factor determined previously.

2.8 Experimental Conditions

For all the experiments reported herein a 100 watt incident power level with a reflected power level of $\leq 5$ watts was used. Table I lists the total gas flow rates, resultant pressures and temperature ranges which were measured for each experiment. The gas compositions for each experiment as determined via the constant volume apparatus are given in Table II. In further discussions the gas composition will be reported as the nominal composition of 75% CF$_4$, 20% O$_2$, 5% Ar.
### TABLE I

Experimental Total Gas Flow Rates, Pressures, Linear Gas Flow Velocities and Temperature Ranges

<table>
<thead>
<tr>
<th>Experiment Number</th>
<th>Total Gas Flow Rate (SCCM)</th>
<th>Pressure (torr)</th>
<th>Temperature Range (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>3</td>
<td>1025</td>
<td>1.697</td>
<td>83 - 136</td>
</tr>
<tr>
<td>22</td>
<td>1072</td>
<td>1.810</td>
<td>73 - 152</td>
</tr>
<tr>
<td>21</td>
<td>1072</td>
<td>1.792</td>
<td>73 - 162</td>
</tr>
<tr>
<td>26</td>
<td>1218.4</td>
<td>2.022</td>
<td>50 - 164</td>
</tr>
<tr>
<td>27</td>
<td>1218.4</td>
<td>2.033</td>
<td>66 - 163</td>
</tr>
<tr>
<td>28</td>
<td>1218.4</td>
<td>2.094</td>
<td>66 - 161</td>
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<tr>
<td>14</td>
<td>502.6</td>
<td>0.946</td>
<td>44 - 166</td>
</tr>
<tr>
<td>15</td>
<td>502.6</td>
<td>0.951</td>
<td>98 - 136</td>
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<td>9</td>
<td>67.1</td>
<td>0.207</td>
<td>39 - 83</td>
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<td>67.1</td>
<td>0.209</td>
<td>69 - 88</td>
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<td>5</td>
<td>62.6</td>
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<td>50 - 79</td>
</tr>
<tr>
<td>7</td>
<td>53.5</td>
<td>0.199</td>
<td>33 - 82</td>
</tr>
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<td>6</td>
<td>53.5</td>
<td>0.195</td>
<td>54 - 70</td>
</tr>
<tr>
<td>11</td>
<td>50.4</td>
<td>0.188</td>
<td>70 - 81</td>
</tr>
<tr>
<td>12</td>
<td>50.4</td>
<td>0.190</td>
<td>53 - 80</td>
</tr>
<tr>
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<td>50.4</td>
<td>0.180</td>
<td>57 - 93</td>
</tr>
<tr>
<td>23</td>
<td>13.62</td>
<td>0.105</td>
<td>67 - 90</td>
</tr>
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<td>13.45</td>
<td>0.104</td>
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</tr>
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<td>13.45</td>
<td>0.102</td>
<td>56 - 70</td>
</tr>
<tr>
<td>Experiment Number</td>
<td>Gas Composition (% Total SCCM)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>-------------------</td>
<td>-------------------------------</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>$O_2$</td>
<td>$CF_4$</td>
<td>$Ar$</td>
</tr>
<tr>
<td>24</td>
<td>75.1</td>
<td>19.7</td>
<td>5.2</td>
</tr>
<tr>
<td>25</td>
<td>75.1</td>
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</tr>
<tr>
<td>5</td>
<td>73.7</td>
<td>21.0</td>
<td>5.3</td>
</tr>
<tr>
<td>9</td>
<td>73.3</td>
<td>19.8</td>
<td>4.9</td>
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<td>74.6</td>
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<td>74.6</td>
<td>20.4</td>
<td>5.0</td>
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<td>75.3</td>
<td>19.8</td>
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<td>5.0</td>
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</tr>
<tr>
<td>27</td>
<td>75.0</td>
<td>20.0</td>
<td>5.0</td>
</tr>
<tr>
<td>28</td>
<td>75.0</td>
<td>20.0</td>
<td>5.0</td>
</tr>
</tbody>
</table>
3.0 RESULTS AND DISCUSSIONS

3.1 The Effect of Pressure and Time on the Substrate Temperature

The temperature of the PI substrates during etching experiments was seen to increase with elapsed time. Figures 10 and 11 show the increase in temperature for pressures of ~0.1 torr (Experiments 24, 25) and ~1.0 torr (Experiments 14, 15) respectfully. Other additional figures showing the PI substrate temperature as a function of time for the remaining experimental pressures are given in Appendix I. These figures have shown that the temperature increase has a linear dependence with time at a given pressure during PI etching.

Table III contains data of the change in temperature with time (ΔT/Δt) for all the experimental PI etching pressures examined. The ΔT/Δt was determined by the slope of a least squares linear regression of the experimental temperature time data. The error reported was associated with the 95% confidence interval about the slope of a given regression. The correlation coefficients for all the experiments were above 0.92 with the majority above 0.98 at the higher pressures > 0.2 torr, see Appendix II.

Figure 3 reveals the experimental relationship found between the temperature rise with time and pressure for the
TABLE III

Resultant Temperature Increases with Time

<table>
<thead>
<tr>
<th>Experiment Number</th>
<th>( \Delta T/\Delta t^* ) (C/min.)</th>
<th>Pressure (torr)</th>
</tr>
</thead>
<tbody>
<tr>
<td>21</td>
<td>10.536 ± 0.332</td>
<td>1.792</td>
</tr>
<tr>
<td>22</td>
<td>10.833 ± 0.229</td>
<td>1.810</td>
</tr>
<tr>
<td>26</td>
<td>8.718 ± 0.467</td>
<td>2.022</td>
</tr>
<tr>
<td>27</td>
<td>8.726 ± 0.420</td>
<td>2.038</td>
</tr>
<tr>
<td>28</td>
<td>9.311 ± 0.444</td>
<td>2.094</td>
</tr>
<tr>
<td>14</td>
<td>10.490 ± 0.186</td>
<td>0.956</td>
</tr>
<tr>
<td>15</td>
<td>9.653 ± 0.069</td>
<td>0.947</td>
</tr>
<tr>
<td>9</td>
<td>4.545 ± 0.676</td>
<td>0.207</td>
</tr>
<tr>
<td>13</td>
<td>4.961 ± 0.953</td>
<td>0.180</td>
</tr>
<tr>
<td>24</td>
<td>4.410 ± 0.475</td>
<td>0.104</td>
</tr>
<tr>
<td>25</td>
<td>2.765 ± 0.210</td>
<td>0.102</td>
</tr>
<tr>
<td>23</td>
<td>1.613 ± 0.142</td>
<td>0.105</td>
</tr>
</tbody>
</table>

* Determined by linear regression of the temperature versus time curves for the respective experiments. The error represents the 95% confidence interval of slope determined by least squares regression.
Temperature vs Time 0.1 Torr

Exp. 25

Figure 10
Temperature vs Time 0.1 Torr

Exp. 24

Figure 11

Temperature as a function of time during PI etching at approximately 1.0 torr. Experiments 14 and 15, total gas flow rate 502.6 sccm.
Change in Temperature with Time as a Function of Pressure

Figure 12
Temperature/Time gradient (\(\Delta T/\Delta t\)) as a function of pressure.
data given in Table I. This figure shows that as pressure was increased from 0.1 to 1.0 torr. The $\Delta T/\Delta t$ also increased at a greater rate than that seen above 1.0 torr. At pressures greater than 1.0 torr, the $\Delta T/\Delta t$ was seen to increase only slightly with increasing pressure and was actually seen to drop at between 1.8 and 2.0 torr. However, in all cases, the absolute value for $\Delta T/\Delta t$ above 1.0 torr were approximately 6 - 8 °C/min. greater that at 0.1 and 0.2 torr.

This effect can be attributed to a change in the heat conductivity between the discharge and substrate surface. With increasing pressures up to ~1.0 torr, the increase in $\Delta T/\Delta t$ is explained by the increasing number of neutral and ionic species impacting the substrate, which thereby increases the amount of energy transferred from the discharge. Above ~1.0 torr, the effect of gas flow velocity and a lowering of the energies for ion, and neutral species are seen to offset the effect of an increased density of neutral and ion species impacting the surface. This therefore, lowers the rise in $\Delta T/\Delta t$ with respect to the $\Delta T/\Delta t$ seen at lower pressures, although the magnitude of $\Delta T/\Delta t$ is still higher.

The etch rate of PI was effected by temperature. Figure 13 shows a representative plot of etch rate with temperature at a pressure of ~2.0 torr. (Experiment 14). The etch rate was found to increase with temperature.
Figure 13
PI etch rate as a function of temperature for Experiment 14. Total gas flow rate 502.6 sccm, pressure 0.946 torr.
indicating the mechanism(s) of PI etching are enhanced with greater thermal energy. This topic shall be further discussed in Section 3.5.

3.2 System Characterization; Pressure, Gas Flow Rate and Linear Gas Flow Velocity

In order to examine the effect of pressure on the etch rate of PI, gas flow rate was increased to increase the systems pressure. The gas composition was held to a nominal 75% O₂ - 20% CF₄ - 5% Ar mixture. The gas composition for all the experiments conducted were reported in Table II. The gas flow rates and resultant chamber pressure are also given in Table IV. Figure 14 reveals that the chamber pressure had a linear dependence on the total gas flow from 13.45 to 1218.4 sccm, resulting in pressures of 0.103 to 2.04 torr respectfully. Further discussion will reference to the effects of pressure with the knowledge that a direct comparison can also be made for the total gas flow rate due to its linear relation with pressure.

The linear gas flow velocity (Vr) can be related to pressure, total gas flow rate, and reactor geometry assuming a cylindrical model for approximation of the reactor by the equation:
<table>
<thead>
<tr>
<th>Experiment Number</th>
<th>Flow Rate (sccm)</th>
<th>Pressure (torr)</th>
<th>$V_f$ (cm/min.)</th>
</tr>
</thead>
<tbody>
<tr>
<td>24</td>
<td>13.45</td>
<td>0.104</td>
<td>2149</td>
</tr>
<tr>
<td>5</td>
<td>62.60</td>
<td>0.205</td>
<td>5008</td>
</tr>
<tr>
<td>9</td>
<td>67.10</td>
<td>0.207</td>
<td>5403</td>
</tr>
<tr>
<td>13</td>
<td>50.40</td>
<td>0.180</td>
<td>4667</td>
</tr>
<tr>
<td>10</td>
<td>67.10</td>
<td>0.209</td>
<td>5351</td>
</tr>
<tr>
<td>7</td>
<td>53.40</td>
<td>0.199</td>
<td>4481</td>
</tr>
<tr>
<td>12</td>
<td>50.40</td>
<td>0.188</td>
<td>4468</td>
</tr>
<tr>
<td>11</td>
<td>50.40</td>
<td>0.190</td>
<td>4421</td>
</tr>
<tr>
<td>14</td>
<td>502.60</td>
<td>0.956</td>
<td>8762</td>
</tr>
<tr>
<td>21</td>
<td>1072.10</td>
<td>1.790</td>
<td>9970</td>
</tr>
<tr>
<td>22</td>
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<td>2.038</td>
<td>9966</td>
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<tr>
<td>28</td>
<td>1218.40</td>
<td>2.094</td>
<td>9697</td>
</tr>
</tbody>
</table>
Figure 14
Relationship between total gas flow rate and pressure for RF system.
\[ V_r = \frac{FR(760/PR)}{T/273} \left(1/ r^2\right) \]  

(4)

where:

\( FR \) = Total Gas Flow Rate (scm)

\( PR \) = Pressure (torr)

\( T \) = Temperature of Gas (°K)

\( r \) = Radius of Cylinder (cm)

In using this equation, \( T \) was assumed to be 298K. The radius of the cylinder used in the reactor, as discussed in Section 2.1 (Radio Frequency Etching System), was taken as 3.81 cm. Calculated values for \( V_r \) at the various experimental gas flow rates and pressures are shown in Table IV. It shall be noted that at the various relative pressure the \( V_r \) was varied by use of a throttle valve controlling the vacuum system conductance. This allowed examination of the effects of \( V_r \) on PI etching to be discussed in section 3.4.

The graph in Figure 15 shows the effect of pressure on \( V_r \). Here it was found that, at the higher pressures (>1.0 torr), changes incurred to the \( V_r \) were less than those of pressures below 1.0 torr. This plateau of \( V_r \) with higher pressures is attributed to the limited conductance of the vacuum system.
Figure 15
Linear gas flow velocity as a function of experimental pressures.
3.3 The Related Effects of Pressure and Plasma Voltages on Polyimide Etch Rate

The etch rate of PI was seen to vary with pressure and temperature. Figure 16 graphically illustrates PI etch rate as a function of pressure at 60°C and 70°C. Table V contains the experimental data of etch rate at 60°C, 70°C and 80°C with the corresponding pressures. The examination of this data reveals that the PI etch rate rose quickly from a pressure ~ 0.1 to 0.2 Torr, where it reached a maximum, the decrease at a lower rate than the initial rise with increasing pressure.

Comparison of the etch rate curves in Figure 16 indicated an increase in etch rate with higher temperatures (70°C versus 60°C) for pressures above 0.1 Torr. The etch rate at ~0.1 Torr was seen to remain relatively constant, ~5200 Å/min., between the two temperatures. At the lower pressures, i.e. 0.1 Torr, the change in temperature with time (ΔT/Δt) and etch rate increase with temperature was the lowest for all the experimental pressures examined, thereby offering an explanation for constant etch rate seen at 0.1 Torr. The increase in etch rate above 0.1 Torr between 60°C and 70°C was ~500 Å/min. at 0.2 and 2.0 Torr. The main variation noted in the etch rates at different pressures was due to purposely induced changes in the linear gas flow velocity, which
<table>
<thead>
<tr>
<th>Experiment Number</th>
<th>Pressure (torr)</th>
<th>Flow Rate (sccm)</th>
<th>Linear Gas Flow Velocity (cm/min.)</th>
<th>Etch Rate 60 C. 70 C. 80 C. =&lt; (Å/min.)</th>
</tr>
</thead>
<tbody>
<tr>
<td>21</td>
<td>1.790</td>
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<td>9871</td>
<td>2682  3374  4065</td>
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<tr>
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<td>2149</td>
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<td>4437  4709  4980</td>
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<td>5008</td>
<td>8263  8776  8289</td>
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<td>9535  10157 10778</td>
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<td>50.40</td>
<td>4421</td>
<td>---   7520  8170</td>
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<td>5321</td>
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<td>4481</td>
<td>---   8160  8930</td>
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<td>8762</td>
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<td>10042</td>
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<td>2.094</td>
<td>1218.40</td>
<td>9697</td>
<td>3033  3451  3869</td>
</tr>
</tbody>
</table>
will be discussed in Section 3.4.

The plasma voltages were also seen to vary with pressure. Table VI shows the resultant plasma voltages: RF peak to peak ($V_{pp}$), self-bias ($V_{dc}$), and target sheath voltage ($V_p + |V_{dc}|$), for the various experiments conducted. The measured $V_{pp}$ was found to decrease with increased pressure from a maximum value of $\sim 2500$ volts at 0.1 torr to a minimum of $\sim 400$ volts at 2.0 torr as shown in Figure 17. The $|V_{dc}|$, $V_p$ and target sheath voltages are shown as a function of pressure in Figure 18. The $V_p$ and target sheath voltage were found to decrease over the entire pressure region examined. These decreases are explained by an increase in target sheath impedance from scattering and ionization collisions as noted by Keller et al [25]. The $|V_{dc}|$, however, was found to increase between 0.1 and 0.2 torr then decrease above 0.2 torr with increasing pressure. The maximum in $|V_{dc}|$ also corresponded to the maximum in etch rate seen at 0.2 torr.

The target sheath voltage ($V_p + |V_{dc}|$) and PI etch rate are shown as a function of pressure at $70^\circ C$ in Figure 19 (data taken from Table V). This target sheath voltage indicated the relative energy that incident positive ions will have on our PI substrate [19]. Although reference is made to an energy, there is actually a range of impacting energies. These curves have shown that the etch rate of PI at pressures below $\sim 0.2$ torr was not
TABLE VI
Resultant Plasma Potentials

| Experiment Number | Pressure (Torr) | Vpp (Volts) | Vp (Volts) | |Vdc| (Volts) | Vp +|Vdc| (Volts) |
|------------------|----------------|-------------|------------|----------------|----------------|----------------|
| 22               | 1.810          | 433.9       | 87.1       | 42.7           | 129.8          |
| 21               | 1.792          | 441.0       | 87.7       | 45.0           | 132.7          |
| 26               | 2.022          | 474.8       | 82.9       | 34.4           | 117.3          |
| 27               | 2.038          | 394.8       | 84.3       | 28.7           | 113.6          |
| 28               | 2.094          | 390.9       | 83.4       | 28.7           | 111.2          |
| 14               | 0.946          | 563.7       | 90.9       | 100.0          | 190.9          |
| 15               | 0.951          | 545.4       | 95.5       | 81.8           | 177.3          |
| 9                | 0.207          | 981.7       | 109.1      | 272.7          | 381.8          |
| 10               | 0.209          | 1036.3      | 136.3      | 245.5          | 381.3          |
| 5                | 0.205          | 945.4       | 181.3      | 290.9          | 472.7          |
| 7                | 0.199          | 976.0       | 116.8      | 254.4          | 371.2          |
| 11               | 0.188          | 1027.1      | 138.7      | 236.1          | 374.5          |
| 12               | 0.190          | 994.0       | 127.2      | 243.0          | 370.2          |
| 13               | 0.180          | 1059.0      | 147.0      | 236.4          | 383.4          |
| 23               | 0.105          | 2289.6      | 471.8      | 201.3          | 673.1          |
| 24               | 0.104          | 2511.6      | 556.2      | 193.5          | 749.65         |

For all Experiments: 100 watts Incident Power
<5 watts Reflected Power
Figure 17
Peak to peak plasma potential (Vpp) as a function of experimental pressure.
PLASMA POTENTIALS AS A
FUNCTION OF PRESSURE

Figure 18
Mean plasma potential ($V_p$), bias potential ($|V_{dc}|$) and target sheath potential
Figure 19

Co-Plot of PI etch rate at 700°C and target sheath potential (Vp + Vdc) as a function of experimental pressures.
directly correlated to the energy of positive ions impacting the bias electrode substrate. If the positive ion energy were to be directly correlated to the PI etch rate one would expect an increase in etch rate below 0.2 torr where the target sheath voltage increased. The decrease in positive ion energy \((V_p + |V_{dc}|)\) with increasing pressure relates to the increased number of collisions processes that may be expected at higher pressures. These collisions are seen to reduce the average ion energy due to energy transfer and possible recombinations [27].

Figure 20 reveals the relationship of PI etch rate as a function of target sheath voltage. This figure reveals that up to ~400 volts for the target sheath voltage the etch rate appeared to show a linear dependence on this voltage. Above a target sheath voltage of ~400 volts the etch rate was seen to decrease. This, thereby, indicated that etch rate was not directly correlated to the impacting ion energy.

Further examination of the data presented in Table VI shows that PI etch rate and \(|V_{dc}|\) varied in a similar manner with pressure. Figure 21 graphically presents this data. Both the \(|V_{dc}|\) and etch rate reach a maximum at 0.2 torr. A linear relationship is therefore seen between etch rate and \(|V_{dc}|\) as shown in Figure 22. The \(|V_{dc}|\) can be related to the ion current density striking the substrate.
Etch rate of PI as a function of target sheath potential (Vp + |Vdc|).

Figure 20
Figure 21

Co-Plot of PI etch rate at 70°C and bias potential (|Vdc|) as a function of experimental pressures.
if one assumes that the ion current emission from the discharge is approximated by Child - Langmuir equations for either the space charge or mobility limited cases.

\[ J_i = k \cdot |V_{dc}|^x \]  \hspace{1cm} (5)

Where \( J_i \) is the ion current density, \( x \) and \( k \) constants dependant on whether dealing with the mobility or space charge limited cases [21,27]. The \( x \) in these equations is above one. The use of this assumption then indicates the ion current density directly influences the PI etch rate for a reactive ion etching, as was used in this investigation. However, it should be noted that the above assumption is not truly valid due to the dependence of the constant in the equations on a number of operating parameters including pressure [27].

The ratio of the plasma voltages \( |V_{dc}| \) and \( V_{pp} \) can be used to attain a relative measure of the ion bombardment energies between the two electrodes of a RIE System. This ratio, \( |V_{dc}/V_{pp}| \) was shown to have a direct correlation to that of the ion bombardment energies as postulated for the capacitive and resistive RF discharge models proposed by Horwitz [23]. For the work presented here, the assumption of a capacitive discharge a sine-wave excitation was made for fitting the experimental data. This model was chosen since the RF discharge was cathode coupled by design and
the resultant RF discharge waveforms as measured were sinusoidal in nature at 13.56 MHz.

Table VII presents the calculated ion bombardment energies, as dictated by the above assumption, and the $|V_{dc}/V_{pp}|$ for the various experimental pressures. This data indicates that a direct relationship exists between the $|V_{dc}/V_{pp}|$ and the experimental ion bombardment energies determined via modeling, as shown in Figure 23. Examination of this figure shows that at a $|V_{dc}/V_{pp}|$ of 0.3 the ion bombardment energy striking the target electrode (PI substrate) was approximately four times greater than the energy of ions striking the grounded electrode chamber, while at a $|V_{dc}/V_{pp}|$ of 0.1 it was only 1.5 times as great. Therefore, a change in the symmetry of ion energies striking the respective electrodes was found as pressures were varied. The experimental data mentioned above also shows agreement with that presented by Horwitz for the capacitive discharge model with sine wave excitation shown in Figure 24, (the C Model), indicating validity to the capacitive discharge model assumption above.

The examination of the ion bombardment energy ratio, $|V_{dc}/V_{pp}|$, at the various experimental pressures reveals a relationship to pressure for $|V_{dc}/V_{pp}|$ that was similar for that shown by the PI etch rate and $|V_{dc}|$. Figure 25 shows the etch rate and $|V_{dc}/V_{pp}|$ as a function of pressure. The $|V_{dc}/V_{pp}|$ was found to increase from 0.1 torr to 0.2 torr,
Table VII

Ion Energy Ratio Data

| Experiment Number | Pressure (torr) | \( \frac{|V_{dc}|}{V_{pp}} \) | \( \frac{V_{pp} - 2|V_{dc}|}{V_{pp} + 2|V_{dc}|} \) |
|-------------------|----------------|-----------------|-----------------------------|
| 21                | 1.790          | 0.102           | 1.513                       |
| 22                | 1.810          | 0.098           | 1.490                       |
| 24                | 0.104          | 0.077           | 1.364                       |
| 23                | 0.105          | 0.088           | 1.427                       |
| 5                 | 0.205          | 0.308           | 4.200                       |
| 9                 | 0.207          | 0.278           | 3.500                       |
| 13                | 0.180          | 0.223           | 2.613                       |
| 11                | 0.188          | 0.230           | 2.701                       |
| 12                | 0.190          | 0.244           | 2.913                       |
| 10                | 0.209          | 0.236           | 3.178                       |
| 7                 | 0.199          | 0.261           |                             |
| 14                | 0.956          | 0.177           | 2.178                       |
| 15                | 0.951          | 0.150           | 1.857                       |
| 26                | 2.022          | 0.072           | 1.339                       |
| 27                | 2.038          | 0.073           | 1.340                       |
| 28                | 2.094          | 0.073           | 1.344                       |

Figure 23
Experimentally calculated ion bombardment energy ratios $S = \frac{(V_{pp} - 2V_{dc})}{(V_{pp} + 2V_{dc})}$ versus the voltage ratio $V_{dc}/V_{pp}$ for a capacitive modeled discharge with sine-wave excitation.
Ion bombardment energy ratio $\xi = (V_1 - V_2)/(V_1 - V_2)$ versus the voltage ratio $-V_b/V_{pp}$ for three voltage waveforms, assuming a resistor/inductor model. The square wave model also corresponds to the capacitive sine-wave excitation model with high leakage resistances.
Figure 25
Co-Plot of PI etch rate at 60°C, 70°C and 80°C and the voltage ratio |Vdc/Vpp| as a function of pressure.
where it reached a maximum, then decrease at higher pressures. This behavior was similar to that shown for the PI etch rate. The PI etch rate was therefore seen to have a linear dependence on the \( \left| \frac{V_{dc}}{V_{pp}} \right| \) as shown in Figure 26.

The effect of increasing etch rate with higher temperatures was also seen by comparison of the maximum etch rates at the temperature shown. The maximum etch rate at 60°C was ~9600 Å/min., at 70°C ~100 Å/min. and at 80°C ~10,800 Å/min.

The curves shown in Figure 27 represent the approximated RF waveforms at the respective pressures of 0.1, 0.2 and 2.0 torr. The voltage asymmetry was seen to be largest at 0.2 torr and smallest at 2.0 torr, also (see RF waveforms in Appendix III). This change in voltage asymmetry with pressure was also in agreement with that noted for the maximization of \( \left| \frac{V_{dc}}{V_{pp}} \right| \) and PI etch rate.

The change in voltage asymmetry with pressure corresponds to a change in the respective time for electron and ion bombardment of the target electrode surface per given RF cycle. The ion and electron bombardment times can be estimated from the RF approximated waveforms by noting the length of time (measured distance) a given full cycle is above the ground potential (electrons) and below (ions) on the graphed waveforms. Table VIII shows these derived time as well as the ratio of ion bombardment time to that electrons for the various experimental pressures. The ratio
Etch Rate vs $|V_{dc}/V_{pp}|$

- Etch Rate vs $|V_{dc}/V_{pp}|$ @ 60°C
- Etch Rate vs $|V_{dc}/V_{pp}|$ @ 70°C
- Etch Rate vs $|V_{dc}/V_{pp}|$ @ 80°C

**Figure 26**
Etch rate of PI, at 60°C, 70°C and 80°C as a function of the voltage ratio $|V_{dc}/V_{pp}|$. 

62
Approximated RF waveform at pressures of 0.1, 0.2 and 2.0 torr, experiments 24, 10 and 26 respectfully. Experiment 24 total gas flow rate 67.1 sccm-pressure 0.209 torr. Experiment 26, total gas flow rate 1218.4 sccm-pressure 2.02 torr.
TABLE VIII

Fraction of Time for Ion and Electron Bombardment

<table>
<thead>
<tr>
<th>Experiment Number</th>
<th>Pressure (torr)</th>
<th>Ion* Time</th>
<th>Electron* Time</th>
<th>Ratio T(i):T(e)</th>
<th>Etch Rate (Å/min.) at 70°C</th>
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</thead>
<tbody>
<tr>
<td>24</td>
<td>0.104</td>
<td>6.70</td>
<td>5.55</td>
<td>1.207</td>
<td>5095</td>
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<td>10</td>
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<td>3.20</td>
<td>4.20</td>
<td>1.954</td>
<td>8990</td>
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<td>14</td>
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<td>7.10</td>
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<td>1.670</td>
<td>6337</td>
</tr>
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<td>26</td>
<td>2.022</td>
<td>6.75</td>
<td>5.56</td>
<td>1.214</td>
<td>3162</td>
</tr>
<tr>
<td>7</td>
<td>0.199</td>
<td>8.45</td>
<td>3.95</td>
<td>2.139</td>
<td>8160</td>
</tr>
<tr>
<td>12</td>
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<td>4.05</td>
<td>2.037</td>
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<tr>
<td>27</td>
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<td>6.80</td>
<td>5.60</td>
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</tbody>
</table>

* per measured distance on RF waveform
of ion to electron bombardment time (T(i):T(e)) represents the symmetry of the discharge as related to the effect of ion bombardment duration. Figure 28 shows this ion bombardment time ratio and PI etch rate at 70°C as a function pressure. This figure shows that the asymmetry variations in ion bombardment duration follow the same behavior as that note previously for PI etch rate. This also infers the PI etch rate has a linear relationship with that of ion bombardment duration as shown in Figure 29.

3.4 The Effects Of Linear Gas Flow Velocity On Polyimide Etch Rate At A Constant Pressure

The parameters effecting the linear gas flow velocity (Vr) were seen to be pressure, flow rate and vessel geometry, as discussed in Section 3.2. The effects of Vr on PI etch rate were examined by using a vessel of a fixed geometry, Section 2.1, at a constant pressure with a varying flow rate. The pressure of the system was controlled by the use of manual throttle valve connecting the reaction chamber to the vacuum system. With increasing Vr at a constant pressure the number of discharge species reaching the target is expected to increase.

Experimental conditions for a constant pressure of ~2.0 torr are given in Table IX. Figure 30 shows the
Etch Rate and The Ratio $[T(i):T(e)]$

(A X 1000) vs Pressure @ 70°C

0 0.8 1.6 2.4
Pressure (torr)

0 4 8 12 16 20
Etch Rate (Å/min.)

1.2 1.4 1.6 1.8 2 2.2
$[T(i):T(e)]$

Figure 28
Co-Plot PI etch rate at 70°C and the ratio of ion bombardment time to electron bombardment time $[T(i):T(e)]$ during a RF cycle.
Figure 29

Etch rate of PI as a function of the ion bombardment time to electron bombardment time ratio, \([T(i):T(e)]\) during a RF cycle.
TABLE IX

Etch Rate Results with Changing Linear Gas Flow Velocity and Plasma Potentials at a Constant Pressure of ~ 0.2 torr

| Experiment Number | Vr (cm/min.) | Pressure (torr) | $|V_{dc}|$ (volts) | Vp (volts) | Vpp (volts) | Vp+|V_{dc}| (volts) | Vpp (volts) | Etch Rate* (A/min.) |
|-------------------|--------------|----------------|------------------|---------|------|----------------|------|------------------|
| 9                 | 5403         | 0.207          | 272.7            | 109.1  | 981.7| 381.8          | 0.278| 10050            | 11200          |
| 10                | 5351         | 0.209          | 245.5            | 136.3  | 1036.3| 381.3          | 0.237| 8990             | 9480           |
| 11                | 4468         | 0.188          | 231.1            | 138.7  | 1027.1| 374.5          | 0.230| 8100             | 8700           |
| 12                | 4421         | 0.190          | 243.0            | 127.2  | 994.0 | 370.2          | 0.244| 7520             | 8170           |
| 13                | 4667         | 0.180          | 236.4            | 147.0  | 1059.0| 383.4          | 0.223| 8410             | 9240           |
| 7                 | 4481         | 0.195          | 254.4            | 116.8  | 976  | 371.2          | 0.261| 8170             | ---             |
| 6                 | 4578         | 0.195          | 327.2            | 72.8   | 945.4| 400.0          | 0.346| 9980             | ---             |

* Data gathered off Etch Rate Verses Temperature Graphs for the respective experiment.
Etch Rate vs Plot

(x1000) Linear Gas Flow Velocity @~0.2 torr

Etch Rate (Å/min.)

11.5
10.5
9.5
8.5
7.5

44 48 52 (x 100)
Linear Gas Flow Velocity (cm/min.)

Figure 30

Etch rate of PI as a function of linear gas flow velocity at a constant pressure of 0.2 torr.
dependence of PI etch rate on $V_r$ at 70°C and 80°C. The etch rate was found to increase with both increasing temperature and $V_r$. The plasma voltages; $V_p$, $|V_{dc}|$ and target sheath voltage ($V_p + |V_{dc}|$) are shown as function of $V_r$ in Figure 31. These plasma voltages are seen to be constant with increasing $V_r$. The ion bombardment energy ratio as a function of $V_r$ is shown in Figure 32. This indicated that at 0.2 torr the PI etch rate was directly influenced by the increasing $V_r$'s and not by the above plasma voltages, which were previously seen to shown a correlation to etch rate changes with pressure variations.

Further examination of the effects of changing $V_r$'s at other constant pressures, (0.1, 1.8, 2.0 torr), and varying temperatures indicated a constant or possibly a decrease in PI etch rate as shown in Figures 33 to 37. The experimental conditions and resultant etch rates for these figures are shown in Table X. The available data regarding the plasma voltages of these experimental pressures is limited, Table XI. The lines drawn on these figures were drawn to aid interpretation and may not definitively represent the actual etch rate behavior with $V_r$. Therefore, no definitive conclusions as to the effect of plasma voltages, $V_r$ and PI etch rate can be made outside those stated above.
Bias potential ($|V_{dc}|$), plasma potential ($V_p$) and target sheath potential ($V_p + |V_{dc}|$) as a function of linear gas flow velocity at a
Figure 32

Voltage ratio $|V_{dc}/V_{pp}|$ as a function of linear gas flow velocity at a constant pressure of 0.2 torr.
TABLE X

Etch Rate Results With Changing Linear Gas Flow Velocities (Vr)

<table>
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<tr>
<th>Experiment Number</th>
<th>Pressure (torr)</th>
<th>Vr (cm/min.)</th>
<th>50C.</th>
<th>60C.</th>
<th>65C.</th>
<th>70C.</th>
<th>80C.</th>
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<td>) (Volts)</td>
<td>( V_p ) (Volts)</td>
<td>( V_{pp} ) (Volts)</td>
<td>( V_{dc} +</td>
<td>V_{dc}</td>
<td>) (Volts)</td>
<td>Etch Rate @70°C (A/min.)</td>
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</table>
Etch Rate vs Linear Gas Flow Velocity
@ 0.1 torr (50 C - 70 C)

Etch rate of PI at 50°C, 60°C, 65°C, and 70°C as a function of linear gas flow velocity at a constant pressure of 0.1 torr.
Etch Rate vs Linear Gas Flow Velocity
at 1.7-1.8 torr (70°C, 80°C)

Figure 34
Etch rate of PI 70°C and 80°C as a function of linear gas flow velocity at a constant pressure of 1.7 - 1.8 torr.
Etch Rate vs Linear Gas Flow Velocity
@ 1.7-1.8 torr (90°C - 140°C)

Figure 35
Etch rate of PI at 90°C, 100°C, 120°C and 140° as a function of linear gas flow velocity at a constant pressure of 1.7 - 1.8 torr.
Etch Rate vs Linear Gas Flow Velocity at Constant Pressure 2 torr (70°C-140°C)

Etch rate of PI at 70°C, 80°C, 90°C, 120°C and 140°C as a function of linear gas flow velocity at a constant pressure of 2.0 torr.
3.5 Activation Energies for Etching Polyimide

The etch rate of PI was examined as a function of the substrate temperature increase for each experiment. The incident power level was held at 100 watts with ≤ 5 watts reflected for all the experiments. The apparent activation energy (Ea) for PI etching was determined by the use of an Arrhenius plot for ln etch rate versus 1/temperature, in degrees Kelvin for each experiment.

Table XII shows the Ea's for the various experiments. The Ea as calculated from the linear regression of the data fitted for each experiment. The error stated in Table XII was determined from the ninety five percent confidence interval about the slope of the linear regression. For each experiment, the linear regressions were determined ≤ 140°C, except for experiment 14. In the case of experiment 14, Figure 38, a change in slope was noted at ~140°C. Therefore two linear regressions were conducted, one above 140°C and one below 140°C which was used for comparisons with the other experiments. The change in Ea shown in experiment 14 (~0.061 ev ≤ 140°C and ~0.077 ev > 140°C) has indicated that the mechanism(s) of PI etching can be effected by temperature. This effect of changing Ea was not seen for the other experiments, however most of these experiments did not reach temperatures > 140°C. Appendix IV contains the
Table XII

Activation Energies of Experimental Results at Various Pressures

| Experiment Number | $E_a^*$ (ev) | Temp Range ($^\circ$C) | Pressure (torr) | $V_\theta - |V_{dc}|$ (Volts) | $|V_{dc}| - V_{pp}$ (Volts) | $V_{dc}$ (Volts) |
|------------------|-------------|------------------------|----------------|-----------------------------|-----------------------------|----------------|
| 26               | 0.0778±0.0040 | 59.4-137.6             | 2.022          | 117.3                       | 0.072                       | 34.4           |
| 27               | 0.0890±0.0120 | 66.0-134.0             | 2.038          | 113.0                       | 0.073                       | 28.7           |
| 28               | 0.0893±0.0036 | 65.8-133.0             | 2.094          | 112.1                       | 0.073                       | 28.7           |
| 9                | 0.0623±0.0098 | 39.2- 80.0             | 0.207          | 331.8                       | 0.278                       | 272.7          |
| 13               | 0.0603±0.0145 | 66.6- 92.0             | 0.180          | 333.4                       | 0.223                       | 236.4          |
| 14               | 0.0610±0.0027 | 44.0-135.1             | 0.956          | 190.9                       | 0.177                       | 160.0          |
| 15               | 0.0614±0.0128 | 100.5-134.3            | 0.975          | 177.25                      | 0.150                       | 81.3           |
| 24               | 0.0690±0.0221 | 50.2- 68.3             | 0.104          | 749.65                      | 0.077                       | 193.5          |
| 25               | 0.0589±0.0190 | 56.3- 69.5             | 0.102          | ---                         | ---                         | ---            |
| 14               | 0.3149±0.0339 | 145.0-168.1            | ---            | ---                         | ---                         | ABOVE-         |

* Activation Energies determined by least squares linear regression of the respective experimental Arrhenius plots. The error represents the 95% confidence interval about calculated slope.
Arrhenius Plot

PI Etch Rate vs Temperature

Exp.14 ~1torr

$\epsilon_a = 0.315 \pm 0.034 \text{ eV}$

$\epsilon_a = 0.061 \pm 0.003 \text{ eV}$

Figure 37

Arrhenius plot for PI etch rate versus reciprocal temperature (1/°K).

Experiment 14 - approximately 1.0 torr.
Activation energy (Ea) for etching PI as a function of experimental pressures.
Activation energy (Ea) for etching PI as a function of PI etch rate for the various experimental pressures (0.1, 0.2, 1.0, 1.8 and 2.0 torr).
Arrhenius plots for the remaining experiments shown in Table XII.

The $E_a$ for the experiments in Table XII indicated that the $E_a$ increased with increasing pressure as shown in Figure 39. The $E_a$ was found to remain relatively constant from ~0.1 to ~1.0 torr. Figure 40 reveals the relationship of $E_a$ as a function etch rate for temperatures <140°C. The $E_a$ was found to decrease with increasing etch rate up to ~6000 A/min. where it remained relatively constant at ~0.060 ev with higher etch rates.

The $E_a$ was also found to show a similar relationship to that of etch rate which the relative ion bombardment energy $|V_{dc}/V_{pp}|$, bias voltage $|V_{dc}|$ and target sheath voltage $(V_p - |V_{dc}|)$ as shown in Figure 41, 42 and 43 respectively. It shall be noted that above ~400 volts for the target sheath voltage the $E_a$ was seen to increase to ~0.069 ev at ~750 volts from ~0.060 ev at ~400 volts which corresponds to the decrease in etch rate previous noted for this target sheath voltage in Section 3.4. This indicated that the conditions which favored voltage asymmetry and high relative ion bombardment energies on the cathode results in a lowering of the etch rate $E_a$ leading to higher etch rates to a point. Above this point, the increases in the voltage asymmetry and relative ion bombardment energy are seen to increase PI etch rates without further decreasing the resultant $E_a$. 
Figure 40: Activation energy (Ea) for etching P1 as a function of the voltage ratio |Vdc/Vpp|.
Activation energy (Ea) for etching PI as a function of bias potential |Vdc/Vpp|.
Activation Energy (Ea) for Etching PI
vs $V_p + |V_{dc}|$

Figure 42

Activation energy (Ea) for etching PI as a function of the target sheath potential $V_p + |V_{pp}|$. 
The maximum etch rate condition at ~0.2 torr corresponded to an Ea for PI of ~ 0.061 ± 0.003 ev for this experimental RIE system. Others have reported an Ea for PI at 0.14 ± 0.03 ev [30] without the presence of ions in a downstream microwave system using the same ternary gas mixture, O₂/CF₄/Ar, as was used in this investigation. Lamontagne et al [29] has determined the Ea of PI in a microwave discharge at 0.21 ± 0.02 ev. It should be noted that the method from which the Ea were determined was not the preferred method since it was determined from the rise in etch rate with temperature for each experiment instead of a measurement of etch rate at various constant discrete temperatures for given experiments.
4.0 CONCLUSIONS

The etching of PI in the presence of ions using a RF excitation source was seen to reach a maximum at ~0.2 torr as pressure was varied from 0.1 to 2.0 torr. This maximum etch rate also corresponded to the maximum in the bias voltage \(|V_{dc}|\) and relative ion bombardment energy \(|V_{dc}/V_{pp}|\) at ~0.2 torr as seen as a function of pressure. The polyimide etch rate was also shown to have a linear dependence on \(|V_{dc}/V_{pp}|\). The target sheath voltage, \(V_{p+}|V_{dc}|\), was seen to decrease with higher pressures indicating that the ion bombardment energy decreased with increasing pressure.

Interpretation of approximated RF waveforms at various pressures has revealed that at low and high pressures (0.1 and 2.0 torr) the resultant waveforms were more symmetrical as compared to the asymmetric waveform seen at a pressure of ~0.2 torr. The asymmetric waveform at ~0.2 torr corresponded to the pressure where the maximum in etch rate and \(|V_{dc}/V_{pp}|\) occurred. This maximum in asymmetry indicated that the PI film was bombarded by ions for a longer period of time as compared to that for the more symmetric waveforms seen at the other pressures. Such a result is also in agreement with the voltage asymmetry maximum inferred from the \(|V_{dc}/V_{pp}|\) ratio.

Therefore, the information presented here suggests
that relative ion bombardment energy ratio as well as the time duration per RF cycle for ion bombardment play major roles in PI etching and can be used for a means of characterizing and controlling a RIE process. The ion energy, as determined by $V_p + |V_{dc}|$, was not seen to have a direct correlation to PI etch rate as pressure was varied below 0.2 torr. Although not examined here, the concentration of atomic oxygen and fluorine may be impacted by the various plasma voltages and pressures, thereby effecting the etch rate and should be further examined.

The etch rate of PI was also impacted by changing linear gas flow velocity ($V_r$) at a relative constant pressure. At ~0.2 torr, increasing the $V_r$ resulted in an increase in the PI etch rate while the various plasma voltages and voltage ratio $|V_{dc}/V_{pp}|$ remained constant. While varying $V_r$ at high and low pressures, 2.0 and 0.1 torr respectfully, the PI etch rate remained relatively constant or possibly decreased with increasing $V_r$. Therefore, it was concluded that, when near the maximum etch rate condition increasing the number of active species reaching the target surface assisted in enhancing the etch rate of PI in the presence of ions.

Etching of PI was seen to increase with increasing temperature. The temperature rise of the substrate with time was influenced by the heat conductivity of the plasma as pressure was varied. At higher pressures, the fastest
rise in temperature occurred when the heat conductivity of
the plasma was high as compared to that at lower pressures.

The activation energy for etching PI in the presence
of ions was seen to correlate to the resultant etch rates
in that with higher etch rates the lower activation
energies were seen (~ 0.061 eV). This trend was also seen
for the relative ion bombardment energy ratio, \( \frac{V_{dc}}{V_{pp}} \).
The range of activation energies was found to vary from
0.0893 \( \pm 0.0036 \) eV at ~2.0 torr to 0.0603 \( \pm 0.0145 \) eV at
~0.2 torr. These values were at least one half of that
reported for PI etching in a microwave plasma system
without the presence of ions. However, it should be noted
that the activation energies shown were calculated from the
relationship between etch rate and the temperature rise
within a given experiment and not from discrete experiments
conducted at various stabilized temperatures.
APPENDIX I

Experimental Plots Of Temperature
As A Function Of Time
## APPENDIX II

Correlation Coefficients Of The Rise In Temperature With Time ($\Delta T/\Delta t$)

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<th>Experiment Number</th>
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APPENDIX III

Approximated RF Voltage Waveforms
EXP. 13

EXP. 7
RF WAVEFORM 0.2 torr

RF WAVEFORM 1.8 torr
APPENDIX IV

Arrehenius Plots—Polyimide Etch Rate Versus Reciprocal Temperature
References


12. E. Matuszak, "Etching and Passivation Down Stream of an \( \text{O}_2 - \text{CF}_4 - \text{Ar} \) Microwave Plasma", M.S. Thesis, Materials Science and Engineering, Rochester Institute of Technology, June 1986


