

On the Role of Ions in Electron Cyclotron Resonance Plasma-Enhanced Chemical Vapor Deposition of Silicon Dioxide

Karen Seaward, Solid State Technology Laboratory
John Turner, K. Nauka, Andre Nel
Integrated Circuit Business Division
HPL-94-94
October, 1994

silicon dioxide, ECR,
PECVD, electron
cyclotron resonance,
plasma

Silicon dioxide films were deposited in an electron cyclotron resonance plasma onto substrates at temperatures ranging from 65 to 200°C. In order to determine the effect of ions on the deposition, both ion flux and ion energy were investigated. The ratio of ion flux to deposition flux was found to be a significant parameter and this "flux ratio" was varied between 8 and 100. Mean ion energy was investigated at 10 eV, 50 eV and 75 eV. The silicon dioxide films were characterized by measurement of wet etch rate, density, composition, and stress, and by infrared spectroscopy. It was found that above a flux ratio of about 20, high-quality SiO₂, was deposited whether or not the substrate was thermally floating or at 65 to 200°C, indicating that the flux ratio was dominant over the temperature. Use of rf bias to increase the mean ion energy to 50 eV or above was effective in producing high-quality SiO₂ when the flux ratio was below 20, but not as effective as using a high flux ratio. Thus high ion flux and low ion energy were found to be useful in producing SiO₂ at temperatures as low as 65°C with properties close to that of thermal silicon dioxide. The role of ions in the deposition process was found to be densification, removal of -OH (or hydrogen) and alteration of Si-O bonding.

Internal Accession Date Only

To be Published in the *Journal of Vacuum Science and Technology B*, February, 1995

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1. INTRODUCTION

Electron cyclotron resonance (ECR) plasmas have been studied and applied to deposition of silicon dioxide since 1978¹⁻⁶. These plasmas are of interest because higher-quality material can be deposited at lower temperature (~100°C) by ECR than by other techniques that are presently the standard in the semiconductor industry, such as parallel-plate rf plasmas (~250°C) or thermally-activated chemical vapor deposition (~400°C). There has been a long-standing interest in the "mechanism" of the deposition and, recently, in how it differs between high-density (e.g., ECR) plasmas and lower-density parallel-plate rf discharges. In particular, there is interest in the plasma energetics that appear to dominate temperature. Numerous studies have examined plasma properties and SiO₂ film properties to elucidate the importance of ions and chemically reactive neutral species for ECR deposition at low temperature⁶⁻¹². Primarily the effect of ion energy has been studied and it has been found that film quality approaches that of thermal SiO₂ (900°C) with the application of rf bias to the deposition surface. The effect of ion flux may also be important and the question remains whether or not to choose low ion flux and high ion energy or high ion flux and low ion energy for the highest quality silicon dioxide deposited at low temperature.

In comparing ECR and rf parallel-plate plasmas, the most striking difference is the ratio of excited neutral species to ions. Some parameters used to make estimates are listed in Table I. For example, in the case of a parallel-plate rf deposition plasma operating at a pressure of 0.5 Torr, we estimate that about 10% of the neutrals are in an excited state. The degree of ionization is around 10^{-6} so the ratio of excited neutrals to ions is about 100,000. From a similar estimate for an ECR plasma operated at 2 mTorr, and assuming that around 50% of the neutrals are excited and that the degree of ionization is 10^{-2} , the ratio of excited neutrals to ions is about 50. Since the ion component during deposition in a high density plasma is so much greater than it is in a parallel-plate rf plasma, in this work the role of ions is investigated. To do this, either ion energy is changed, by using rf bias, or ion flux is changed, by altering the plasma density near the deposition surface. Ion flux is measured with a Langmuir probe and silicon dioxide depositions are characterized by Auger electron spectroscopy, Rutherford backscattering spectroscopy (RBS), Fourier transform infrared spectroscopy (FTIR) and measurement of stress and wet etch rate. The best measure for comparing the depositions in which ion flux is varied turns out to be the ratio of ion flux to deposition flux, which will be called the "flux ratio" and denoted J_i/J_{ox} .

Estimates of J_i/J_{ox} , which can also be described as the number of ions per molecule of deposited SiO₂, can be derived from some recent publications. For example, from the work of Fukuda and coworkers employing an ECR system with a mirror magnetic field^{11,13}, ion flux is derived from Langmuir probe measurement of the ion current density at 7.5 mA/cm². Using a deposition rate of 1500 Å/min, for which high-quality SiO₂ was obtained without using rf bias, J_i/J_{ox} is about 8. From Andosca and coworkers¹⁰, also employing a mirror-type ECR source, estimating a lower limit of 1×10^{10} ions/cm³ and an upper limit of 1×10^{11} ions/cm³ from measurements in our system,

and using the published deposition rate of 70 Å/min, the flux ratio is 4 to 40. Using a multipolar ECR source, Buckle and coworkers⁹ deposited SiO₂ at a rate of 400 Å/min; estimating the ion density¹⁴ to be $1-4 \times 10^{11}/\text{cm}^3$, a flux ratio of 2 to 8 is derived. In addition, in their work no difference was found between depositions at 80°C and at 200°C. In another style of multipolar ECR source, a flux ratio of 27 is derived from the work of Plais et al¹⁵; this estimate uses their values, for deposition at 800W and 1 microbar, of 1.9 mA/cm² for ion current density and 120 Å/min for the silicon dioxide deposition rate. All of these estimates for J/J_{∞} are greater than 1 and were derived from data on depositions without substrate bias. The mean ion energy in these cases is estimated¹⁶⁻¹⁸ to be tens of eV, for example below 20 eV for pressures above 1 mTorr, which are typical of ECR deposition.

In our work, a range of flux ratios from 5 to 100 and mean ion energies of 10 eV (the plasma potential), 50 eV, and 75 eV are investigated. As well, four fixed temperatures are evaluated, 65, 100, 150, and 200°C, plus a "floating temperature" that corresponds to plasma heating, in order to determine whether or not the plasma energetics at the deposition surface override these temperatures. From this work, as well as published work using similar deposition conditions wherein similar and higher ion energies were investigated, some guidelines are established for the flux ratios and ion energies needed to produce high-quality silicon dioxide at low temperature.

2. EXPERIMENTAL METHODS

Silicon dioxide films were deposited in three different plasma reactors. One is a parallel-plate rf system shown in Fig. 1. This was used to deposit rf PECVD silicon dioxide for comparison with ECR deposited SiO₂. The ECR system was built by Oxford Plasma Technology and is shown in Fig. 2 with a source that uses a mirror magnetic field and in Fig. 3 with a divergent field source and a trim magnet downstream from the deposition surface. The mirror source configuration was used to deposit SiO₂ with J/J_{∞} varying between 6 and 35 by changing the current in the lower magnet and by altering the deposition rate by changing the silane flow; rf bias was also used to change ion energy. In this reactor, the deposition temperature was floating and typically increased up to 120°C. The oxygen to silane ratio was about 3 and depositions were done at 2 mT. The configuration in Fig. 3 was used to produce increased deposition rates and the ion flux was varied by operating the trim magnet between conditions for magnetic field collimation and cusp formation; for this configuration, J/J_{∞} varied between 8 and 100, the oxygen to silane ratio was also about 3, and the deposition pressure ranged from 2 to 10 mT. Helium backside cooling was employed to control the deposition temperature at 65, 100, 150, or 200°C; this was accomplished by first heating the deposition surface using a plasma containing O₂ and argon then relying on a calibrated balance between plasma heating and helium cooling measured with a Luxtron probe. In both configurations, a Plasma and Materials Technologies fast-injection Langmuir probe was used to measure ion saturation current near the deposition surface. From values in mA/cm², ion flux was calculated by dividing by the electronic charge. Deposition flux,

J_{ox} , was calculated from the deposition rate and included the density as measured by RBS. Used this way, J_{ox} is the net deposition flux.

Silicon dioxide films were analyzed by a General Ionics RBS system using He^{++} at 2.0 MeV. Auger analysis using a Perkin-Elmer PHI 660 was used to determine atomic composition; data were evaluated using sensitivity factors measured on thermally oxidized silicon. The presence of -OH groups and the nature of bonding in the oxides were evaluated on doubly-polished GaAs wafers with FTIR on a Nicolet 60SX. Thin film stress was measured on a FleXus 2-300i (Tencor Instruments) and etch rate was determined in 10:1 $H_2O:HF$. Additional measurements of temperature during deposition were done with Omegalabels (Omega Engineering, Inc.) firmly attached to a silicon wafer; measurements made with these indicators on the back side of wafers were the same as measurements made on the side facing the plasma and are therefore thought to be a reasonable estimate of wafer temperature, free of artifacts due to the presence of the plasma. Most ECR depositions were done with O_2 and 10% silane in argon. The rf PECVD depositions were done with N_2O and 2% SiH_4 in N_2 .

3. DATA AND INTERPRETATION

In Fig. 4 is shown a comparison of the FTIR spectra of an ECR silicon dioxide deposition and an rf PECVD deposition. The ECR film was grown using N_2O and 10% SiH_4 in N_2 with the temperature floating up to 120°C during the deposition. The rf PECVD film was grown using N_2O and 2% SiH_4 in N_2 with the temperature fixed at 200°C. The Si-O stretching absorption is nearly the same for these films, 1071 cm^{-1} for the ECR and 1068 cm^{-1} for the rf sample. However, the value of the full-width-at-half-maximum (FWHM), which is a qualitative estimate of the distribution of bonding arrangements¹⁹, is smaller for the ECR film, 97 cm^{-1} , than for the rf film, 127 cm^{-1} . In addition, the rf film shows absorption at 3390 cm^{-1} corresponding to adsorbed H_2O , and absorption at 3620 cm^{-1} corresponding to Si-OH²⁰. The ECR film also contains Si-OH but far less than the rf film; when grown with more oxygen and a temperature floating to 200°C, Si-OH does not appear in the FTIR spectra of the ECR oxide. Water is a byproduct of the reactions that produce silicon dioxide from N_2O and SiH_4 and the ECR deposition environment more effectively removes this byproduct from the deposited oxide.

Figures 5-7 show data from silicon dioxide films grown in the mirror ECR configuration depicted in Figure 2. The ion saturation current at the deposition surface was changed by changing the current in the second magnet from 0 to 30, 60, 90, and 120 Amps, as shown in Fig. 5. Also indicated in the figure is the flux ratio of ions to deposited SiO_2 and for these experiments this ratio varied from 7 to 35. The density of the deposited oxide, as measured by RBS, changed from 1.8 g/cm^3 to 2.2 g/cm^3 as the flux ratio was increased, approaching the value of 2.27 g/cm^3 for thermally grown silicon dioxide. Over this range of flux ratios, the oxygen/silicon ratio remained constant, as indicated in Fig. 6. However, the thin film stress changed from tensile (at about 40 MPa) to compressive, saturating at about 200 MPa for J_i/J_{ox} .

greater than about 25. The corresponding wet etch rates in 10:1 H₂O:HF went from 250 Å/sec at a flux ratio of 7 down to 8 Å/sec at a flux ratio of 35; this latter etch rate is a factor of two higher than what is measured for thermal silicon dioxide and a factor of 5-10 lower than a diverse collection of rf PECVD films that were evaluated.

Also shown in Figure 6 are the changes that occur by increasing the mean ion energy. For a flux ratio of 8, the compressive stress increases from 20 MPa with no rf bias to 150 MPa with 50 eV ions and 220 MPa with 75 eV ions. At even higher ion energies, the compressive stress continues to increase. The wet etch rate decreases as the ion energy is increased, also shown in Figure 6; at a flux ratio of 8, a decrease from 135 to 35 Å/sec is measured when the mean ion energy is increased from 10 to 50 eV and a further decrease to 20 Å/sec occurs when the mean ion energy is increased to 75 eV.

In Figure 7 are shown the changes in FTIR spectra as the flux ratio is changed. The Si-O stretching frequency increases and the full-width-at-half-maximum of this absorption decreases as the flux ratio increases. At the highest flux ratio of 35, the Si-O stretch frequency is 1074 cm⁻¹ and the FWHM is 91 cm⁻¹, to be compared to the values measured for thermal silicon dioxide which are 1077 cm⁻¹ and 83 cm⁻¹. Typical values for rf PECVD oxide are 1060-1070 cm⁻¹ for the stretching frequency with the FWHM >120 cm⁻¹. Again, the use of higher ion energy improves the oxide; by increasing the ion energy from 10 to 50 eV at a flux ratio of 8, the stretching frequency is increased from 1067 cm⁻¹ to 1071 cm⁻¹ and the FWHM is reduced from 110 cm⁻¹ to 101 cm⁻¹. However, it appears that use of a high flux ratio may produce higher-quality material. For example, by using a flux ratio of 28, a stretching frequency of 1072 cm⁻¹ with a FWHM of 93 cm⁻¹ is measured. Even though the stretching frequency is similar to that of the deposition at a flux ratio of 8 with rf bias generating 50 eV ions, the FWHM is considerably smaller for the material grown at the higher flux ratio without rf bias.

The -OH content of the depositions is compared by measuring the relative peak area of the absorptions occurring between 3350 and 3750 cm⁻¹. As expected, the peak area decreases as the flux ratio increases, as shown in Figure 7. There is a large decrease in -OH content when the flux ratio is increased from 8 to 16. Increasing the mean ion energy from 10 to 50 eV decreases the -OH content by 70% when the flux ratio is 8.

In Figure 8, the wet etch rate of silicon dioxide films deposited in both ECR configurations is shown as a function of the flux ratio. The etch rate values have been normalized to that of thermal silicon dioxide. For the mirror ECR configuration where the deposition surface was 19 inches from the ECR zone, the sample temperature started at 21°C and increased to about 120°C; etch rate data are shown as triangles in Fig. 8. For the high rate configuration depicted in Figure 3 where the deposition surface was 8 inches from the ECR zone, the samples reached about 200°C; etch rate data are shown as squares in Fig. 8. The wet etch rate is high at low flux ratio, and for flux ratios above about 22, the wet etch rate becomes constant at a value of two times that of thermal silicon dioxide.

More depositions were analyzed at $J/J_{ox} = 17$ or 31 at a fixed temperature of 65 , 100 , 150 , or 200°C . The data for wet etch rate and stress are listed in Table II. As expected, an increase in temperature results in reduced wet etch rate and increased compressive stress for both flux ratios. However, the etch rate at $J/J_{ox} = 31$ and 65°C is similar to that of the deposition at $J/J_{ox} = 17$ and 150°C , indicating that the higher flux ratio is effective at “replacing” temperature for reducing the etch rate.

In Figure 9, the -OH peak area from FTIR is shown as a function of flux ratio. As for the case of etch rate, the -OH peak area decreases as the flux ratio increases and it also becomes constant above a flux ratio of about 20. For flux ratios of 17 and 31, data are shown for depositions done at 65°C and at 200°C . The -OH peak areas are the same for these two temperatures, which is somewhat surprising. This suggests that -OH content depends primarily on flux ratio and not on temperature, up to 200°C .

4. DISCUSSION AND CONCLUSIONS

From the relationships shown in Figures 8 and 9, we conclude that above a flux ratio of about 20, high-quality silicon dioxide is deposited. Above this flux ratio, the wet etch rate is at its lowest at about twice that of thermal silicon dioxide. The -OH content is also at its lowest value. By fixing the deposition surface temperature at 65 , 100 , 150 or 200°C , it was found that very similar silicon dioxide films were produced when the flux ratio was high ($J/J_{ox} = 31$). This indicates that the flux ratio dominates the temperature over the range investigated.

With regard to ion energy, only a few experiments were conducted. It was found that the use of 50 eV mean ion energy at a flux ratio of 8 increased the Si-O stretch frequency considerably but that for a comparable value of stretch frequency, the FWHM was narrower by using a flux ratio of 28 without rf bias. In addition, as ion potentials rose higher than 50 eV, the oxide films became more and more compressive, whereas by increasing the flux ratio, the compressive stress saturated at a reasonable value of 200 MPa.

Taking a look at published work investigating the effect of ion potential on ECR-deposited silicon dioxide, similar conclusions are drawn about the effect of rf bias. For example, use of 50 eV ion energy in a distributed ECR system reduced the wet etch rate of SiO_2 films by a factor of 10 over the value for films grown with 10 eV ions; however, etch rate and fixed oxide charge remained the same for films grown with ions from 50 to 150 eV¹². For deposition conditions where we have estimated $J/J_{ox}=2-8$ in another distributed ECR system, use of rf bias from -20 to -100 V_{ac} produced films with the same value of Si-O stretching frequency⁹. In a mirror ECR deposition system for which we have estimated $J/J_{ox}=4-40$, the FTIR spectra of silicon dioxide films deposited with different values of rf bias were examined in detail¹⁰. An optimum rf-

induced dc bias of minus 50 V was found, based on the Si-O stretching absorption and its minimum FWHM. In another mirror ECR deposition system, the conclusion was drawn that use of rf-induced dc bias above -50 V produced SiO₂ films comparable to those deposited by LPCVD; the comparison was based on wet etch rate, refractive index, and FTIR analysis²¹.

Going back to the depositions described earlier, for which lower-quality SiO₂ was deposited with a flux ratio of 8 and ion energy of 50 eV and higher-quality SiO₂ was deposited with a flux ratio of 28 and ion energy of 10 eV, the suggestion is that raising the ion flux is more beneficial than raising the ion energy. By looking at these depositions in terms of average energy deposited per atom,²² where $E_d = E_i \times J/J_{\infty}$, we find that the lower-quality oxide is deposited with $E_d = 350$ eV and the higher-quality oxide is deposited with $E_d = 280$ eV. In other words, there is a difference depending on the energetic pathway that is chosen for deposition. This conclusion was reached earlier for a completely different material system, a study of reactive magnetron sputtering of polycrystalline TiAlN.²²

In conclusion, from our work investigating the importance of the ratio of ion flux to deposition flux in ECR deposition, a strong trend was found. For values above about 20 for this flux ratio, high-quality silicon dioxide films were deposited from 65 to 200°C. Addition of rf bias was also studied. From our work and from published literature there is some consensus that low energy ions, on the order of 50 eV, are sufficient for producing the highest-quality silicon dioxide, based on thin film characterization by measurement of wet etch rate, stress, and composition, and FTIR analysis. Returning to the question proposed in the introduction, that of choosing between a high flux of low energy ions or a low flux of high energy ions, the trend shown in our work and other literature cited is that a high flux of low energy ions is preferable for producing high-quality silicon dioxide at low temperature in high density plasmas. As for the implicit question of the role of ions in ECR deposition, the data indicate that ions densify, change the bonding of, and drive off -OH (or hydrogen) from depositing silicon dioxide. Lastly, although it does not add a lot to our understanding of the mechanism of silicon dioxide deposition in a high density plasma, the flux ratio itself is seen as an effective parameter for process development.

ACKNOWLEDGMENTS

We wish to thank, from Hewlett-Packard, Francoise Mertz for extensive assistance with experiments, Bill Collins for hardware support, and Paul Soo for Auger Analysis. Also, we are grateful to Vince Mazzi at Stanford University for setting up the Langmuir probe, and to Professor J. Leon Shohet at the University of Wisconsin for motivating discussions.

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Table I. Comparison of rf and ECR plasmas.

PARAMETER	rf plasma	ECR plasma
pressure (mTorr)	500	2
n (cm^{-3})	2×10^{16}	7×10^{13}
n^* (reactive species)	$< 10\% n$	$> 50\% n$
n_i (ions)	$10^6 n$	$10^2 n$
n^*/n_i	100,000	50

Table II. Comparison of $J_i/J_{ox} = 17$ and $J_i/J_{ox} = 31$ at 65, 100, 150, and 200°C.

Temperature	<u>$J_i/J_{ox} = 17$</u>		<u>$J_i/J_{ox} = 31$</u>	
	Etch rate	Stress	Etch rate	Stress
65°C	45 A/sec	50 MPa tens	17 A/sec	160 MPa comp
100°C	34 A/sec	tens/comp	10 A/sec	120 MPa comp
150°C	20 A/sec	90 MPa comp	10 A/sec	150 MPa comp
200°C	9 A/sec	200 MPa comp	6 A/sec	210 MPa comp

notes: Tens is tensile; comp is compressive; tens/comp is at the transition and too low to measure.

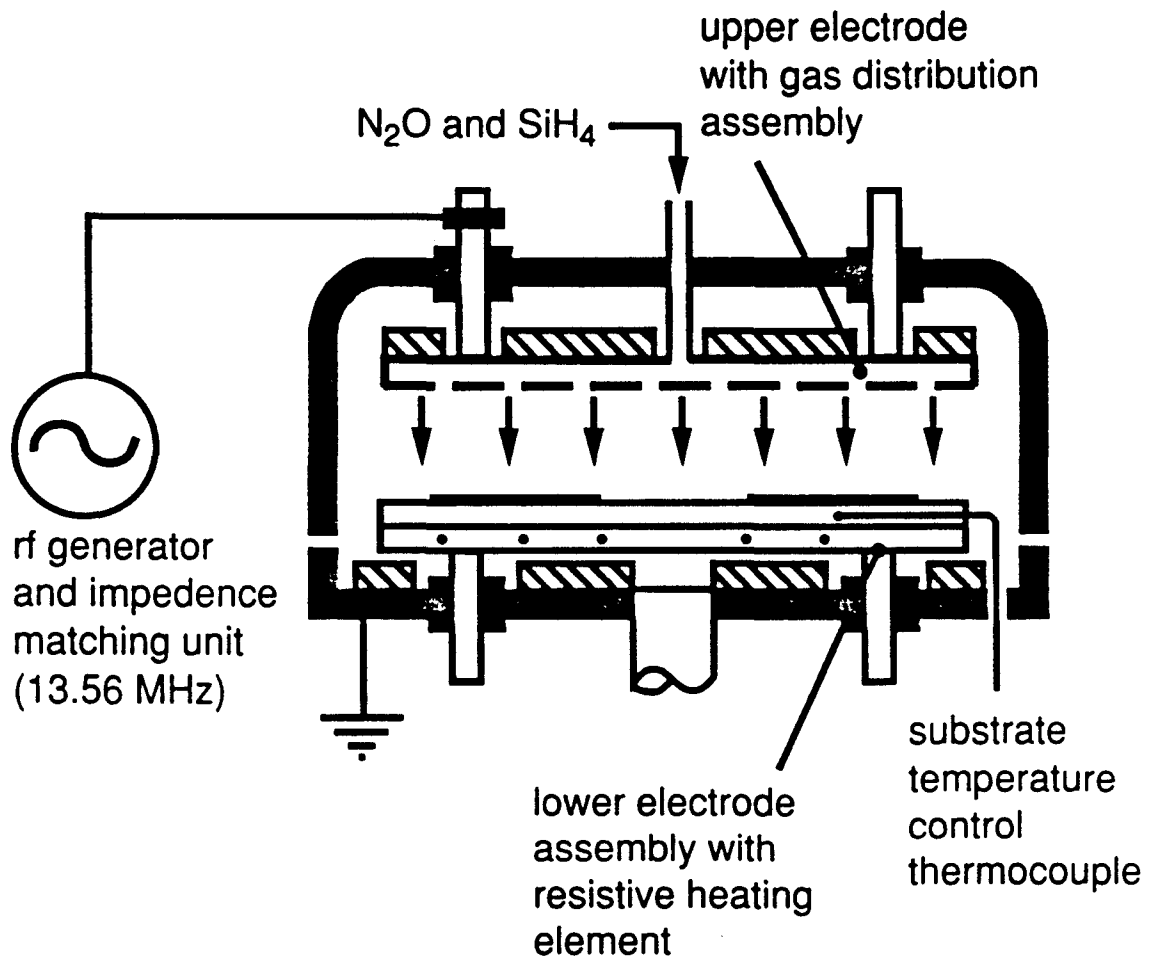


Figure 1. Schematic of rf PECVD system, from A. Kiermasz, A.A. Chambers, A. McQuarrie and M. Stephens, Semicon East, September 1986, Boston, MA.

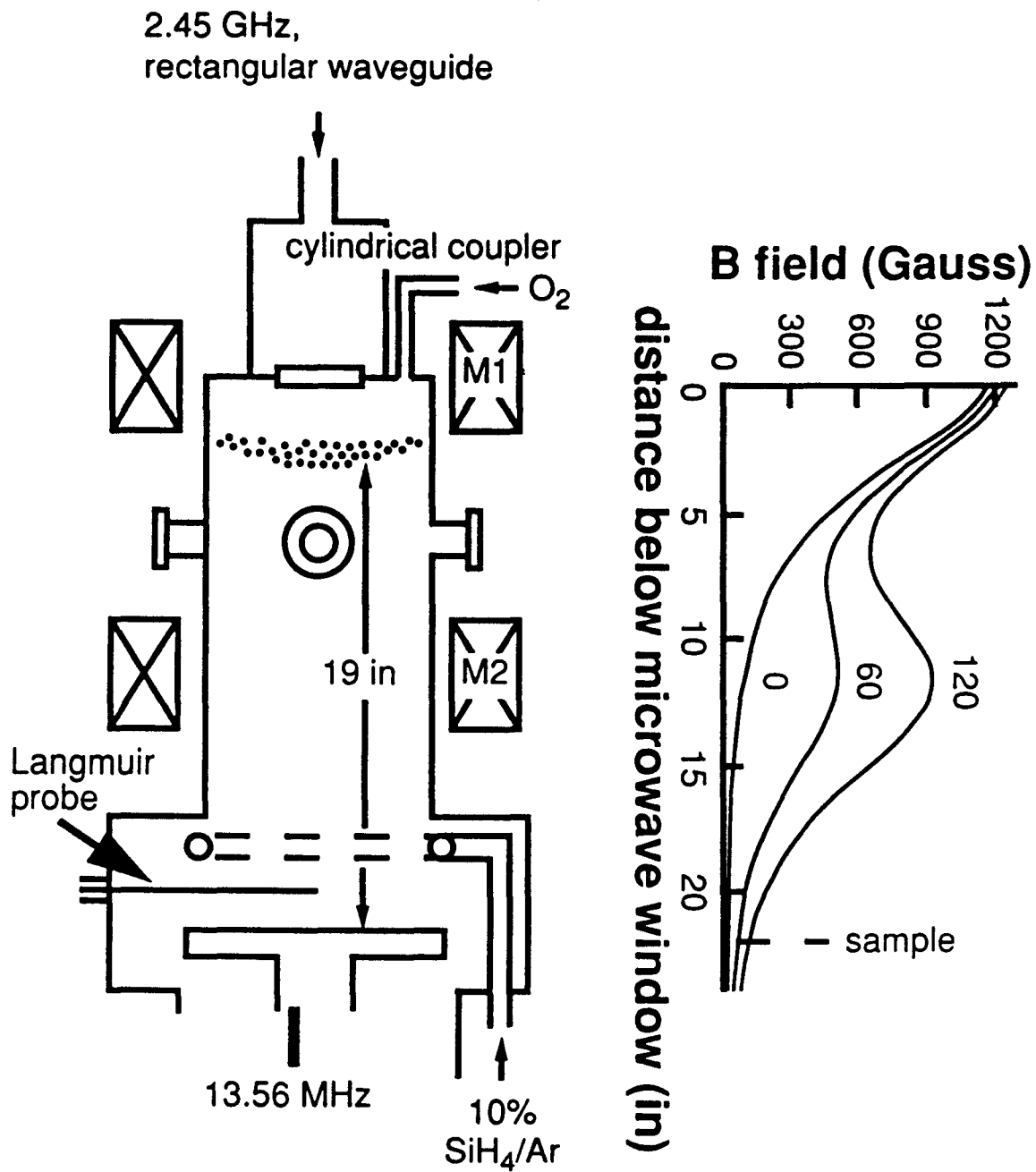


Figure 2. Schematic of ECR PECVD system with a mirror source configuration. The axial magnetic field corresponding to 170 Amps in M1 and varying the current in M2 from 0 to 60 to 120 Amps is included.

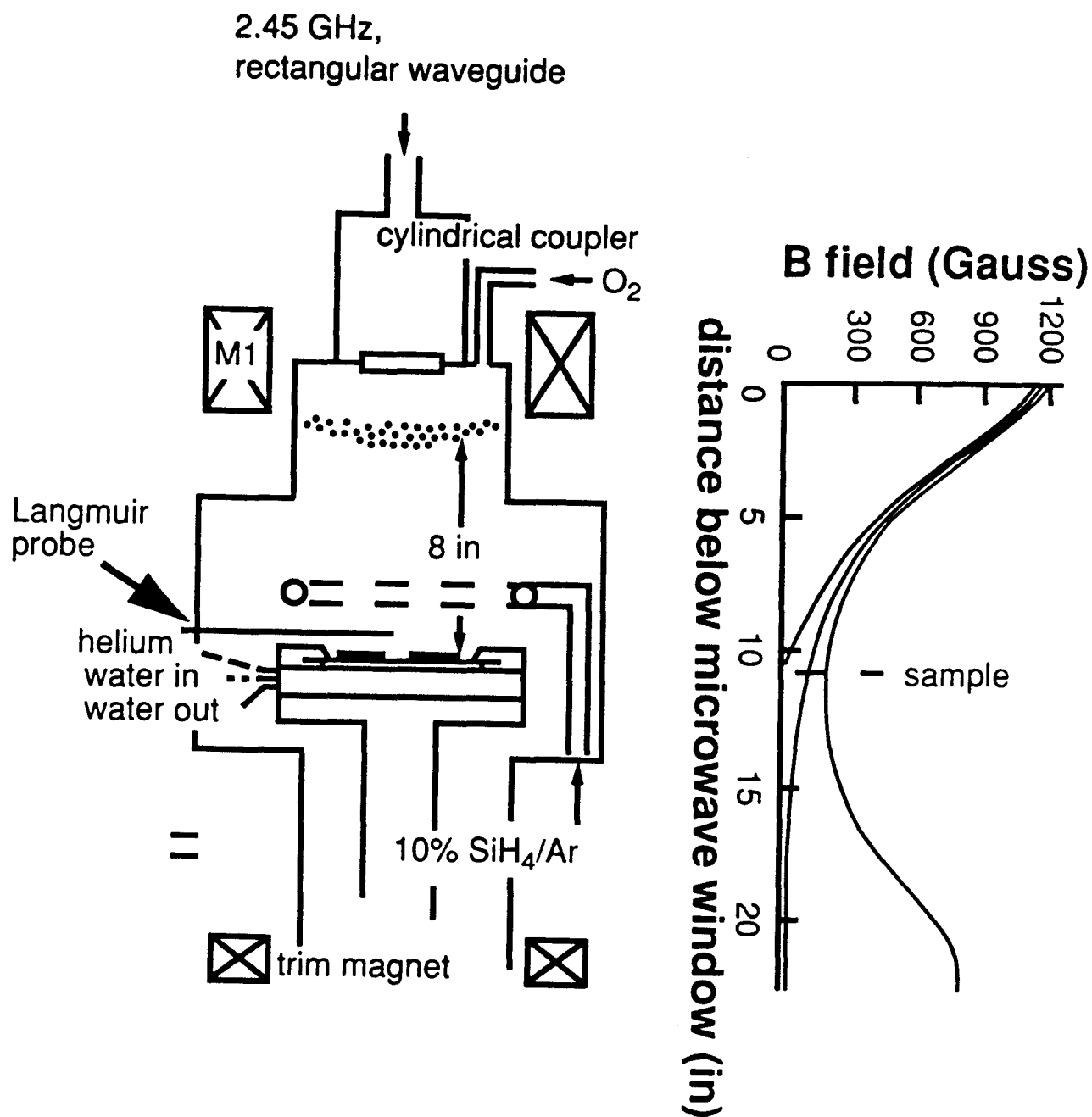


Figure 3. Schematic of ECR PECVD system with a divergent magnetic field source for deposition at high rate. The axial magnetic field corresponding to divergence, collimation and cusp configurations is included.

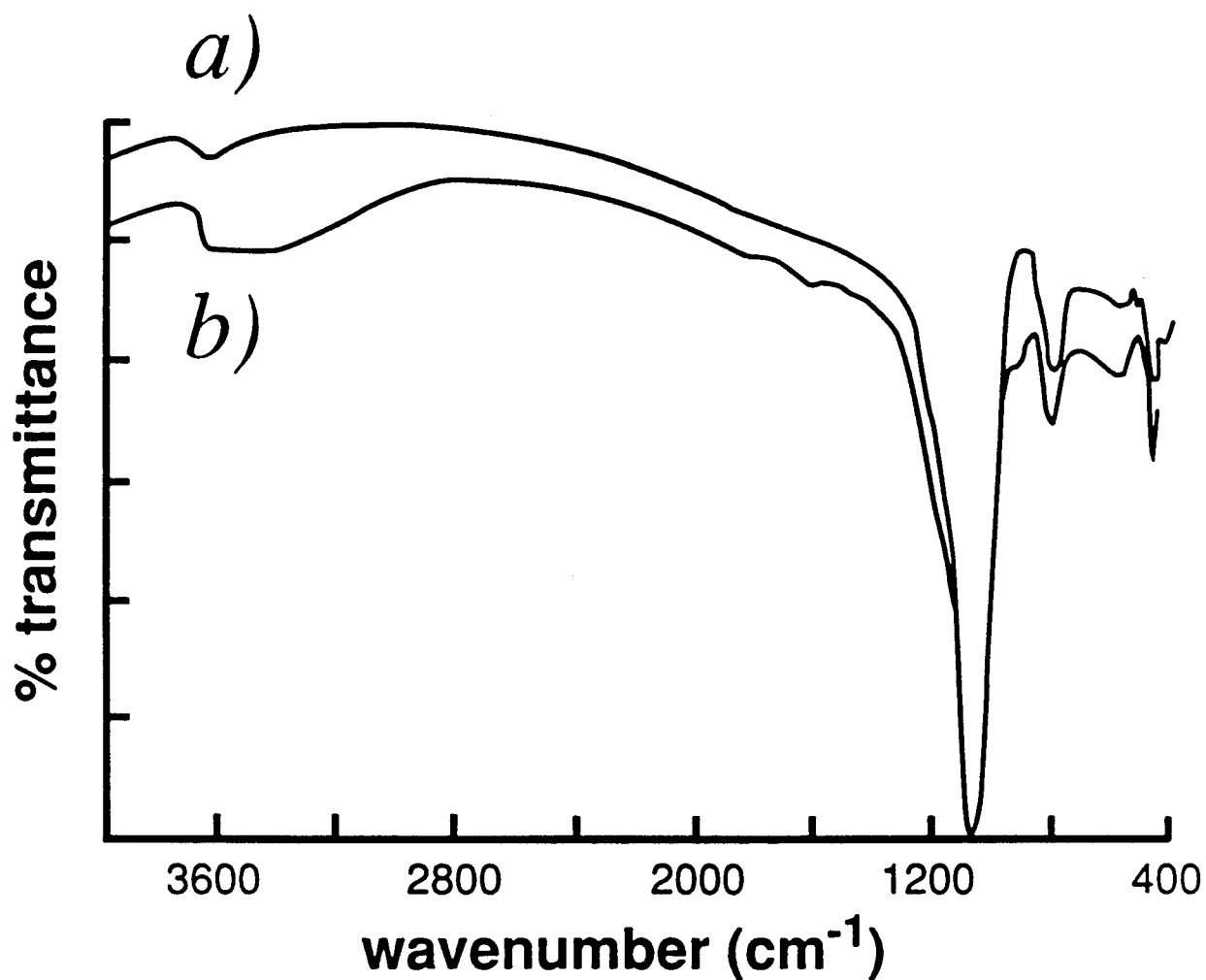


Figure 4. Fourier Transform Infrared Spectra of a) an ECR oxide grown with N₂O and 10% SiH₄ in N₂ with temperature floating up to 120°C and b) an rf PECVD oxide grown with N₂O and 2% SiH₄ in N₂ with temperature fixed at 200°C.

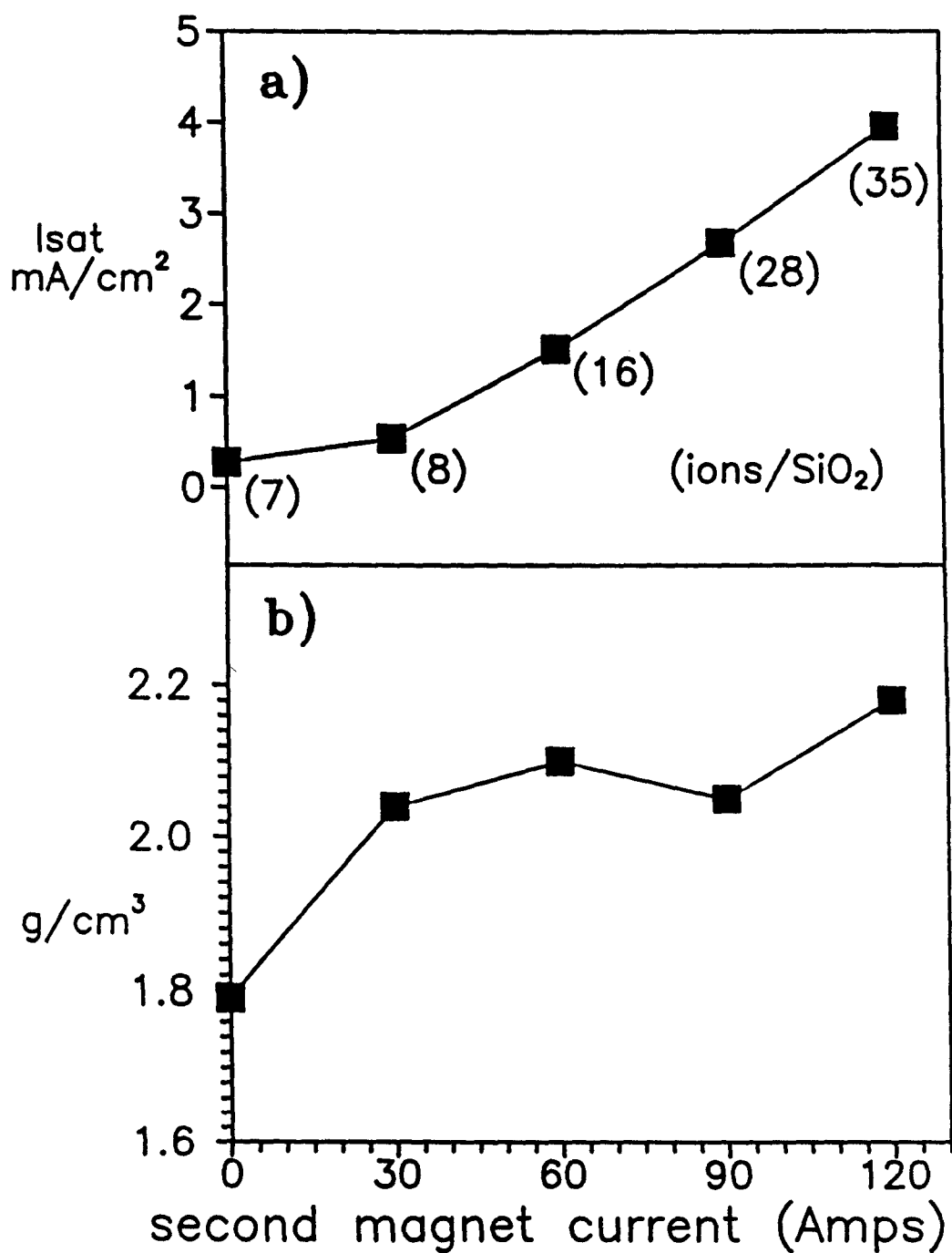


Figure 5. Measurement of a) ion saturation current and b) ECR oxide density as the current is increased in the second magnet of the ECR configuration shown in Figure 2. Ion saturation current is measured with a Langmuir probe and oxide density is measured by RBS. Also shown (in parentheses) is the flux ratio, J/J_{α} , as a function of the current in the second magnet.

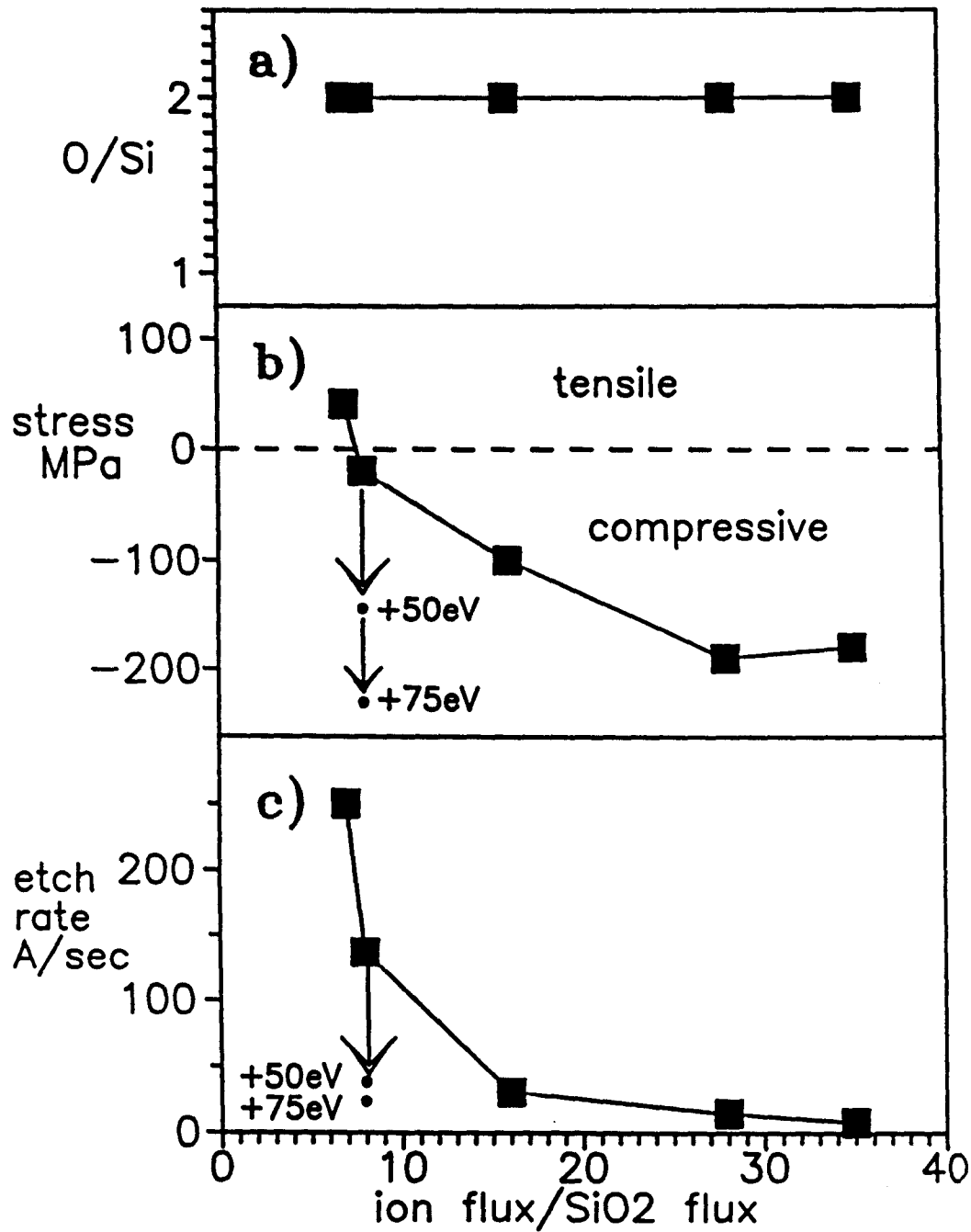


Figure 6. Measurement of a) oxygen-to-silicon ratio, b) oxide stress, and c) wet etch rate as a function of the flux ratio. Oxygen-to-silicon ratio was determined by both Auger and RBS. Stress was measured with a FleXus 2-300i and wet etch rate corresponds to a 10:1 $\text{H}_2\text{O}:\text{HF}$ solution. Also shown are the effects of increasing the mean ion energy to 50 and 75 eV by using rf bias during deposition.

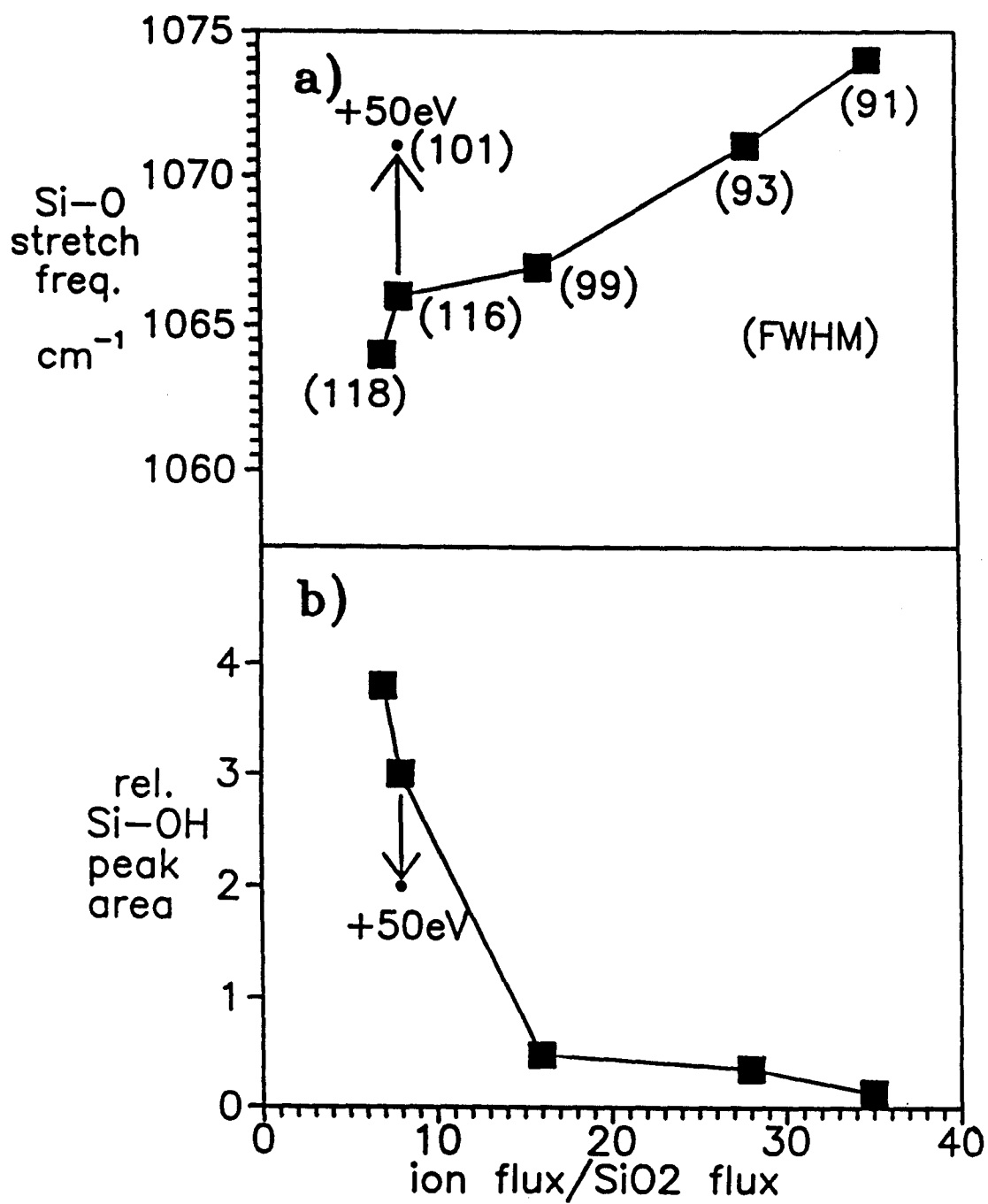


Figure 7. Changes, as the flux ratio is increased, in a) the Si-O stretching frequency and its full-width-at-half-maximum (shown in parentheses), and b) relative Si-OH peak area. Included are changes that occur by increasing the mean ion energy to 50 eV by using rf bias during deposition.

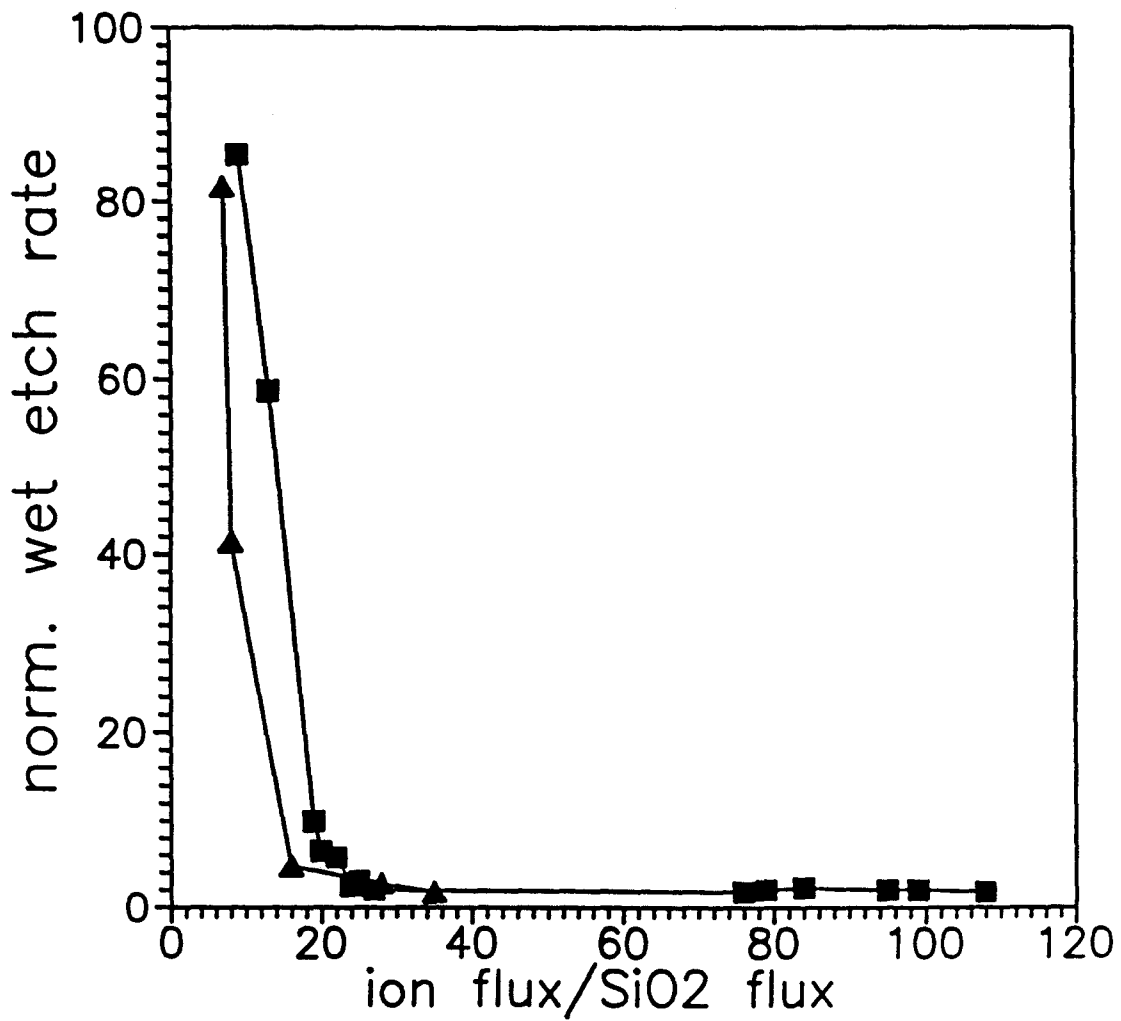


Figure 8. Decrease in wet etch rate in 10:1 H₂O:HF as the flux ratio is increased. Wet etch rate values are normalized to that of thermal silicon dioxide. Data for the mirror ECR configuration are shown as triangles; the temperature floated to 120°C for these depositions. Data for the high rate ECR configuration are shown as squares; the temperature floated to 200° C for these depositions.

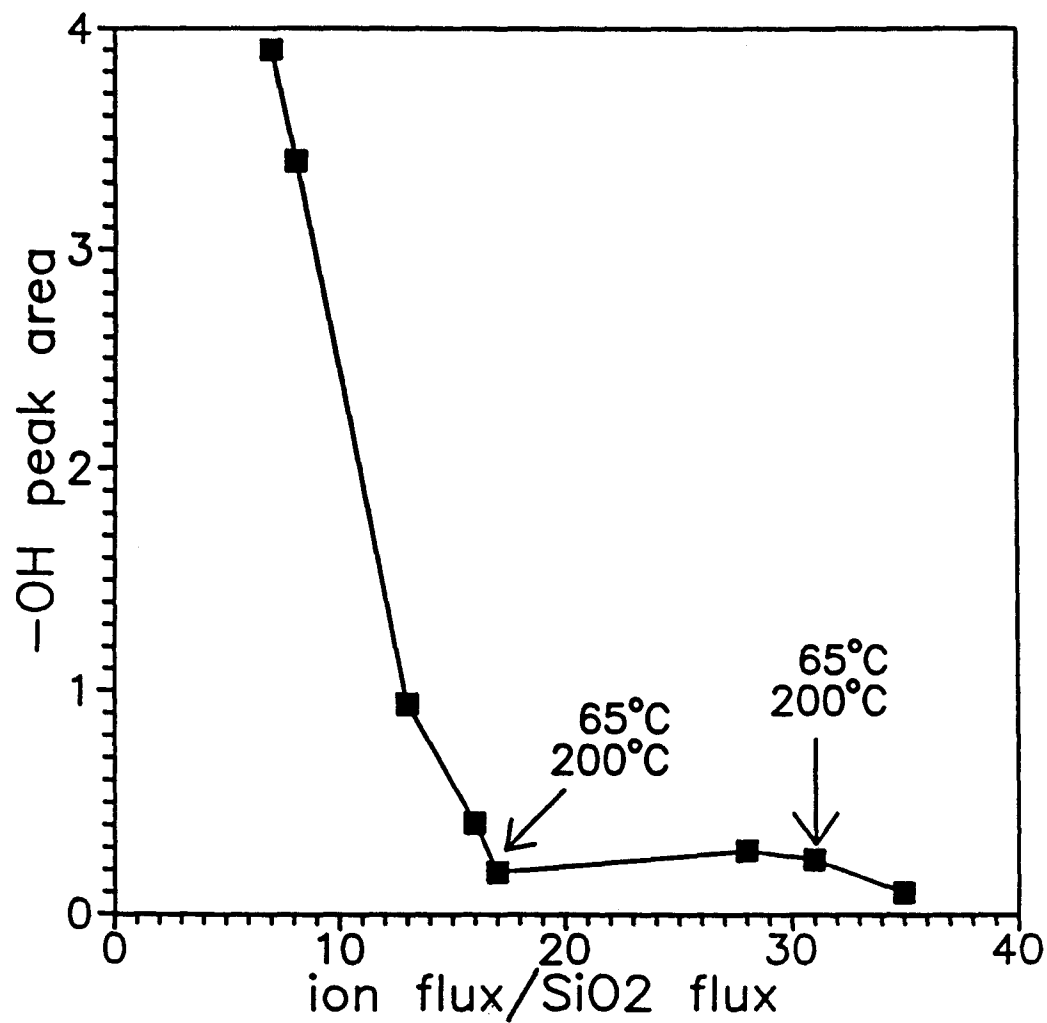


Figure 9. Decrease in the relative -OH peak area, measured on FTIR spectra, as the flux ratio is increased. Where temperature is indicated, the temperature was fixed. All other data are for a floating temperature of 120°C.