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The Role of Heat Transfer During Reactive-Ion Etching of Polymer Films

by

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The Role of Heat Transfer During Reactive-Ion Etching of Polymer Films

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ABSTRACT

A study of the kinetics of O₂ and Ar reactive-ion etching (RIE) of organic polymer films and an analysis of substrate heat transfer was carried out. Radiative heat transfer played a significant role in determining the substrate temperature profile during RIE. At the relatively low pressure of 5 mTorr, where anisotropic etch profiles are typically achieved, radiative heat transfer accounted for nearly 85% of the total energy (heat) flux away from the substrate. RIE processing time (substrate temperature) drastically affected the RIE rate of chain-scissioning polymers, which included poly(methyl methacrylate) and poly(α -methylstyrene), while processing time had absolutely no effect on etch rates of cross-linking polymers. To confirm the role of radiative heat transfer during RIE, the underside of a silicon wafer was painted flat-black, which increased the total radiative surface emittance and lowered the steady-state substrate temperature for a given set of RIE conditions. Ultimate etch rates of poly(methyl methacrylate), PMMA, were measurably lower for films on the black-silicon substrates. Thermal bonding alleviated substrate heating and greatly improved etch rate control, but the practical application of this method to a state-of-the-art auto-loading system remains highly suspect.

INTRODUCTION

Reactive-ion etching (RIE) is a critical plasma etching process used in the fabrication of fine-line semiconductor devices. The popularity of RIE stems from the ability to produce anisotropic etch profiles, accurately and reliably, using this dry etching technique. However, in order to extend the anisotropic etching capabilities of RIE to future generations of evermore complex chip-making processes, an improved understanding of the fundamental degradation processes, which take place during etching, is required.

In general, the RIE rate of organic materials, normally employed as resist materials in the integrated circuit industry, is influenced by the complex and often synergistic behavior of physical (ion) and chemical etching effects. At moderately low film temperatures ($T < 100^{\circ}\text{C}$), the RIE rate of

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polymer films appears to be limited by the ion energy flux (ion power density) to the surface [1], while the etch rate depends only to a small degree, if any, on substrate temperature. However, when processing times are increased, as in the etching of "thick" organic planarizing layers used in multi-layer resist (MLR) schemes [2,3], substrate temperature may become increasingly important. This is because RIE rates, in many cases, become more sensitive to temperature above some threshold temperature where, presumably, polymer degradation is influenced by other removal processes - i.e. thermally-activated depolymerization, thermal decomposition, and thermal oxidative degradation.

A recent study of the substrate heat transfer process taking place during RIE showed that substrate heating is due, almost exclusively, to ion bombardment [4]. The ion bombardment energy flux can raise the temperature of a silicon wafer (placed on the cathode) to well over 150°C in just a few minutes. Excessive surface heating can adversely affect the performance and processing latitude of a polymer resist films by increasing polymer etch rate during a process cycle and/or causing the polymer resist to "flow." The latter effect can result in some or complete loss of physical (image) integrity of the film, depending on the thermal dimensional stability of the polymer.

Previously, it was believed that substrate heating effects could be reduced by placing the substrate on a temperature-controlled electrode. However, in situ substrate temperature measurements showed that the quality of substrate-to-cathode thermal contact in this configuration is generally poor and insufficient to maintain set-point temperatures.

In light of these observations, it is surprising to note that very little work has been done in studying the effects of elevated temperature (T

> 100°C) on RIE rates of polymeric materials. A better understanding of these processes will help minimize the adverse effects of substrate heating thus leading to improved process control.

The goal of this work was to study, quantitatively, the relationships between the ion power density at the cathode, substrate temperature profile, and RIE kinetics of organic polymer films. This required the development and use of techniques that were capable of making accurate measurements of ion power density and polymer etch rate. These measurements were, in turn, used as input parameters to a mathematical model of the substrate heat transfer process, formulated earlier by Visser [4].

EXPERIMENTAL

The etching experiments were carried out using a custom-built, asymmetric, parallel-plate reactive-ion etching apparatus described elsewhere in the literature [5,6]. The aluminum cathode temperature was maintained at 25°C using a circulating coolant bath in all of the experiments. A He-Ne laser interferometer was used for accurate and non-intrusive determination of polymer etch rate and ion power density at each set of RIE conditions. The ion power density measurements ($\pm 10\%$) were made using the in situ substrate temperature measurement technique [1], in which the temperature of plate-glass substrates were monitored via laser interferometry [6,7].

The polymers studied included poly(methyl methacrylate) (PMMA), poly(styrene) (PS), poly(α -methylstyrene) (P α MS), and poly(chloromethylstyrene) (PCMS). PMMA was obtained as a 6% polymer solution in chlorobenzene and used as delivered (KTI Chemicals, MW=950k). Polymer solutions (6%) of the three styrenyl-based polymers, PS, P α MS, and PCMS, were prepared by dissolving the polymers in appropriate casting solvents.

Thin polymer films were obtained by spin-casting the polymer solutions onto 3 in. diameter silicon wafers at 1000 rpm for 60 seconds, followed by a hard-bake at 160°C for 60 minutes in a convection oven. Final film thicknesses, determined using an alpha-step stylus profilometer (Tencor Instruments, Inc.), were 1.3 to 1.5 μm . In some cases, "thick" films (3 to 5 μm) were used to ensure that the times for polymer stripping were comparable to the time for the substrate temperature to reach its saturation condition. The "thick" films were obtained by spin-casting and hard-baking three or four successive polymer film layers on the same substrate (one spin and one bake per layer).

To fully assess the effect of substrate heating, reactive-ion etch rates of films on thermally-bonded substrates were compared to etch rates of films on unbonded substrates (substrate placed in casual contact with the cathode) in each case. A thin film of high-vacuum grease (Dow-Corning) was applied in between the silicon wafer and cathode surface to thermally-bond the substrates. The nominal rf power density, pressure, and gas flow rate were 0.75 Watts/cm², 35 mTorr, and 20 SCCM, respectively, unless otherwise specified.

RESULTS AND DISCUSSION

Temperature Effects on PMMA Etch Rate in O₂ and Ar

Elevated substrate temperatures had a profound effect on the etch rate of PMMA, while thermal bonding of substrates to the cathode greatly improved etch rate control (figure 1). The etch rate (ER) of films on unbonded substrates increased two to three times relative to the initial rate, while those on thermally-bonded substrates were constant for all time.

Unfortunately, thermal bonding seems to be impractical for any high throughput

process since wafer handling is slow, complicated, and messy, and contamination levels (from the bonding agent) are unacceptably high.

Referring to the argon RIE results in figure 1, the ER increased from 250 to 550 nm/min after only one minute. Note also that the maximum ER in argon was comparable to the initial ER, but still less than the maximum ER in oxygen. These results are in partial agreement with those of Visser and de Vries, who reported that maximum etch rates of PMMA in argon RIE were equal to maximum rates in oxygen RIE [8].

These results demonstrate that thermal depolymerization can, under high (substrate) temperature conditions, contribute significantly to the RIE rate of PMMA. It has been suggested that the high-vacuum conditions during RIE processing should shift the equilibrium in favor of the "unzipping," or chain-depolymerization, reaction to lower temperatures, thus leading to higher etch rates [1]. However a set of crude experiments, in which film loss of PMMA was monitored with a stylus profilometer as a function of temperature under moderate vacuum conditions ($P \approx 100$ mTorr), failed to show any measurable film loss in the 0 to 270°C range [9]. Apparently, ion bombardment plays a role in promoting chain-depolymerization.

There was also a distinct difference in the laser interferometer waveform patterns, generated during etching, between thermally-bonded and unbonded films. In the case of a thermally bonded film, the laser reflectance amplitudes remained nearly constant throughout argon and oxygen RIE, indicating maintenance of film homogeneity. However, interferograms from unbonded film surfaces exhibited a monotonically decreasing amplitude with time due to a gradual increase in light scattering.

An earlier study, in which laser interferometry was used to study the RIE of polymer films, found that small changes in reflected intensity amplitude were due to small (0.1 to 0.2 μm) surface fluctuations on the film surface [10]. However, amplitude reductions measured at these conditions were much greater in magnitude than those one would expect from surface roughening alone, implying further that these effects were related to changes in film physical integrity. This idea is consistent with visual inspections of films that were subjected to two-minute and three-minute oxygen and argon RIE treatments, which showed that unbonded films turned opaque while thermally bonded films remained translucent.

Heat Transfer Analysis

To gain further insight into the heating effects observed for films on unbonded substrates, a study of the heat transfer process for a substrate subjected to RIE was carried out. When a substrate is placed in casual contact with the cathode surface and immersed in an rf plasma, the substrate temperature, T , begins to increase due to a constant bombardment flux of high-energy positive ions, q_+ . The rate of substrate temperature rise, dT/dt , depends upon the difference between the ion energy (heat) flux to the substrate and the rate of heat loss from the substrate to the surrounding, $q_L(T)$. This can be expressed as,

$$q_n(T) = \rho C_p L (dT/dt) = q_+ - q_L(T) \quad (1)$$

where $q_n(T)$ is the net heat flux to the substrate, and ρ , C_p , and L are the substrate mass density, specific heat, and thickness, respectively.

The heat loss function during the RIE process, $q_L(T)$, was found to be well-represented by the expression [4],

$$q_L(T) = 2\sigma_{SB}\epsilon(T^4 - T_o^4) + -k_g(dT/dz) + \alpha\Lambda_o P(T - T_o) \quad (2)$$

where σ_{SB} is the Steffan-Boltzmann constant for radiative heat transfer, ϵ is the surface radiation emissivity constant, T_o is the wall temperature (assumed constant), k_g is the thermal conductivity of the gas, dT/dz is the temperature gradient across the electrode gap (=5.0 cm), α is the accommodation coefficient for molecular heat transfer, Λ_o is the molecular thermal conductivity of the gas, and P is the gas pressure.

The first term on the right-hand side of equation 2 represents heat transfer from the substrate to the surroundings by radiation, while the second and third terms account for heat transfer by viscous (independent of P) and molecular (proportional to P) heat conduction to the gas. As noted earlier, solid-state heat conduction from silicon substrate-to-cathode during RIE processing was found to be negligibly small [4]. This was also the case for 0.10 cm thick plate-glass substrates [6].

Upon combining equations 1 and 2, the substrate temperature dynamics and equilibrium conditions can be determined to within the accuracy of the physical constants and q_+ . In this study, the values for q_+ were determined experimentally. In addition, 2ϵ for a silicon wafer was assumed equal to 0.30 [1]. For oxygen, the constants α , Λ_o , k_g , were assumed equal to 0.51, 1.19×10^{-4} W/cm²-K-Pa, and 2.6×10^{-4} W/cm-K [1,11].

Figure 2 shows that the heat loss function from a silicon wafer during oxygen RIE is a combination of radiative and molecular gas conduction. At low pressure (5 mTorr), the radiative heat flux term is about a factor of ten greater than either the molecular or viscous heat conduction terms. The molecular heat conduction term becomes comparable to the radiative term at 35 mTorr, and is about 2.5 times greater than the radiative term at 100

mTorr. The viscous heat conduction term remains small and its magnitude, relative to molecular heat conduction, diminishes rapidly above 100 mTorr.

RIE processing is often carried out at low pressure (< 35 mTorr) to ensure a high degree of etch anisotropy by minimizing inter-sheath collisions between fast ions and other gas molecules. Substrate heating effects are seen to be most pronounced in this regime, and thus, it is vitally important to maintain and control substrate temperature during RIE processing.

Relation of Substrate Temperature Dynamics to ER of PMMA

For unbonded films of PMMA in oxygen, there was very close agreement between ER and estimated temperature-time profiles. To aid in the analysis, ER and T (estimated) were expressed in terms of the fractional normalized response, defined as $[x(t)-x_0]/[x_{max}-x_0]$, where $x(t)$ is the value of ER or T at time t , x_0 is the initial value at time zero, and x_{max} is the maximum, or steady-state, value.

When normalized values of ER and T were plotted versus time, steady-state etch rates, which were obtained over a broad range of processing conditions, occurred consistently between 0.8 and 0.9. Thus, a first-order time constant, representing the time for the normalized temperature to rise to a value of 0.63, is a good predictor for estimating the point at which the effect of temperature on etch rate becomes significant.

Substrate Emittance Effects

Painting the underside of a silicon wafer flat-black increased the total surface emittance, 2ϵ , from an average of 0.3 to 0.96 [1]. As the constant 2ϵ is increased, the temperature difference between substrate and surroundings required to radiate away a constant heat flux is reduced.

Thus, surface emittance should also influence the etch rate of thermally-sensitive resists at elevated temperatures.

Figure 3 is a plot of oxygen RIE rate versus time for PMMA at two power levels. Silicon wafers (3 in. diameter) were used as substrates and were prepared in two ways: (a) substrate placed on electrode with no thermal bond, and (b) substrate placed on electrode with no thermal bond but with the underside painted flat-black. Numerical simulations predict that increasing 2ϵ from 0.3 to 0.96 will decrease saturation temperature from 160 to 105°C at 0.50 W/cm² rf power and from 200 to 130°C at 0.75 W/cm² rf power, respectively.

Etch rates were in line with expectations. Ultimate etch rates were measurably lower for the black-silicon substrates. PMMA etch rates on silicon were 18% and 65% greater than PMMA etch rates on black-silicon at 0.50 and 0.75 W/cm², respectively. A plot of $\ln(\text{ER})$ versus $1/T$, or an Arrhenius plot of the data, showed that etch rates became somewhat more sensitive at higher substrate temperatures. Activation energies, determined from the slope of the plot, were on the order of 0 to 1 kcal/mol in the low temperature regime, and increased by at least an order of magnitude above some break point. However, even in the high temperature regime, the activation energies were still much less than those normally associated with plasma-less thermal decompositions and/or autooxidations. The break point temperature, denoting the temperature at which the etch mechanism apparently changes, was not entirely obvious but it was certainly located between 100 and 150°C.

Styrenyl Polymers

As a rule, polymers containing some aromatic functionality are among the most RIE resistant organic materials. This superior etch resistance is attributed to the energy stabilizing effect of the phenyl ring as well its ability to act as a free radical "sink," which further inhibits oxidative degradation.

As was the case with PMMA, the thermal stability of aromatic-based polymers contributed significantly to the RIE durability. The oxygen RIE behavior of PS, PCMS, and P α MS are shown in figure 4. The physical integrity of the styrenyl-based polymers was, in general, much better than PMMA for both thermally-bonded and unbonded films. Laser interferograms, obtained for films of PS, P α MS, and PCMS on unbonded (silicon) substrates, did not show any of the excessive amplitude reduction that was commonly observed with PMMA.

Referring to figure 4, for a substrate temperature of 25°C, ER for P α MS was 500 nm/min, or 1.5 times faster than PS or PCMS films, which both etched at 330 to 350 nm/min. PCMS contains a labile chlorine atom which apparently led to the faster rate of etching.

The most striking feature to note is the distinct difference in ER for films on unbonded substrates between P α MS, for which ER doubled after 2.5 min., and PS and PCMS, for which ER remained independent of time (and substrate temperature). A plausible explanation for the superior RIE resistance of PS and PCMS over P α MS at elevated substrate temperature is that the degradation rate mechanism(s) for cross-linking polymers (PS and PCMS) are temperature-insensitive, while those for chain-scissioning polymers (PMMA and P α MS) are, or become, temperature-sensitive above 100°C or so.

These results were also somewhat correlated to the ceiling temperature, T_c , since T_c of PMMA (220°C) and PαMS (60°C) are both less than T_c of PS (300°C) [12]. The relationship between T_c and ER(T) would imply that rate-limiting step for the etching of PMMA and PαMS films was ion bombardment energy-limited for $T < T_c$ and rate-limited thermal depolymerization for $T > T_c$. It is well known that thermal degradation of PαMS occurs by random chain scission followed by polymer depropagation at elevated temperatures [13]. In fact, earlier studies of the thermal degradative stability of PαMS have reported zip lengths - the number of monomer units liberated per polymer molecule - in excess of 200 and monomer yields in excess of 95% [14].

SUMMARY

Heat transfer cannot be ignored during reactive-ion etching (RIE) of polymer resist materials. Substrate heating during RIE processing drastically affected the RIE rates of chain-scissioning polymers [poly(methyl methacrylate) and poly(α-methylstyrene)], while it had absolutely no effect on the RIE rate of cross-linking polymers [poly(styrene) and poly(chloromethylstyrene)]. Etch rates of chain-scissioning polymers increased 2 to 3 times over the initial rate in less than 3 min. compared to etch rates of cross-linking polymers, which were constant for all time (substrate temperature).

Thermal bonding the (silicon) substrates to the cathode greatly improved control of substrate temperature, polymer etch rate, and polymer film integrity, but the practical application of this technique to a state-of-the-art auto-loading system is highly suspect.

Ion energy fluxes were measured experimentally and used in an analysis of heat transfer, which showed that the main determining factors controlling substrate temperature were radiation and molecular heat conduction to the

gas-phase. Painting the underside of a silicon substrate was a remarkably effective way to control the etch rate of poly(methyl methacrylate).

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Figure 1: Effect of thermal bonding on argon and oxygen RIE rate of PMMA versus time. RIE conditions: 0.75 W/cm², 35 mTorr, 20 SCCM, -600 Vdc.

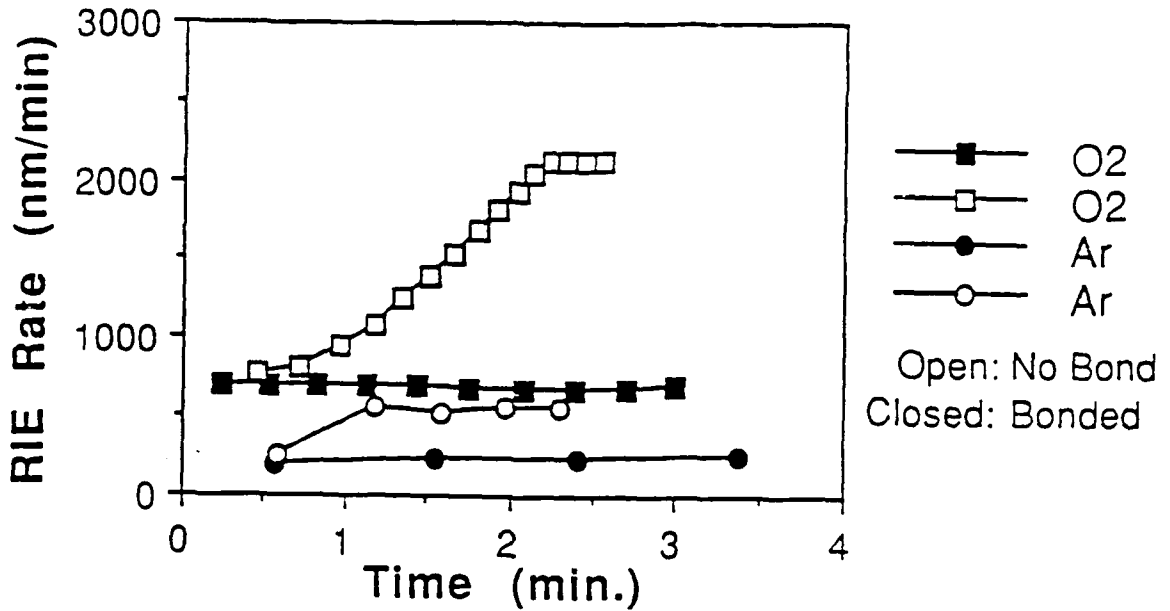


Figure 2: Calculated heat loss contributions as a function of pressure for a substrate cooling under typical RIE conditions. Total heat fluxes were determined experimentally.

Substrate Heat Flux vs. Pressure

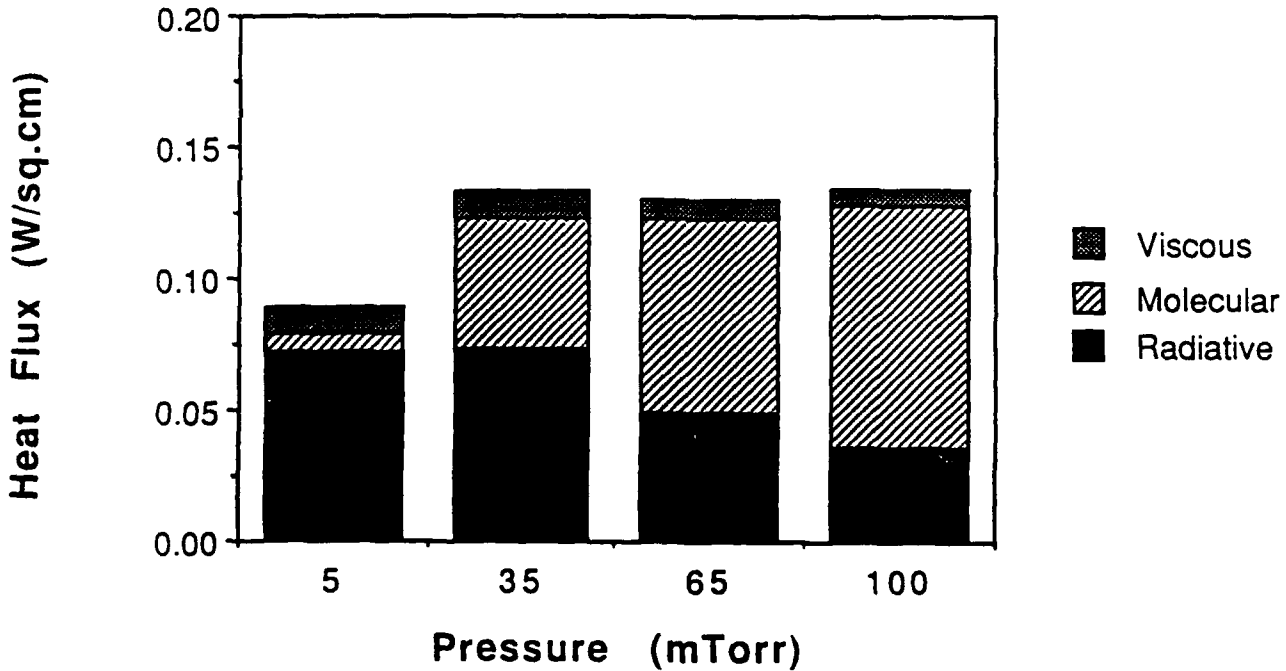


Figure 3: Effect of substrate emittance on oxygen RIE rate of PMMA. RIE conditions: 35 mTorr, 20 SCCM, 575 Vdc @.75W/cm² (circles) and 600 Vdc @.50 W/cm² (squares).

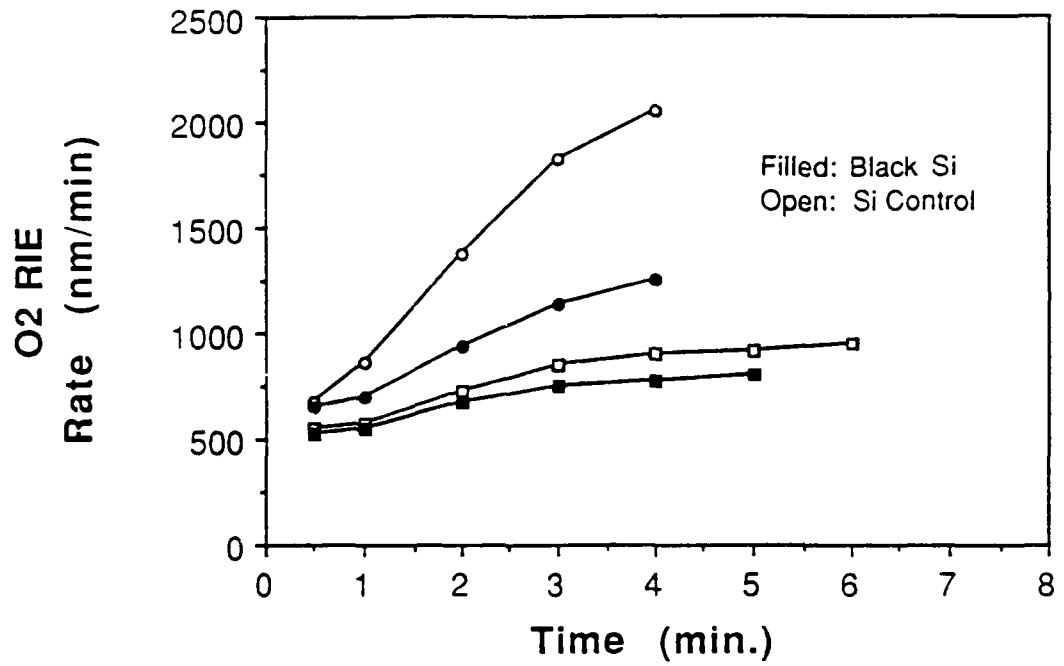
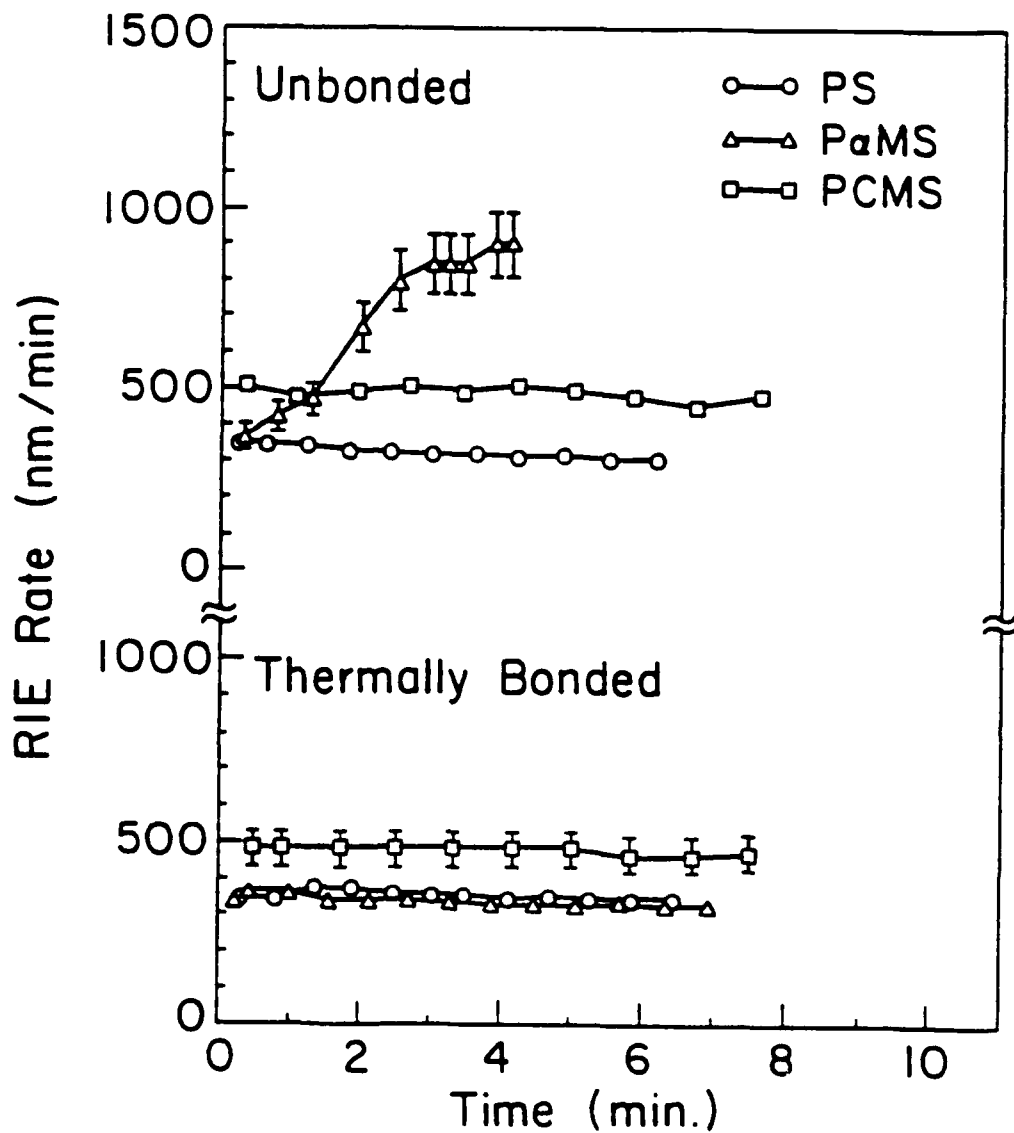


Figure 4: Etch rate versus time of styrenyl-based polymers. RIE conditions: 0.75 W/cm², 35 mTorr, -685 Vdc, T* ≈ 200°C.



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