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13. ABSTRACT *(Maximum 200 words)*

A collaborative effort to synthesize amino-substituted polyphosphazenes and examine their sensitivity to radiation has recently beeen undertaken. The objective was to determine the value of using such polymers as new and better resist materials for microlithographic applications. An initial study was carried out using elastomeric phenoxy-substituted polyphosphazenes as models for radiation and grafting.[1] However, it became necessary to synthesize several new amino-substituted polyphosphazenes for several reasons. First, the usefulness of elastomeric polymers for resist applications is severely limited by their inability to remain dimensionally stable at normal temperatures. Glassy polymers have been shown to provide the necessary thermal properties required for resist films deposited on SiO_2 wafers.[2-5] Second, the amino-substituted polyphosphazenes are excellent film-forming polymers with high molecular weights. Finally, it seemed possible that these polymers would exhibit similar reactive ion etching resistance to those of a phenoxy-substituted counterpart,[6,7] and experimental work was performed to determine reactive ion etching values for the most radiation-sensitive of the new polymer candidates.

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Radiation-Induced Modifications of Allylamino-Substituted Polyphosphazenes

by

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I. INTRODUCTION

A collaborative effort to synthesize amino-substituted polyphosphazenes and examine their sensitivity to radiation has recently beeen undertaken. The objective was to determine the value of using such polymers as new and better resist materials for microlithographic applications. An initial study was carried out using elastomeric phenoxy-substituted polyphosphazenes as models for radiation and grafting.[1] However, it became necessary to synthesize several new amino-substituted polyphosphazenes for several reasons. First, the usefulness of elastomeric polymers for resist applications is severely limited by their inability to remain dimensionally stable at normal temperatures. Glassy polymers have been shown to provide the necessary thermal properties required for resist films deposited on SiO_2 wafers.[2-5] Second, the amino-substituted polyphosphazenes are excellent film-forming polymers with high molecular weights. Finally, it seemed possible that these polymers would exhibit similar reactive ion etching resistance to those of a phenoxy-substituted counterpart,[6,7] and experimental work was performed to determine reactive ion etching values for the most radiation-sensitive of the new polymer candidates.

II. EXPERIMENTAL

Syntheses of allyl amino-substituted polyphosphazenes were accomplished using nucleophilic substitution techniques described previously[8-10] and represented in Figure 1. Following the synthesis of these polymers, molecular and materials' characterization was accomplished by means of ^{31}P and ^{1}H NMR, and elemental analysis, followed by GPC for molecular weight determination, and thermal analysis by DSC for T_g measurements. Films of 0.0045-0.0065 inch thickness, prepared from solutions of the polymer in THF, were cast on clean glass plate using a precision blade to spread the 20 wt.% solution. Deionized water was used to remove the dried film from the glass substrate. To determine their sensitivity to radiation, the polymer films were irradiated in a Gammacell 220 ^{60}Co γ-ray source manufactured by Atomic Energy of Canada Ltd. Films weighing approximately 0.0100 g were placed in sealed glass evacuated vials ($1 \times 10\text{-}6^{-6}$ torr) before exposure. The dose rate was 0.52 kGy/hour. Extraction of the soluble portion was performed by placing irradiated films in fritted glass vials (20-50 $\mu\mu$ pore size) and refluxing with THF to constant weight.

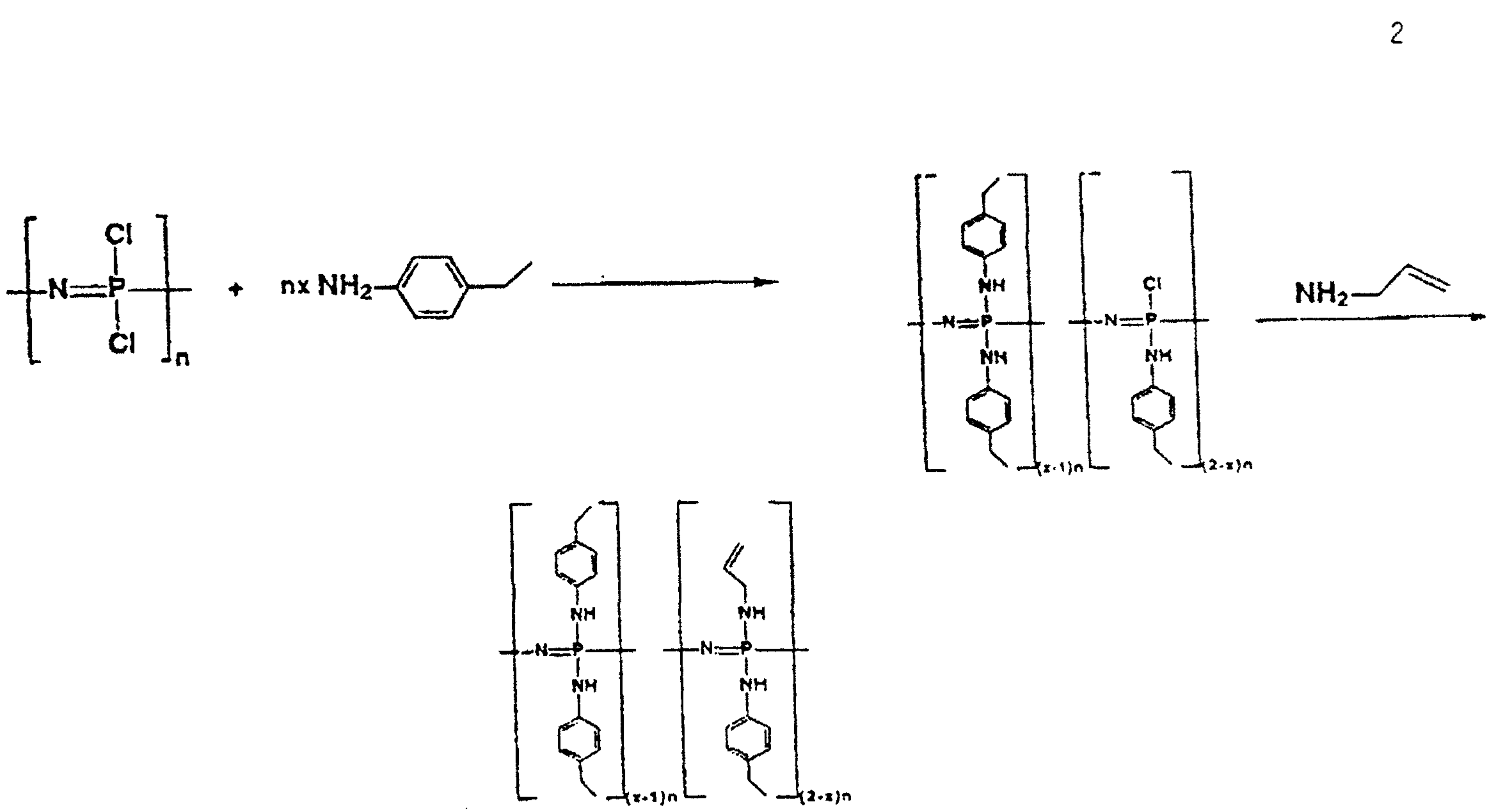

Figure 1. *Nucleophilic Aminolysis Reaction for Synthesis of Allyl-Amino Substituted Polyphosphazenes.*

II. EXPERIMENTAL

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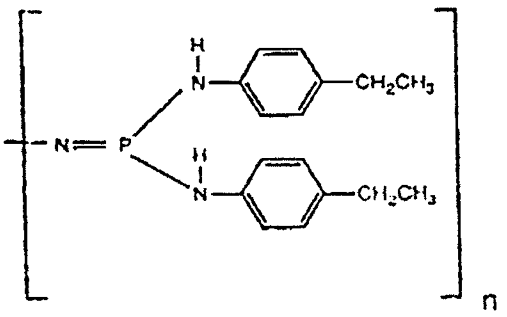
III. RESULTS AND DISCUSSION

Initially, model polymers were investigated for their sensitivity to both E-beam and gamma-radiation. Experimental determination of the G(X) (the number of crosslinks/100 eV) values for chemically different poly(organophosphazenes) was used as an initial indicator of their behavior as resist materials. In that study,[1] it was found that the presence of an allylic substituent (8 mol%) could greatly enhance not only gelation, but also the grafting to the elastomers of reactive monomers. The first attempt to synthesize an amino-substituted polyphosphazene specifically

A-1

tailored to provide better resist properties was unsuccessful because the polymer was quite insensitivie to radiation. Incorporation of the allylic groups into the amino-substituted polymers allowed for sufficient gelation after irradiation to suggest possible negative resist applications.

Experience with the irradiation of polyphosphazenes in any form (rubbery elastomer, fibrous glassy, or film) has been limited to that of only two or three groups of researchers, including those from both of these laboratories.[11-16]. Stannet et al[15,16] investigated eight different poly(organophosphazenes) and obtained Dg (the dose where the gel is first formed), G(X), and G(S) values for each. Depending on the nature of the substituent, the G(X) values found ranged from 0.050 to 2.49. The smallest G(X) value was found for the amino-substituted phosphazene, possibly due to the stabilizing influence of the "hindered amines" within its structure.[17] Studies by Beggiato.[18] Hiraoka,[19] and Lora[20] have reviewed the different aspects of irradiating polyphosphazenes, but neglected the use of irradiation grafting techniques. Recently, a study[21] was conducted where dimethylaminoethyl methacrylate monomer was grafted to various poly(organophosphazenes) to increase biocompatibility. Synthesis and characterization of allyl amino-substituted polyphosphazenes were carried out using previously described techniques.[8-10] Initially, the polymer shown in Figure 1, containing only 4-ethyl anilino substituents, was synthesized, characterized, and exposed to varying dosages of gamma radiation.



Tg = 86 C

Figure 2. *Structure of 4-ethyl-anilinophosphazene homopolymer.*

Unfortunately, this polymer was found to be very radiation-insensitive when exposed to gamma rays under vacuum (10^{-6} torr) even at temperatures of 95°C--well above the T_g. This stabilizing influence has recently been confirmed by adding a small amount (1.25 - 2.5 wt %) of the polymer to styrene monomer. This resulted in small but significantly retarded polymerization yields and molecular weights of the polystyrene. Thus, several syntheses were subsequently performed to obtain polymers with the three structures shown in Figures 3 (a), (b), and (c) below.

Work has been performed for two of the polymers to determine G(X) values. This involves the use of the Charlesby-Pinner (CP)[21-23] treatment which describes the determination of the gel fraction of the polymer as a function of radiation dose. Figures 4(a) and 4(b) illustrate that polymers with structures such as those shown for Polymers **1** and **2** are suitable for crosslinking when exposed to ^{60}Co radiation. Polymer **1** has been synthesized with approximately 7% of the allylic substituent, while the synthesis of polymer **2** allowed for 3-4% of the same allylic group.

The Charlesby-Pinner equation used to determine the G(X) values for these polymers is given as: $s + s^{1/2} = p_0/q_0 + 2/q_0MwD$ (1)

where: p_0 = density of main chain fractures per unit dose
D = radiation dose
q_0 = density of crosslinks per unit dose
s = soluble fraction of the polymer

and reduces to allow the simple determination of the G(X) value when p_0 is close to zero:

$$G(X) = \frac{4.52 \times 10^6}{Dg \times Mw} \qquad (2)$$

where; Dg = dose at which the gel first appears
Mw = weight average molecular weight as determined by GPC

This equation is very dependent on proper determination of Dg, which can be facilitated by using a method made popular by Lyons[24]--that of a log-log plot of $s + s^{1/2}$ vs. dose. Other restrictions in using this technique: (a) crosslinks are distributed randomly along molecular chains during irradiation, and (b) crosslinks are assumed independent of the absorbed dose. This was confirmed by the plots in Figures 4 (a) and (b), which indicate the best straight-line fit of the data goes through the origin.

Grafting experiments with purified acrylic acid have also been performed. We speculate that the stabilizing influence of the substituent has hindered the graftability of the allyl-amino-substituted polyphosphazenes. Attempted grafting with a 50/50 mixture of acrylic acid and water has resulted in little or no success. Pure acrylic acid was found to dissolve the polymer. The best polymer film candidate based on sol/gel analysis performed as described, was dissolved (5 wt.%) in methyl-isobutyl ketone (MIBK) and spin-coated onto a 4 in. SiO_2 wafer at 2000 r.p.m. using hexamethyldisilazane (HMDS) as a primer. Small sections of the optimum wafers were sectioned and subjected to a 15 KeV electron beam power source using an electron beam/scanning electron microscope lithography tool, as shown in Figure 5. The exposure was performed at 2×10^{-6} torr. Subsequent development was achieved in 10 seconds, again using THF as the solvent.

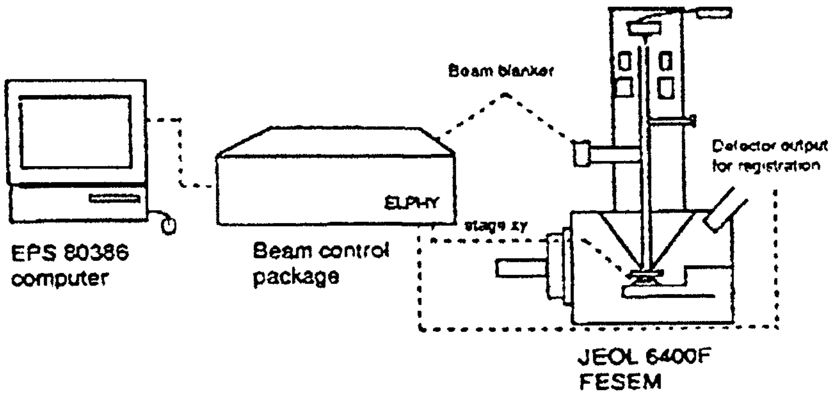
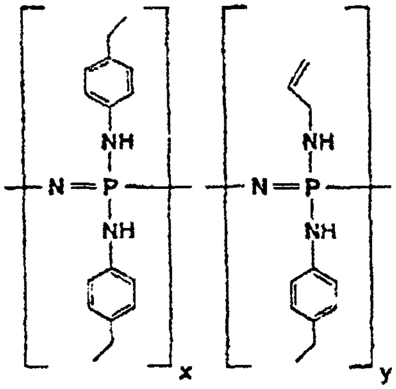
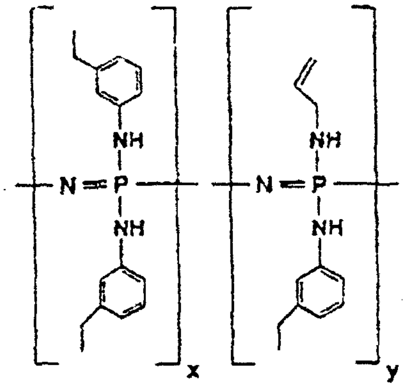


Figure 5. *Schematic Diagram of ERC Electron Beam Lithography Tool.*



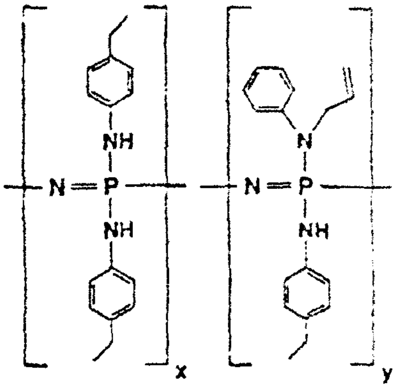
Tg = 87 C

Polymer 1
Figure 3(a).



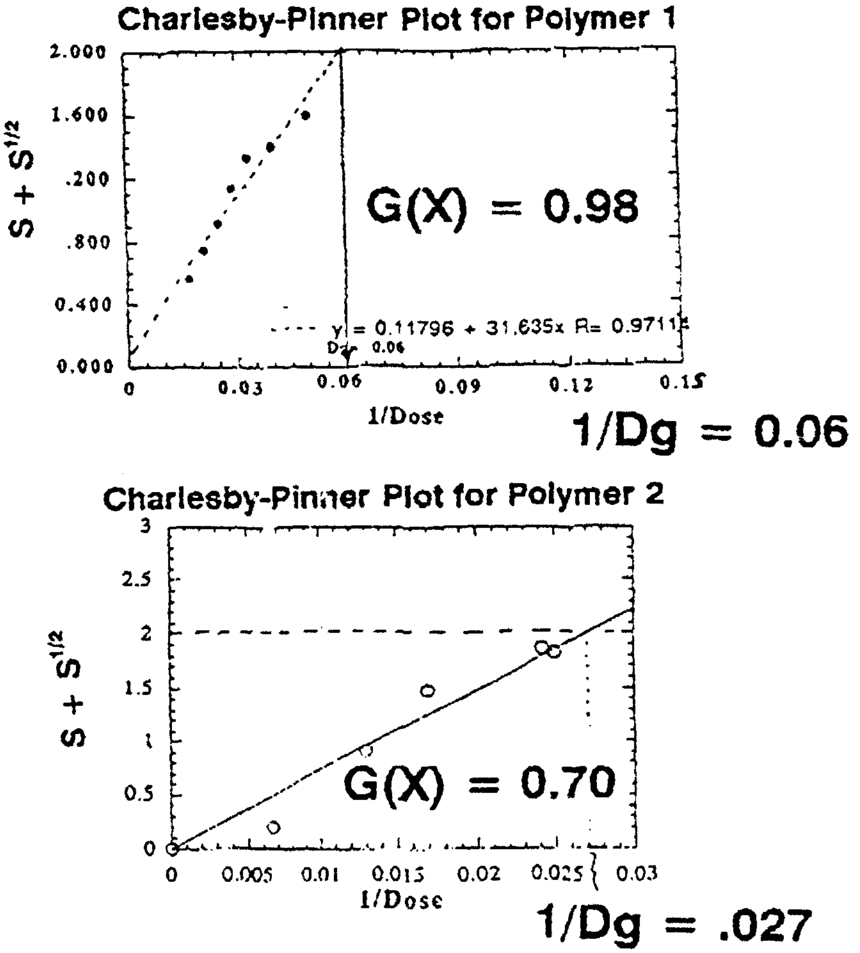
Tg = 52 C

Polymer 2
Figure 3(b).



Tg = ?

Polymer 3
Figure 3(c).



Figures 4(a) and 4(b). *Charlesby-Pinner Plots for Polymer 1 and Polymer 2*

The Charlesby-Pinner equation used to determine the G(X) values for these polymers

is given as: $s + s^{1/2} = p_0/q_0 + 2/q_0MwD$ (1)

where: p_0 = density of main chain fractures per unit dose
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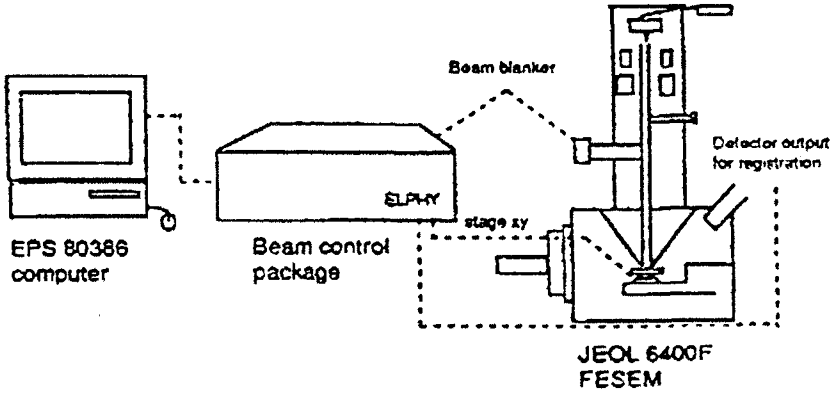

Figure 5. *Schematic Diagram of ERC Electron Beam Lithography Tool.*

Figure 6 illustrates a cross-section of an SiO_2 wafer with a 1780 Å film thickness of Polymer 1 spin-coated on its surface. Figure 7 is an E-beam lithographic pattern using a 15 KeV power source at 6 $\mu C/cm^2$ of the same wafer. Submicron resolution (0.1 - 0.5 μm) was achieved using THF as a developing solvent--development time was 10 seconds. No pre- or post baking of the wafer/resist was performed, yet adhesion of the polyphosphazene film was excellent.

Reactive ion etching rates as low as 585 Å/min. have been determined for this same water/film combination. These rates are compared in Tables 1 and 2 from a previous study and from the results of our work. The effect of the differences in vacuum and two types of equipment used for plasma etching is reflected in the etch rate differences shown.

Table 1. **Reactive Ion Etching Rates for Various Negative Resist Materials Based on Work by Hiraoka [6]. (-250 V Bias, 40 SCCM, 60 mTorr, 0.35 W/cm.^{2}).**

Resist Type	Etch Rate
Poly(diphenoxyphosphazene)[a]	30 A^0/min.
AZ-1350J	1300 A^0/min.
Silyated AZ-1350J	30 A^0/min.
Poly(chloromethylstyrene)	1400 A^0/min.

[a] UV-hardened films

Table 2. **Reactive Ion Etching Rates (RIE) for Allyl-amino Sunstituted Polyphosphazenes and Commercially Available Resist - (-250 V Bias, 40 SCCM, 900 mTorr, 0.08 W/cm.^{2})**

Resist Type	Etch Rate
Shipley 1400-31 Novolak Resin	4,224 A^0/min.
Polymer 1 - Exposed w/E-beam	585 A^0/min.
Polymer 1 - Unexposed	830 A^0/min.

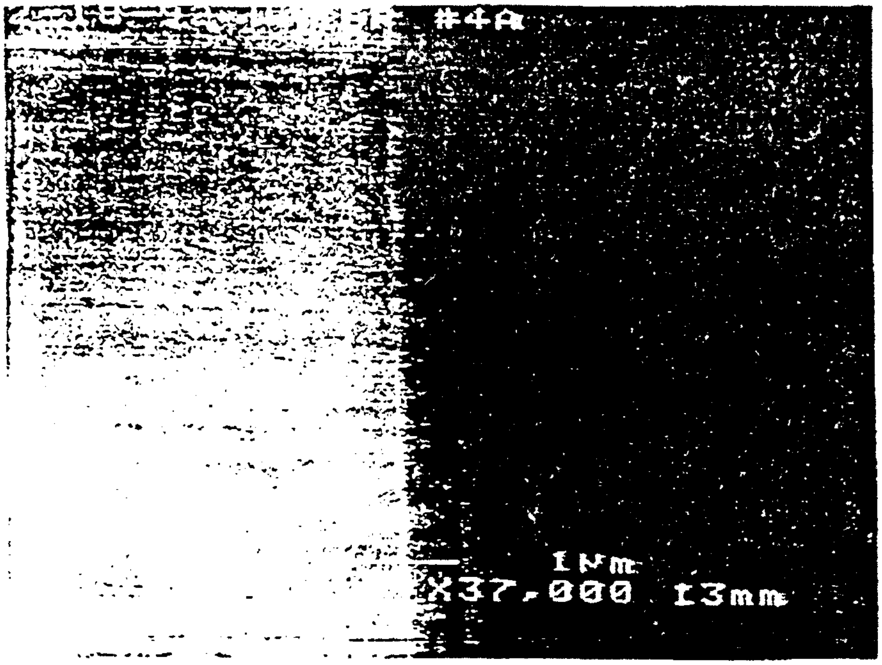

Figure 6. *Cross-section Microphotograph (37,000 X Magnification) of Polymer 1 w/7% Allylic Substituent on SiO_2 Wafer Indicating 1780 A° Film Thickness.*

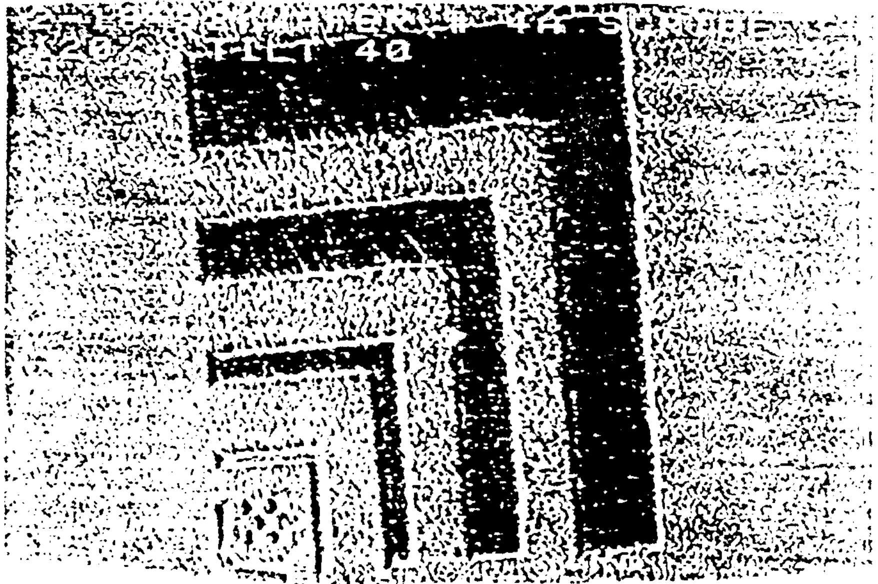

Figure 7. *Microphotograph (5,500 Magnification) of Patterned Wafer Using 15 KeV Electron Beam/SEM at 6 µC/cm.^{2} illustrating submicron (0.1-0.5 µm) resolution for Polymer 1. Development solvent/time is THF/10 seconds.*

IV. SUMMARY

It has been shown that allyl-amino substituted polyphosphazenes, specifically tailored to form films from suitable solvents can be used for negative resist/mask applications for microlithography. As suggested by earlier model studies, the addition of allylic double bonds to amino-substituted polyphosphazenes increases the sensitivity to radiation-induced crosslinking. However, it is probable that the stabilizing influence of the hindered amine substituent has reduced the graftability of these polymers. In addition, the inherent RIE resistance of the phosphazene family has been demonstrated, which underscores the potential usefulness of these systems for future resist work. Currently, other related polymer structures with higher allylic loadings are being investigated for enhanced radiation sensitivity.

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