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A review describing efforts to synthesize allylamino-substituted polyphosphazenes and characterize their sensitivity to radiation, and determine the value of using such polymers as new and better resist materials for microlithographic applications.

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RADIATION-INDUCED MODIFICATIONS OF ALLYLAMINO-SUBSTITUTED  
POLYPHOSPHAZENES

by

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# Radiation-Induced Modifications of Allylamino-Substituted Polyphosphazenes

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Efforts to synthesize allylamino-substituted polyphosphazenes and characterize their sensitivity to radiation and determine the value of using such polymers as new and better resist materials for microlithographic applications continues. An initial attempt to synthesize an amino-substituted polyphosphazene specifically tailored toward providing better resist properties was unsuccessful in that the polymer was quite radiation insensitive. The elastomeric models of an earlier study led us to the realization that the addition of allylic substituents might enhance the radiation sensitivity of such polymers significantly. Also, these amino-substituted polyphosphazenes were found to exhibit the glassy, thermal, and film forming properties required for conventional resists. Other potentially favorable resist characteristics of these polyphosphazenes includes relatively high RIE and high temperature resistance and good adhesion to SiO<sub>2</sub> substrates. This work indicates our progress involving the use of these polymer systems including results for G(X) values, (a measure of the efficiency of crosslinking) and grafting attempts to induce complete solubility changes. Silicon wafers coated with thin films of the allylamino-substituted polyphosphazenes were patterned with E-beam lithographic techniques and illustrated excellent sensitivity ( $<6\mu\text{C}/\text{cm}^2$ ) and resolution (to 0.1  $\mu\text{m}$ ).

A collaborative effort to synthesize amino-substituted polyphosphazenes and examine their sensitivity to radiation has been recently 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. In contrast, glassy polymers have been shown by many (2-5) to provide the necessary properties required for resist film formation on silicon wafers. Second, the amino-substituted polyphosphazenes are excellent film forming polymers with high molecu-

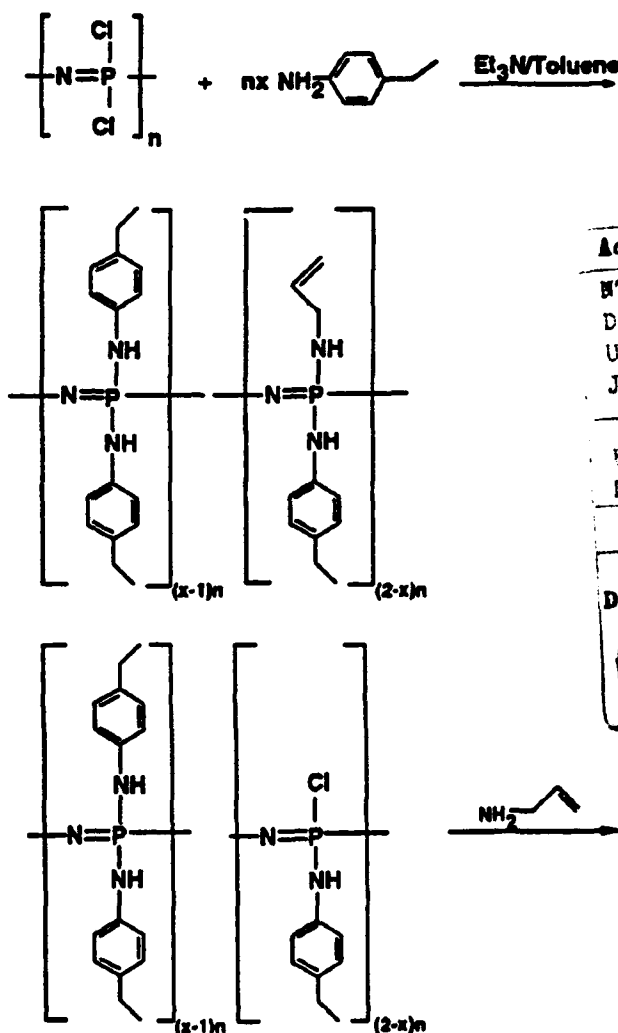
lar weights. Finally, it seemed possible that these polymers would exhibit similar RIE resistance to those of a phenoxy-substituted counterpart (6, 7), and experimental work was performed to determine RIE values for the most radiation sensitive of the new polymer candidates.

## EXPERIMENTAL

Synthesis of several allyl amino-substituted polyphosphazenes were accomplished via nucleophilic substitution techniques described previously (8-10) and are represented in Figure 1.

Following the synthesis of these polymers, molecular and materials characterization was accomplished by means of  $^{31}\text{P}$  and  $^1\text{H}$  NMR, and elemental analysis, followed by GPC for molecular weight determination and thermal analysis by DSC for Tg measurements.

Films of .0045-.0065" thickness, prepared from solutions of the polymer in THF, were cast on a clean glass plate using a precision blade to spread the 20



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Figure 1. Nucleophilic aminolysis reaction for synthesis of allyl-amino substituted polyphosphazenes.

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  $^{60}\text{Co}$   $\gamma$ -ray source manufactured by Atomic Energy of Canada Limited. Films weighing approximately .0100 g were placed in sealed evacuated glass vials ( $1 \times 10^{-6}$  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  $\mu\text{m}$  pore size) and refluxing with THF to constant weight.

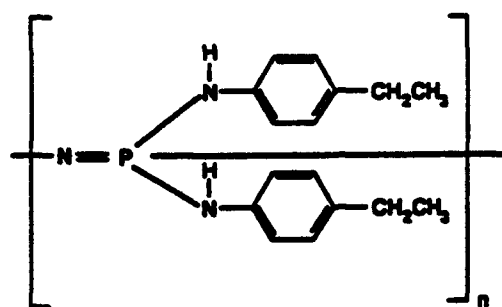
## RESULTS AND DISCUSSION

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 (8.5 mol%) could greatly enhance not only gelation, but also the grafting of reactive monomers to the elastomers. The first attempt to synthesize an amino-substituted polyphosphazene specifically tailored to provide better resist properties was unsuccessful because the polymer was quite insensitive to radiation. Incorporation of the allylic groups into the amino-substituted polymers, resulted in 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). Stannett et.al. (15, 16), investigated eight different polyorganophosphazenes and obtained  $D_g$  (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.

Grafting experiments with purified acrylic acid have also been performed in our laboratories. We speculate that the stabilizing influence of the amine substituent has hindered the graftability of the allyl-amino substituted polyphosphazenes. It is possible that the amine acts as an internal radical scavenger, thus trapping radicals formed during gamma or electron-beam irradiation. 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 these polyphosphazene polymers.

Synthesis and characterization of allyl amino-substituted polyphosphazenes were carried out using previously described techniques (8–10). Initially, the polymer shown in Figure 2, containing only 4-ethyl anilino substituents, was synthesized, characterized and exposed to varying doses of gamma radiation.

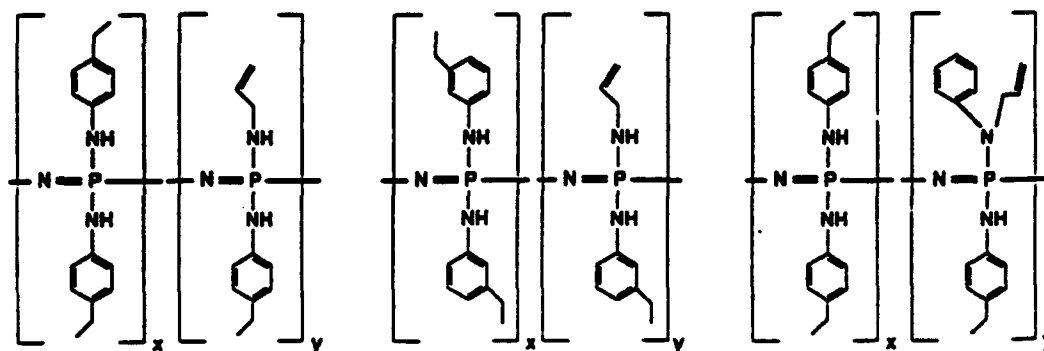


$T_g = 86\text{ C}$

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  $95^\circ\text{C}$ —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 lower molecular weights of the polystyrene. Subsequently, polymers with the three structures shown in Figures 3 (a), (b), and (c) were obtained, and  $G(X)$  values for two of the polymers were determined.

This involves the use of the Charlesby-Pinner (CP) (22–24) 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 as those shown for Polymers 1 and 2 crosslink when exposed to  $^{60}\text{Co}$  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. Glass transition temperatures for these polymers are not quite that of conventional resins such as polymethyl methacrylate ( $>100^\circ\text{C}$ ), and it is speculated that as allyl content is increased,  $T_g$  will decrease slightly.



$T_g = 87\text{ C}$

$T_g = 52\text{ C}$

$T_g = ?$

Figure 3. Structures of polymers 1, 2, and 3.

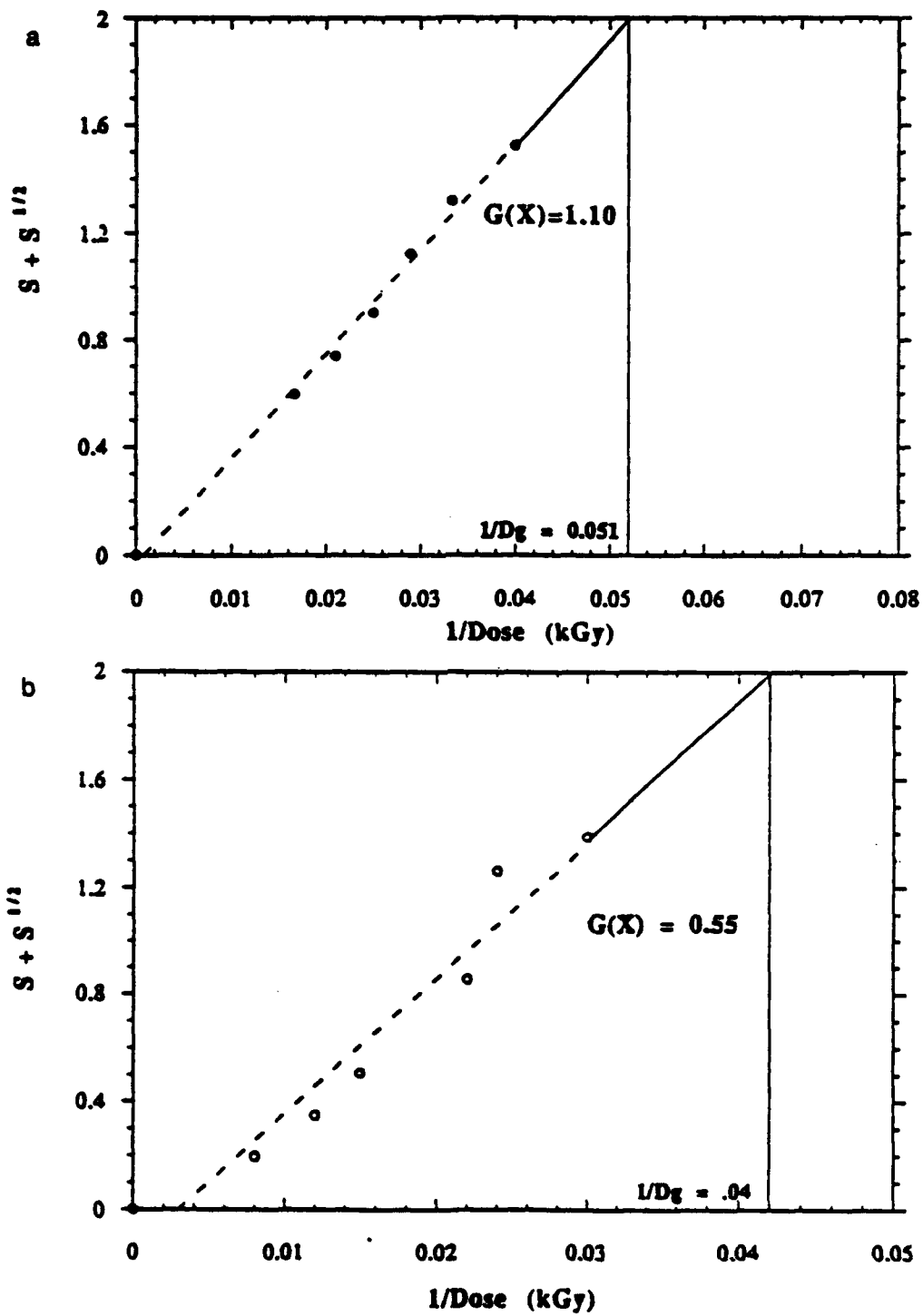


Figure 4. Charlesby-Pinner Plots for a, Polymer 1 and b, Polymer 2.

The Charlesby-Pinner equation used to determine the  $G(X)$  values for these polymers is given as:

$$s + s^{1/2} = p_0/q_0 + 2/q_0 M_w D \quad (1)$$

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:

$$G(X) = \frac{4.52 \times 10^6}{D_g \times M_w} \quad (2)$$

where  $D_g$  = dose at which the gel first appears in kGy

$M_w$  = weight average molecular weight

This equation is very dependent on proper determination of  $D_g$ , which can be facilitated by using a method made popular by Lyons (25)—that of a log-log plot of  $s + s^{1/2}$  vs. dose. Other restrictions in using this technique include:

a) crosslinks are distributed randomly along molecular chains during irradiation

b) crosslinks are assumed independent of the absorbed dose

This was confirmed by the data in Figures 4(a) and (b), which indicate the best straight-line fit of the data goes through or close to the origin.

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" silicon wafer at 2000 rpm using hexamethyldisilazane (HMDS) as a primer. Small sections of the more homogeneous wafers were sectioned and exposed to a 15 keV electron beam using an electron beam/scanning electron microscope lithography tool. The E-beam exposure of the wafer was performed under vacuum at  $2 \times 10^{-6}$  torr. Subsequent development was achieved in 10 seconds, again using THF as the solvent.

A cross-section of a spin-coated silicon wafer and SEM analysis indicated a 1780 Å film thickness of polymer 1 on its surface. Figure 5 is an E-beam lithographic pattern obtained using a 15 keV exposure at a dose of  $6 \mu\text{C}/\text{cm}^2$ . Submicron resolution (to 0.1  $\mu\text{m}$ ) was achieved using THF as a developing solvent and a development time of 10 seconds. No pre- or postbaking of the resist was performed, yet adhesion of the polyphosphazene film was excellent. Solvents other than THF were used for this system, however it was THF that was found not to cause the swelling found with conventional crosslinked resists.



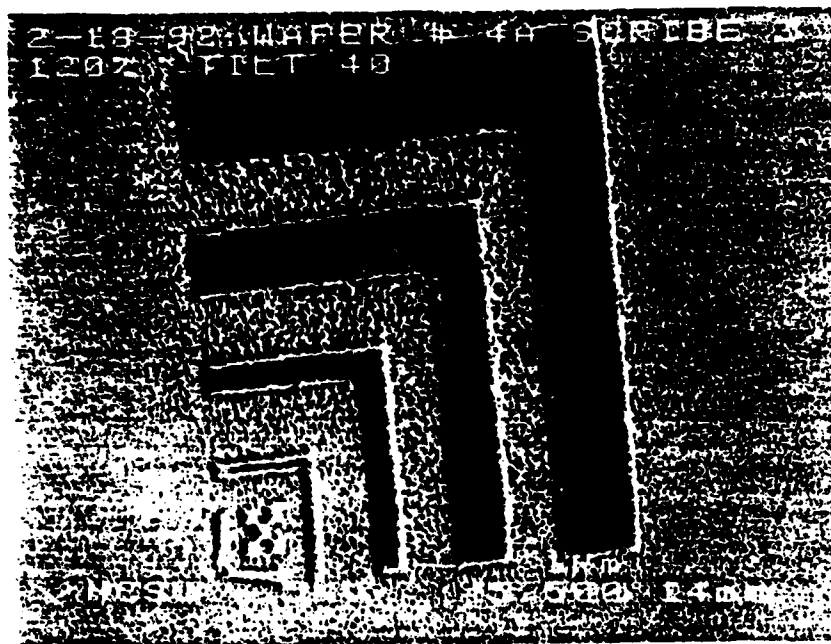


Figure 5. Microphotograph (5,500 magnification) of patterned wafer using 15 keV electron beam/SEM at  $6 \mu\text{C}/\text{cm}^2$  illustrating submicron ( $0.1 \mu\text{m}$ ) resolution for Polymer 1. Development time in THF was 10 seconds.

Reactive ion etching rates as low as 585 Å<sup>0</sup>/min. have been determined (after E-beam irradiation and subsequent crosslinking) for this film. These rates are given 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 between the diphenoxyphosphazene and the allyl-amino substituted derivatives. A more common description of the process performed at 900 mTorr would perhaps be "high pressure oxygen plasma etching", and is given simply for comparison purposes based on the only other work in this area performed by Hiraoka and Chiong (6).

A problem with the E-beam patterning of polymer 1 on the silicon substrate (SiO<sub>2</sub> over silicon) is the "growth" of unexposed regions or other regions near the unexposed areas, which were purposely "written" with the computer driven E-beam unit. These effects are universal when using E-beam writing tools, and are commonly referred to as "proximity effects". These effects are the major problem associated with establishing E-beam lithography for industrial use in the large scale production of microelectronic devices.

One type of proximity effect—the interproximity effect—involves the exposure of features near the ones that are intentionally exposed, referred to as near neighbors. Unexposed spaces between lines are exposed by backscattered electrons. This effect is seen in the work performed for this study. The interproximity effect causes unexposed spaces in positive resists to become thinner and narrower. For negative resists, such as ours, the unexposed region is filled with "scumming" resist.

Reduction of interproximity effects involves a series of trade-offs depending on the geometry of the desired patterned features. One of many methods to substantially reduce proximity effects for positive and negative resists, is by using a membrane 5-10 fold thinner than the dimension of the feature to be used (26, 27) over the actual resist layer. Because the proximity effect is almost directly proportional to resist thickness, multilayer resists are similarly practical solutions to this problem as well. Top surface imaging, thus again plays an important role in the future use of E-beam lithographic imaging. The use of a 6-Hema monomer at the very top surface of an already highly sensitive RIE resistant polyphosphazene, may be one method of overcoming the interproximity effects found in this study and is discussed in a future publication.

## SUMMARY

It has been shown that allyl-amino substituted polyphosphazenes, specifically tailored to form glassy films from suitable solvents can be used for negative resist/mask applications for microlithography. As suggested by 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 possible usefulness of these systems for future resist work. Currently, other related

**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<sup>2</sup>)**

Resist Type	Etch Rate
Poly(diphenoxyphosphazene) <sup>a</sup>	30 Å <sup>3</sup> /min.
AZ-1350J	1300 Å <sup>3</sup> /min.
Silyated AZ-1350J	30 Å <sup>3</sup> /min.
Poly(chloromethylstyrene)	1400 Å <sup>3</sup> /min.

<sup>a</sup> UV-hardened films

**Table 2. Reactive Ion Etching Rates for Allyl-Amino Substituted Polyphosphazenes and Commercially Available Resist Materials (-250 V Bias, 40 SCCM, 900 mTorr, 0.08 W/cm<sup>2</sup>)**

Resist Type	Etch Rate
Shibley 1400-31 Novolak Resin	4,224 Å <sup>3</sup> /min.
Polymer 1 - Exposed w/E-beam	585 Å <sup>3</sup> /min.
Polymer 1 - Unexposed	830 Å <sup>3</sup> /min.

polymer structures with higher allylic loadings are being investigated for enhanced radiation sensitivity.

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