Study of Base Additives for Use in a Single Layer 193 nm Resist Based Upon Poly(norbornene/maleic anhydride/acrylic acid/tert-butyl Acrylate)

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We report on a study of the chemical and lithographic behavior in a 193 nm single layer resist of two types of base additives, aminosulfonates onium and tetrabutylammonium carboxylate salts. These additives were examined because of their low potential for undesirable reaction with maleic anhydride or acrylic acid repeat units in norbornene/maleic anhydride/acrylate based 193 nm resins. For ammonium carboxylate additives it will be shown that using these gives comparable lithographic performance to a standard formulation containing an amine additive. For aminosulfonate onium salts a study was done of the relationship between chemical structure of these additives and their thermal stability and the lithographic performance imparted by these to a 193 nm single layer resist system. It will be shown that the decomposition temperature is a function of the basicity (nucleophilicity) of the counter anion decreasing with increasing basicity but can be improved by going from an iodonium to a sulfonium chromophore. Cyclamate and cysteate onium salts will be shown to provide good lithographic performance with good post-exposure bake delay latitude.

Keywords: Base additive, Photodecomposable base, 193 nm

1. Introduction

We have evaluated over the last few years the properties of a class of single layer 193 nm chemically amplified resists\cite{1-4} based upon an alternating copolymer of norbornene (NB) and maleic anhydride (MA) which also contains a small proportion (8 to 20\%) of another component such as tert-butyl esters of acrylic acid (TBA) and/or acrylic acid (AA). Among our studies have been that of the effect of various additives, photoacid generators (PAG’s)\cite{1,2} dissolution inhibitors (DI’s)\cite{3,4} base additives and photodecomposable bases (PDB’s)\cite{2}. This work will describe our recent work on base additives such as tetraalkylammonium carboxylates and also some further studies which we have done on photodecomposable bases such as the sweet PAG\cite{2}.

Base additives (either photochemically inert or photodecomposable) are important \cite{5,6} in imparting resistance to deleterious lithographic imaging arising from either acid diffusion, low residual acid concentrations in nominally unexposed areas, and base poisoning of acid at the resist air or resist substrate interface. Examples of deleterious behavior alleviated by base additives include T-topping, line footing, line slimming and other phenomena which limit either resolution or post-exposure delay latitude.

Many basic additives which have been discussed for use with 193 nm single layers
resists are based upon amine additive. Such materials may have a propensity to react with the MA repeat unit itself or with pendant carboxylic acid moieties (Scheme 1 and 2).

Scheme 1

\[
\text{R}_3\text{N} + \text{RCO}_2\text{H} \rightarrow \text{RCOO}^- + \text{R}_3\text{N}^- \text{H}
\]

In addition certain amino compounds can act as potential catalysts for ring opening of a maleic anhydride moiety. Indeed our earlier work on the addition of imidazole and dimethylaminopyridine to resist developers has shown an acceleration of dissolution rate attributed to anhydride ring opening\[7\]. Consequently, it was of interest for us to choose a less reactive (nucleophilic) basic moiety. Since lower nucleophilicity often parallels low basicity we have studied various low pKa additives containing carboxylate base additives, amino sulfonate, and combinations of the two (i.e. cysteate). We have looked at such additives either as non-photoreactive or photodecomposable base components.

2. Experimental.

2.1 Synthesis

The synthesis of poly(norborene/maleic anhydride/acrylic acid) (P(NB/MA/AA)), poly(norborene/maleic anhydride) (P(NB/MA)), and poly(norborene/maleic anhydride/acrylic acid/tert-butyl acrylate) polymers\[1\] and oligomeric cholate based dissolution inhibitors\[1c,d\] monomeric cholate dissolution inhibitor\[2\], and the cyclohexanecarboxylates, adamantane carboxylate DI’s were described elsewhere\[4\]. The Synthesis of bis(tert-butylphenyl)iodonium nonaflate and tris(tert-butylphenyl)sulfonium nonaflate were carried out similarly to previously described procedures\[4\]. Tetrabutylammonium hydroxide 1M solution, cholic acid, deoxycholic acid, lithocholic acid, adamantane carboxylic acid and trifluoroacetic acid were obtained from Aldrich Chemicals. The cholic, deoxycholic, lithocholic and adamantane carboxylic acid were dried under vacuum at 60°C overnight prior to use. The photodecomposable bases were prepared\[2\] in a manner similar to what has already been described for the sweet PAG starting from materials commercially available from Aldrich Chemicals. Characterization data for select samples follows:

**Preparation of tetrabutylammonium carboxylates**

These materials were prepared by mixing 10 mL of 1 M tetrabutylammonium hydroxide (actual titrated molarity 1.0493 M) with an equimolar quantity of a dry carboxylic acid. The reactions were stirred for 2 days in stoppered emmery flask to give in all cases clear solutions. Water was removed by freeze-drying (All yields >95% yield). The carboxylates were tested for solubility in 2-heptanone and all were read soluble to the extent of 1 wt % in 2-heptanone except for the cholate which was barely soluble enough to prepare the resist solution.

**Tetrabutylammonium cholate**

\[^1\text{H-NMR (DMSO d}_6\text{): 4.2(m, 1H), 3.68(m, 1H), 3.5(broad m, 4H), 3.18(m, 8H), 1.48(m, 8H), 0.71(s, 3H), 0.48(s, 3H)}\]

**Tetrabutylammonium deoxycholate**

\[^1\text{H-NMR (DMSO d}_6\text{): 4.2(m, 1H), 3.75(m, 1H), 3.35(m, 1H), 1.48(m, 8H), 0.82(t, 12H)}\]

**Tetrabutylammonium lithocholate**

\[^1\text{H-NMR (CDC1}_3\text{): 3.04(m, 8H), 1.28(m, 8H), 0.82(t, 12H)}\]

**Tetrabutylammonium trifluoroacetate**

\[^1\text{C-NMR (CDC1}_3\text{): 3.04(m, 8H), 1.28(m, 8H), 0.82(t, 12H)}\]
Preparation of photodecomposable bases

Synthesis of tris(tert-butylphenyl)sulfonium cyclamate
A solution was prepared consisting of 2 g (2.23 mmol) of tris(tert-butylphenyl)sulfonium 4-methoxybenzenesulfonate dissolved in 15 mL of ethylacetate which was mixed with a solution of 3.5 g sodium cyclamate in 15 mL distilled water. After stirring overnight the organic layer was separated away and stirred with another fresh aliquot of sodium acetate in water for another 24 hours. This was repeated two more times. Finally, the organic layer was washed with distilled water and stripped of solvent. In this manner 1.5 g of white crystals were isolated.

'H-NMR (CDCl₃): 1.26 (s, 12 H), 13C-NMR (CDCl₃): 157.0, 131.1, 128.3, 122.2, 52.6, 35.3, 34.5, 30.9, 25.9, 24.1.

NMR Characterization of PDB derivatives

PDB₂ 'H-NMR (CDCl₃): 8.13 (d, 4H), 7.52 (d, 4H), 3.3 (d, 3H), 2.5 (s, 2H), 1.27 (s, 18H).

PDB₃ 'H-NMR (CDCl₃): 7.84 (d, 4H), 7.36 (d, 4H), 3 (m, 2H), 1.8 (m, 4H), 1.27 (s, 18H).

PDB₄ 'H-NMR (CDCl₃): 8.0 (m broad, 1H), 7.89 (d, 4H), 7.28 (d, 4H), 4.1 (m, 1H), 3.2 (m, 2H), 2.35 (m, 2H), 1.27 (s, 18H).

PDB₅ 'H-NMR (CDCl₃): 8.10 (d, 4H), 7.50 (d, 4H), 1.26 (s, 18H).

PDB₆ 'H-NMR (CDCl₃): 7.8 (d, 4H), 7.51 (d, 2H), 7.37 (d, 4H), 6.51 (d, 2H), 1.26 (s, 18H).

PDB₇ 'H-NMR (DMSO d₆): 8.70 (d, 1H), 8.13 (d, 4H), 8.02 (d, 1H), 7.53 (d, 1H), 7.51 (d, 4H), 7.3-7.4 (m, 3H), 6.50 (d, 1H), 3.10 (m, 2H), 1.27 (s, 18H).

PDB₈ 'H-NMR (CDCl₃): 7.83 (d, 4H), 7.40 (d, 4H), 2.96 (m, 3H), 2.42 (m, 2H), 2-1.5 (m, 4H), 1.5-0.8 (m, 7H), 1.27 (s, 18H).

2.2 Lithography

Preparation of resist solutions for tetrabutylammonium experiments

Resist materials were prepared by combining tris(tert-butylphenyl)sulfonium nonaflate, a resist polymer, a dissolution additive, and the base additive. Specifically, the formulation contained about 15% by weight solids (e.g., polymer, dissolution, inhibitor, base additive and PAG). The amount of PAG in the formulations was 2% of solids (i.e., the PAG, polymer, base additive and dissolution additive). The total amount of dissolution additive was 25 wt% of the solids consisting of 1 wt% tert-butyl cholate and 24 wt% tert-butylcholate glutarate dimer and 0.36% of tetrabutylammonium cholate (or an equimolar amount of one of the other bases for other formulations. The formulation solutions in 2-heptanone were passed through PTFE filter membranes (0.2-0.45 micron) prior to casting. Films were formed on HMDS primed silicon wafers by spinning the wafers at a speed (1,100-2,000) so as to obtain a film thickness of 0.4017 microns and then baking the wafers at 145°C for 90 seconds. The films were exposed to radiation with a wavelength of 193 nm and a dose which range from 1 to 50 ml/cm². The exposure tool used was an ISI 0.6 NA (numerical aperture) small field catadioptric exposure system. The tool was used with optics that reduced the image by a factor of ten, and the tool had a partial coherence of 0.7. After exposure the films were baked at 160°C for 120 seconds.

Preparation of resist solutions for photodecomposable base experiments

The formulation was similar to above except that that bis(tert-butylphenyl)iodonium nonaflate was employed as PAG (2 wt% loading.) In this instance also the total DI loading consisted of 34% total DI (5% tert-butyl cholate, 30% tert-butyl cholate glutarate dimer) For base the loading of PDB1 (sweet PAG) was 0.36% (other formulations were prepared with equimolar amounts of base). Resist films were evaluated on spun on silicon (~0.5 μm). Exposures were done with ISI 9300 (0.60 NA, 0.70 sigma). The softbake condition (PAB) was 140°C for 90 seconds; the post exposure bake (PEB) was 155°C for 90 seconds. Development was done with OPD-262 (8 second stream/24 second puddle).

3. Results and Discussion

One effect of using strong base additives in a norbornene/maleic anhydride/acrylate (P(NB/MA/TBA/AA) based 193 nm resin is seen if one compares the clearing doses of this type of resist system to that of a phenolic based 248 nm chemically amplified resist (Fig. 1). From this study (Fig. 1) it is apparent that there is little variation in clearing dose for the latter with changing pKₐ. This is probably because the former system is being leveled to the pKₐ of a carboxylic acid either through reaction with MA or AA repeat units.
Amines can also have a deleterious effect on stability towards hydrolysis (Fig. 2) during long term study exposure to moisture. Fig 2 shows an IR study done of the accelerated aging of resists.
solution after 6 weeks at 40°C. This study shows that 0.2 wt % of triphenylimidazole dramatically accelerates the hydrolysis of anhydride with water. This change in hydrolysis rate is also evident from the increase it causes in the rate of dark erosion after aging. Specifically, although formulations with or without the amine initially have a similar dark erosion of ~2.7 Angstroms/sec, after 6 weeks of aging at 40°C, the formulation containing the amine additive has increased its dark erosion to 26.0 Angstroms/sec while the formulation without this additive has only increased to 5.3 Angstroms/sec. Consequently, it is probable that the amine is acting as a catalyst for the hydrolysis of the resin anhydrides in the resist solution in a manner similar to what we have previously observed upon addition of amine additives to TMAH based developers [7]. Since, as just discussed, strong amine bases level resists to the carboxylate pK_a weaker bases were studied as base additives. We have studied two classes of materials with lower pK_a's carboxylate tetrabutylammonium salts and aminosulfonate opium salts. Furthermore, since both of these species are generally less nucleophilic than most simple amines they do not ring open MA repeat unit either through direct reactions or by acting as catalysts.

Tetrabutylammonium carboxylates
Table 1 list the carboxylates chosen for evaluation and gives some important physical properties of both the carboxylates and their corresponding free carboxylic acids. Table 2 lists the lithographic properties of different formulations containing these tetrabutylammonium carboxylates, a P(NB/MA/TBA/AA) resin, tert-butylcholate DI additives and a tris(tert-butylphenyl)sulfonium nonaflate PAG and compares them to the properties of a standard formulation containing an amine additive. Comparing the resist containing different non-volatile carboxylate (i.e. based upon high boiling point acids) additives there is a slight increase in resist sensitivity as follows:
lithocholate>deoxycholate>cholate>adamantane carboxylate. There is also a slight increase in dark erosion concurrent with the increase in resist sensitivity. Since the formulations contain equimolar quantities of the carboxylate additives this would suggest a corresponding increase in basicity of the carboxylates. Table 1 lists the predicted pK_a values for each carboxylate. The predicted pK_a's for lithocholate and the adamantane carboxylate bear out the differences in lithographic sensitivities but does not account for the differences observed for the cholate, deoxylate and lithocholate formulations since these additives should have similar pK_a's. However, since the decrease in thermal stability of these derivatives corresponds to their decrease in effectiveness as base additives, thermal decomposition during PAB and PEB
processing may be a better explanation for differences in lithographic sensitivity.
Two mechanisms (Schemes 3 and 4) are plausible to account for thermal decomposition
of these additives. Interestingly, the resist with the highest sensitivity also showed the highest
contrast by nearly a factor of two. However, this higher contrast does not result in any significant
improvement in lithographic performance (Table 2).

Scheme 3

Tetrabutylammonium trifluoroacetate was not discussed with the other carboxylates because its
behavior is anomalous. This carboxylate has a very low pKa (only 0.67 compared to the other
carboxylates at ~4) and it was expected to impart the least buffering ability and
consequently lead to resists with the highest sensitivity and greatest dark erosion. However, the
trifluoroacetate formulation gave a dark erosion and sensitivity more in line with a much
more powerful buffering agent. However, this can be explained by the high volatility of
trifluoroacetic acid which can be lost from the resist film during post-exposure bake effectively
removing acid from the resist film (Scheme 5). Thus although trifluoroacetate is a poor base, the
high volatility of trifluoroacetic acid makes this material a far better resist buffering agent than
would be expected from its pKₐ. Fig. 3 compares the imaging capability of a formulation containing the trifluoroacetate to
that of a formulation containing a more conventional high basicity amine component,
tri phenyl imidazole (TPI) Evidently, the performance is quite comparable without use of a
strong amine which might undergo undesirable reaction with the MA repeat unit.

Scheme 5

Photodecomposable bases.
Fig. 4 gives the structure of bis(4-t-butylphenyl)iodonium cyclamate, the “sweet
PAG.” This nomenclature is based on the fact that one of the precursors to this material is
sodium cyclamate, a well-known artificial
sweetener, and that this molecule photogenerates
cyclamic acid (PAG=photoacid generator) upon
exposure to 248 or 193 nm light. Scheme 6
shows the photolysis of a typical aminosulfonate
PDB. Previously its use in single layer
chemically amplified resists has been
reported[2] for resists incorporating either low or high activation energy protecting groups. In a low activation energy resist the sweet PAG has the role of a typical PAG component in a chemically amplified resist but releases an acid which only affect deprotection during post-exposure bake. In such systems it also acts as a PDB component limiting diffusion of acid and eliminating small background concentrations of it in nominally unexposed resist areas.

For resist containing high activation energy groups, such as in the 193 nm single layer resist based on P(NB/MA/AA/TBA), the role of sweet PAG is restricted to that of a PDB and another onium salt which photo-releases a stronger super acid must be used as PAG component. However, the use of sweet PAG in high activation energy resist formulations drastically reduces both line slimming and increases post-exposure delay (PED) latitude[2]. Moreover, the use of such PDB additives gives a much lower resolution doses than employing a non photo-active basic analog[2]. Specifically, comparing two formulations of P(NB/MA/TBA/AA) base containing equimolar amount of bis(tert-butylyphenyl)iodonium cyclamate or dicyclohexyl ammonium cyclamate (a non-photoactive basic analog) shows that the use of this PDB additive decreases the clearing dose by almost half [2]. Fig 5 shows the structure of the other anionic moieties now evaluated for their properties as PDB's as the bis(tert-butylyphenyl)iodonium salt derivatives.

These new materials were evaluated in a resists consisting of P(NB/MA/TBA/AA) resin, tert-butylcarboxylates DI's and bis(tert-butylyphenyl)iodonium nonaflate PAG. The new PDB's similarly to the sweet PAG all were aminosulfonate but had different basicities due to either different spacing groups between the sulfonate and amino moieties or in the case of PDB4, the cysteate anion, the presence of a carboxylate functionality.

For use in resist formulations as a PDB these materials had to pass two screening test, adequate solubility (at least 0.2 wt % in 2-heptanone containing 18 wt % P(NB/MA/TBA/AA) polymer) and they had to have a thermal stability of at least 150°C to be able to withstand typical resist thermal processing.

Table 3 summarizes the results of these screening tests showing that only five materials had both enough solubility and were sufficiently thermally stable for further evaluation. Interestingly the thermal stability of the aminosulfonate PDB's appears to be related to their basicity with the more basic materials less thermally stable. As seen in Fig. 6, pKa's above 5 lead to materials which had TGA onsets below 150°C. Such a trend can be explained by the electrophilic nature of the onium cation which can undergo increasing degrees of nucleophilic attack as basicity(nucleophilicity) increases.

Scheme 7 shows a plausible scheme for such a nucleophilic attack. The cysteate PDB4 has a TGA onset of decomposition of 152°C and a pKa of 1.89; it was not listed in Fig. 5 because it contains both an aminosulfonate and a nucleophilic carboxylate moiety which may explain its lower degree of stability.
Fig. 5 Structure of anions of bis(tert-butylphenyl)iodonium salts studied as photodecomposable bases.

Table 3 Summary of screening tests for photodecomposable bases.

<table>
<thead>
<tr>
<th>anion</th>
<th>Solubility*</th>
<th>Thermal stability at least 150°C</th>
</tr>
</thead>
<tbody>
<tr>
<td>PDB1 (sweet PAG)</td>
<td>yes</td>
<td>Yes</td>
</tr>
<tr>
<td>PDB2</td>
<td>yes</td>
<td>Yes</td>
</tr>
<tr>
<td>PDB3</td>
<td>yes</td>
<td>No</td>
</tr>
<tr>
<td>PDB4</td>
<td>yes</td>
<td>Yes</td>
</tr>
<tr>
<td>PDB5</td>
<td>yes</td>
<td>Yes</td>
</tr>
<tr>
<td>PDB6</td>
<td>yes</td>
<td>Yes</td>
</tr>
<tr>
<td>PDB7</td>
<td>yes</td>
<td>Yes</td>
</tr>
<tr>
<td>PDB8</td>
<td>yes</td>
<td>No</td>
</tr>
<tr>
<td>PDB9</td>
<td>yes</td>
<td>No</td>
</tr>
<tr>
<td>PDB10</td>
<td>no</td>
<td>Yes</td>
</tr>
</tbody>
</table>

a) at least 0.2 wt % in 2-heptanone containing 18 wt % P(NB/MA/TBA/AA) polymer
Table 4 Lithographic properties of resists formulated with PDB's

<table>
<thead>
<tr>
<th>Base</th>
<th>$E_0$</th>
<th>Contrast</th>
<th>Dark Erosion</th>
<th>$E_{1.1}$ 0.16 Features (mJ/cm²)</th>
<th>$pK_a$*</th>
</tr>
</thead>
<tbody>
<tr>
<td>PDB6</td>
<td>3.8</td>
<td>4.7</td>
<td>0.791</td>
<td>11.25</td>
<td>3.2</td>
</tr>
<tr>
<td>PDB7</td>
<td>5.2</td>
<td>4.0</td>
<td>0.892</td>
<td>9.1</td>
<td>3.5</td>
</tr>
<tr>
<td>PDB1 (SWEET PAG)</td>
<td>9.4</td>
<td>4.5</td>
<td>0.917</td>
<td>22.7</td>
<td>1.7</td>
</tr>
<tr>
<td>PDB4</td>
<td>6.4</td>
<td>4.2</td>
<td>0.907</td>
<td>13.7</td>
<td>1.9</td>
</tr>
<tr>
<td>PDB5</td>
<td>4.4</td>
<td>3.4</td>
<td>0.800</td>
<td>10.95</td>
<td>1</td>
</tr>
<tr>
<td>TPI</td>
<td>8.8</td>
<td>5.6</td>
<td>0.938</td>
<td>18.6</td>
<td>4.8</td>
</tr>
</tbody>
</table>

$E_0$, $E_{1.1}$, and $pK_a$* are predicted using ACD/pKa DB software from Advanced Chemistry Development Inc.

![Graph showing TGA onset of decomposition versus the pKa of select photodecomposable bases](image)

Fig. 6 TGA onset of decomposition versus the pKa of select photodecomposable bases

Scheme 7

Table 4 summarizes the lithographic properties of the screened PDB derivatives and compares them to a standard formulation containing a photochemically inert amine, triphenylimidazole (TPI). Most of the formulations containing the PDB additives have a higher sensitivity than that of the TPI standard. This probably arises both because of their lower basicity and also due to photodecomposition of base in the exposed area. The only exception is for the cyclamate which shows a sensitivity slightly lower than that of the TPI formulation. The higher than expected sensitivity of the TPI formulation may perhaps be explained by reaction of TPI with MA or AA repeat units on the polymer resulting in a resist system which contains not just TPI but a complex mixture of products.

Unlike the photochemically inert ammonium carboxylates differences in sensitivity between...
different PDB's does not appear to simply reflect differences in thermal stability of the base additives. This is expected since sensitivity in this case is probably a complex function of differences of $pK_a$, thermal decomposition and photodecomposition tendencies among the different PDB's. The best imaging capability are seen for the cysteate and cyclamate formulations (PDB1 and PDB4 respectively). Fig. 7 shows a study of post-exposure delay latitude study[8] for a P(NB/MA/TBA/AA) resist containing PDB4 in which it can be seen that a good PEB latitude of up to two hours.

Finally, thermal stability of these PDB's can be improved by using a tris(tert-butylphenyl)sulfonium chromophore instead of bis(tert-butylphenyl)iodonium with the tris(tert-butylphenyl)sulfonium cation. For instance the onset of decomposition for tris(tert-butylphenyl)sulfonium cyclamate is $289^\circ$C compared to $204^\circ$C for the bis(tert-butylphenyl)iodonium analog(PDB1) (Fig. 8). Fig. 9 shows images obtained in a P(NB/MA/TBA/AA) resist formulated with this new sulfonium material.

4. Conclusion

Carboxylate ammonium salts can be used to replace amines as components in P(NB/MA/TBA/AA) 193 nm resist formulations. Of particular interest is trifluoroacetate which despite being a poor base is actually quite a good base quencher because it forms a volatile acid which is effectively removed during post-exposure bake. A number of different photodecomposable bases were also examined. It was found that thermal stability of bis(tert-butylphenyl)iodonium based PDB decreased as their basicity increased. The most useful materials in terms of solubility and imaging power were the cyclamate and the cysteate. This latter material was shown to confer a post-exposure bake latitude of at least 2 hours to a P(NB/MA/TBA/AA) resist formulation. Finally, the use of sulfonium instead of iodonium salts improved thermal stability of these materials while maintaining similar imaging capability. The use of the sulfonium chromophore allows the use of more basic materials. However, the inherent reactivity of the matrix with basic additives still may place a fundamental limit of what maximum benefit that can be confirmed by these.

Fig. 7 Post-exposure delay study of P(NB/MA/TBA/AA) resist containing bis(tert-butylphenyl)iodonium cysteate. The delay study was done in an controlled environment with 14 ppb ammonium as detailed in reference 8.
Fig. 8 Comparison of thermal stability of tris(tert-butyphenyl)sulfonium to bis(tert-butyphenyl)iodonium cyclamates

Fig. 9 Images obtained for P(NB/MA/TBA.AA) formulation containing tris(tert-butyphenyl)sulfonium cyclamate and tris(tert-butyphenyl)sulfonium nonaflate. The resolution dose was 28.9 mJ/cm²

References
2. a)F.M. Houlihan, J.M. Kometani, A.G. Timko, R.S. Timko, RA. Cirelli, E.


