

Simulation Based Formulation of a Non-Chemically Amplified Resist for 257 nm Laser Mask Fabrication

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ABSTRACT

The demand for smaller and more uniform features on photomasks has inspired consideration of a DUV (257 nm) resist process for optical pattern generation. Chemically amplified resists require storage and exposure in carbon filtered environments, and they require post-exposure bakes. Few mask facilities are set up to handle chemically amplified resists commonly used in deep UV wafer fabrication process. Hence, it is appropriate to explore the lithographic performance of non-chemically amplified resist materials for 257 nm laser photomask lithography.

Resist characterization and lithography simulation were used to formulate a 257 nm resist from DNQ/ novolak materials provided by a commercial resist supplier. Diazonaphthoquinone (DNQ)/ novolak resists have not been used for DUV Integrated Circuit (IC) applications mainly due to the low sensitivity and the strong absorbance of the DNQ photoactive compound (PAC) at 248 nm. However, a 2,1,4 DNQ based resist has been characterized that bleaches at 257 nm and inhibits novolak. The photoproduct of the 2,1,4 DNQ PAC is much more transparent at 257 nm than 248 nm. Novolak resin also has an absorbance minimum in the DUV at 257 nm that provides transparency similar to poly (hydroxystyrene).

Traditional photoresist formulation requires tedious, iterative, and expensive manufacturing trials. Resist characterization and lithography simulation can be used to relate lithographic performance (resolution, sidewall and process latitude) to resist formulation parameters (PAC concentration, developer concentration, etc.), thereby supporting the formulation optimization. An exposure system using a 257 nm frequency doubled argon laser system has been constructed to study the resist photokinetics. Dill exposure parameters (A, B and C) have been extracted for a 2,1,4 DNQ/ novolak based resist. Dissolution rate measurements have been made with a development rate monitor developed at the University of Texas at Austin. Simulation using the exposure and development rate models were used to determine the resist formulation that maximizes the sidewall angle and exposure latitude for isolated resist spaces. Preliminary experiments reveal that a DNQ/ novolak resist is capable of resolving 0.30 μm linewidths using a 257 nm optical pattern generator.

Keywords: DUV non-chemically amplified photoresist, lithography simulation and photomask

1. INTRODUCTION

Several diazonaphthoquinone (DNQ) photoactive compounds (PACs) and novolak resins were screened to determine the most inhibiting and bleaching PAC at 257 nm¹. A trifunctional 2,1,4 diazonaphthoquinone photoactive compound was found that has better bleaching characteristics than the 2,1,5 isomers at 257 nm. A novolak resin was selected that was the most strongly inhibited by this 2,1,4 PAC. The optical, exposure (Dill A, B and C) and dissolution properties were extracted as a function of PAC concentration for this specific PAC/ resin system. Dill's exposure parameters (A and B) quantify the PAC and resin absorbance and the level of bleaching of the PAC at 257 nm. The exposure rate constant (Dill C) of the resist formulation was approximately 3 times higher than that of 365 nm resists. A quantum efficiency analysis confirmed that the photospeed of the selected PAC is higher at 257 nm than 365 nm largely because of the higher molar absorptivity at the shorter wavelength.

The dissolution properties of resist formulations are dependent on the amount of formulated PAC (M), the fraction of PAC exposed (m) and developer concentration for a specific resin and PAC combination. Dissolution functions

($R(m)$) were measured for the 2,1,4 DNQ/ novolak system at multiple PAC to resin ratios and developer concentrations. These dissolution functions establish the contrast and the dissolution threshold for each resist formulation. This analysis allows expansion of the dissolution modeling to create an $R(m, M, OH)$ function, an experimental surface that describes the development properties throughout the formulation space.

Simulations were used to study the lithographic performance of the resist system throughout the formulation space based on sidewall angle, exposure latitude, and dose to size. The dissolution contrast (slope of $R(m)$) increases significantly with PAC concentration. The sidewall angle is strongly dependent on the latent image and dissolution contrast. The simulations revealed that the sidewall angle actually increases slightly with PAC concentration in this formulation range because the increase in bleaching and dissolution contrast overtakes the higher overall absorbance of the resist. Finally, simulations predicted a dose to size in the range of 70 mJ/cm^2 to 140 mJ/cm^2 over the selected PAC formulation space for $0.35 \text{ }\mu\text{m}$ isolated spaces in films developed with 0.23N TMAH developer concentration and processed without a post-exposure bake (PEB).

2. RESULTS AND DISCUSSION

2.1 Optical and exposure properties of 257 nm NCA photoresists

Finding the optimum DNQ/ novolak photoresist formulation for 257 nm lithography requires understanding and modeling the influence of PAC concentration on the optical, exposure and dissolution properties of the photoresist. A frequency doubled argon ion laser exposure system operating at 257 nm was used to expose resists for bleaching and dissolution experiments¹. The 2,1,4 DNQ chosen for these studies shows a significant level of bleaching at 257 nm (Figure 1).

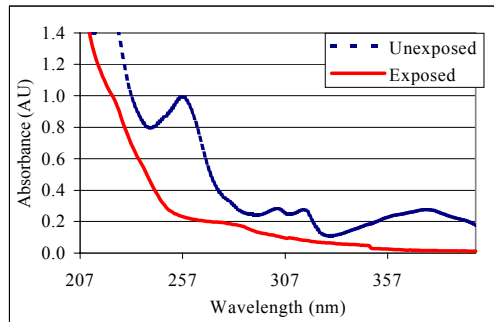


Figure 1: The 2,1,4 DNQ bleaches significantly at 257 nm

Dill's A and B values were calculated using the complex index of refraction from exposed and unexposed resist films². Index measurements were made with a J. A. Woollam ellipsometer (WVASE software version 2.92). The index of refraction was measured on resist films coated at approximately 440 nm and baked for 5 minutes at $90 \text{ }^\circ\text{C}$. The bleachable component (A) of the photoresist increased from $0.81 \text{ }\mu\text{m}^{-1}$ to $1.46 \text{ }\mu\text{m}^{-1}$ over a PAC concentration range from 0.10 molal to 0.20 molal (m) (Figure 2). However, the non-bleachable component of the resist (B) also increased from $1.55 \text{ }\mu\text{m}^{-1}$ to $1.95 \text{ }\mu\text{m}^{-1}$ over this PAC range. The non-bleachable absorption is due to novolak ($0.94 \text{ }\mu\text{m}^{-1}$) and PAC photoproduct (ranging from $0.61 \text{ }\mu\text{m}^{-1}$ to $0.82 \text{ }\mu\text{m}^{-1}$ over the 0.10 m to 0.20 m PAC range). The 2,1,4 DNQ PAC bleaches to approximately 60 % of its unexposed absorbance at 257 nm. This is far less than a typical 365 nm resist such as IP3600, but the final absorbance is only about 50 % more than typical non-bleachable chemically amplified resists at 257 nm.

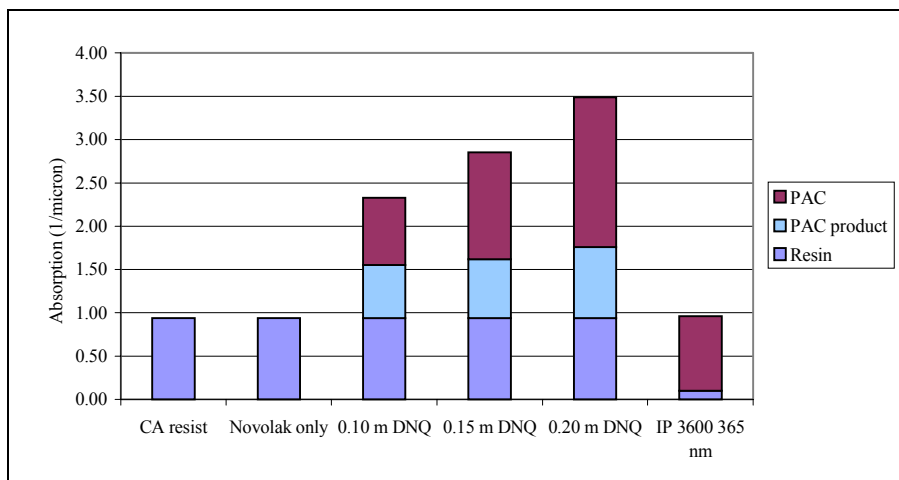


Figure 2: Absorbance of several 2,1,4 DNQ/ novolak resist formulations and a CA resist at 257 nm relative to an I-line resist

The kinetic rate constants (Dill C) of the 257 nm resists were calculated from transmittance measurements made on resist coated (440 nm) quartz substrates that were exposed to incremental doses. The transmittance was measured with a Hewlett Packard 8452A UV-VIS spectrophotometer. The bleaching curves (Figure 3) reveal that resist formulations based on the 2,1,4 PAC bleach with low exposure doses. The 0.10 m (9.9 wt % PAC), 0.15 m (14.2 wt % PAC) and 0.20 m (18.0 wt % PAC) 2,1,4 DNQ resists fully bleach with approximately 50 mJ/cm², 100 mJ/cm² and 150 mJ/cm² doses, respectively. These resist formulations contain less PAC than standard I-line resists (~18 wt %) to maximize transparency, while maintaining dissolution inhibition.

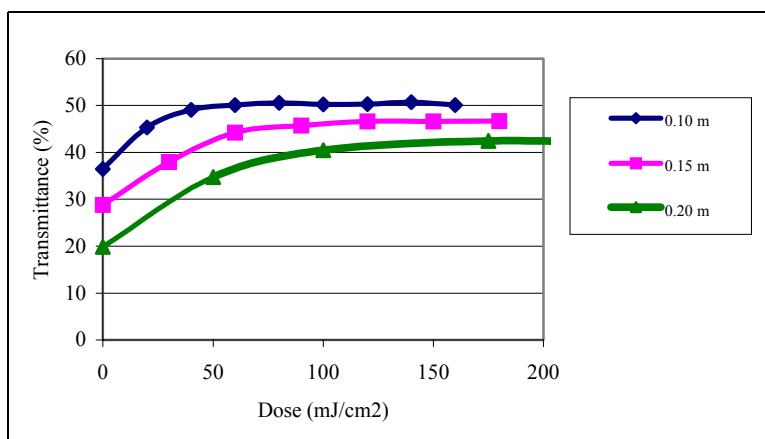


Figure 3: Bleaching curves for 2,1,4 DNQ resist formulations at 257 nm

The rate constant was calculated from the change in the real and complex index of refraction of the resist as a function of dose using PROABC³. The influence of the change in the real index of refraction on the C value is minimal since its change with exposure is small. The 0.15 m DNQ/ novolak resist real index of refraction only changes from 1.827 unexposed to 1.845 with full exposure. The experimental C values for the 0.10 m, 0.15 m and 0.20 m DNQ resists are 0.056 cm²/mJ, 0.042 cm²/mJ and 0.048 cm²/mJ, respectively (Table 1). The C value should be independent of PAC concentration assuming that the molar absorptivity and quantum efficiency are independent of PAC concentration. The range in C values is attributed to the precision of the measurements of the bleaching curve through dose.

Table 1: Index of refraction and Dill parameters for 2,1,4 DNQ based resists at 257 nm and 365 nm

PAC (m)	wavelength (nm)	n (unexp)	k (unexp)	n (exposed)	k (exposed)	Dill A (1/micron)	Dill B (1/micron)	Dill C (cm ² /mJ)
0.10	257	1.833	0.0477	1.8461	0.0317	0.78	1.55	0.056
0.15	257	1.831	0.0583	1.8535	0.0330	1.23	1.62	0.042
0.20	257	1.824	0.0713	1.8633	0.0360	1.73	1.76	0.048
AZ 1024	257	1.841	0.0629	1.8620	0.0356	1.33	1.74	0.042
novolak	257	1.850	0.0196				0.96	
0.10	365	1.681	0.0115	1.6850	0.0046	0.24	0.16	0.021
0.15	365	1.684	0.0163	1.6887	0.0052	0.38	0.18	
0.20	365	1.686	0.0208	1.6990	0.0055	0.53	0.19	
novolak	365	1.683	0.0012				0.04	

These resist formulations have much higher photospeed than 365 nm resists containing similar photoactive compounds. I-line resists typically have a C value between 0.01 - 0.02 cm²/mJ. The C value of the 0.1 m 2,1,4 DNQ/ novolak formulation was measured at 365 nm (Figure 4) and found to be 0.02 cm²/mJ, which is reasonable for a DNQ based resist exposed at 365 nm. Thus, the photospeed of the resist is 2.5 times faster at 257 nm than 365 nm.

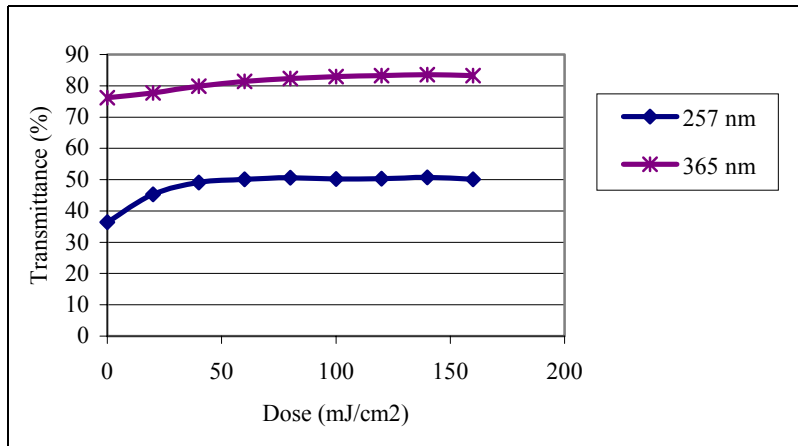


Figure 4: Bleaching curves for the 0.1 m DNQ resist at 257 nm and 365 nm

2.2 Quantum efficiency analysis

A quantum efficiency analysis as a function of PAC concentration explains this wavelength dependent photospeed. The quantum efficiency describes the probability of PAC conversion per absorbed photon (Equation 1).

$$\Phi = \frac{\text{molecules of PAC converted to photoproduct}}{\text{number of photons absorbed by the PAC}} \quad (1)$$

The quantum efficiency is linearly dependent on the kinetic rate constant C and inversely dependent on the molar absorptivity (base e) of the PAC. The quantum efficiency is described by the following measurable properties (Equation 2)⁴.

$$\phi_T(\lambda) = \frac{N_A hcC(\lambda)}{\lambda a_m(\lambda)} \quad (2)$$

where

C = kinetic rate constant (J/m²)
 ϕ_T = quantum efficiency
 a_m = molar absorptivity (m²/mole)

λ = wavelength of light (m)
 N_A = Avogadro's number (molecules/mole)
 h = Planck's constant (Js)
 c = speed of light (m/s)

The quantum efficiency as a function of PAC concentration was calculated from the experimental rate constant (Dill C) and solid phase molar absorptivity measurements at 257 nm and 365 nm. Molar absorptivity describes the probability that a PAC molecule will absorb light at the exposure wavelength. The molar absorptivity was calculated from the solid phase absorbance of unexposed resist films and the PAC concentration of the film. The solid phase absorbance was determined from the complex index of refraction that was measured at 257 nm and 365 nm. The solid phase PAC concentration was calculated from the formulated PAC concentration (9.9 wt% or 0.1 molal), the residual PGMEA weight percent (16 wt%) of a baked resist film at 90 °C^{5,6}, approximate density (1.1 g/ml) of the baked resist film⁴ and molecular weight of the PAC. The PAC molecule has 3 chromophores and the molar absorptivity was calculated on a per chromophore basis (Equation 3).

$$a_m = \frac{A}{[DNQ]} = \frac{A \times MW_{pac}}{w_{PAC} \rho n} \quad (3)$$

where

- a_m = molar absorptivity (m²/mole)
- MW_{pac} = molecular weight of PAC (g/mole)
- A = absorbance per meter from ellipsometry (1/m)
- ρ = density of baked photoresist film (1.1 g/ cm³)
- w_{PAC} = weight fraction of PAC
- $[DNQ]$ = concentration of chromophores (moles/ m³)
- n = number of chromophores per PAC molecule

The molar absorptivity of the 2,1,4 DNQ is 5500 m²/mole at 257 nm and 2010 m²/ mole at 365 nm. The 2.5 time increase in the molar absorptivity is responsible for the increase in photospeed at 257 nm compared to 365 nm. The absorbance for the PAC in acetonitrile supplements this result, since the unexposed PAC has an absorbance of 1.0 AU at 257 nm and 0.3 AU at 365 nm (Figure 1). The molar absorptivity in baked films is essentially independent of PAC concentration, which confirms Beer's law in this range of PAC concentrations.

The quantum efficiency of DNQ at 365 nm was calculated to be 0.34, which is very close to a referenced quantum efficiency of 0.37 (Table 2)⁴. This supports that the C value of 0.02 cm²/mJ for the 0.1 m DNQ resist appears valid at 365 nm. The quantum efficiency for these resists at 257 nm range from 0.37 to 0.47 (average of 0.42). The quantum efficiency of the 2,1,4 DNQ at 257 nm seems reasonable compared to 0.34 for the same PAC at 365 nm.

Table 2: Molar absorptivity and quantum efficiency calculations for DNQ at 257 nm and 365 nm

PAC (m)	Wavelength (nm)	Molar Absorptivity (m ² /mole)	Quantum Efficiency	Dill C (cm ² /mJ)
0.10	257	5500	0.47	0.056
0.15	257	5300	0.37	0.042
0.20	257	5550	0.40	0.048
0.10	365	2010	0.34	0.021

The exposure rate constant (C) is directly proportional to the molar absorptivity, quantum efficiency and wavelength (Equation 4). The C value increases significantly at 257 nm due to the 2.5 times increase in molar absorptivity. The average C value of the 2,1,4 DNQ resists was 0.048 cm²/mJ.

$$C(\lambda) = \frac{\phi_T a_m(\lambda) \lambda}{N_A h c} \quad (4)$$

2.3 Dissolution rate modeling

Dissolution functions (R(m)) were determined for 3 formulations of 2,1,4 DNQ/ novolak at two developer concentrations. These data describe the development properties of the resist materials through formulation space. The dissolution rate of the photoresists was measured using a development rate monitor (DRM) developed at the

University of Texas at Austin⁷. Dissolution rate measurements were made on 900 nm resist films coated on 4 in. diameter antireflective (AR3) photomask substrates. The resist films were post-application baked for 5 minutes at 90 °C (mask surface temperature) and post-exposure baked for 5 minutes at 110 °C (mask surface temperature) to minimize standing waves. R(m) functions have been generated for 3 resists, PAC concentrations of 0.10 m, 0.15 m and 0.20 m, at both 0.23N and 0.26N TMAH developer concentrations (TOK NMD-W) (Figures 5 – 10). The experimental dissolution rate data were fit with the "Notch" model used in the commercial lithography simulator PROLITH⁸. The contrast or slope of the dissolution function increased dramatically with PAC concentration and the location of the dissolution notch was shifted to a lower relative PAC concentration and higher dose to size as the developer concentration was reduced.

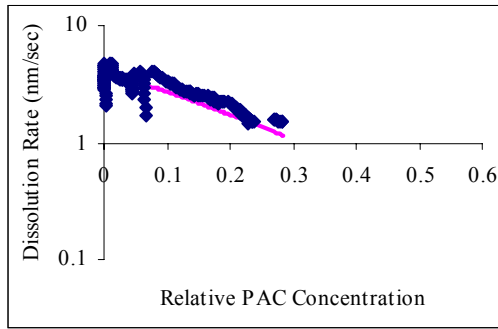


Figure 5: 0.10 m DNQ with 0.23N TMAH

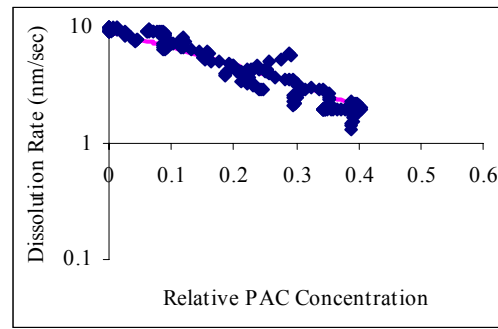


Figure 6: 0.10 m DNQ with 0.26N TMAH

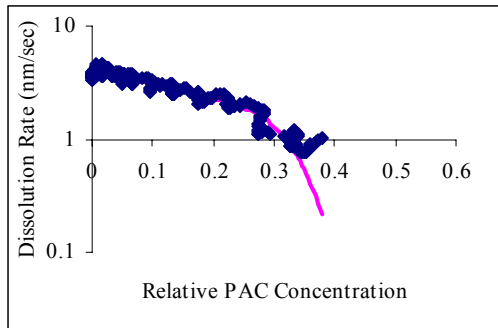


Figure 7: 0.15 m DNQ with 0.23N TMAH

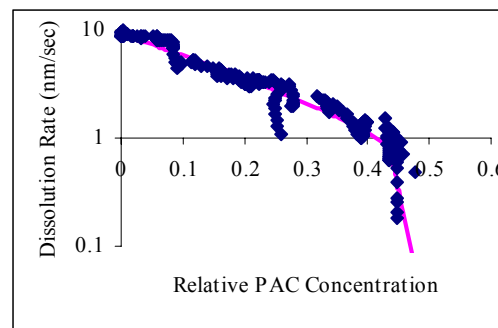


Figure 8: 0.15 m DNQ with 0.26N TMAH

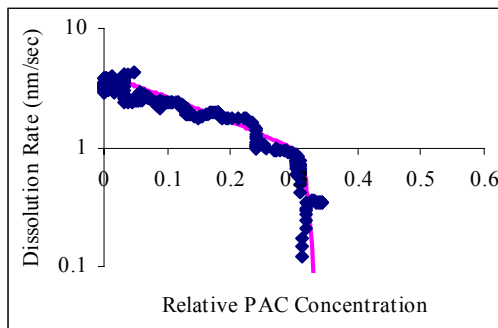


Figure 9: 0.20 m DNQ with 0.23N TMAH

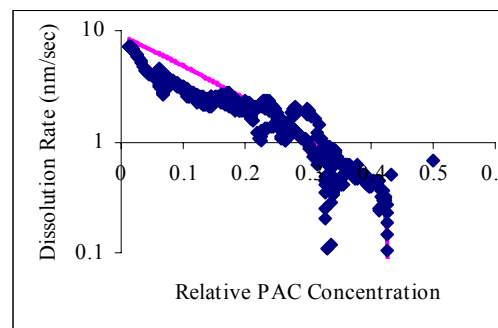


Figure 10: 0.20 m DNQ with 0.26N TMAH

This shift in the development notch moves the initiation of development to a PAC concentration closer to the location where the PAC gradient is optimized as a function of dose⁹. Slight oscillations in the R(m) curves indicate that standing waves still exist in the resist. The R(m) function was created from a "smoothed" PAC profile that included a diffusion length of 25 nm for the PEB.

The dissolution rate curves for the 2,1,4 DNQ/ novolak resist were plotted as a function of formulated PAC concentration, M , and exposed relative PAC concentration, m . These graphs reveal the non-linear resist development response to dose that link the extremes of fully exposed and unexposed resists that are normally represented by a Meyerhoffer plot (Figure 11 and 12). The dissolution rate of the fully exposed and unexposed resists must ultimately converge to the dissolution rate of the pure resin without PAC. More measurements are needed to fully converge the R_{max} and R_{min} Meyerhoffer trends at the limit of zero PAC concentration.

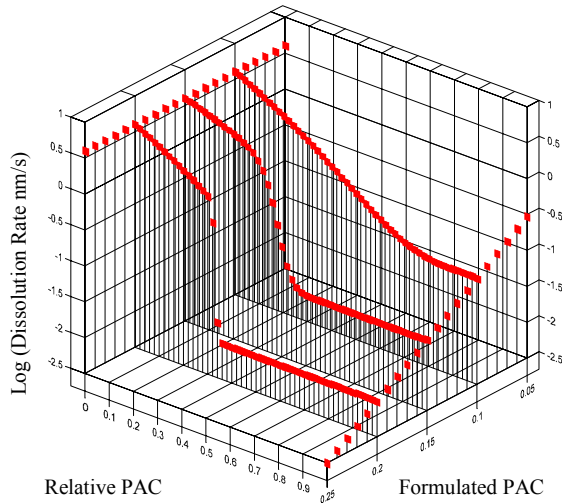


Figure 11: $R(m, M)$ developed with 0.23N TMAH

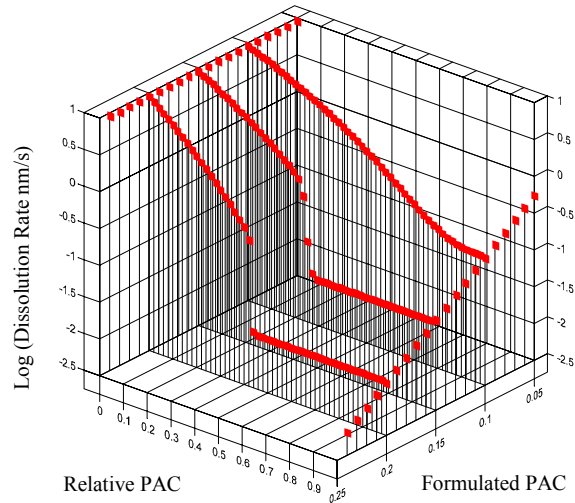


Figure 12: $R(m, M)$ developed with 0.26N TMAH

The $R(m, M)$ curves highlight the increase in contrast with increased formulated PAC concentration as well as the shift in the development notch that results from lower developer concentration. The overall dissolution rates of the formulations are shifted lower with lower developer concentration. These $R(m, M)$ curves for multiple developer concentration enable us to simulate lithographic performance through formulation space.

2.4 Simulation based formulation of a 257 nm NCA photoresist

The lithographic performance (CD sensitivity to dose and sidewall angle) was studied as a function of the image transfer function (PAC gradient) and dissolution response ($R(m, M, OH)$) in the resist. A performance tradeoff exists between the transparency and contrast of the resist as a function of PAC concentration. Addition of PAC makes the resist more opaque, which diminishes the image transfer and reduces the sidewall angle. However, the addition of PAC increases the contrast and the PAC gradient. PROLITH 6.1 has been used to model the sidewall angle and change in linewidth with dose over the formulated PAC concentration range for a 0.35 μm space developed in 0.23N TMAH developer (No PEB) (Figure 13). The dose to size for each formulation is labeled at the appropriate PAC concentration. The low PAC (0.1 molal) formulation has a low sidewall angle of 76.5° and has a high linewidth sensitivity to dose ($9 \text{ nm}/(\text{mJ}/\text{cm}^2)$) due to low contrast. The mid-PAC formula (0.15 molal) shows a much steeper sidewall angle (79.5°) and a major decrease in linewidth sensitivity to dose ($2 \text{ nm}/(\text{mJ}/\text{cm}^2)$). This resist formulation has a much higher contrast. A formulation with high PAC concentration (0.20 molal 18 wt% PAC) has a slightly lower sidewall angle (79°) and lower linewidth sensitivity to dose (below $2 \text{ nm}/(\text{mJ}/\text{cm}^2)$). This resist has about the same amount of PAC as a typical 365 nm resist and a dose to size of $140 \text{ mJ}/\text{cm}^2$. These simulations suggest that an optimal 2,1,4 DNQ/ novolak resist formulation exists that maximizes the sidewall angle and minimizes the CD sensitivity to dose.

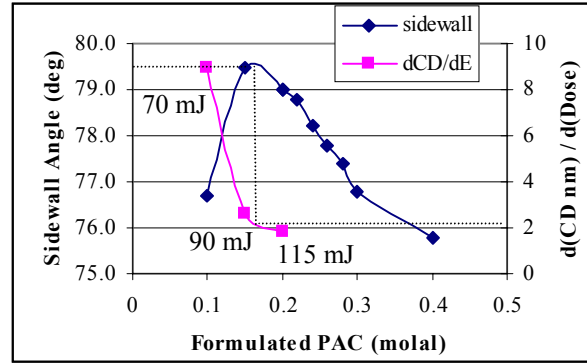


Figure 13: Simulation of the optimal 2,1,4 DNQ/ novolak resist formulation at 257 nm

Figure 13 show how contrast gained from the addition of PAC concentration to the formulation maximizes the sidewall angle at 79.5° and that the minimum in the CD sensitivity to dose occurs in the same formulation range for this set of materials. The gain in resist contrast with PAC concentration allows the final resist profile to match the iso-PAC concentration contour at the sidewall. Simulations that extrapolate the sidewall angle to a PAC loading above 0.2 molal were based on a Beer's law approximation for Dill's A and B values. The contrast (R(m)) is assumed not to change significantly with PAC concentrations higher than 0.20 molal. At PAC concentrations above 0.2 molal, it can be seen the sidewall angle decays quickly due to the increased absorbance (Figure 13).

Simulations of 0.35 μm resist profiles (isolated spaces) using aerial images from an experimental pattern generator supplied by ETEC Systems were run over the formulation space for 0.23N TMAH and 0.26N TMAH with and without a PEB (Table 4) including empirical surface inhibition models. Resists developed with the 0.23N TMAH required a longer development time (240 seconds) than the same resists developed in 0.26N TMAH (120 seconds). The post-exposure baked resists show less resist loss (< 1.5 nm) than the non post-exposure baked films (1 nm < r < 15 nm). The films that were not post-exposure baked show a slight decrease in dose due to the faster development rates. Underexposure leads to non-optimal image transfer (low PAC gradient at resist feature edge) and a process with a high CD sensitivity to dose and development time. The high contrast resist process containing PAC concentrations around 0.15 molal shows low CD sensitivity to dose and development time. The 0.2 molal and the 0.15 molal PAC resists that were not post-exposure baked gave the steepest sidewall angles (79°-80°). The CD sensitivity to dose was minimized in all cases with the resists containing the highest PAC concentration (0.20 molal). In summary, lithographic simulations through the process space indicate that DNQ resists with PAC concentrations between 0.10 – 0.20 molal have the highest sidewall and lowest CD sensitivity to dose for this set of resist materials.

Table 4: Simulated process latitudes for 0.35 μm spaces as a function of formulated PAC concentration, developer concentration and post-exposure bake conditions at 257 nm

Parameter	PEB						no PEB					
	0.23N TMAH			0.26N TMAH			0.23N TMAH			0.26N TMAH		
	0.1 m	0.15 m	0.2 m	0.1 m	0.15 m	0.2 m	0.1 m	0.15 m	0.2 m	0.1 m	0.15 m	0.2 m
develop time (s)	240	240	240	120	120	120	240	240	240	120	120	120
resist loss (nm)	1.5	0.5	0.1	1.4	0.4	0.1	4.7	1.3	0.4	15	3.3	1.1
dose (mJ/cm²)	87	104	138	74	100	137	67	89	116	54	80	99
sidewall (deg)	76.0	78.3	78.9	74.8	77.5	78.5	76.7	79.5	79.0	77.2	79.2	78.4
dCD/dE	5.2	4.4	1.8	5.8	3.0	2.7	9.0	2.6	1.8	10.4	4.5	2.9

Process latitude simulations were run for the 0.1 m, 0.15 m and 0.20 m DNQ/ novolak resists developed at 0.23N (No PEB). They reveal that the resist with the highest PAC concentration (0.20 m) has the largest exposure latitude

(Figures 14-16). The highlighted images show the isolated resist features that have sidewall angles above 75° and linewidth within 10% of 350 nm. The 0.20 m DNQ resist has such high contrast that the linewidth is relatively insensitive to development time. The 0.1 m DNQ resist has a strong dependence of linewidth on development time due to the low contrast. The linewidth of the 0.15 m DNQ resist shows low CD sensitivity to dose, but it still does not have as much exposure latitude as the 0.20 m resist.

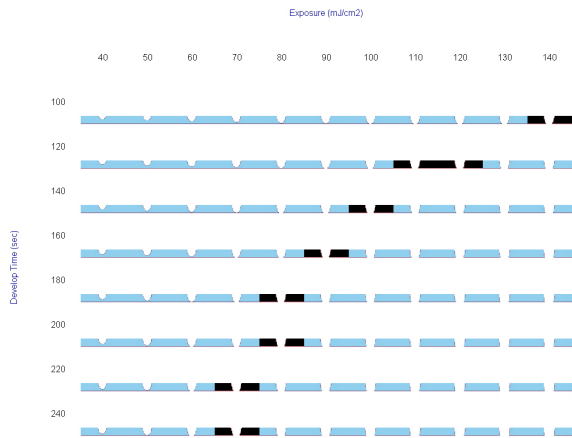


Figure 14: 0.1 m DNQ/ in 0.23 N TMAH

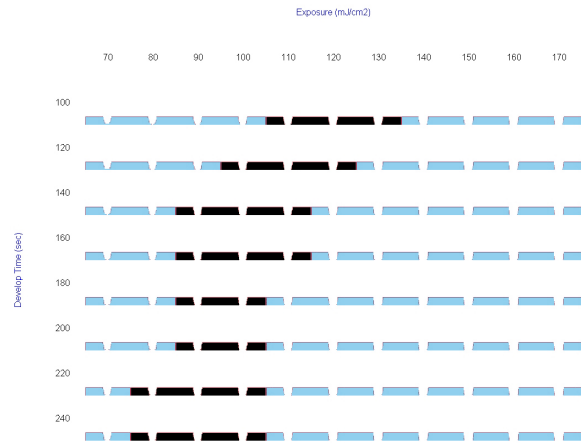


Figure 15: 0.15 m DNQ in 0.23 N TMAH

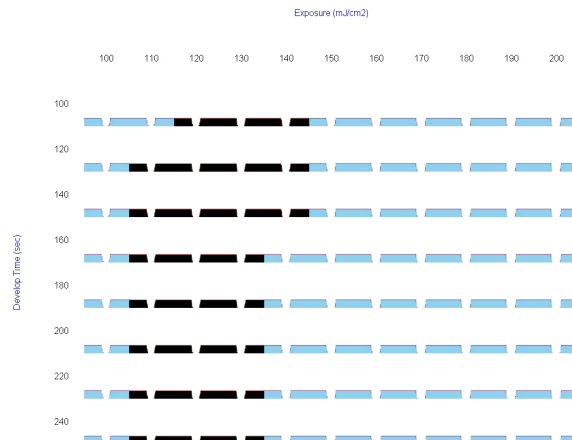


Figure 16: 0.20 m DNQ in 0.23 N TMAH

Experiments including exposure of these resist formulations with a 257 nm laser pattern generator have just begun. The 0.1 m 2,1,4 DNQ resist (440 nm) was post-application baked at 90°C for 420 seconds, post-exposure baked at 110°C and developed for 300 seconds with 0.26N TMAH developer (NMD-W). Scanning electron micrographs of $0.3\ \mu\text{m}$ and $0.5\ \mu\text{m}$ line/ space pairs printed in the resist are provided as Figures 17 and 18. These are very encouraging results, since DNQ/ novolak at 248 nm show very poor sidewall angles. The profiles appear to have a small foot at the bottom of the spaces. This could be attributed to the low exposure dose node of the standing waves close to the substrate, incomplete development, and/ or incomplete exposure due to the attenuation of light in an absorbing resist. Experiments are in progress to gather focus-exposure latitude, linearity and sidewall angle data for the 0.1 m and 0.15 m resist formulations.

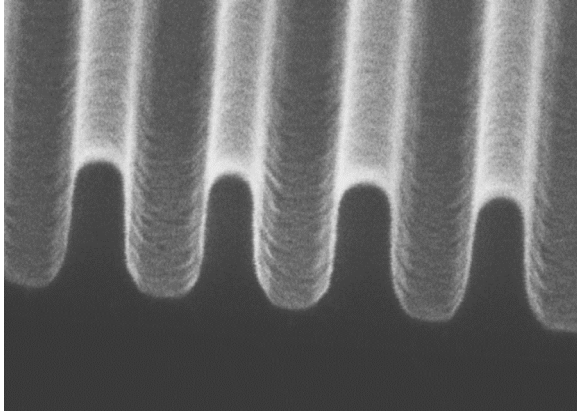


Figure 17: 0.3 μm l/s pairs for 0.1 m DNQ/ novolak

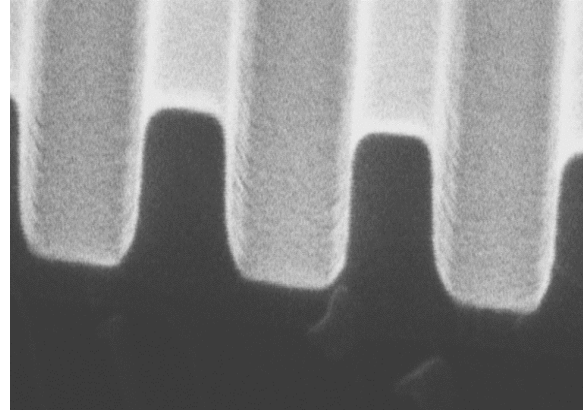


Figure 18: 0.5 μm l/s pairs for 0.1 m DNQ/ novolak

3. CONCLUSIONS

Lithographic performance including sidewall angle, exposure latitude and development latitude was simulated as a function of PAC concentration. These simulations reveal that an optimum resist formulation exists that maximizes the sidewall angle and minimizes the CD sensitivity to dose as a function of PAC concentration. Experiments are now underway to fully tune simulated image profiles, dose and development responses. Preliminary experiments have demonstrated the ability to image 0.3 μm line/space pairs at 257 nm in a DNQ/ novolak resist. These results demonstrate the ability to efficiently screen resist materials and optimize process variables using simulation.

4. ACKNOWLEDGEMENTS

This work was supported by ETEC Systems, Inc. and a SRC/ Texas Instruments Fellowship to B. M. Rathsack. The authors thank Peter Tatersall, Chris McAdams, Shipley Company (Cheng Bai Xu) and Clariant (Ralph Dammel) for providing resin and PAC samples. We thank FINLE Technologies (Chris Mack and Mark Maslow) for modeling discussions and the gift of PROLITH/2; and Ron Synowicki at J. A. Woollam for help in modeling the index of refraction of our photomask substrates

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