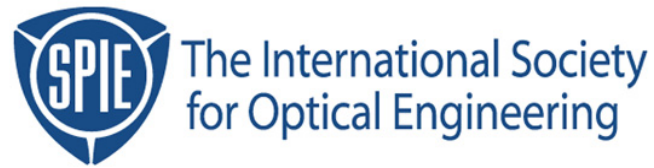


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Diffusivity Measurements in Polymers II: Residual Casting Solvent Measurement by Liquid Scintillation Counting

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ABSTRACT

Simulation of the microlithographic process plays an important role for manufacturing integrated circuitry. Unfortunately, most simulations still lack fundamental relationships that link the resist chemistry and the final resist image. This study is directed towards generating the data necessary to quantify the effects of residual casting solvent on the resist image. The amount of casting solvent was measured directly by using liquid scintillation counting to determine the mass of residual solvent as a function of the post apply bake temperature and time for several casting solvents. These measurements have been carried out on four identical diazonaphthoquinone-novolac resist formulations cast with different radio-labeled casting solvents (ethyl cellosolve acetate, PGMEA, diglyme, and ethyl lactate). From this data we have estimated the diffusion coefficients for the solvents and the dependence of these coefficients on temperature. These data are then convolved with dissolution parameters and Dill parameters to isolate and establish the relationship between these parameters and the post apply bake process that controls the amount of residual casting solvent.

Keywords: Residual Casting Solvent, Dissolution Kinetics, Dill Parameters, Post Apply Bake, Scintillation Counting

1. INTRODUCTION

Microlithography involves many interdependent processing steps. Due to the complexities of these steps and their interdependency, simulation has become an increasingly important part of process optimization. The ultimate goal is to have simulations that can predict the resist image for a wide range of process conditions and formulations without having to perform parameter extraction experiments for each processing condition and formulation. Several simulator tools^{1,2} can, with varying degrees of ease and sophistication, accurately model the effect of changes in the illumination systems, focus, lens characteristics, etc.. The influence of these variables on the free space image are accurately described by calculations based on fundamental physical relationships. With this free space image information and certain photochemistry principles, the latent image in the resist can also be very accurately modeled.

Unfortunately, current simulations tools do not include all of the fundamental relationships between the dissolution rate and process or formulations variables. Several studies have looked at the influence of polymer^{3,4}, photosensitive compound⁵, casting solvent^{6,8}, and developer solution⁹ on rate. One particular relationship that needs further investigation is the relationship between residual casting solvent and the resist image. The dissolution rate of resist films has been shown to steeply depend on both the amount of residual casting solvent and on the type of casting solvent^{6,8}. The exposure parameters are also affected by the pre-exposure bake conditions¹⁰, which serves to drive off residual casting solvent. These relationships between casting solvent and resist image, however, have not been quantitatively related in a way that allows their incorporation into a simulation model. This study quantifies the residual casting solvent content as a function of post apply bake temperature and time. These data, the dissolution rate, and the optical properties are then related to the post apply bake process.

The amount of residual casting solvent (RCS) in resist films is measured as a function of post apply bake (PAB) by scintillation counting. This method provides a more direct means of determining RCS content in resist films than the methods of FTIR spectroscopy and gravimetric analysis that are used by Beauchemin *et al.*⁶ and Ouano *et al.*⁸. These methods require either external standards for the FTIR analysis or assumptions that the weight loss is exclusively from the solvent loss. Utilizing scintillation counting eliminates the need for any external standard or assumptions, provides a more direct method for determining RCS content, and allows measurement of the solvent mass with extremely high sensitivity and precision.

These measurements have been carried out on four identical diazonaphthoquinone-novolac resist formulations with different radio-labeled casting solvents. Knowledge of the RCS content as a function of PAB information allows calculation of the diffusion coefficients of the solvents and the dependence of these coefficients on temperature. These data have been then convolved with dissolution parameters and Dill parameters for one resist to isolate and establish a quantitative relationship between these parameters and the soft bake process that controls the amount of residual casting solvent.

2. EXPERIMENTAL

2.1 Resist Formulations

Four positive tone novolac-diazonaphthoquinone resist solutions were made using novolac resin ($M_w=9370$, $P_d=4.880$) from Schenectady International, diazonaphthoquinone (DNQ) from IBM Corp., and ^{14}C -radio-labeled solvents. The four resist solutions were identically formulated; except that each had a different casting solvent: cellosolve acetate, diglyme, PGMEA, or ethyl lactate. The DNQ loading for each resist was 12 wt. % relative to the solids, and the solvent content for each resist solution was 77 wt. %. The resist solutions were pre-filtered by pressure filtering through Whatman Puradisc 25 TF 1.0 μm filters and then through MSI Cameo 50F 0.22 μm filters.

Cellosolve acetate, diglyme, PGMEA, and ethyl lactate were radio-labeled with ^{14}C (Figure 1) using the synthesis routes from Hinsberg *et al.*¹¹ and Gardiner *et al.*¹². The radioactivity of each solvent and resist film was measured using a Beckman 1801 liquid scintillation counter (LSC) and Fisher ScintiVerse II scintillation cocktail. The liquid scintillation counter was calibrated using the "H-Number" technique developed by D. Horrocks¹³. This calibration corrects for the counting variation from quenching and allows direct comparison between samples containing materials with dissimilar quenching effects. Quenching is extremely important for this study because these four casting solvents have different quenching effects. The specific activity ($\mu\text{Ci}/\text{gram}$) of each solvent was measured prior to mixing the solids and filtering the solution.

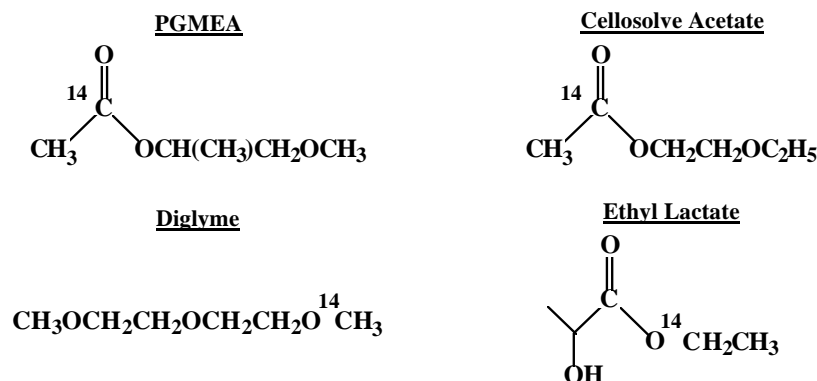


Figure 1: Radio-labeled casting solvents.

2.2 Resist Film Casting and Collection

The resist solutions were spin coated on 4 in. silicon wafers using Headway Research Inc. Model PM101DT spin coater. The spinning time was held constant, but the spinning speed was adjusted for each baking temperature and casting solvent to produce film thicknesses close to 1 μ m. Six different baking times were used at each PAB temperature of 70°C, 90°C, and 110°C. Since the PAC is thermally labile and the amount of RCS has been shown to depend albeit weakly on PAC concentration^{8,14}, the maximum baking time was decreased with increasing temperature to prevent more than 4 wt. % PAC decomposition.

The thickness of each film was measured using an SC Technology multi-wavelength reflection interferometer. In order to determine the Cauchy coefficients for each formulation, four non-radio-labeled resist solutions were identically formulated and filtered. These non-labeled resist formulations were each processed similar to the radio-labeled formulations and sent to J.A. Woollam Co. Inc. for measurement of the Cauchy coefficients.

The PAB was performed on a Thermolyne HP1151B hot plate that is controlled by a proportional - integral - differential controller and fitted with a vacuum chuck. Exactly 2 minutes after finishing the PAB, the film was dissolved in a collection dish by 5 mL of non-radio-labeled casting solvent. The dish was then washed with 14 mL of Fisher ScintiVerse II scintillation cocktail. These solutions were placed in a scintillation vial for counting. During this 2 minute period, the films were cooled for 15 seconds, the thickness was measured, and the coated wafer was then weighed on a Denver Instruments A-200DS balance.

2.3 Dill Parameter Measurement

The apparatus used for the Dill parameter measurements is described by Henderson *et al.*¹⁵. The index of refraction at 365 nm was measured for the non-bleached and fully bleached films that were baked for 90 seconds at 70°C, 80°C, 90°C, 100°C, and 110°C, using a 2010 Metricon prism coupler instrument. The Metricon instrument utilized an Oriel 500W Hg-Xe (Model: 66032) exposure source whose radiation passed through an 365 Hg line interference filter. Films were cast again with this resist and baked for 90 seconds at 70°C, 80°C, and 90°C. These films were used to establish Dill's A, B, and C values. These data were analyzed using FINLE Technology's ProABC v 1.0 software. From the analysis, the A, B, and C parameters were determined as a function of bake temperature.

2.4 Dissolution Rate Measurement

The dissolution rate as a function of exposure dose, R(E), was measured. The resist films were cast and baked at the three bake temperatures for 90 seconds. These films were then exposed with different doses at 365 nm using an Oriel 500W Hg-Xe (model: 8089) exposure lamp system. The radiation passed through an 365 Hg line interference filter. After exposure, the wafer is placed in a Perkin-Elmer DRM and puddle developed. The developer was a solution of 70% by volume Hoechst Celanese AZ 300 MIF developer and 30% distilled water. The dissolution rate was measured for each bake condition and dose, which gave the full range of DNQ concentrations. The data was analyzed using FINLE Technology's ProDRM v1.0a software. The R_{max} and R_{min} values were measured independently. The R_{max} value was determined from a completely exposed film that was puddle developed in the SC Technology interferometer. The R_{min} value was determined by subjecting an un-exposed film to developer for 45 minutes. The thickness was measured before and after development to establish the R_{min} rate.

3. RESULTS AND DISCUSSION

3.1 Residual Casting Solvent Results

The four resists were spin coated at different spinning speeds to maintain film thicknesses close to 1 μ m. Ideally, the films should be very close to the same thickness to allow direct comparison between the diffusion coefficients of the different solvents. By keeping the film thicknesses close, the transport from the film during baking can be compared without concern for a dependence on thickness, that can occur with non-Fickian transport. The viscosities of the solutions covered a wide range; therefore, the spinning speeds were varied. The spinning speeds and average baked film thickness are in Table 1.

Table 1. Spin speeds and resulting thicknesses for different solvents and PAB temperatures for a 90 second bake.

PAB Temperature	Diglyme	PGMEA	Cellosolve Acetate	Ethyl Lactate
70°C	2500 RPM 1.01 μm	4000 RPM 1.16 μm	2700 RPM 1.09 μm	4000 RPM 1.15 μm
90°C	2350 RPM 1.01 μm	3600 RPM 1.18 μm	2450 RPM 1.12 μm	3550 RPM 1.22 μm
110°C	2250 RPM 1.00 μm	3470 RPM 1.15 μm	2320 RPM 1.14 μm	3300 RPM 1.22 μm

The wafer weight was measured before and after coating. The RCS mass was determined by the LSC and the specific activities of the solvents. The resists' RCS weight percents were calculated using the total film weight and the RCS mass as follows:

$$\text{RCS wt. \%} = \frac{\left(\text{DPM}_{\text{Film}} \right) / \left(\text{Specific Activity}_{\text{solvent}} \right)}{\text{mass}_{\text{coated wafer}} - \text{mass}_{\text{un - coated wafer}}} \times 100 \left[= \right] \frac{(\mu\text{Ci}) / (\mu\text{Ci} / \text{grams})}{\text{grams}} \quad \text{Eqn.(1)}$$

The Denver Instruments A-200DS balance is capable of measuring mass to ± 10 mg; however, wafer measurements were reproducible to only ± 0.2 mg. Film weights varied from 9.5-11 mg for the different resists. The DPM measurements for the resist films had 2σ counting errors values ranging from ± 0.3 -2.0%. The error in the solvent specific activity comes from errors in solvent mass measurement and counting. These errors were less than $\pm 0.1\%$ for both the mass error and 2σ counting errors.

The weight percent of RCS in the films was measured for each of the four resists as a function of PAB (Fig #2-5). The resist solutions made with diglyme and cellosolve acetate cast thinner films and have relatively low RCS content; whereas PGMEA casts thicker films that contain higher amounts of RCS. The resist formulated from ethyl lactate deviates from the expected trend that thicker films retain more casting solvent. Specifically, the ethyl lactate formulation casts the thickest films, but it contains the least amount of solvent. There are at least two plausible explanations for this observation: (1) physical properties of ethyl lactate are significantly different from the other solvents (2) the radio-label on ethyl lactate was lost during film processing. The first explanation derives from the analysis of spin coating by Bornside *et al.*¹⁶. Bornside shows that there is a complex relationship between the physical properties of the solution and the dynamics of spin casting, which can cause the films to lose solvent and still cast thick films.

It is also possible that the radio-labeling could cause this effect. Despite the fact that radio-labeling can give direct, high precision measurements for diffusion studies, one of its draw backs is that the molecule being studied must retain the radio-label during the experiment. If the molecule undergoes some reaction that involves the radio-labeled site, then it is possible to acquire misleading results. The ethyl lactate is labeled on the ethyl site. It has been well documented that ethyl lactate can undergo a self condensation (dimerization) reaction which produces radio-active ethanol. If this reaction occurs, the label would be lost from the lactate molecule. Both hypothesis are currently under investigation.

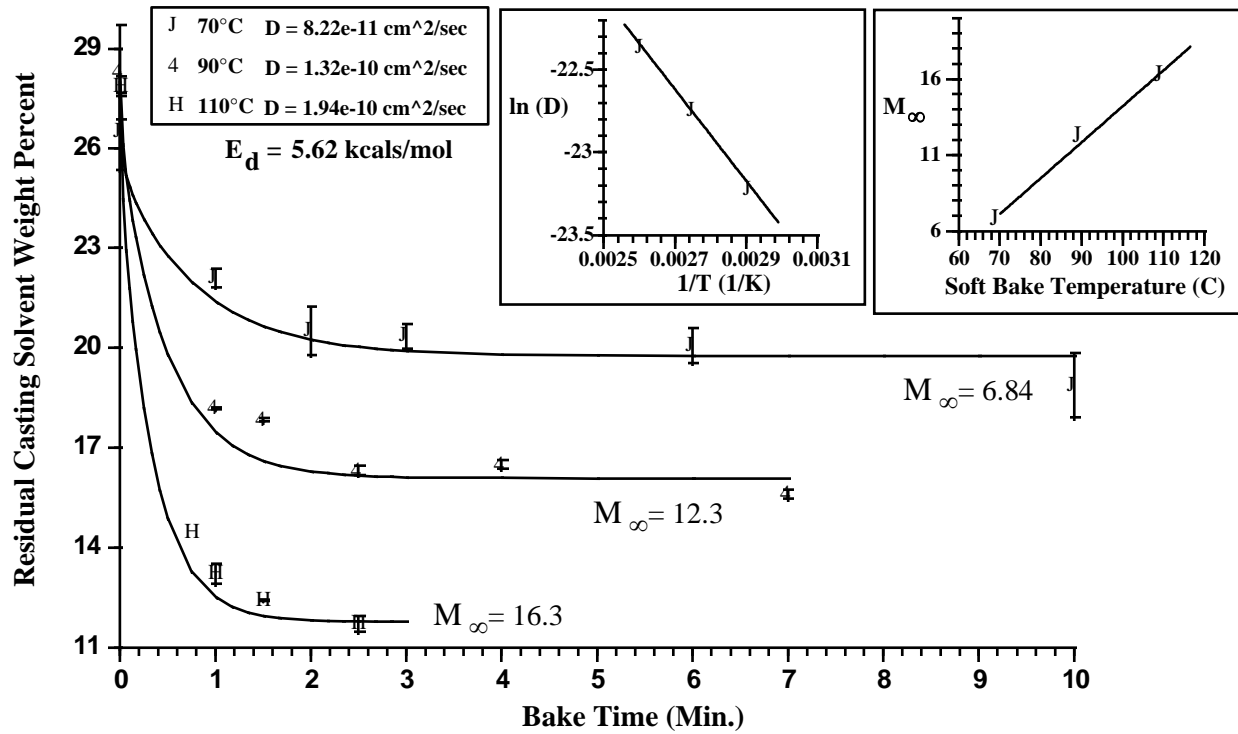


Figure 2: Weight percent of PGMEA in positive resist films for different PAB conditions.

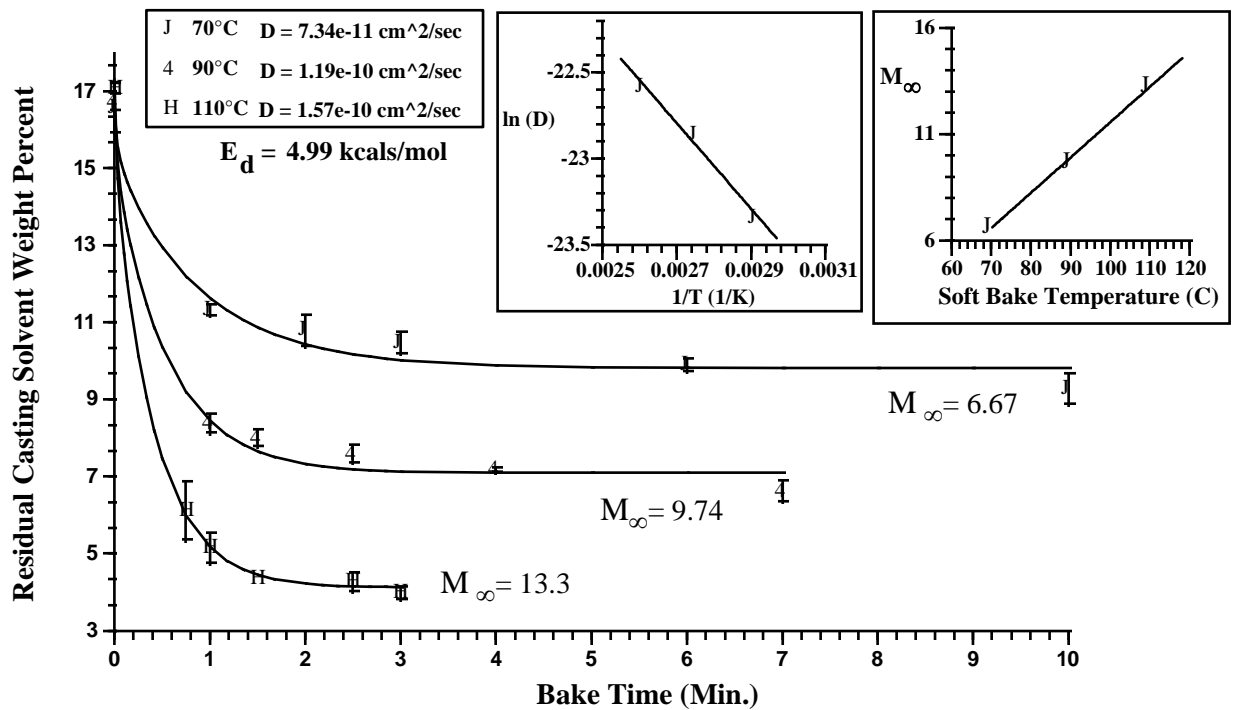


Figure 3: Weight percent of diglyme in positive resist films for different PAB conditions.

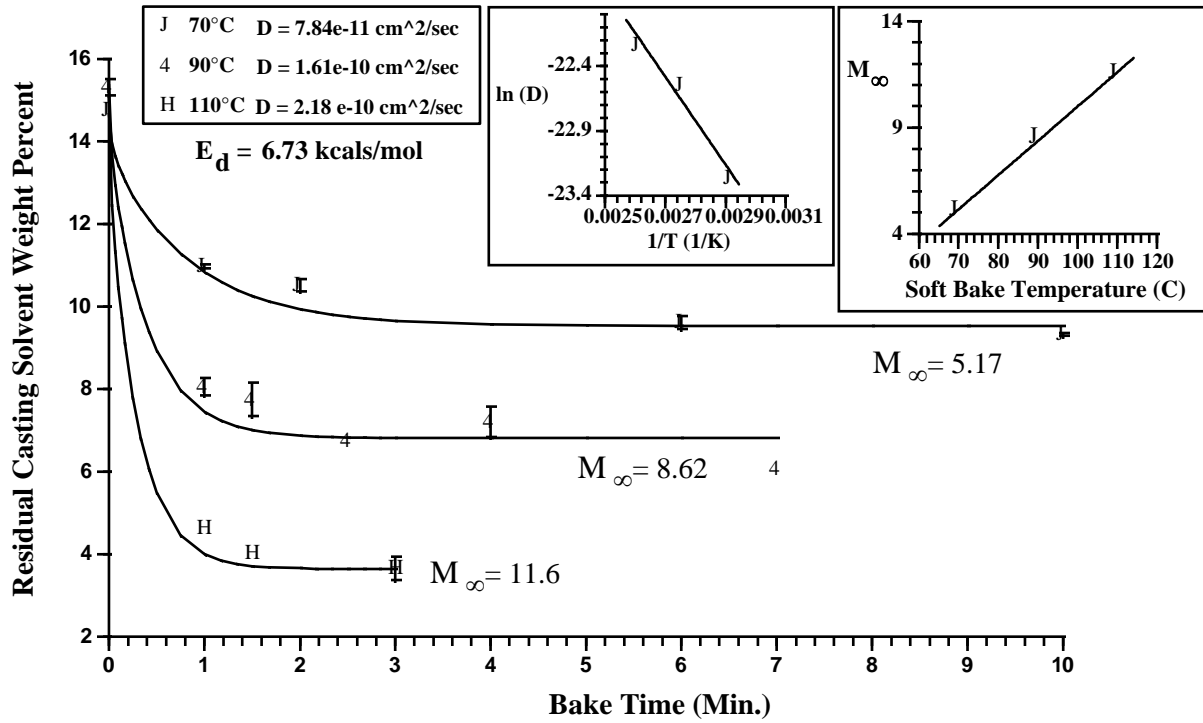


Figure 4: Weight percent of ethyl lactate in positive resist films for different PAB conditions.

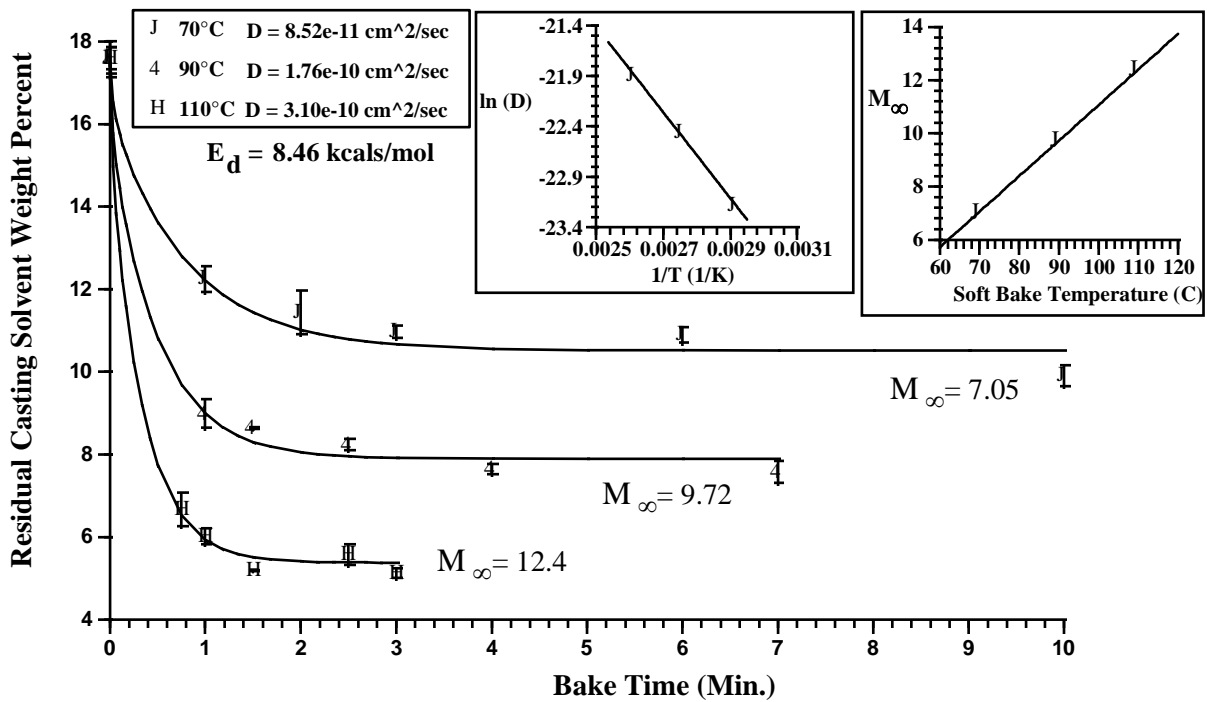


Figure 5: Weight percent of cellosolve acetate in positive resist films for different PAB conditions.

The mass loss data (Figures 2-5) can not be fit by the simple constant diffusion coefficient model (eqn 2) for inert penetrants in thin films,

$$\frac{M_t}{M_\infty} = 1 - \frac{8}{\pi^2} \sum_{n=0}^{\infty} \frac{1}{(2n+1)^2} \exp\left[-\frac{(2n+1)^2 \pi^2 D t}{4l^2}\right] \quad \text{Eqn.(2)}$$

where M_t is the desorbed from the film at time t ; M_∞ is the amount of solvent desorbed from the film at infinite time; D is the constant diffusion coefficient; and l = film thickness. Clearly, the transport is more complex than the constant diffusion coefficient model for which this expression was derived. There appear to be two different transport processes involved in this system, one of which is quite fast and one of which is relatively slow on the semiconductor processing time scale. This is not a new observation. Ouano *et al.*⁸ report a fast and slow regime for chlorobenzene in poly(methyl methacrylate); and Dill and co-workers¹⁴ used mass spectroscopy to demonstrate what they describe as “bound” and “unbound” states for solvent in resist films. They concluded from their studies that the solvent in the “bound” state is not in equilibrium with that in the “unbound” state. Therefore, that solvent which is in the bound state does not depopulate to the unbound state as the unbound solvent is remove. Mack *et al.*¹⁷. postulated a very strong concentration dependence of the diffusion coefficient of casting solvent in resist films.

This equation fits the experimental mass loss data fairly well only if M_∞ is set not to the initial solvent mass but to the measured amount of solvent desorbed from the film after long times relative to the semiconductor process cycle. In theory, all of solvent in the film should escape at infinite bake time since the resist is losing solvent to an infinite volume. The diffusion coefficient calculated from this equation follow an Arrhenius relationship for which an activation energy for diffusion can be determined. In addition to predicting intermediate temperature diffusion constants, the value of M_∞ must be determined for intermediate temperatures. Figures 2-5 show that M_∞ is linear with respect to temperature. Using these two relationships, the casting solvent can be predicted for intermediate PAB conditions. Clearly, a more sophisticated analysis is demanded to accurately model the amount of solvent remaining in the films after long times at elevated temperatures. We are continuing our work toward a more complete description of this process. However, from an engineering perspective, it is valuable to have the ability to accurately calculate the solvent concentration over different conditions that would be typically used by the semiconductor industry. These data and our "modified" diffusion coefficients allow that calculation. These fits are valid only over the time frame of the measurements from which they are derived, but these times are long relative to processing time.

3.2 Optical Parameter Results

The index of refraction of the PGMEA formulation was measured at 365nm for three different baking temperatures for both un-bleached and completely bleached samples (Table 2). These data are required to properly model the transmission vs. dose curves from which the Dill parameters are calculated. The Dill parameters for the three bake temperatures are in the Table 3.

Table 2. Refractive indices for the PGMEA resist as a function of PAB temperature for a 90 second bake.

PAB Temperature	Un-Bleached index of refraction at 365nm	Bleached index of refraction at 365nm
70°C	1.676	1.668
80°C	1.677	1.673
90°C	1.682	1.676
100°C	1.687	1.680
110°C	1.689	1.684

Table 3. Measured Dill parameters for the PGMEA resist as a function of PAB temperature for a 90 second bake.

Temperature (90 seconds)	A	B	C
70°C	0.3221	0.0657	0.0065
80°C	0.3283	0.0435	0.0065
90°C	0.3455	0.0518	0.0064

As reported by Henderson *et al.*¹⁰, the A parameter increases as bake temperature increases. The B value is unaffected over the temperature range studied; and the C value is constant. The C value is related to the quantum efficiency of the photoactive compound (PAC); therefore, the PAB and solvent content should not effect the C value. The B value is the exposure-independent parameter related to the absorption of the resin and the exposure by-product compounds, which should not be greatly influenced by solvent concentration. The A value is the exposure-dependent absorption parameter. This parameter is affected by the PAB in two ways. For high temperature, decomposition of the DNQ results in a decrease the A parameter¹⁸. For low temperature, solvent evaporation does not affect absorption by the DNQ, but the density of the film increases. This densification causes the A parameter to increase because of its dependence on the absorption per micron of the film.

3.3 Dissolution Rate Results

R_{min} and R_{max} measurements (Figure 6) show that both R_{min} and R_{max} decrease with increasing baking temperature. This trend has been seen in studies of solvent effects on dissolution rate. An increase in the solvent concentration generates a larger free volume as the solvent interferes with the polymer-polymer packing. An increase in free volume enables faster diffusion of developer solution into the resist and thereby presumably generates a higher dissolution rate.

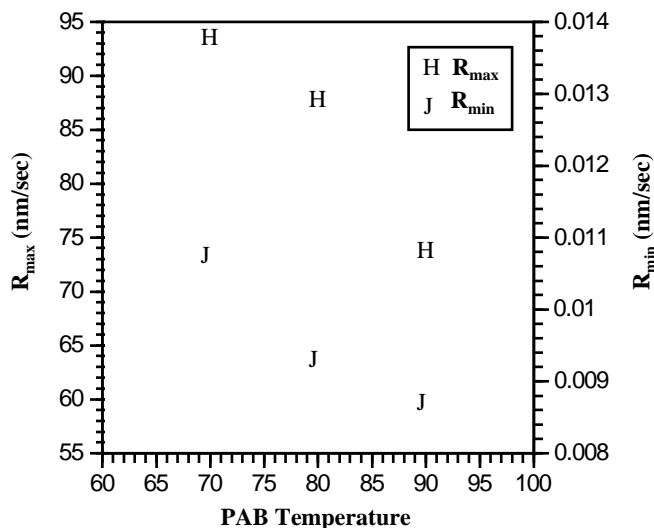
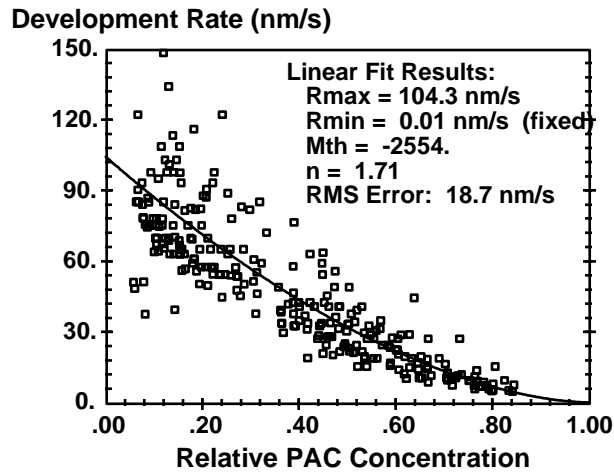


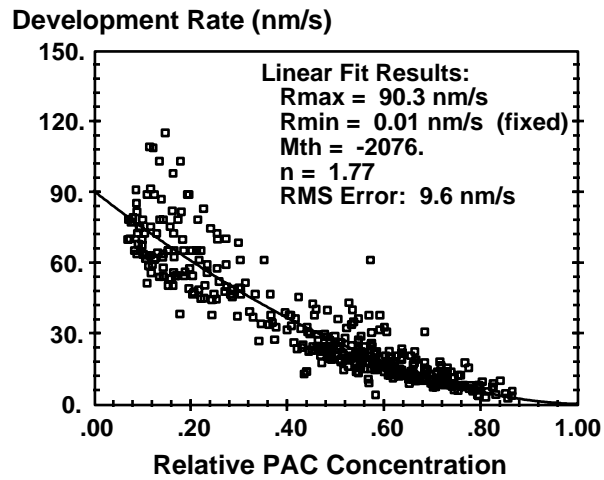
Figure 6: R_{min} and R_{max} .

The dissolution rate as a function of dose, $R(E)$, was measured at the three bake temperatures (Figures 7a-c) and the data was fit with the original Mack Model¹⁹,

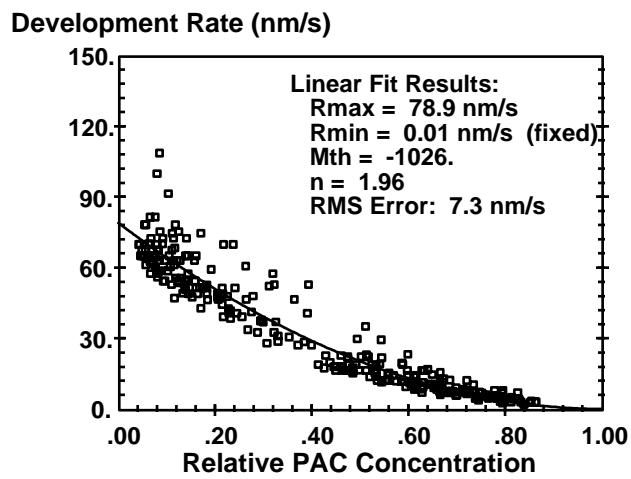
$$R = R_{max} \frac{(a+1)(1-m)^n}{a+(1-m)^n} + R_{min} \quad a = \frac{(n+1)}{(n+1)} (1-m_{TH})^n \quad \text{Eqn.(3)}$$



(a)



(b)



(c)

Figure 5: ProDRM analysis of the dissolution rate data of PGMEA solvent-based resist for a 90 second PAB with (a) 70°C, (b) 80°C, and (b) 90°C temperatures.

The n value can be thought of as a selectivity of the developer towards the exposed resist. The m_{TH} value is related to the position of the inflection point in the development rate vs. PAC concentration curve. Figure 7 shows that increasing of PAB temperature increases the n value and therefore the selectivity. The m_{TH} value decreases with increasing PAB temperature. Dill *et al.*⁵ baked identical resist at 70°C and 100°C PAB in a convection oven for 1 hour; and he witnessed R_{max} and n decreased, m_{TH} was unaffected, and R_{min} increased with PAB temperature. This effect of PAB on the n , R_{min} , and m_{TH} values for Dill are different from our results.

This difference in result can be explained by the relative influence of both film drying and PAC decomposition. Both n and m_{TH} values are independent of the developer concentration; however, they are dependent on the initial amount of PAC in the resist. The PAB serves to dry the resist film by driving the casting solvent out of the film and to anneal the film. Unfortunately, baking the film at elevated temperatures can cause the PAC to decompose. At our baking temperature range and time, the difference in the amount of PAC decomposition between PAB conditions should be much smaller than 1 wt. %; and the drying mechanism should dominate. Baking different films, as Dill did, for 1 hour at 70°C and 100°C would cause significant differences in the amount of PAC decomposition. Chen *et al.*²⁰ have also demonstrated that the photospeed is affected by drying at temperatures lower than 120°C and by PAC decomposition higher than 120°C. We are currently studying higher temperatures to demonstrate this influence of both PAC decomposition and film drying mechanisms on dissolution rate parameters.

4. CONCLUSIONS

Liquid scintillation counting provides a measurement technique that can measure residual solvent content with high accuracy and precision. It also directly measures the amount of solvent content without the need for any assumptions or external standards.

The desorption of casting solvent from resist has a complex transport mechanism that can not be modeled with a constant diffusion coefficient expression. This solvent transport from resist films is characterized by an initial fast desorption during the time of most industrial post apply bake processes. The solvent desorption then decreases significantly compared to the initial transport period. We are currently working on a model to explain this transport mechanism. However, the post apply bake process can be modeled over typical industrial bake times fairly well using an empirical model based on the constant diffusion expression. This empirical expression assumes that there is a non-zero finite amount of solvent left in the film at infinite bake time. This model also assumes that the total amount of solvent desorbed from the film at infinite bake time for different temperatures follows a linear relationship with respect to post apply bake temperature. This model allows accurate calculation of the solvent content at temperatures intermediate to the temperatures studied.

Both the dissolution and optical properties of resist films are affected by film drying. Dill's A parameter is dependent on PAC concentration and film density. For our PAB conditions, variation in bake temperature did not cause a significant difference in PAC decomposition; and the increase in the A parameter as bake temperature increased was attributed to film densification. The dissolution rate parameters from the Original Mack model showed that the n increased and m_{TH} value decreased with increasing bake temperature. These changes were attributed to film drying. It was also mentioned that at high baking temperatures the PAC decomposition mechanism also starts to influence these parameters. So, the optical and dissolution rate parameters depend on the relative influence of both film drying and PAC decomposition which is related to the bake temperature and, to a lesser extent, bake time.

5. ACKNOWLEDGMENTS

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