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Thermal Effects on the Photoresist AZ1350J

Abstract: An experimental study is reported on the various effects produced by the baking steps normally used in processing positive photoresists for application in microelectronics. The particular material investigated is AZ1350J, made by the Shipley Company, Inc. The thermal effects are studied in terms of a newly modified model that characterizes the exposure and development processes in photoresist. The changes in performance of the photoresist as a result of prebake, post-exposure bake, and post-development bake are discussed and are related to the parameters in the model that govern exposure and development. The model is derived from physical rather than chemical measurements.

Introduction

Heat is a process input normally used in photolithography to dry photoresist prior to exposure, to minimize standing wave effects after exposure, and to improve adhesion after development. Baking conditions vary radically depending on tradition and local judgment of optimum conditions. Unfortunately, decisions relating to bake cycles are sometimes made without knowledge of how they affect the entire lithography process.

Thermal processing affects photoresist in several ways:

1. Partial removal of solvents
2. Thermal destruction of the photoactive inhibitor
3. Diffusion of the results of exposure
4. Changes in development properties
5. Softening and flow of developed images
6. Changes in chemical resistance and/or adhesion.

These effects occur together, not independently, and they can make significant changes in many aspects of resist performance.

In a study of thermal processing effects, we attempt to find physical measurements that quantitatively or qualitatively describe a particular aspect of the problem. As far as possible we view thermal effects in terms of the resist parameters as defined in the photoresist model [1-3]: A, B, and C for exposure and $R(M)$ for development. These parameters, discussed in the section following, physically describe the resist well enough to allow simulation of the resist exposure and development process for specific environments [4]. It is important that they be understood conceptually before an attempt is made to use them to describe new effects.

Photoresist parameters A, B, C and $R(M)$

All the positive photoresist materials commonly used in microelectronics are based on the photosensitivity of diazo-oxide materials carried in optically insensitive base resins [5]. The strong optical absorption of these films due to the diazo material is bleached by exposure, making it possible to use optical absorption as a tag to describe the exposure state of the resist material. Although the modeling studies based on optical absorption have a relationship to quantitative resist chemistry, this relationship has not been measured on the microchemical thin-film samples used in practice.

The basis of photoresist modeling is a set of physically measurable parameters. For exposure, A, B, and C are used to describe the optical destruction of the photoactive component of the resist. In development, a rate curve $R(M)$ describes the development rate associated with a given degree of destruction of the photoactive component.

The optical absorption coefficient of the resist at exposing wavelengths is related to the amount of photoactive compound remaining in the resist material. Since we do not measure this quantity chemically and know it only relative to the unexposed state, we choose to refer to it as M, the relative inhibitor concentration, to emphasize its role in preventing dissolution of the resist by the developer. M has a value of unity for unexposed resist and zero for completely exposed resist. The optical absorption of the photoresist is given by

$$\alpha = AM(z, E) + B, \tag{1}$$

where A is the exposure-dependent absorption parameter

that we associate largely with the photoactive compound; B is the exposure-independent parameter associated with the absorption of the base resin and exposure by-product compounds; and M, the relative inhibitor concentration, is a function of position in the resist film, z, and exposure energy, E. The two parameters A and B describe the optical absorption of the resist during exposure. A third parameter, C, is needed to describe the sensitivity of the resist in terms of its change in optical properties,

$$\partial M/\partial E = -I(z, E)\ M(z, E)\ C, \qquad (2)$$

in which I is the intensity of the illumination exposing the resist. The intensity I is a function of position because of the strong optical absorption of the resist, and of exposure energy because of the dependence of the absorption on the photoactive compound which is destroyed by exposure.

Because A, B, and C are measured on films prepared for exposure, they relate to the resist at exposure time. Their values can be changed by pre-exposure processing, such as aging, incorporating additives in the resist, or thermal processing of the resist film.

We emphasize that M, the fraction of remaining photoactive compound, is not measured chemically. It is a quantity implied from the changing optical properties of the resist film. Relative inhibitor concentration does not represent a fundamental chemical parameter of the resist system but is calculated from parameters that depend on resist processing. It can have chemical meaning only if one can follow the chemical changes that take place in processing between the liquid resist as supplied by the vendor and the thin resist film as prepared for exposure. As far as the exposure model is concerned, any chemical changes in the photoactive compound prior to exposure are contained in the A, B, and C values. The quantity M is always defined as unity at exposure time.

The index of refraction of the resist, its dynamic absorption constant, and the exposure environment, including thin-film effects on reflective substrates and the exposing image, comprise a sufficient set of parameters on which to base the exposure model. An exposed photoresist film can be described in terms of the inhibitor distribution within the film. This represents the fraction of the photoactive compound present before exposure which remains afterward. The computation is purely optics, based on the solution of Maxwell's equations with an exposure-dependent absorption term [3].

The development rate curve $R(M)$ is the link between the description of an exposed photoresist film and its removal by a developer solution. This curve is determined by measuring the removal rate of photoresist films with known relative inhibitor distributions under specified development conditions, and by assuming that development is described as a surface-limited dissolution reaction. Automated measurement techniques allow $R(M)$ to be measured routinely [6].

Since the inhibitor distribution of an exposed film is relative to the $M = 1$ state just prior to exposure, the $R(M)$ curve will be altered by any pre-exposure process, such as heating, that changes the resist chemistry. In addition, $R(M)$ is specific to a particular developer chemistry, typically concentration and temperature. It also includes any changes in resist solubility induced between exposure and development. Our only knowledge of the exposure state of a resist film is its inhibitor distribution immediately after exposure. Changes induced between exposure and development must be interpreted in terms of redistribution of inhibitor or change in development rate.

Our photoresist model assumes that the material has isotropic properties before exposure, that a single photon process is involved in exposure, that there is a single photoreaction taking place in the material, and that development is a surface-limited etching reaction without measurable swelling or induction period. These assumptions all seem to be adequate for resists processed with moderate thermal steps, not exceeding 70°C. The motivation for this thermal effects study was the observation that many users subject the resist to significantly higher temperatures in processing. This appears to be done often without much understanding of the implications of high temperature thermal processing.

In attempting to understand thermal effects, we see modification of photoresist properties before and after exposure, including redistribution of inhibitor after exposure. In addition we see deviations from the assumptions of the original resist model such as surface effects which apparently do not satisfy the requirement of isotropic films before exposure. These effects must be handled as additions to or deviations from the model. Incorporation of such deviations as second-order effects is an important step in growth of the modeling techniques.

Bulk thermal effects

Heat can destroy the photoactive compound in the photoresist being studied, AZ1350J [7]. The thermal process is not the same as the optical process, as evidenced by measurements of A, B, and C. This destruction takes place at temperatures commonly used for pre-exposure bake and can significantly alter resist performance.

Figure 1 shows the optical transmission of three samples of AZ1350J photoresist on optically matched glass substrates measured during exposure. This is the measurement used to determine the A, B, C exposure constants for positive photoresist. Samples were baked prior to the measurement at 70, 100, and 130°C for one hour. Note the extreme differences in transmission and bleaching during exposure.

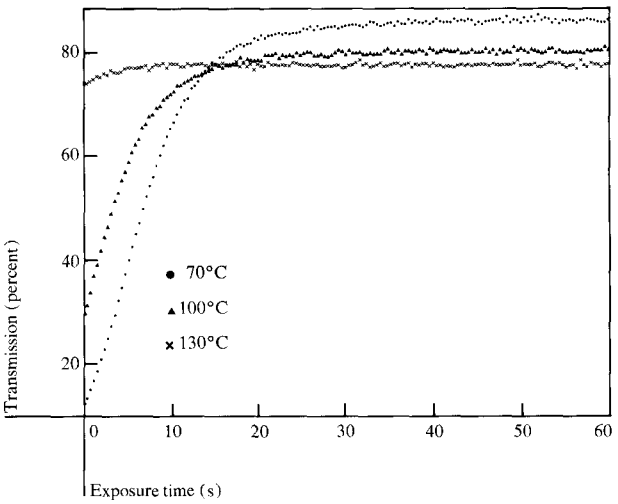

Figure 1 Optical transmission of three 2μm AZ1350J photoresist film specimens on matched substrates, measured during exposure to 5 mW/cm^2 light at 404.7 nm wavelength. Measured for three different prebake temperatures.

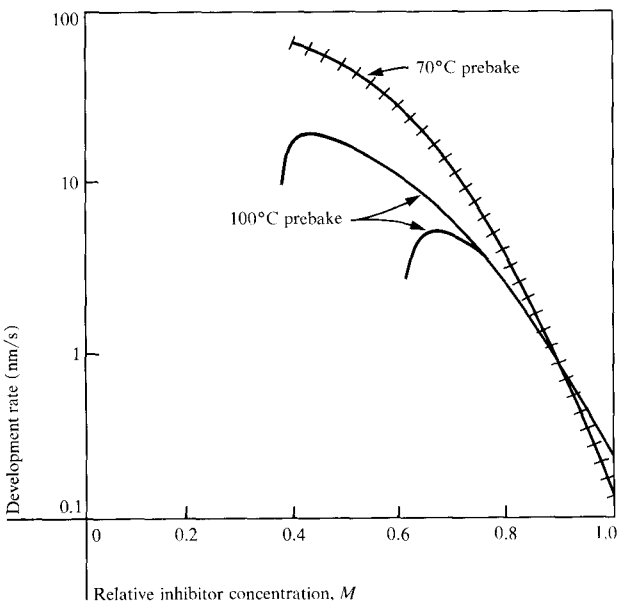

Figure 2 $R(M)$ development rate curves for AZ1350J photoresist in 1:1 AZ developer:water at 20°C. Prebakes at 70°C and 100°C.

The A, B, C values derived from these measurements are shown in the following tabulation.

	Parameter		
Prebake temperature, °C	A (μm^{-1})	B (μm^{-1})	C (cm^2/mJ)
70	0.89	0.077	0.28
100	0.48	0.114	0.29
130	0.026	0.131	0.30

Change of the exposure-dependent absorption term, A, indicates that the 100°C bake destroys half of the inhibitor (as related to the 70°C sample); the 130°C bake leaves only three percent of the inhibitor. The remaining inhibitor is unaltered, as indicated by a lack of significant change in the optical sensitivity term, C.

The thermally produced by-product compounds are optically insensitive, i.e., they are not changed by exposure to ultraviolet light. They are clearly different from optically produced by-products, as evidenced by the large increase in the exposure-independent absorption term B with increased bake temperature. Our physical measurements do not give us any direct insight into chemical processes such as cross-linking in the resist film.

Development rate curves also reflect the effect of prebake processing. Figure 2 shows the $R(M)$ curve for AZ1350J in 1:1 AZ developer:H_2O at 20°C with a 70°C prebake and also a curve for a 100°C prebake. The high temperature prebake slightly increases the development rate for lightly exposed films ($M > 0.8$) but shows a significant 3× decrease for heavily exposed films ($M = 0.4$).

High temperature bake gives a low solubility range between heavily and lightly exposed regions, and results in somewhat different image edge profiles than are seen for low temperature prebakes. This can be seen in Fig. 3, where A, B, C and $R(M)$ are used to model line-edge profiles [3] for a monochromatic projection printing environment. The high temperature prebake gives smaller standing-wave fringes on the line edge. This may or may not be a better process operating point: that decision should be based on careful sensitivity studies and not just a change in one parameter.

It is generally well known that a high enough bake temperature can eliminate the photosensitivity of AZ1350J. The manufacturer implies this in the product literature, and it is verified by our measurements of thermal destruction of inhibitor at 130°C. Our data show no measurable removal of a resist film baked at 130°C for one hour during a 15-minute development. This can be a desirable attribute, particularly when using a high temperature post-bake of resist images before etching an underlying surface with an alkaline etchant.

Surface thermal effects

In addition to destruction of the inhibitor, with associated exposure and development consequences in the interior of a resist film, heat can also cause surface effects. These cannot yet be as clearly quantified through A, B, C and $R(M)$ measurements as bulk effects, but we can identify significant effects on resist development and resultant image profiles. These surface effects, which violate our assumption that the resist film is an isotropic

medium, are seen in development, where they are not accompanied by any measurable swelling but only by a reduced dissolution rate.

Figure 4 shows measured curves of thickness versus development time for Alnoval 429k resin films developed in 1:1 AZ developer. This is a typical Novolak resin used in photoresist. With a 70°C bake, the resin starts developing almost immediately and continues at a nearly constant rate. The two 100°C bakes show an induction period during which the films develop very slowly, followed by development through the film at a rate only slightly lower than for the lower temperature bake. The induction is longer for a lengthier bake time at 100°C. This induction effect, or reduction of development rate near the resist surface, is seen in resist films as well. In measurement of the development rate curve $R(M)$ using a thick resist film (about 2 μm) on a matching substrate, the development rate is lower near the resist surface, causing the apparent drop-off of development rate at low inhibitor concentration seen in Fig. 2. No surface effect is evident where a 70°C prebake was used.

The surface rate reduction can be evaluated in a semiquantitative way by taking the ratio of the measured rate data near the surface to a smooth fit through all of the data (for the same M value). For the 100°C bake of Fig. 2 this gives the following characteristic:

Depth (nm)	*Rate multiplier*
0	0.2
25	0.5
50	0.7
100	0.8

This surface rate reduction can be added onto the model for photoresist development. Figure 5 shows modeled line-edges for a monochromatic projection environment calculated with and without the surface development rate reduction. The surface effect causes a significant steepening of the line-edge profile. At higher temperatures or other baking conditions it can become a significant process variable.

Thickness loss due to baking

It would be convenient to presume that photoresist films were isotropic two-component mixtures of base resin and associated inhibitor. However, dried films clearly contain some residual solvent, if one takes as evidence the fact that increasing bake temperatures decreases resist thickness and weight at temperatures below that necessary for thermal decomposition of the photoactive compound. The accompanying paper, "Thermal Analysis of Positive Photoresist Films by Mass Spectrometry" [8], uses mass spectrometric techniques to study solvent retention in resist films.

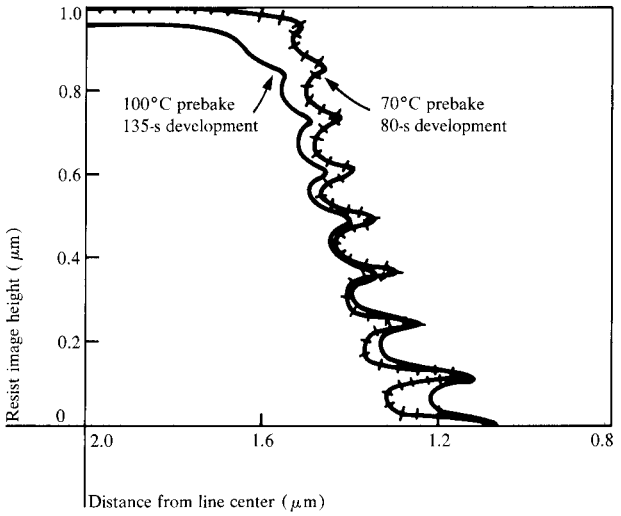

Figure 3 Calculated resist image profiles for one μm thick AZ1350J on bare silicon developed in 1:1 AZ developer:water. 2 μm projection printed through a N.A. 0.23 lens at 404.7 nm wavelength with $I_0 = 60$ mJ/cm^2. Images shown for 70°C and 100°C prebake conditions.

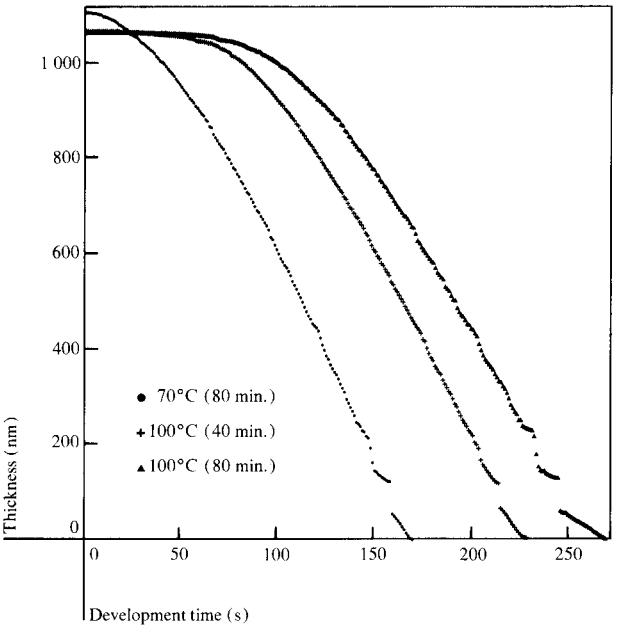

Figure 4 Thickness of Alnoval 429 K resin films as a function of development time in 1:1 AZ developer:water. Films prebaked at 70°C for 80 min and 100°C for 40 and 80 min.

In this paper, we concentrate on film thickness. Figure 6 shows measured thickness of resist films as a function of bake time and temperature. This was done using the IOTA automated spectrophotometer [6] to measure thickness of films on a hotplate. The hotplate measurements may not compare exactly with the oven bakes used for other experiments in this paper.

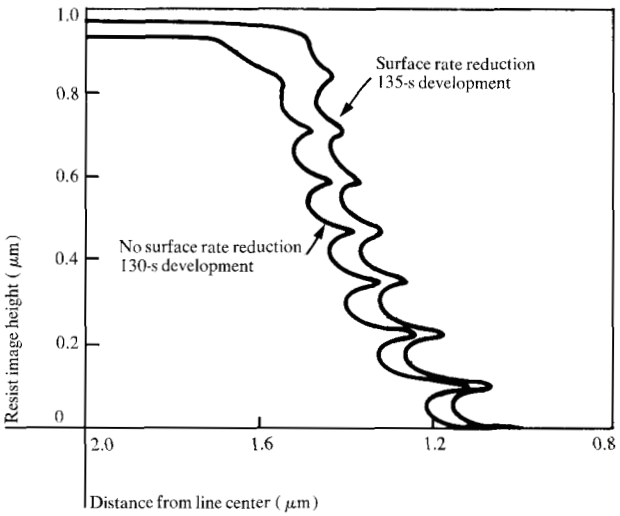

Figure 5 Calculated resist image profiles showing the effect of surface development reduction due to 100°C bake for 1/2 hour.

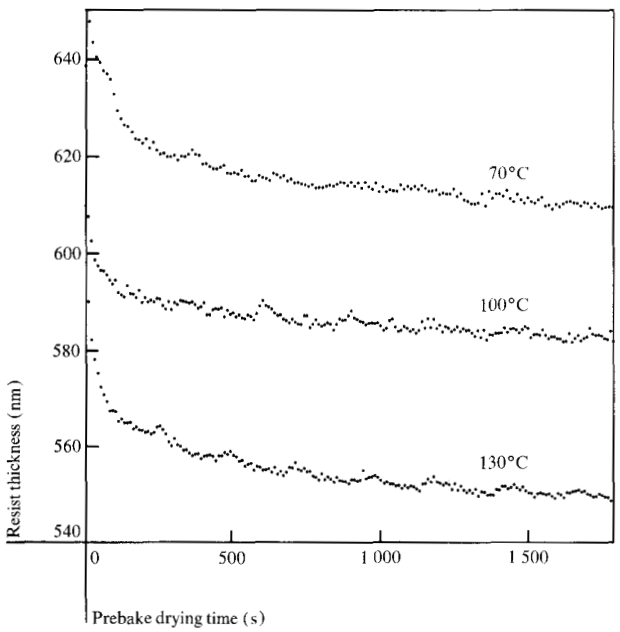

Figure 6 Effect of three different prebake temperatures (70, 100, and 130°C) on the drying rate of photoresist films. Initial thickness of all three resist-coated wafers was 678 nm.

Since resist thickness is a particularly important parameter in monochromatic projection exposure of positive photoresist, it is important that this be known at exposure time. We cannot assume that two samples of equal thickness before prebake at different temperatures will be equivalent at exposure time. Such assumptions have often led to unexplained scatter in experimental results.

Thermal diffusion effects after exposure

A post-exposure bake process has been shown [9] to dramatically reduce standing-wave fringes on monochromatic projection printed images. This does not imply elimination of interference effects and their critical implications relating exposure control to resist and dielectric layer thickness—only the appearance of interference effects is eliminated.

Measurement of resist thickness during development for monochromatic exposure of films on bare silicon shows the effects of post-exposure bake. Figure 7 gives thickness of developing AZ1350J films on silicon for different post-exposure bake conditions. Prebake was 70°C for one hour. The 100°C post-exposure bake for 20 minutes essentially eliminated the stair-step development characteristic. We attribute this "washout" to diffusion of the effects of exposure (inhibitor and/or byproducts) which eliminate the very close-spaced variations in development rate due to interference. These curves cannot be accounted for by changes of the $R(M)$ curve alone.

The inhibitor distribution after exposure for these resist films is calculated in Fig. 8. The close spacing of inhibitor minima and maxima (60 nm) makes diffusion possible normal to the film plane. Lateral inhibitor gradients due to images take place over much larger distances, so that diffusion can probably be ignored in this direction.

Preliminary indications are that the amount of diffusion taking place in post-exposure bake depends upon the degree of pre-exposure bake. This may be due to solvent-carrier effects, but in any case it constitutes a reduction in diffusion constant for films prebaked at higher temperatures. Because of this apparent dependence of the diffusion effects on processing temperatures, we have not attempted to measure what might be termed a diffusion "constant." It seems likely that the diffusion constant changes during the post-exposure bake process.

Knowledge of a diffusion constant is needed only for computation of intermediate stages in the diffusion process. Where diffusion has completely washed out some structure such as variation in inhibitor concentration due to interference effects, we can compute the new distribution. In the resist model this is easily done by applying an appropriate least-squares fit to the inhibitor concentration in the vertical direction. We can use this analytic curve for inhibitor concentration after diffusion, shown in Fig. 8, to model the resist thickness as a function of development time, but we must also know the change of development rate due to the post-exposure bake process.

Figure 9 shows a $R(M)$ curve measured with resist films on optically matched substrates baked at 100°C

after exposure. The exposure gradients are small in the optically matched environment, and diffusion due to the post-exposure bake was assumed to be small enough to be ignored in the rate measurement. This curve closely matches Fig. 2, where the bake occurred before exposure.

Another factor that must be considered in modeling post-exposure bake is the decrease in resist thickness that occurs during post-exposure bake. In order to correctly calculate the inhibitor concentration after exposure, we must know the resist thickness at exposure time. This is particularly important for the monochromatic exposure environment that produces strong effects of standing-wave interference. One thickness must be used for exposure and another for development. Note that the thickness at exposure is 0.64 μm, whereas the measured thickness after post-exposure bake is 0.6 μm, as shown in Fig. 8.

Figure 10 shows experimental and modeled thickness versus development time for post-exposure baked AZ1350J photoresist. Because our previous measurement for the 100°C bake showed induction was relatively unimportant we have not included it in this calculation; however, agreement is good. Extension of this model to two-dimensional line-edge contour calculations shows complete washout of the standing wave fringes, as is observed in practice for post-exposure baked images. This is shown in Fig. 11(a), which indicates good correspondence to the experimental images observed by Walker [9].

Higher temperature post-exposure bake processes can be expected to introduce more significant developer induction effects. Figure 11(b) shows line-edge contours calculated for post-exposure bake with a large (10× reduction) surface initiation effect. This results in steep edge profiles, particularly for overdeveloped images. Such effects have been seen by H. Moritz [10] in projection printed resist patterns, where 110°C bake temperatures were used. These steep profiles might be used for lift-off metallization with single-layer optical resist processing.

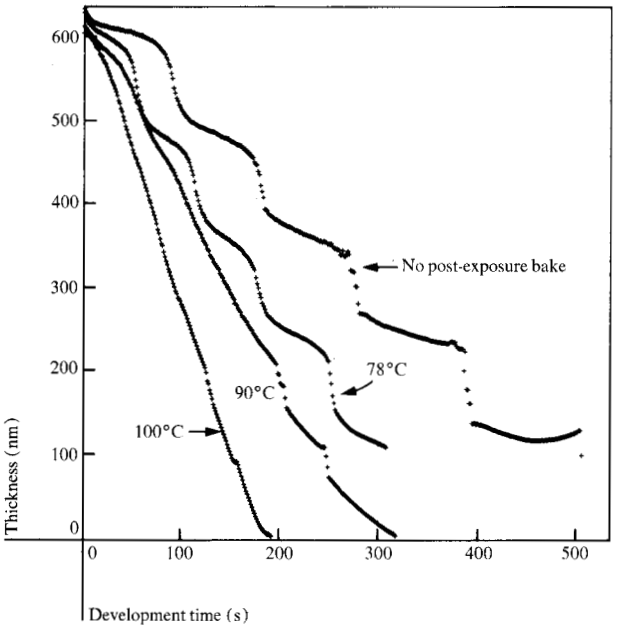

Figure 7 AZ1350J films on bare silicon exposed to 15 mJ/cm^2 at 404.7 nm and measured during development in 1:1 AZ developer:water at 20°C. Post-exposure bake at 78, 90, and 100°C for 20 min.

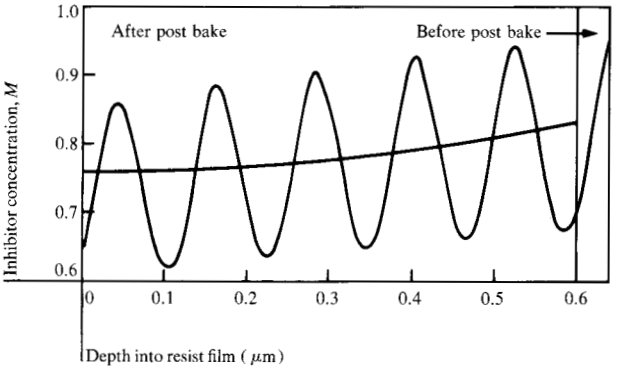

Figure 8 Calculated relative inhibitor concentration in a 0.64 μm AZ1350J film on bare silicon exposed to 15 mJ/cm^2 at 404.7 nm before post-bake and after post-bake with thickness reduction to 0.6 μm caused by the bake.

Post-development bake effects

A high temperature bake (about 140°C) is commonly used after development on the presumption that it improves resist adhesion or durability under etching conditions. Although this process is not included in modeling resist image profiles, it does frequently produce significant image modification. In this section we discuss effects that might be quantified to model post-bake and propose some measurements to help characterize adhesion and durability.

In post-bake, the temperatures used are high enough to cause modification of resist image profiles. The resist softens sufficiently that surface and interfacial tension forces modify the image shape, for they operate to minimize the energy associated with them. Both can be affected by adhesion promoters and by additives to the resist. We do not currently have any measurements of these forces or of the resist mechanical properties (e.g., yield strength or viscosity) at post-development bake temperatures.

In spite of the lack of quantitative data, we can observe some effects. Surface tension acts to minimize sur-

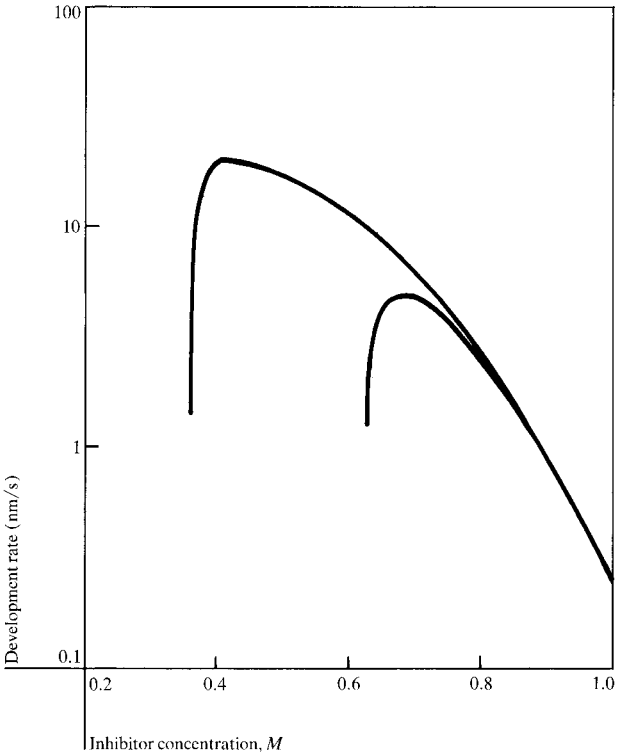

Figure 9 $R(M)$ Post-exposure bake at 100°C for 20 min.

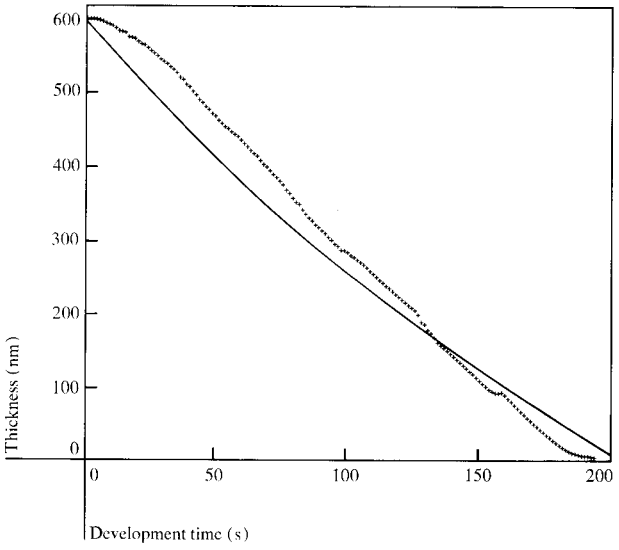

Figure 10 Thickness of an AZ1350J film developed in 1:1 AZ developer:water. Post-exposure baked for 20 min at 100°C. Experimental and modeled data.

face area, limited by factors such as gravity and flow properties of materials. There is a net tendency to round sharp contours and gradually smooth surfaces. Figures 12(a) and 12(b) are scanning electron micrographs of small line images before and after post-bake. The immediate effect is the spreading of the fringes on the line edge. The bottom fringe stays down because of interfacial adhesion to the substrate. The fringes would eventually pull in and disappear with continued baking, but this takes place slowly because of the amount of flow required.

In post-bake, it is quite possible for image detail to be lost and for small defects to heal. It should be a complex but reasonably straightforward fluids calculation to model image redistribution in post-bake after the appropriate parameters have been determined.

Resist adhesion can be treated in a quantitative way for some etching environments. In these environments the films under the resist appear to etch laterally on the surface at a higher rate, v_s, than in the bulk, v_b. This results in an identifiable etched edge contour. This contour can be seen in Fig. 12(b), which had the underlying oxide film etched.

An etching profile is shown in detail in Fig. 12(c), where the angle θ of the flat sidewall with the resist-substrate interface can be related to v_s and v_b by $v_b/v_s = \sin\theta$.

The larger θ is, the better the adhesion. From an etch control standpoint we should like v_s to be as close to v_b as possible, but the sharp edge contour may not be desirable for other reasons.

Etch modeling and electron microscopy (e.g., Fig. 12) are powerful tools for looking at the usefulness of postbake processes and adhesion promoters. They have been little used to date. We need to understand the effect of adhesion promoters on resist exposure and development parameters as well as their influence on adhesion during etching.

Measurement of resist thickness as a function of time in the etchant is another useful quantitative tool in understanding photoresist performance while etching. Alkaline etchants, for example, can be very similar to developer solutions. A high temperature bake can reduce the rate of removal of resist by alkaline developers of etchants. Measurement of this rate is simple using IOTA [6], and can be used as a measure of the durability of the resist in the etchant.

Developer concentration effects

The effects discussed in this paper are for AZ1350J photoresist developed in 1:1 AZ developer:H_2O at 20°C. This development condition gives a very high solubility range, about 1000:1, which tends to emphasize standing-wave fringes on line edges and induction effects. More dilute developers are reported to show even greater induction effects.

For development in concentrated AZ developer the solubility range is markedly reduced to 100:1 or less.

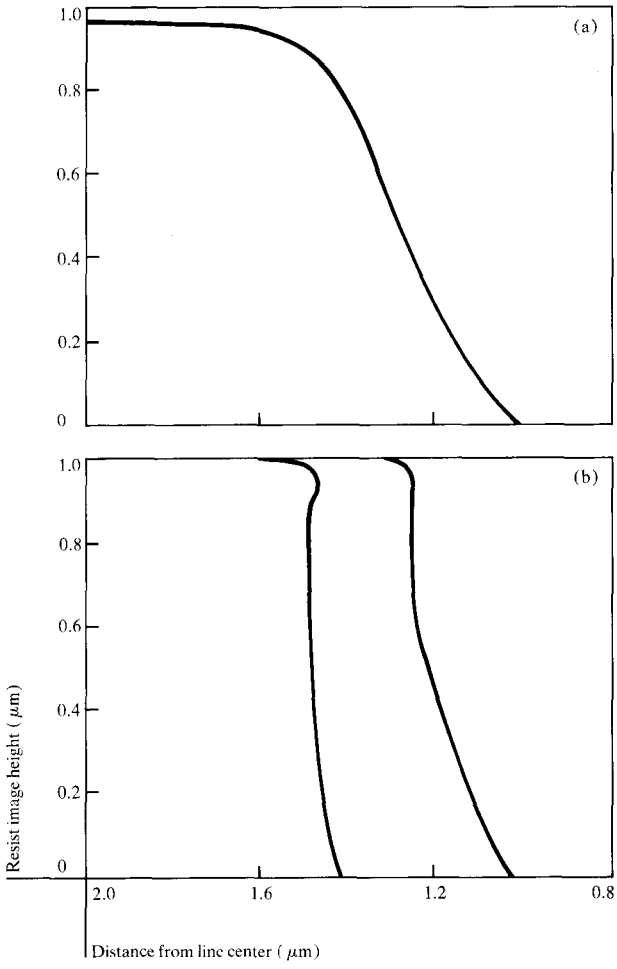

Figure 11 Calculated resist image profile. (a) The effect of a 100°C post-exposure bake for 20 min. (b) The effect of post-exposure bake, extreme surface rate reduction, and overdevelopment (up to 1.4 times nominal linewidth).

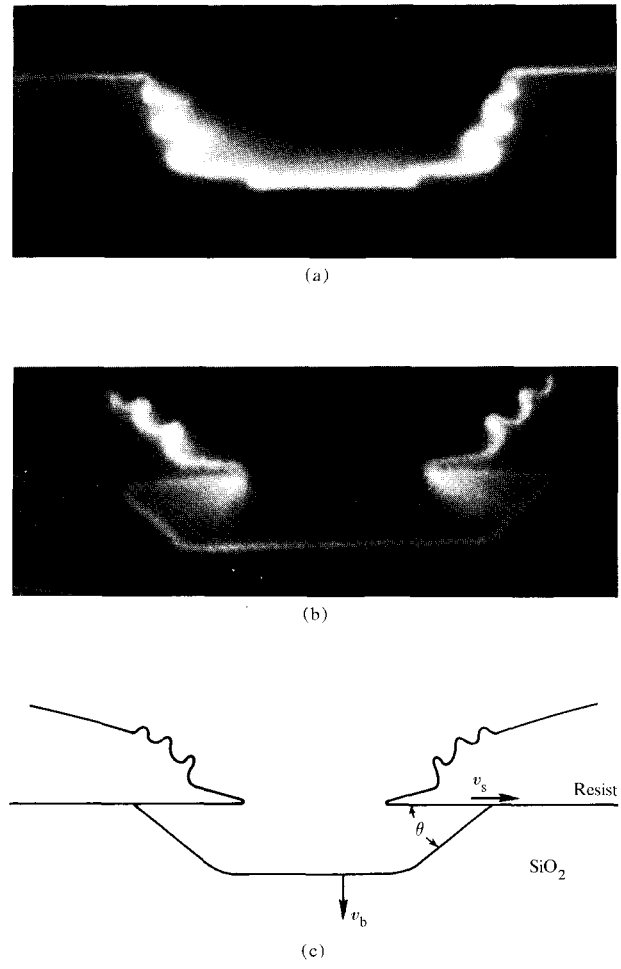

Figure 12 Etching profiles of small line images. Scanning electron photographs (a) before post-bake and (b) after post-bake. (c) Sketch of profile showing the etch interface angle.

What appears as fringes in our line-edge contours would show up as steps in the edge profile for concentrated developer. Initiation can probably be ignored for concentrated developer, except under extreme bake conditions.

Conclusions

• *Prebake*

Higher prebake temperatures can significantly modify the chemistry and performance factors of AZ1350J. Most significant is the loss of photosensitivity, resulting in much lower contrast. It can also introduce a significant initiation delay in development, particularly with less concentrated developers.

• *Post-exposure bake*

Baking after exposure can cause thermal redistribution of the effects of exposure which can eliminate standing wave fringes from a monochromatically printed pattern. It does not eliminate the most serious problem of monochromatic exposure: a variation of the total amount of energy coupled into the resist film by as much as a factor of two due to a quarter wavelength (60 nm) change in resist film thickness.

In spite of this limit, post-exposure bake can be a useful process, particularly over reflective metal substrates. Elimination of standing-wave artifacts, especially for the first exposure minimum above the substrate, can lead to significantly improved process control for both monochromatic and polychromatic exposure. It is this strongest

minimum, near the substrate, that develops most slowly and is responsible for untold numbers of unopened patterns.

The diffusion associated with post-exposure bake is not a parameter of the resist normally subject to quality control by the manufacturer. Thus there is some risk that this process could suddenly stop working, a risk we take any time we use an unspecified parameter. There is a history of such problems in photoresist, but usually in the past we were unaware of the unspecified parameter until after the process ran out of control.

Changes in exposure or development time required for post-exposure baked resist can occur in either direction, depending on process details. The slowly developing exposure minima are removed, but thermal decomposition of inhibitor and initiation effects can offset this, making prediction impossible without modeling.

• *Post-bake*

The post-bake process has a well-established tradition. There has been little work, other than looking at etched images, that gives any real measure of the effectiveness of the process for adhesion improvement or of image distortions created by it. We need further study in order to include this within those areas of photoresist processing that can be understood and optimized as a system.

Acknowledgments

Many have contributed important clues to unlocking the mysteries of positive photoresist. H. Levine, D. Ilten, D. Havas, and others contributed important observations. L. Kaplan pointed out many effects including post-exposure bake, initiation, and etch angle. J. Benz showed us initiation effects in development of base resin. E. Walker investigated reduction of fringes on projection printed lines with post-exposure bake. H. Moritz has recently shown steep profiles in post-exposure baked line edges. A. Neureuther, J. Tuttle, and E. Walker have helped build the model from which we are now able to look for deviations. W. Hornberger developed experimental techniques for characterization, for which P. Hauge provided theory and programs. K. Konnerth has provided tools and programs necessary for automated in-situ measurements that give us important new experimental capabilities.

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