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RECEIVED August 1, 1985

Physical and Chemical Modifications of Photoresists

Peter C. Sukanek

Department of Chemical Engineering, Clarkson University, Potsdam, NY 13676

The physical changes which occur in a resist during spin coating and the chemical changes which may occur during various image stabilization techniques are discussed. To predict the film thickness in spin coating, macroscopic mass balance equations are written for a resist system which contains a nonvolatile component and a volatile component which evaporates during the spinning. The model predictions are in qualitative agreement with experimental data. The film height is always proportional to the inverse square root of the spin speed. The factors which may influence resist image deformation at high temperature are also reviewed. Some of the proposed stabilization methods and the mechanisms by which they are thought to work are presented. Unknown factors in both coating and stabilization are highlighted.

This paper investigates two problems frequently encountered in the processing of resist materials. These are, first, the physical transformation of the resist from a viscous liquid to a solid-like film on the substrate (usually a wafer) surface. The second problem is how this film is chemically altered during the various image stabilization steps suggested in the literature. What often happens in this step is that the resist is transformed from a soluble material to an insoluble one. The extent of the image deformation on heating probably depends on the properties of the film, specifically the softening point (and hence the molecular weight) and the extent of retained solvent after spinning. Despite the widespread use of both of these processes in the industry today, several unknowns remain.

Spin Coating: Development of the Equations

The first published analysis of spin coating is due to Emslie, Bonner and Peck (1), who treated the problem of coating a single component, non-volatile, newtonian fluid on a disk. Film height

Continuously decreases with time as the fluid is "pumped" by centrifugal force along the disk. Since the viscosity of the fluid assumed constant, this pumping mechanism is always present, and film thickness eventually approaches zero. Acrivos, Shah and Arsen (2) later treated the problem of coating a power-law fluid. Unlike the newtonian result, a uniform film thickness cannot be obtained in this case. A layer which is initially uniform becomes non-uniform as a result of the spinning. The more the nature of the fluid's behavior from newtonian, the more non-uniform the film. Again, the film height approaches zero at 3 times. In order to achieve a finite film height, a constant force of fluid must be supplied at the center of the disk. Whereas above two investigators used as the starting point in their analyses macroscopic mass balance equations, Washo (3) attempted to solve the full Navier-Stokes equations from the start. Unfortunately, an error in his simplification of these equations invalidates his theoretical results. (Ignoring the small but important vertical velocity leads to an incorrect mass balance.)

Meyerohofer (4) examines the problem of spinning a two component film which is composed of "solids" and a volatile "solvent". He includes the effect of solvent evaporation and a viscosity which varies with time not only because of the centrifugal pumping, but because of the evaporation from the surface. As the film is leted in solvent, the viscosity increases and less fluid is lost due to pumping. Eventually a finite film thickness can be achieved because all of the solvent evaporates, and the film consists only an immobile solid phase. The present analysis is similar to rhofers's. His treatment of the evaporation is corrected, as is conclusion on the effect of spin speed (which apparently is due numerical error on his part), and some of his assumptions in plotting the equations are relaxed.

The fluid is assumed to consist of two components, a volatile component with concentration C_V and a nonvolatile component with concentration C_N . Mass balances on a cylindrical control volume on disk yield:

$$\frac{\partial}{\partial t} (C_N h) = -\frac{1}{r} \frac{\partial}{\partial r} (r C_N q) \quad (1)$$

$$\frac{\partial}{\partial t} (C_V h) = -\frac{1}{r} \frac{\partial}{\partial r} (r C_V q) - e \quad (2)$$

Flow rate, q , is given by (1, 2):

$$q = r \Omega^2 h^3 / (3\nu) \quad (3)$$

The evaporation rate, e , can be expressed in terms of a mass transfer coefficient, k :

$$e = k(C_V^* - C_{V\infty}) \quad (4)$$

where C_V^* is the concentration of the volatile component in the gas phase in equilibrium with the film, and $C_{V\infty}$ is the concentration in the gas far away from the disk. In most cases, this latter value can be taken as zero. If the distribution coefficient (or equilibrium constant) K is introduced, equation (4) becomes:

$$e = kK C_V \quad (5)$$

Finally, provided the air flow above the disk is laminar (i.e., the air Reynolds number is less than about 2×10^5) the Sherwood number varies with the square root of the air Reynolds number, and the mass transfer coefficient, k , is given by (5):

$$k = a \Omega^{1/2} \quad (6)$$

Two final assumptions concerning the system are made. First, we look for a solution which depends only on time; that is, the height does not vary with radial position on the disk. Second, we assume that the density of the film is a constant and is independent of the concentration of either component.

With these assumptions, equations (1-3, 5 and 6) can be combined, rearranged and put in dimensionless form to give:

$$\frac{\partial y}{\partial t} = -\frac{2}{3} \frac{y^3}{\nu/\nu_0} - \alpha x \quad (7)$$

$$\frac{\partial x}{\partial t} = \alpha x(x-1)/y \quad (8)$$

where the dimensionless parameters are defined by:

$$y = h \Omega^{1/2} / (\nu_0^{1/2} \nu^{1/4}) \quad (9)$$

$$x = C_Y/\rho \quad (10)$$

$$\tau = t\Omega a/\nu_0 \quad 1/2 \quad (11)$$

$$\alpha = \nu_0 \quad 1/4 K/a \quad 1/2 \quad (12)$$

Note that with these definitions, the spin speed is removed as a parameter from the defining equations.

The kinematic viscosity ratio is assumed to be of the same form as that used by Meyerhofer (4):

$$\nu/\nu_0 = \nu_s/\nu_0 + (1-\nu_s/\nu_0) \left(\frac{1-x}{1-x_0} \right)^n \quad (13)$$

Equations (7), (8) and (13) are solved using a Runge-Kutta integration. At sufficiently large values of τ , the dimensionless height, Y , approaches a constant. From the definition of Y , equation (9), the final height is given by:

$$h_f = Y_f a \quad 1/2 \quad 1/4 \Omega^{-1/2} \quad (14)$$

Hence, the height depends on the inverse square root of the spin speed. Further, provided the dependence on Ω is not strong, the height also depends on the one-fourth power of the initial kinematic viscosity. These conclusions are in good agreement with experimental data.

Spin Coating: Results and Discussion

It should be noted that the governing equations do not account for any differences between static and dynamic dispense methods. Neither is the spinner acceleration included. While there is no simple way to incorporate these effects in this model, the experimental investigation of Daughton and Givens (6) shows that the final height is independent of the dispense speed over a range of 0 to 500 rpm and over an initial viscosity range of 28 to 1100 cp. Also, they find that the final height is independent of the acceleration from dispense speed to final spin speed in the range of accelerations of 3000 to 30000 rpm/sec. These values are typical of modern spin coaters.

The initial height of the fluid on the disk is unknown and does

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not appear in this analysis other than as the initial condition for the integration. Figure 1 shows the effect of this initial height, Y_0 , on the final film height. Above a critical value of about 0.1, the final height is independent of the initial value. This result is in agreement with the experiments reported in (6), which show no effect of the dispense volume on Y_f . Using representative values for the parameters, Y_0 is about 0.2 for dispense volumes of 1 cm on a three inch wafer or 2 cm on a four inch wafer. This assumes, of course, that the fluid once dispensed uniformly covers the entire disk. While this does not happen (typically, a dispensed photoresist forms a puddle on the wafer surface which extends to about 1 to 2 cm from the wafer edge), photographs in (6) show that the wafer is fully covered in less than two revolutions.

Figure 2 shows that as the evaporation coefficient, α , increases, the film height also increases. This is in qualitative agreement with the work of Chen (7) on the effects of different solvents on the final thickness of spun-on polymer films. It is not possible to obtain a quantitative comparison with Chen's experiments because of the empirical nature of his evaporation term. The more volatile the solvent, the faster the viscosity of the fluid increases, the less important the centrifugal pumping effect and the greater the film height. A similar effect is found with increasing the initial solids content, as shown in Figure 3. Adjusting the initial solids loading by the addition of a thinner to the resist is a common way to control film height. Figure 4 shows the effect of the viscosity exponent, n , on the final height. As reported (4) this parameter has little effect.

The results thus far have assumed that all of the volatile component is removed during the spinning process. However, according to the measurements of Dill, et al. (8,9) the film after spinning can contain significant amounts of solvent. These investigators showed that 20% of the weight of AZ-1350J resist film after spinning is cellosolve acetate solvent. The effect of the residual solvent on final height is shown in Figure 5. The evaporation coefficient was modified for these calculations as:

$$\alpha = \alpha' (x - x_R) \quad (15)$$

where x_R is the mass fraction of residual solvent. As can be seen from Figure, residual solvent causes the final film height to increase, but not linearly. That is, a film with 10% residual solvent is not 10% thicker than one with no solvent. The increased solvent content decreases the viscosity causing more fluid to be pumped off the wafer by centrifugal action.

A number of experimental investigations into spin coating have appeared in the literature. Different investigators correlate the thickness with different parameters. Daughton and Givens (6) and Chen (7) show a dependence on the initial kinematic viscosity to the 0.29 and 0.36 powers, respectively. This is in rough agreement with

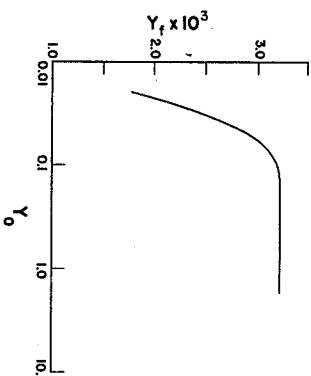


Figure 1. Effect of initial film height, Y_0 , on final height, Y_f . ($x_0=0.9; \nu_s/\nu_o=10^{-2}; n=4; \alpha=10^{-5}$)

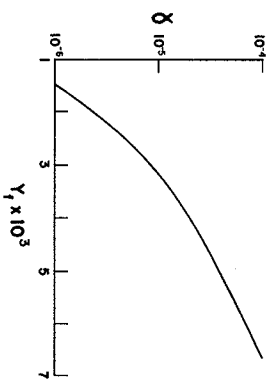


Figure 2. Effect of evaporation coefficient, α , on final film height, Y_f . ($x_0=0.9; \nu_s/\nu_o=10^{-2}; n=4; Y_0=0.2$)

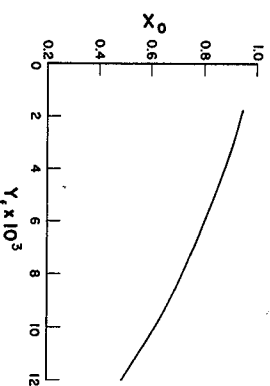


Figure 3. Effect of initial solids content, x_0 , on final film height, Y_f . ($Y_0=0.2; \nu_s/\nu_o=10^{-2}; n=4; \alpha=10^{-5}$)

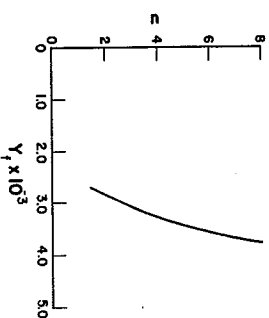


Figure 4. Effect of viscosity exponent, n , on final film height, Y_f . ($Y_0=0.2; \nu_s/\nu_o=10^{-2}; \alpha=10^{-5}; x_0=0.9$)

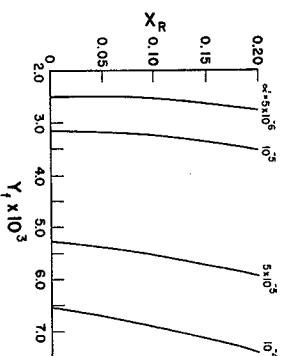


Figure 5. Effect of residual solvent, x_R , on final film height, Y_f . ($Y_0=0.2; \nu_s/\nu_o=10^{-2}; n=4; x_0=0.9; \alpha=\alpha'(x-x_R)$)

the 0.25 power illustrated by equation (14). Others (10,11) show a more complex dependence. This, however, is not surprising considering that initial viscosity and solids content usually cannot be varied independently and that the evaporation coefficient also depends on initial viscosity.

Spin speed is the most common parameter used in the correlation of film height, with most results (3,4,6,7,10,11) showing the inverse square root dependence predicted by equation (14). The major discrepancy is with the results of Daughton and Givens (6) for polyamic acid solutions, where a -0.81 power dependence was found. While it is tempting to attribute this result to possible non-newtonian behavior of the solution, rheological measurements for similar solutions (12) do not support this conclusion. The maximum value of the shear rate (or the velocity gradient), $\dot{\gamma}$, in spin coating occurs at the interface between the wafer and the fluid. For a newtonian fluid, the shear rate at this location is:

$$\dot{\gamma} = 3q/h^2 = \rho r \Omega^2 h / \mu \quad (16)$$

For a 5 μ m thick film spun at 4500 rpm at the edge of a 3 in. wafer:

$$\dot{\gamma} = 40/\mu \quad (17)$$

with viscosity, μ , in Pa s.

From Jenekhe's data (12) on what appears to be the same polyamic acid solution as that used in (6), the newtonian region ($\mu = 1.4$ Pa s) persists out to a shear rate of about 300s⁻¹. This indicates the flow is always in the newtonian region for the experiments reported in (6). It is, of course, difficult to estimate the boundary between newtonian and non-newtonian effects in this flow since as the solids content of the resist increases, non-newtonian behavior may be expected to set in at a lower shear rate. However, from equation (17), the shear rate experienced by the fluid decreases as well due to the increased viscosity. In order to resolve this question, more data is needed on the variation of the rheological behavior with solids content.

The expression for the evaporation rate used in equation (4) is based on laminar air flow. As the size of wafers used in the industry increases from the most common 125mm size used today to the relatively new 150mm and then to the 175mm and 200mm values recently obtained by crystal growers, the air Reynolds number increases. Using a typical value for the kinematic viscosity of air (15 mm²/s), the product of the rotation rate, ω , in rpm and the square of the radius in mm must satisfy the following inequality for the air flow to remain laminar:

$$\omega r^2 \leq 1.43 \times 10^7 \quad (18)$$

For a 125 mm wafer, the air flow at the very edge is no longer laminar once the rotation rate exceeds about 3700 rpm. At 6000 rpm, the transition occurs at a radial position of 49 mm; the outer 14 mm is in the transition or turbulent flow regime on a 125 mm wafer at this speed. In the turbulent region, the evaporation rate depends on radial position and varies with the spin speed to a power other than one-half. The result is expected to be a non-uniform resist thickness. While this effect is probably not important for the wafers in use today, it may become important in the future. The critical speed for a 200 mm wafer is only 1430 rpm. Above this value, at least part of the wafer will be in the turbulent air flow regime.

Image Stabilization: Methods and Mechanisms

In some processes, such as ion implantation and reactive ion etching, the resist can experience high temperature. When heated to temperatures above about 130°C, the resist can "reflow", that is, images present in the resist can deform. The surface forces which act on the film can cause the resist to flow into previously open areas and to pull back from the edges at other locations. In either event, the images are no longer satisfactory. The forces acting on the resist structures are complex, and it is not the intent of this paper to address the causes of this deformation. The "softening point" of typical resins used in resist lie in the range of 90-120°C (13). (The softening point is not a well-defined value.) Solvent still present in the resist after the spinning operation can act as a plasticizer, further reducing the softening point and leading to greater image distortion.

Several methods have been proposed in the literature to avoid or reduce the extent of the deformation. Certain resist systems are specifically marketed based on their stability to high temperature bakes. Other methods which may improve stability include slow heating, vacuum exposure with ultraviolet (UV) radiation, deep UV (at a wavelength of about 250 nm) exposure and exposure of the imaged resist to a plasma. In the remainder of this paper these methods will be discussed with the aim of gaining some insight into the chemical changes which occur in the resist during these operations.

In order to understand the changes, it is necessary first to know the components of the resist and the chemistry of the exposure step. For positive photoresists, the mechanism was elucidated by Paensky and Iyerla (14). Typical resists are a solution of a naphthoquinone diazide photoreactive compound (PAC) and a cresol formaldehyde novolak resin in one or more high boiling point industrial solvents. The number average molecular weight of the resin is quite low, on the order of 1000, with a polydispersity of about 10 (13). During the normal exposure step, the PAC, in the presence of water, absorbs light and is transformed into a

carboxylic acid. When water is absent the PAC reacts with the resin to form an ester. If the PAC contains more than one active group, a crosslinked polyester can result. This is the explanation of Tracy and Mattox (15) for their vacuum exposure stability method. The crosslinked structure, is too rigid to undergo deformation at high temperature.

A similar mechanism can be invoked to explain increased stability as the result of vacuum or programmed (slow) heating (16, 17). In this case, however, two competing phenomena occur. As demonstrated by Allen, et al. (18), when the resist is heated to high temperature, it becomes insoluble in common solvents. However, if the PAC is first deactivated, the resist is still soluble after a high temperature bake. In addition images present in the resist undergo greater distortion at high temperature if the PAC is deactivated prior to bake. This suggests that a crosslinking reaction similar to that discussed above occurs at high temperature. In contrast with the vacuum exposure, during the high temperature bake both image distortion and resist reaction occur simultaneously. At higher temperatures the deformation apparently occurs more rapidly than the chemical reaction, giving a distorted image in an insoluble resist. If the resist is slowly heated, the deformation rate is slower, and the chemical reaction causes some stability of the images.

The explanation for stability after deep UV exposure is not apparent, despite the fact that commercial instruments are available to effect this stability. Different groups have examined resist changes brought about by deep UV exposure (17-20), and it is convenient to examine their results with respect to the wavelength of light used, whether or not water is necessary, whether or not the PAC is required and the resist type.

There is little agreement on which factors are important in deep UV image stabilization. Hiraoka and Pacansky (17, 19) claim that any wavelength is effective in achieving a stable image, provided the PAC is deactivated. On the other hand, Allen, et al. (18) find that this blanket exposure increases the deformation. Vollebrotok, et al. (20) claim that the same mechanism is at work here as in the vacuum exposure method; i.e., the PAC undergoes a photochemical reaction with the resin to form a crosslinked polyester. Therefore, their exposure must be done in the absence of water. The use of 254 nm radiation confines the crosslinks to the top surface of the resist, since the resin absorbs strongly at this wavelength. Allen, et al. (18) were able to show that image stability is linked to the formation of a "skin" on the surface of the resist that results from the deep UV treatment. If 200 nm of the resist surface is removed in a plasma, the images will again deform. They exposed both the resin alone and resist with the deactivated PAC to 254 nm radiation and found a surface skin in both cases, suggesting that the reaction involves the resin only. It seems clear that the deep UV radiation at 254 nm gives the resist a "case" which contains it during subsequent high temperature bakes

and allows the thermal reaction to occur without the thermal deformation.

One final method of stabilization which has been proposed is to expose the resist to a glow discharge from a nitrogen or other gas plasma (21). Hansen and Schonhorn (22, 23) found that polyethylene and other polymers underwent a surface crosslinking reaction when exposed to a glow discharge from any of the inert gases. They termed the process "CASING" (Crosslinking by Activated Species of Inert Gases). The name was subsequently shown to be a misnomer by Hinds (24) who found that the deep ultraviolet radiation from the discharge causes the reaction. Hence it seems likely that the same mechanism which is responsible for the deep UV stabilization is also active in plasma methods.

The reason for the intrinsic temperature stability of certain resists is probably related to the resist components. Assuming that all positive photoresists contain resin, PAC and solvent as discussed above, we can postulate two ways to increase the stability. The first is to use a solvent which evaporates completely during the spinning or post apply bake. Removing the solvent would raise the viscosity and softening point of the resist. Another method of raising these values is to increase the molecular weight of the resin. As mentioned above, the number average molecular weight of typical resist resins is in the neighborhood of 1000. Gipstein, et al. (25) found that the softening point increased from 95 to 122°C when the molecular weight of the novolak resin increased from 900 to 1350.

Summary

Macroscopic balance equations have been used to investigate the dependence of resist thickness on spinning parameters. The final height of the film is a complex function of initial viscosity and solids content, evaporation rate and residual solvent. The predictions are in qualitative agreement with experiments. As initial viscosity (or solids content) increases, or as the solvent evaporation rate increases, the film thickness increases. In addition the theory indicates that as larger wafers are used, variations in film height may become important because of turbulent mass transfer effects. As long as the fluid is newtonian and the air flow above the wafer is laminar, theory predicts that the thickness is inversely proportional to the square root of the spin speed. This is in good agreement with available data on photoresist spinning. Experimental measurements on polyamic acid solutions show a definite deviation from the inverse square root dependence. Since non-newtonian effects appear to be negligible in the experiments, the cause of this deviation is unknown.

Residual spinning solvent left after the post apply bake, as well as the low molecular weight of the resin used in the resist, lead to deformation of the lithographic images when the resist is heated to temperatures in excess of about 130°C. In order to avoid this problem, a number of image stabilization schemes have been

proposed. Both vacuum exposure of the imaged resist to UV light and heating appear to bring about a reaction between the resin and the photoactive compound to produce a high molecular weight material which is insoluble in common solvents and comparatively immobile at high temperatures. If the heating is done slowly, the thermal reaction occurs faster than the image deformation, and some image stability is achieved. Exposing the imaged resist to deep UV radiation or a glow discharge forms a shell around the images which hold them together during the high temperature bake. The mechanisms of these reactions are unknown.

Nomenclature

- a, constant in mass transfer expression ($m/s^{1/2}$)
 - c, concentration (kg/m^3)
 - e, evaporation rate (kg/cm^2s)
 - h, film height (m)
 - k, mass transfer coefficient (m/s)
 - Q, distribution coefficient
 - q, flow rate per unit circumference (m^2/s)
 - r, radial position (m)
 - t, time (s)
 - x, mass fraction
 - y, dimensionless film height
 - α , dimensionless evaporation coefficient
 - γ , shear rate (s^{-1})
 - v, kinematic viscosity (m^2/s)
 - ν , shear viscosity (Pa.s)
 - ρ , total density (kg/m^3)
 - T, dimensionless time
 - ω , spin speed (rpm)
 - Ω , spin speed (s^{-1})
- Subscripts
- F, final value
 - N, non-volatile component
 - 0, initial value
 - R, residual value
 - s, solvent
 - V, volatile component
 - ∞ , ambient atmosphere

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