Characterization of AZ PN114 resist for soft-x-ray projection lithography

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Using 14-nm wavelength illumination, we have imaged 0.1-μm-wide lines and spaces in single-layer thin films of the highy sensitive, negative, chemically amplified resist AZ PN114 by using both a Schwarzschild 20× camera and an Offner ring field 1× optical system. For soft-x-ray projection lithography the approximate 0.2-μm absorption length in resists at 14-nm wavelength necessitates a multilayer resist system. To explore further the requirements of the imaging layer of such a system, we have transferred patterns, exposed by a high-resolution electron beam in a 60-nm-thick layer of AZ PN114, into the underlying layers of a trilevel structure. Significant pattern edge noise and resist granularity were found. It remains to be determined whether the observed noise is dominated by statistical fluctuations in dose or by resist chemistry. We also investigated pinhole densities in these films and found them to increase from 0.2 cm^{-2} for 380-mm-thick films to 15 cm^{-2} for 50-nm-thick films.

Introduction

To be useful for integrated circuit (IC) manufacturing, a resist process must be capable of high throughput and have good pattern transfer capability as well as demonstrate high resolution. In 1990, imaging of 50-nm-wide lines and spaces in poly(methyl methacrylate) (PMMA) resist, with a 20× Schwarzschild camera at λ = 14 nm, was reported.[1,2] PMMA has long been known for its extremely high resolution and good process latitude, but its low sensitivity and poor plasma etch resistance make it an unlikely candidate for an IC fabrication process.

Chemically amplified resists are attractive candidates for use with soft-x-ray projection lithography (SXPL) because of their improved sensitivity and their novolak-type resin matrices, which provide good dry plasma etch resistance during pattern transfer.[3] While a lower exposure dose threshold would allow for higher wafer throughput, increased dose sensitivity usually leads to reduced resolution and process latitude. The crossover point for these two opposing requirements has been addressed theoretically[4–9] but has not yet been firmly established experimentally.

A difficulty encountered with any resist at λ = 14 nm is the short absorption length, typically <0.2 μm.[10,11] Although this absorption length is a factor of 4 greater than that found at λ = 193 nm,[12] it is short enough to mandate a bilevel or trilevel pattern transfer system. In a positive resist such as PMMA, even for a relatively thin (0.1-μm-thick) top layer of a trilevel, significant sidewall angles are found in the developed image in spite of the high modulation transfer function of the imaging system.[10] These sloped sidewalls, which are a by-product of the decrease in dose as a function of depth in the resist film, preclude realizing the ±10% critical dimension (CD) tolerance required in IC manufacturing.

A negative cross-linking resist should exhibit steep sidewalls compared with positive resists, since the most strongly cross-linked region will be at the resist surface. This strong cross linking will tend to counteract rather than add to the effect of the developer gradient with depth in the film. But this slight advantage will not be sufficient to offset absorption effects so as to allow for a single-layer resist process.

Generally IC manufacturers avoid bilevel or trilevel resists because the introduction of additional processing steps increases cost and adds to the difficult task of maintaining high yield. (For thin surface-imaging layers, yield reduction caused by pinholes is of particular concern.) Nevertheless bilayers are cur-

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rently used in production. Two examples are antireflective coatings beneath resists in *i*-like lithography[13] and the diffusion-enhanced silylating resist (DESIRE) process[14,15] now in use for patterning metal interconnects. With the possible exception of x-ray proximity printing lithography,[5–9,16] it is unlikely that any technology for producing 0.1-μm CD IC's will be able to utilize a single-layer resist scheme. And even for proximity printing, at a 0.1-μm CD, linewidth variations caused by Fresnel diffraction over topography on the wafer surface may necessitate a planarizing layer. However, for that technology an advantage will remain in that the absorption length in resists is more than 2 μm at $\lambda \sim 1$ nm, and hence the imaging layer thickness is limited only by mechanical stability. For either SXPL or optical imaging at 193 nm the imaging layer thickness is restricted by the absorption depth within the resist.

In an effort to clarify the prerequisites necessary to achieve 0.1-μm CD's in surface-imaging layers with good process latitude, we have characterized thin films of AZ PN114 resist[3,17–20] with SXPL and an electron beam, and we have pinpointed some of the problems that must be solved if multilayer resists are to be usable in manufacturing. We have imaged 0.1-μm-wide lines and spaces in film thickness ranging from 50 to 200 nm, using both a Schwarzschild 20× reduction camera[1,2] and an Offner 1× optical system.[21,22] The linear absorption coefficient and dose sensitivity at $\lambda \sim 14$ nm have been measured by Kubiak *et al.*[23] We have investigated pinhole densities as a function of resist thickness, and using electron-beam-exposed samples, we have transferred patterns to the underlying films of Ge and hard-backed (HB) resist in a trilevel structure. We do not suggest that this trilevel will be a manufacturable commercial process. We merely use it to illustrate the difficulties involved in imaging in thin surface layers of highly sensitive resist.

Exposures and Processing

Two different types of wafer were used for imaging experiments: resist on bare silicon that had been dehydration baked at 200 °C for a minimum of 30 min and then vapor primed with hexamethyldisilizane to promote adhesion and a trilevel consisting of 60 nm of resist on 20 nm of thermally evaporated Ge on top of 200 nm of HB resist. Thin films were spin cast from resist that had been diluted 1:2 or 1:3 with AZ thinner.[6] Each wafer received a 1-min, 120 °C, solvent-drive-off bake. Resist-on-Si samples were exposed at the U13UB beamline at the National Synchrotron Light Source at Brookhaven National Laboratory by using both the 20× Schwarzschild[1,2] and the 1× Offner ring field[21,22] optical systems. In addition, both types of sample, trilevel and resist-on-Si, were exposed with a 50-keV JEOL JBX-5D II direct-write, electron-beam lithography system.

Immediately after exposure, each sample was baked for 5 min at 105 °C to drive forward the acid-catalyzed, cross-linking reaction. Resist-on-Si samples were immersion developed in 0.3 normality (N) AZ developer.[17] The trilevel samples were developed in Shipley MF312 27CD[24] that was diluted 1:1 with deionized water to yield 0.13N. Electron-beam-generated sensitivity curves for the two developers are shown in Fig. 1. The resist was found to be slightly more sensitive to the MF312. For both developers, in the given processing conditions, with development times of 90 s and 6 min, the gamma values were found to be ~4. Significantly, the heavily exposed material exhibited a negligible dissolution rate for either developer. The metal-ion-free MF312 developer was chosen for use with the trilevel because the NaOH-containing AZ developer was found to etch Ge at a rate of ~0.8–1 nm/min, which was sufficient to cause adhesion failure.

Both the prebake and postexposure bake (PEB) were carried out with a PMC 730 series digital hot plate, to which we added a custom-built vacuum chuck. The chuck temperature was monitored at a point ~0.5 in. (~1.27 cm) beneath the chuck surface. We compared that temperature with the temperature of a probe glued to a wafer surface; the two differed by at most 1 °C. From experiments with electron-beam writing of fine patterns, we determined that PEB temperatures of 110 and 115 °C cause unacceptable pattern distortion for both small and large features.[25] Both the 120 °C prebake and the 105 °C PEB temperatures were controlled to within ±1 °C.

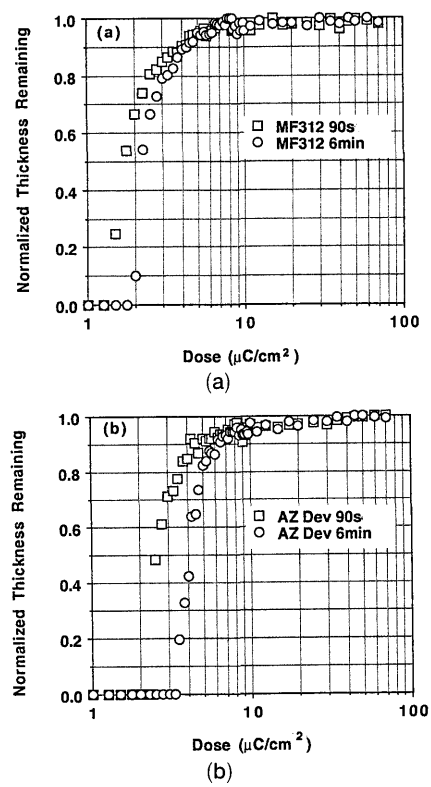

Fig. 1. Sensitivity curves for 0.25-μm-thick films of AZ PN114 exposed by an electron beam and developed in (a) 0.3N AZ developer and (b) 0.13N Shipley MF312.

Trilevel samples were exposed with a 17-nm-diameter beam and 15-pA current; the pixel size was 12.5 nm. Images were transferred to the Ge layer by reactive ion etching (RIE) with CF_3Br at 20 (SCCM denotes cubic centimeter per minute at STP), 10 mTorr, and 100-V dc bias. The Ge:AZ PN114 etch ratio was 4:1; thus the selectivity for this step was 12:1. Pattern transfer to the HB resist was done by RIE in O_2 gas at 20 SCCM, 10 mTorr, and 100-V bias. The Ge:HB-resist etch ratio was > 125:1, leading to a selectivity of better than 12:1 in the etch.

Resolution and Edge Definition

Figures 2 and 3 are scanning electron micrographs of resist patterns imaged with the Schwarzschild camera. The sensitivity of AZ PN114 at $\lambda = 14$ nm was found by Kubiak *et al.*[23] to be ~ 3 mJ/cm^2, roughly an order of magnitude improvement over PMMA. From the measured sensitivity, and from comparisons with typical PMMA exposure times, we estimate the doses for these exposures to be in the range of 6–10 mJ/cm^2. Note that in Fig. 2, while the 0.1-μm lines in the background are resolved, the region in the foreground, corresponding to the 0.05-μm line/space pattern, is slightly modulated but not well resolved. At the time of these experiments the optic numerical aperture was set to 0.08, which corresponds to a resolution limit of ~0.1 μm. However, had the numerical aperture been selected for imaging 0.05-μm features, we doubt that they would have been resolved in AZ PN114 because 0.05 μm most probably exceeds the resist resolution limit. The slight residue between the exposed 0.1-μm lines is believed to be due to overdosing of the resist. In Fig. 3 we show close-ups of 0.1- and 0.15-μm line/space patterns in a 200-nm-thick film. Although we were successful in imaging in this comparatively thick film, the process latitude was not robust because of attenuation. Note the resist granularity, which is exhibited as both surface and edge roughness and which will be replicated in the pattern transfer to an underlying layer.

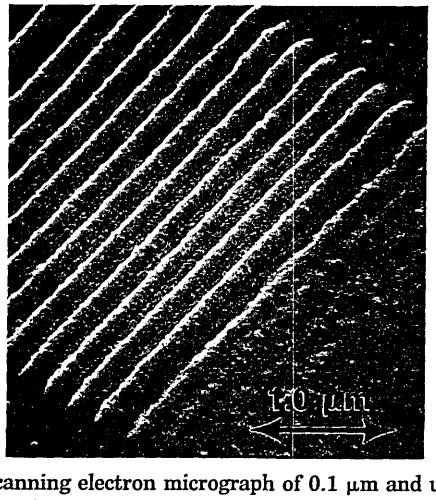

Fig. 2. Scanning electron micrograph of 0.1 μm and unresolved 0.05-μm line/space patterns imaged in 50-nm-thick film of AZ PN114 with the Schwarzschild camera.

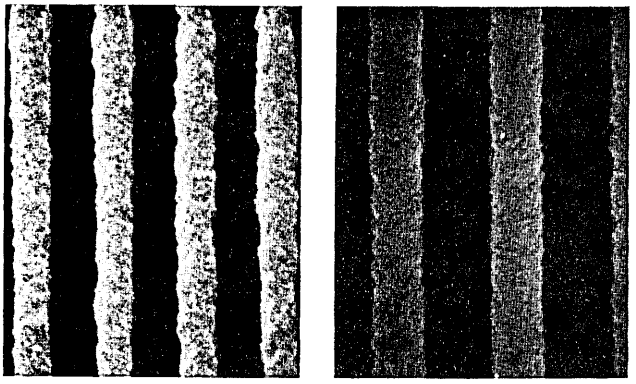

Fig. 3. (a) 0.1-μm lines and spaces and (b) 0.15-μm line and spaces in 200 nm of AZ PN114 imaged with the Schwarzschild camera.

Examples of etched trilevel patterns, exposed by an electron beam with a dose of 8.5 μC/cm^2, can be seen in Figs. 4 and 5. The numbers in Fig. 4 refer to grating periods. The smallest fully resolved period is 0.15 μm, that is, 0.075-μm-wide lines and spaces. Figure 5 is a close-up of a 0.1-μm-wide line etched in Ge on HB resist. The edge noise is ~14 nm peak to valley.

Because of the many parameters influencing the performance of chemically amplified resists,[18–20] and because of the tool dependence of resolution,[5,7,26] we cannot state categorically that the resolution and edge noise seen in these experiments will not be improved on in the future by the use of either SXPL or x-ray proximity printing. Nevertheless, because we believe that this is the highest resolution ever demonstrated in this resist, and because the edge noise seen on the 0.1-μm-wide line in Fig. 5 is typical of the best results that we have been able to produce, it is reasonable to use this empirical evidence to estimate the minimum CD for this resist in a manu-

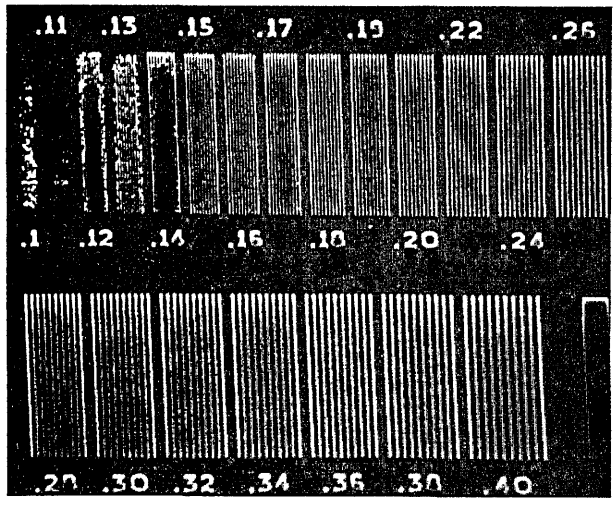

Fig. 4. These gratings were exposed in 60 nm of AZ PN114 by the electron beam. The pattern was then transferred into 20 nm of Ge on 200 nm of HB resist by using RIE. The numbers in the micrograph refer to the grating periods in micrometers. The smallest fully resolved grating period is 0.15 μm.

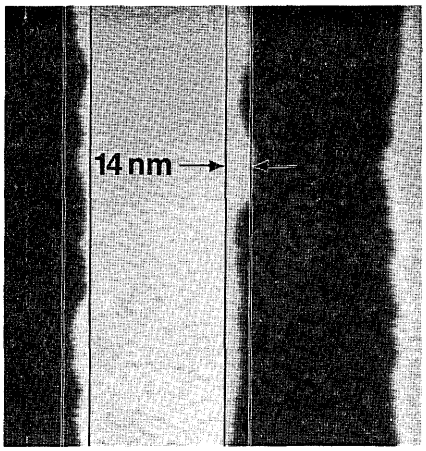

Fig. 5. Close-up of a 0.1-μm-wide line etched in Ge on HB resist. The edge roughness is ~ 14 nm peak to valley.

facturing environment. Using the 14-nm peak-to-valley noise estimate, we take the rms amplitude of the edge noise to be $7/\sqrt{2}$ nm. Assuming that the noise from two edges adds in quadrature, we find that $\sigma = 7$ nm. Thus, to maintain ±10% linewidth control with 3σ confidence, the minimum acceptable CD is 0.2 μm, and for 6σ confidence the CD is 0.4 μm.

Several points concerning the resist roughness and pattern edge noise bear mentioning. For the electron-beam exposure the observed noise is comparable with the beam diameter. From the sensitivity curves for MF312 the clearing dose at the line edge should correspond to ~2 μC/cm², or ~10 electrons/12.5 nm pixel, and thus considerable statistical fluctuation in dose might be expected. The granularity seen in the SXPL exposure in Fig. 3 is of the same order of magnitude as that seen in electron-beam exposures even though the number of primary exposure events (i.e., absorption of 90-eV photons) per unit area is an order of magnitude higher than the number of incident electrons in the electron-beam exposure. As pointed out by Smith,[5] electron-beam statistics are fundamentally two dimensional in nature because few if any incident electrons are absorbed in the resist, but photon statistics is a three-dimensional problem since the photons are absorbed at discrete locations within the film thickness. For an incident dose of 3 mJ/cm² in a 200-nm-thick film with a linear absorption coefficient of $\alpha = (0.22\ \mu\text{m})^{-1}$,[23] the average number of photons absorbed in a cubic volume of 10 nm on a side is found to be ~10 at the top of the resist and ~4 at the bottom.

For the case of an electron-beam, Sutherland *et al.*[4] investigated pixel error rates using the equation

$$P(\text{error}) = [(\pi/2)\bar{n}]^{-1/2} \exp(-\bar{n}/8), \qquad (1)$$

where $\bar{n}$ is the mean number of electrons per pixel and P(error) is the probability that a pixel will be incorrectly exposed because of statistical fluctuation. The theoretically acceptable error rate depends on a number of assumptions. For example, if one assumes that all we are interested in is killer-defect density, where killer defects are assumed to be squares of CD/4, and if we assume a chip size of 2.5 cm² and a CD of 0.2 μm, a dose of 2 μC/cm² would produce an error rate of 4.9*e*–19 or one error per 20 million chips. This suggests that a 0.2-μm CD, AZ PN114 is still far from the shot noise limit of resolution. However, the more stringent criterion is linewidth control.

For proximity printing, Smith[5,7] has analyzed the effect of shot noise on CD control and estimates that 24 photons per volumetric element of CD/10 will be sufficient to maintain ±10% linewidth control. Neureuther and Willson[6] analyzed the error rate for the case of a positive resist and considered the ability of the developer to remove underexposed volume elements. They arrived at a figure of 30 photons per volume element, although they use a spherical volume of diameter CD/3. Based on these analyses, and depending on the volume definition, the minimum estimated CD for the dose discussed here ranges from 0.06 to 0.18 μm. The issue is further complicated by the statistics of chemical amplification, which have been included in simulations,[8,9] but have not yet been analyzed in terms of impact on yield and CD control.

Generally a given resist will have associated with it a characteristic resolution-limiting dimension that is based on its material properties. For PMMA this dimension might be the size of a void created by the removal of a pendant group that will detach and outgas from the resist on irradiation. Ouano[27] has estimated that these voids are ~1 nm in diameter. For a chemically amplified resist the characteristic dimension will be associated with but not limited to the size of the acid catalyst molecule that is generated during exposure.[8] In this case the characteristic dimension will also depend on the number of bond-forming or bond-breaking events caused by each of these molecules and possibly on the acid diffusion length. A separate characteristic dimension can be associated with the incident irradiation: the secondary electron range in the case of electron beam and the photo and Auger electron range for x rays. The effect of these two dimensions, one associated with the resist and another with the radiation source, will add in quadrature, but in general one will dominate. We need more research to determine whether the resolution in chemically amplified resists such as AZ PN114 is dominated by photon and electron-beam statistics or by chemistry.

Pinhole Density

We determined pinhole densities by using a defect magnification technique. Thermally oxidized silicon wafers were spin coated so that resist thicknesses of 50, 100, 250, and 380 nm were obtained. The resist and dilution solvent had been filtered to 0.1 μm by the manufacturer and were deposited with a syringe with a 0.2-μm filter in a class 10 clean room. After the solvent-drive-off prebake the wafers were submerged for 10 min in 1:7 buffered oxide etchant diluted with deionized water. The etchant, attacking the oxide

Table 1. Pinhole Densities Found in Spin-Case Films of AZ PN114

Thickness (nm)	Pinholes (cm^2)
380	0.2
250	1
100	4
50	15

through an infinitesimally small pinhole in the resist, would generate a hole in the SiO_2 of ~1-μm diameter. To make these defects more easily identifiable, the wafers were anisotropically etched in ethylenediamine pyrocathecol. The pits formed in the silicon by the ethylene diamine pyrocathecol etchant take on characteristic shapes that appear as squares or rectangles when viewed at normal incidence through a microscope. Defect densities were established by counting the number of pits found in an area of 9 cm^2 at the center of each wafer. The densities, which are given in Table 1, agree in magnitude with those found by Kuan *et al.*[28] in thin films of AZ 5206. The trend is clearly toward higher densities for thinner films.

To place these numbers in context, we note that the targeted defect density for resist coating in 256-Mbit dynamic random-access memory fabrication is 0.06 cm^{-2}.[29] Even the thickest film tested, 380 nm, failed to approach this target. However, the pinhole detection technique used here should be able to detect pinholes smaller than those of killer-defect dimension. Also, to avoid generating additional defects unrelated to film thickness, we left the resist in this experiment unexposed. This does not accurately reflect real IC manufacturing, in which only a fraction of the chip area is patterned. Pinholes in areas that are etched away in the resist development process will be of no consequence. These two factors suggest that our experiment may overestimate the meaningful pinhole density. Finally, filtration through pores of dimensions smaller than the resist thickness and deposition in a class one environment should reduce the number of pinholes. Further investigation is needed to determine whether more fundamental barriers will prevent pinhole elimination in ultrathin spin-cast films.

Conclusions

We have demonstrated 0.1-μm resolution in thin films of AZ PN114 by using SXPL at doses that are approximately an order of magnitude lower than those required for PMMA to be exposed. Using electron-beam-generated images, we have resolved lines and spaces 75 nm wide, and we have demonstrated pattern transfer into a trilevel structure. Based on empirical evidence of granularity and pattern edge roughness, we believe that AZ PN114 resist, as processed in these experiments, might possibly be used for CD's down to 0.2 μm with ±10% linewidth control and 3σ confidence; for 6σ confidence the minimum CD is likely to be 0.4 μm. Further research is needed to determine whether the resolution limits seen here are dominated by photon and electron-beam statistics or by resist chemistry and processing conditions. Pinhole densities were seen to increase with decreasing film thickness. For 50-nm-thick films we found ~15 defects/cm^2. We anticipate that these numbers can be improved by better clean-room control and finer filtering, although it is unknown whether the targeted 2-order-of-magnitude reduction in defects can be met in ultrathin films.

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