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ANTI-REFLECTIVE POLYMER COATINGS IN OPTICAL MICROLITHOGRAPHY

Binod De[a]; Sanjay Malik[a]; Stephanie Dilocker[a]; Gregory Spaziano[a]; John Biafore[a]; Murrae Bowden[a]
[a] Photoresist Materials Research, Arch Chemicals Inc, East Providence, RI, U.S.A.

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ANTI-REFLECTIVE POLYMER COATINGS IN OPTICAL MICROLITHOGRAPHY

Binod De,* Sanjay Malik, Stephanie Dilocker, Gregory Spaziano, John Biafore, and Murrae Bowden

Photoresist Materials Research, Arch Chemicals Inc, 200 Massasoit Avenue, East Providence, RI 02914

Dedicated to the memory of Professor Sukant K. Tripathy.

ABSTRACT

The use of polymers based on biphenyl methacrylate as antireflective coatings (ARC) in lithographic applications is described. The optimum range of refractive index (n) and complex index (k) resulting in minimal reflectivity, as predicted by Prolith simulation, was 1.56 to 1.76 and 0.125 to 0.275, respectively, which corresponded to polymers containing 50 to 70 mol% of biphenyl methacrylate. ARCs from these polymers were formulated with a melamine crosslinker and a thermally activated catalyst. Optimal lithographic performance was obtained by baking the spin-coated films at 200°C for 90 seconds for crosslinker concentrations less than 7.5% as confirmed by lack of footing and scum at imaging layer/ARC interface.

Key Words: Anti-reflective coating (ARC); DUV lithography; Optical parameters; Curing properties; Etch selectivity

*Corresponding author.

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INTRODUCTION

Microlithography is defined as the process of transferring a pattern into a reactive polymer film (termed as resist), which will subsequently be used to replicate that pattern into an underlying thin film or conductor [1]. As applied to the patterning of microstructures for electronics applications, the lithographic process involves transfer of geometric features or patterns, outlined in a two-dimensional mask, into the surface of a suitable substrate, such as silicon. The basic pattern transfer steps are: (1) coating a suitable semiconductor wafer with a light sensitive polymer film, called a photoresist; (2) exposing the resist to patterned actinic radiation; (3) developing the unwanted photoresist region to define the 3-dimensional resist pattern created in the exposure step, and, (4) etching the pattern into the wafer. These steps are illustrated in Fig. 1, exposure to light induces a chemical change that alters the solubility of the photoresist film in the exposed area.

The control of critical dimension (CD) of resist features across the wafer is affected by several conditions on the wafer surface, notably surface reflection and variation in topography. The former causes standing wave effects, while the latter gives rise to variations in resist thickness across the substrate. Both phenomena result in variations in absorbed energy that translate into line width variations in the developed image. To minimize reflectivity from the wafer surface, an antireflective coating (ARC) is typically applied. This coating consists of a polymer film appropriately formulated with a light-absorbing group (chromophore) designed to absorb most of the light that reaches the interface through the resist [2–5]. Typical chromo-

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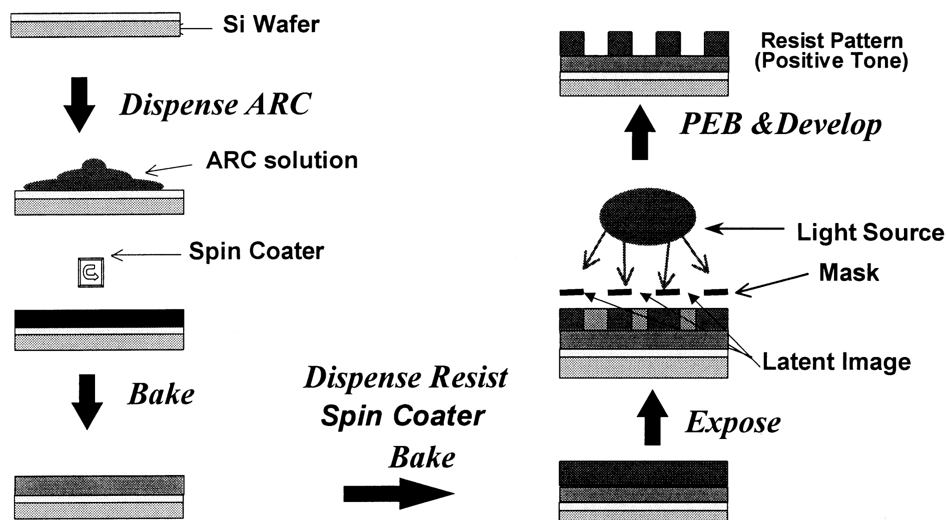

Figure 1. Schematic diagram showing basic patterning process.

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phores suitable for application at 248 nm include anthracene [6], benzophenone [7], and aromatic sulfone moieties. The chromophore can either be physically dispersed throughout the polymer matrix or chemically bound to the backbone.

In order to preserve the optical characteristics of the interface between resist and ARC, it is important that no intermixing of the two layers take place. This condition requires that the ARC be rendered insoluble following spin coating so that no intermixing takes place on subsequent spin coating of the resist. This is accomplished by formulating light-absorbing polymers with a crosslinking agent and curing the film at elevated temperature prior to spin coating the resist layer.

In addition to its dependence on the optical n and k parameters of the ARC and resist film, reflectivity is also influenced by the thickness of the ARC layer. Interference effects lead to a series of maxima and minima in reflectivity as ARC thickness increases, finally reaching a constant reflectivity value that is determined by the optical parameters of both films. The reflectivity remains effectively constant at higher ARC thickness which offers the advantage over thinner ARC films of less susceptibility to small variations in resist thickness. Another advantage of moving to the thicker films is the tendency of the ARC to provide a more planarised coating, which tends to minimize CD variability arising from wafer topography. These advantages are exploited in bilayer resist design which uses a thin imaging resist on top of a thick, planarizing ARC layer properly designed to serve as a pattern etch mask.

In this paper we describe the design of thermally cross-linkable polymeric materials applicable for anti-reflective coatings for bilayer application. The physical, chemical and optical phenomena involved in photoresist and ARC processing will also be discussed.

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EXPERIMENTAL

Materials

All polymers were prepared by standard free radical polymerization in THF at 65°C for 24 hours.

Characterization

Polymer compositions were determined by ^{1}H and ^{13}C NMR spectroscopy using a Bruker spectrometer Model AC250 operating at 250 MHz. Molecular weight of the polymers was determined by GPC against polystyrene standards using a RI detector and a set of four Styragel columns. Weight-average molecular weight (M_w) and polydispersity were calculated using the Millenium^{TM} (v2.15) software package from Waters Corporation.

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Thermal studies of the polymers were performed using Perkin-Elmer Pyris 1 Thermogravimetry Analyzer and Differential Scanning Calorimeter at a heating rate of 10°C/minute.

Lithographic Properties

ARC formulations were prepared by blending polymers with standardized amounts of a thermal acid generator (TAG) and a melamine crosslinker in propylene glycol monomethyl ether acetate (PGMEA). The solutions were sequentially filtered through 0.5 and 0.2 μm Teflon filters prior to spin coating. The films were spun-coated onto four-inch silicon wafers and then cured on a hotplate at a constant temperature. The optical properties (n and k) were calculated from reflection data using an n&k tool. Film thickness was measured on a Prometrix SpectraMap model SM300.

Lithographic performance was evaluated by spin coating ARCH's TIS-2000 photoresist on top of the cured ARC film, and exposing to 248 nm wavelength using a Canon EX6 stepper with a binary mask [8]. The optical settings used were numerical aperture of 0.65 and partial coherence 0.80 (outer)/0.5 (inner).

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RESULTS AND DISCUSSION

Materials Design

The general design concept of our ARC material is shown in Fig. 2. For the dye component, we chose specific monomers based on the 4-biphenyl chromophore e.g., biphenyl methacrylate (BPMA), which shows strong absorbance at 248 nm. As a suitable crosslinking unit we chose monomers with hydroxyl functionality e.g., 4-hydroxy styrene or 2-hydroxyethyl methacrylate. These functional moieties also facilitate solubility of the resulting polymer in the solvents commonly employed for photoresist application. Our third component was chosen to provide etch stability and was selected from

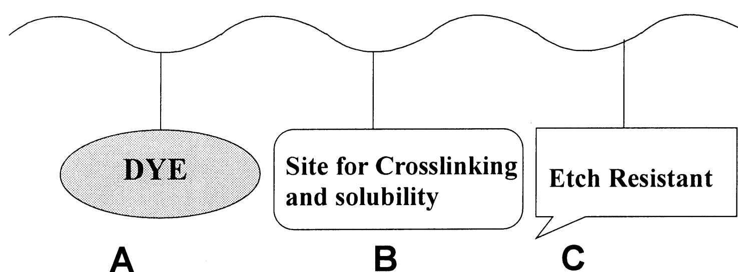

Figure 2. General design concept of anti-reflective polymeric coating.

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groups containing alicyclic or aromatic moieties that do not absorb at 248 nm. By varying the concentration of each component in the polymer, we could optimize the various parameters of importance, e.g., optical absorption, cross-linking density, etch resistance, solubility, etc.

Design Considerations

In order to design an ARC polymer for optimal lithographic performance, it is important to know the corresponding n and k values that will minimize reflections at the interface. These parameters are materials dependent, and can be determined from lithography simulation programs such as PROLITH [9–10] into which the various molecular and optical parameters have been fed. Fig. 3 shows the simulated reflectivity as a function of k, indicating an optimum value of 0.1 to 0.2 for a refractive index of 1.70 and a film thickness of 5,000 A. Fig. 4 extends the parameter space by taking both reflectivity and refractive index into consideration and indicates that intensity of reflected light at the photoresist-ARC film interface is minimum when n is in the range of 1.56 and 1.76 and k is in the range of 0.125 and 0.27.

The importance of selecting the right optical parameters of the ARC on the lithographic properties of the imaging layer is clearly illustrated in Fig. 5. ARC-A, whose k-value (0.41) and substrate reflection (1%) lie well outside the optimal range, shows considerable scum at the resist/ARC interface and strong standing wave pattern in the resist. In comparison, ARC B with a k value of 0.21 and substrate reflection of 0.5% produces much cleaner profiles with squarer tops.

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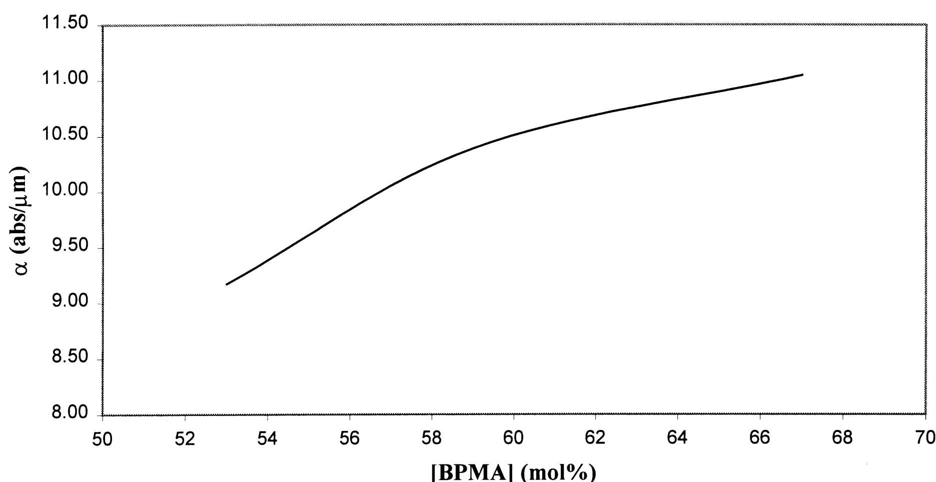

Figure 3. Absorbance vs. BPMA concentration.

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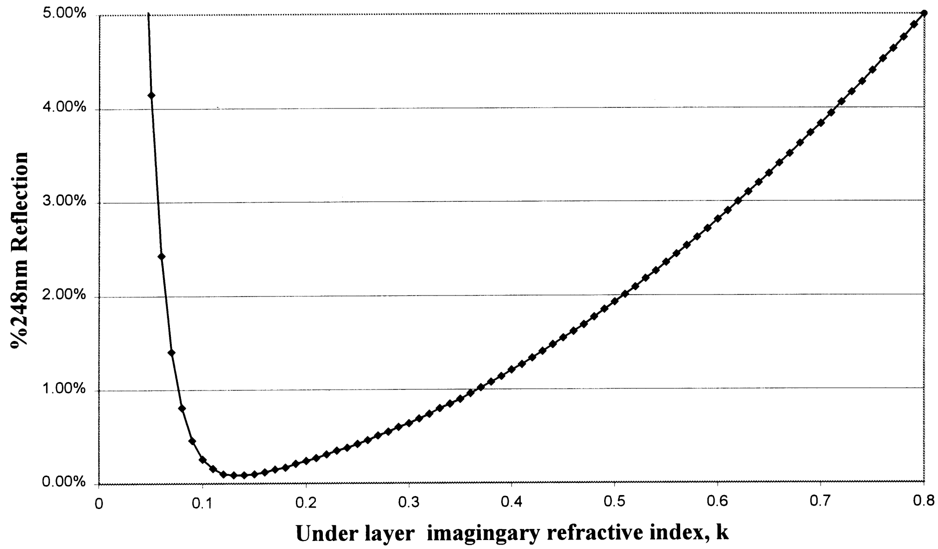

Figure 4. 248 nm reflection as a function of imaginary refractive index (k); ARC film thickness = 5000 Å and n = 1.70.

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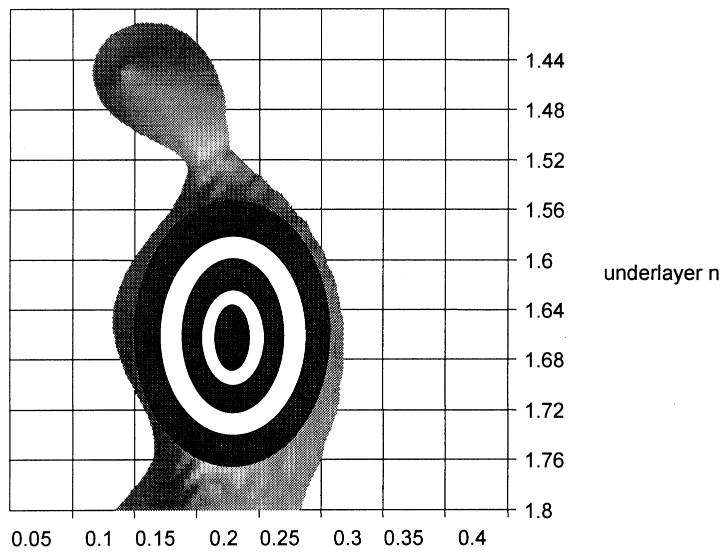

Figure 5. Target values for n and k to achieve desired reflectivity of <0.5% at 248 nm.

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Polymer Composition and Optical Properties

The synthesis and molecular characteristics of terpolymers designed to span the optimized absorption range are summarized in Table 1.

All polymers were soluble in commonly used lithographic solvents such as PGMEA, 2-heptanone, n-butyl acetate and methyl methoxy propionate and formed uniform films by spin coating. The low molecular weight of the polymers (6–7 Daltons) was targeted to ensure both solubility in resist solvents, and optimum film quality for use as a planarizing ARC. The high thermal stability of the polymers (ca. 300°C) permitted a wide process window for curing. The glass transition temperature (Tg) was designed to be on the order of 120°C to facilitate uniform film flow during curing process.

The optical properties of the ARC materials at 248 nm are summarized in Table 2. The reflective index value of 1.718 was essentially the same for all polymers over the BPMA range investigated. However, the k-value increased with increasing amount of BPMA in the polymer as expected.

The absorption coefficient α, (which is related to k through the equation $\alpha = (k \cdot 4\pi/\lambda)$ is plotted in Fig. 6 illustrating the increase in absorption as BPMA content in the polymer is raised from 50 to 65 mol%.

Curing Properties

The organic anti-reflective coating should provide an impervious surface that prevents intermixing of the upper photoresist layer. An improperly crosslinked ARC layer can allow cross-diffusion of small molecules such as acids, bases and other contaminants, resulting in several defects at the photoresist/ARC interface that can be transferred into the semiconductor substrate. For instance, improper curing can lead to a degree of porosity of the ARC underlayer, facilitating diffusion of the photogenerated acid from the exposed photoresist film into the underlayer during post-exposure baking (PEB) which depletes its concentration in the imaging layer. This acid-loss results in an incomplete chemical change in the exposed area at the interface and consequent partial insolubility of the photoresist film, which leaves a

Table 1. Properties of Polymers

Sample ID	[BPMA] Moles	M_w (Daltons)	M_n (Daltons)	T_g (°C)	TGA at 5% Wt Loss (°C)
Polymer 1	53	13,200	6,800	128	309
Polymer 2	59	13,700	6,600	124	305
Polymer 3	67	11,200	6,000	118	300

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Table 2. Optical Properties of Polymer Formulations at 248 nm

[BPMA] Moles	n	k
53	1.718	0.181
59	1.719	0.205
67	1.718	0.218

residue after development. Hence, achieving a high degree of crosslinking is crucial.

The requisite degree of crosslinking can be achieved by choosing an appropriate multifunctional crosslinking agent [11]. The melamine crosslinker used in this study, can potentially provide six crosslinking sites making it an efficient crosslinking agent. The crosslinking reaction requires a thermal acid generator (TAG) such as an alkyl-*p*-toluenesulfonate, which decomposes at elevated temperatures to generate acid [8].

The mechanism of the curing reaction is shown in Scheme 1. Thermally generated acid forms a reactive carbocation on the crosslinker, which either reacts with the hydroxyl group of the functional polymer or attacks the ring of the aromatic group and forms another molecule of acid. This chain reaction produces a network of crosslinked resin [12].

The onset of curing is estimated to be 150°C as indicated by a strong exotherm beginning at around 150°C and extending to 190°C as shown in the DSC thermogram in Fig. 7. The lack of an exotherm during the second heating cycle in the DSC thermogram (see Fig. 7) indicates that the film is fully cured during the first cycle of heating. The temperature at which curing

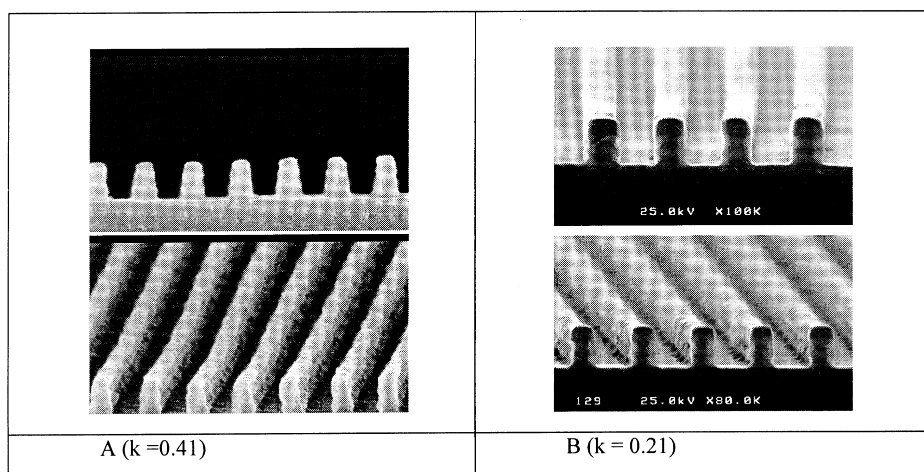

Figure 6. Effect of k on lithographic performance.

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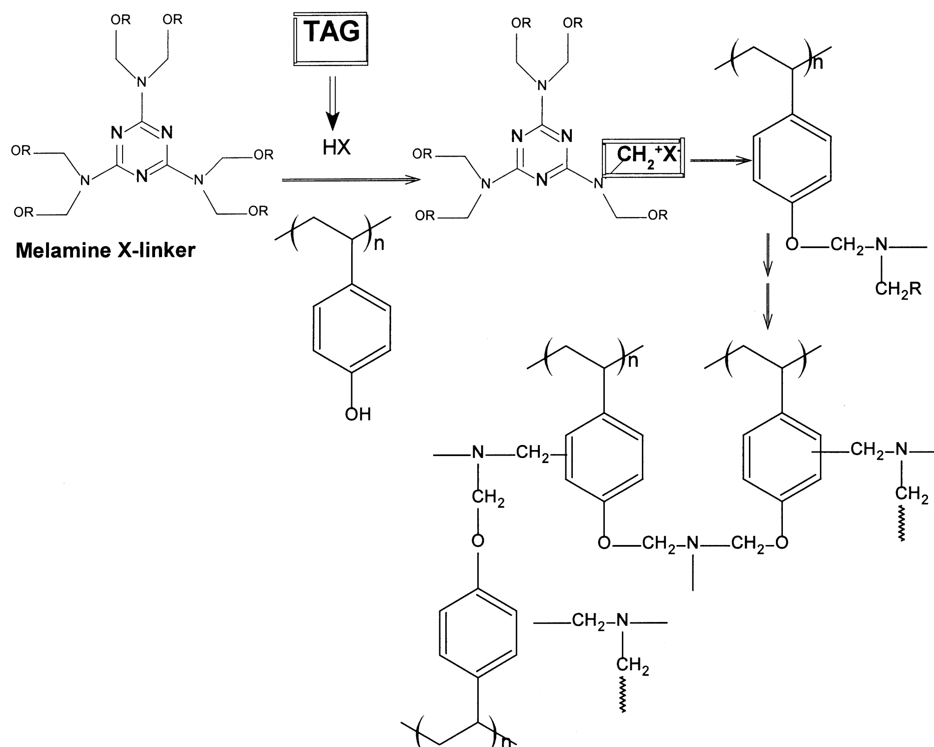

Scheme 1. Curing reaction of ARC.

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commences is ca. 30°C above the Tg of the film ($\sim$120°C) facilitating film flow during post exposure baking to improve planarization.

The dependence of crosslinking on curing temperature is shown in the derivative-TGA plot in Fig. 8. The extent of curing is monitored by first heating the formulated film for 90 seconds at temperatures varying from 100 to 225°C, and then measuring the weight loss over the temperature range of 50 to 400°C. The subsequent extent of weight loss should be reflective of the degree of curing during the initial isothermal heating cycle, decreasing with increasing cure temperature. If the isothermal curing temperature is insufficient to cause full curing, the loss of byproducts of curing reaction should be evident during subsequent heating in the TGA. As seen in Fig. 8, weight loss occurred at all temperatures below 200°C suggesting that full curing under these conditions requires temperature of at least 200°C.

The complete curing of the polymer at this temperature was also confirmed by its insolubility in organic solvents such as, THF, PGMEA and EL, which dissolved the uncured polymer. The results obtained from the derivative TGA were confirmed by isothermal weight loss at 200°C. As seen in Fig. 9, very little additional weight loss is observed after 90 seconds.

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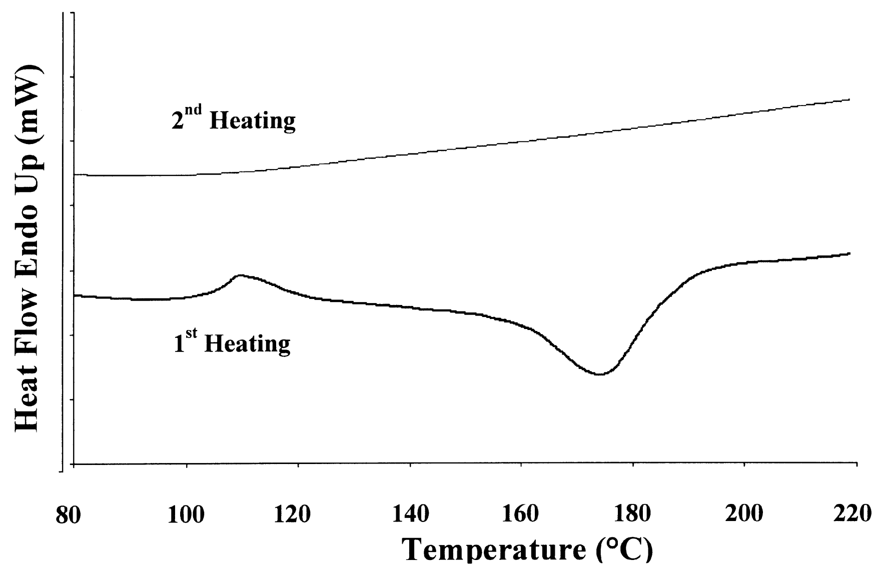

Figure 7. Curing efficiency of ARC (polymer/crosslinker/TAG = 93.5/4/2.5 wt%).

Having established an optimum cure time and temperature, for a fixed acid and crosslinker concentration, we next needed to determine the minimal crosslinker concentration required to fully cure the ARC film within the allotted 90 seconds cure time. The minimum crosslinker

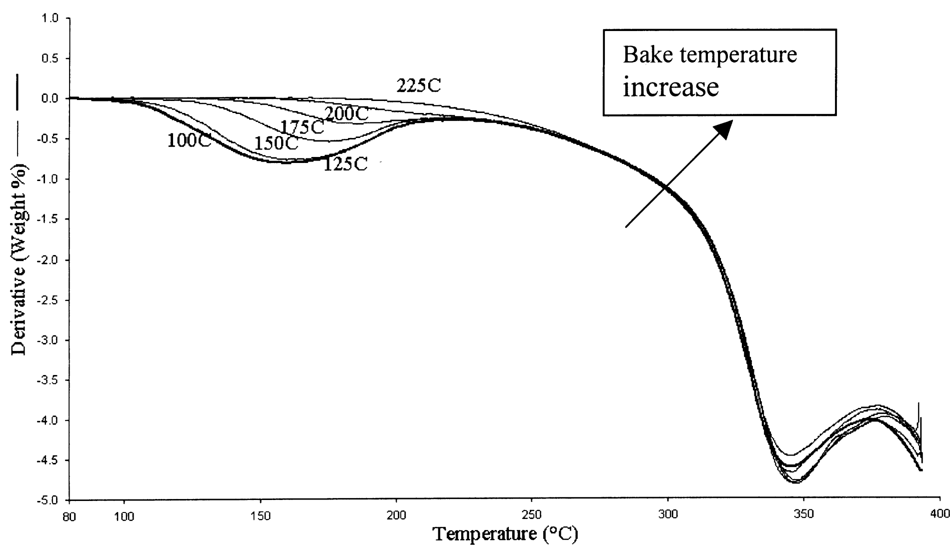

Figure 8. Curing efficiency vs. bake temperature (polymer/crosslinker/acid = 93.5/4/2.5 wt%).

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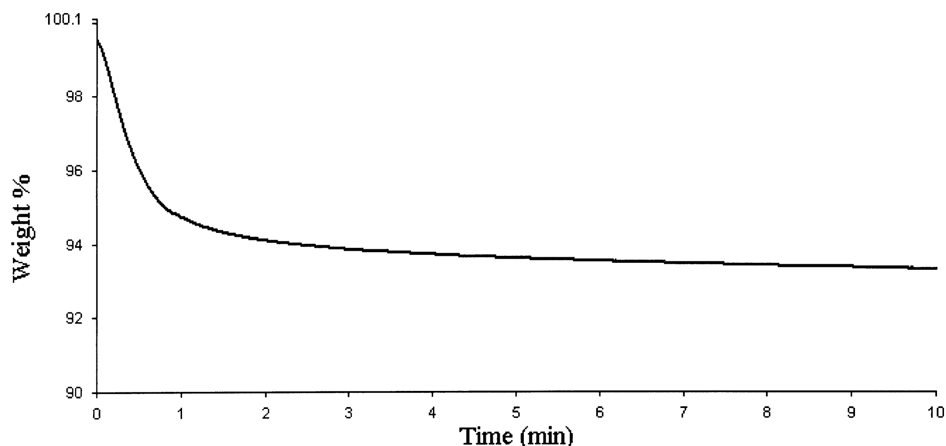

Figure 9. Effect of time on degree of curing (curing temperature = 200°C) (polymer/crosslinker/TAG = 93.5/4/2.5 wt%).

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concentration ensuring complete curing at 200°C was determined by measuring the film thickness loss for different concentration of melamine crosslinker. The change in film thickness of ARC films cured at 200°C following immersion in PGMEA for one minute is shown in Table 3 for four different melamine concentrations.

Considerable film thickness loss was observed at 2.5 weight percent loading of melamine crosslinker curing agent indicating insufficient crosslinking. However, no significant film thickness changes were observed at 5–7.5 weight percentage of the curing agent. Film thickness loss at 10 wt% curing agent may reflect leaching of excess crosslinker from the film. When repeated over a range of curing temperatures, (see Fig. 10), films containing 5–7.5 wt% again showed no further thickness loss up to cure temperatures of 230°C. Those containing 10% showed rather anomalous behavior passing through a minimum loss at about 210°C, but increasing thereafter.

The effect of concentration of curing agent on lithographic performance is evident in Fig. 11. Insufficient curing of ARC at low concentration of

Table 3. Film Thickness Loss as a Function of Melamine Crosslinker Concentration [Curing Temperature = 200°C; TAG Concentration = 2.5 wt%]

Melamine (Wt%)	2.5	5.0	7.5	10.0
% Film thickness loss in PGMEA	0.70	0.04	0.03	0.2

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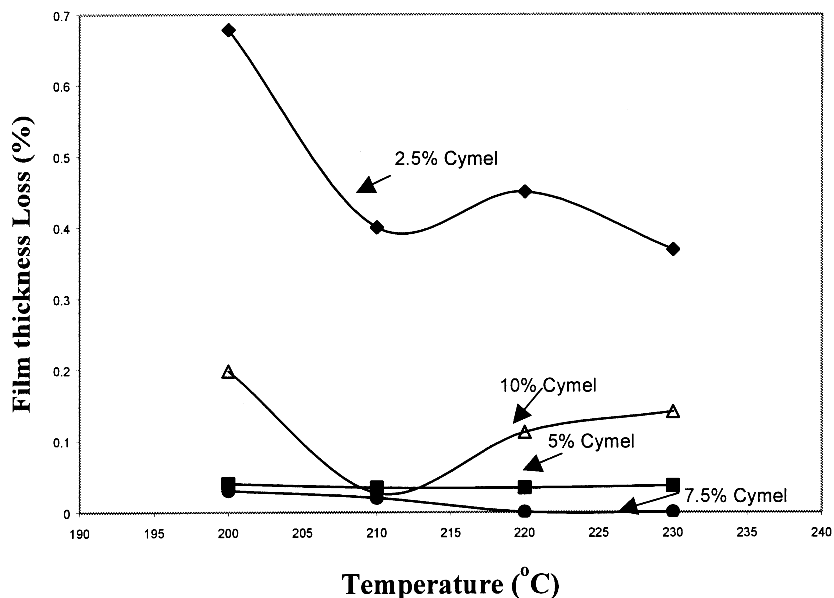

Figure 10. Film thickness loss as a function of bake temperature and curing agent concentration [TAG = 2.5 wt%].

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melamine crosslinker leads to collapsed line patterns, perhaps due to intermixing between ARC and imaging layer. Scum formation and footed profiles were observed at higher Melamine crosslinker concentrations of 10 wt%. As explained before, a footed profile at the interface of the two layers can be explained by the loss of acid due to its neutralization by the residual curing agent migrating at the interface. Similarly, the film thickness loss study at different temperatures in Fig. 11 shows that incomplete curing at lower temperature and excess crosslinker present at higher loading result high film thickness loss which was also reflected in lithographic performance.

Shelf Life Stability of ARC Formulations

The choice of curing agent and TAG could potentially affect the shelf life stability of the formulation. Premature decomposition of TAG could lead to undesirable curing. However, data in Fig. 12 suggest that the optimized formulation is stable when stored at 40°C for 30 days as evident by no change in the molecular weight of the polymer. Likewise, no change in the filterability and coating properties of the solution was observed upon storage, suggesting excellent shelf-life property at ambient storage condition.

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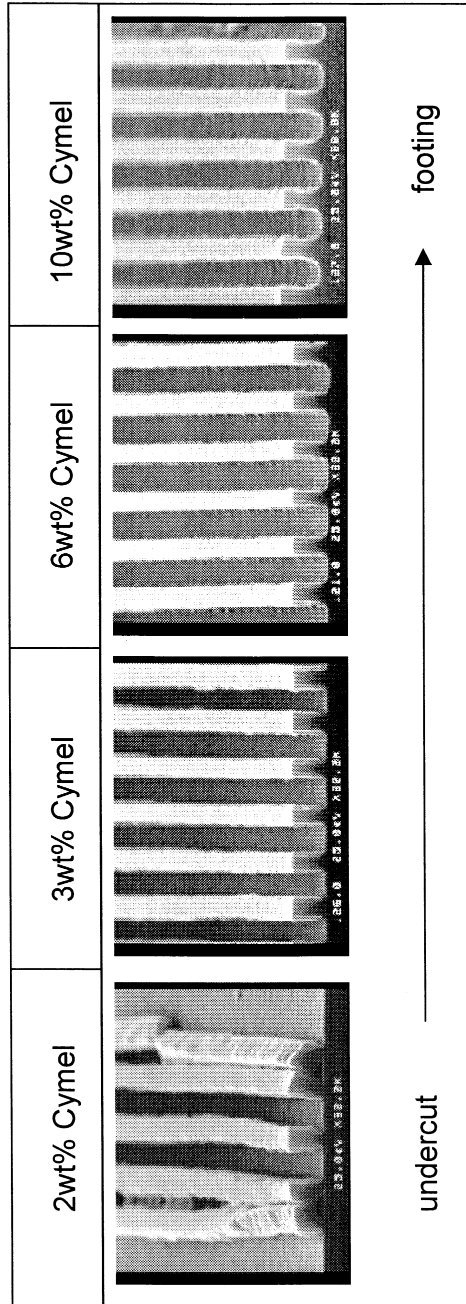

Figure 11. Profile change as a function of curing agent concentration.

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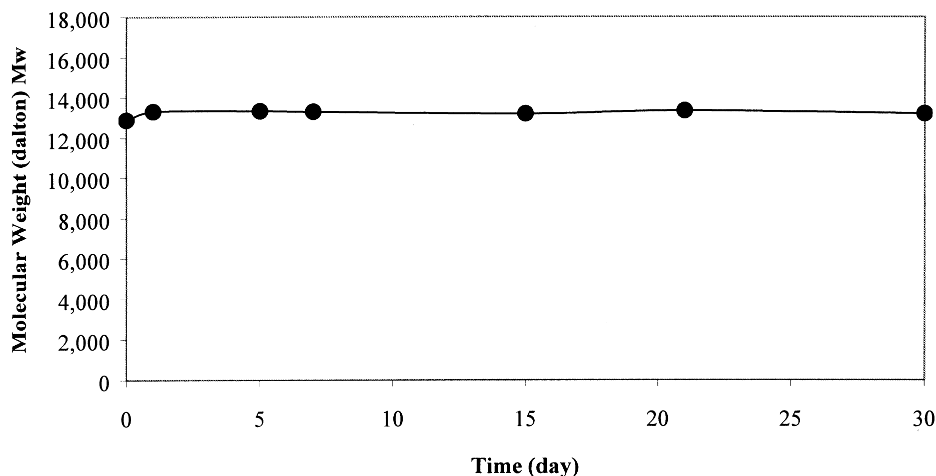

Figure 12. Variation in polymer molecular weight with storage time (storage $T = 40°C$).

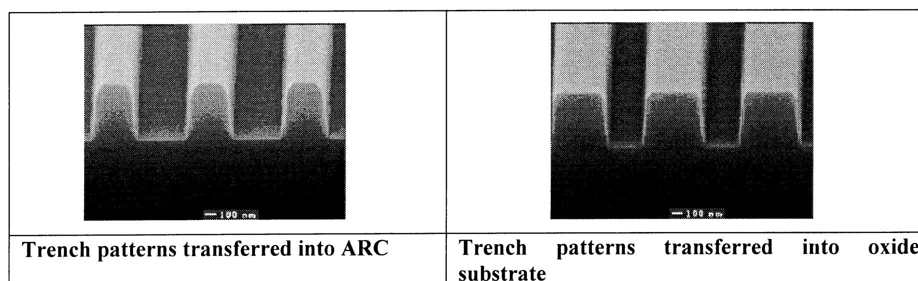

Figure 13. Cross-sectional SEM showing pattern transfer of 250-nm trenches.

Etch Selectivity

The etch resistance of the ARC material was determined by pattern transfer of 250 nm trench features into 600 nm of oxide substrate. The profiles of the transferred features are shown in Fig. 13. The good etch selectivity of the ARC is attested to by the fact that it lost only 40% of its original film thickness during the etching process. The good etching properties of this material are attributed to high aromatic content of the polymer.

CONCLUSION

An anti-reflective coating based on biphenyl methacrylate absorbing units can be designed with desired optical and thermal properties suitable for a bilayer process in microlithography. Proper considerations must be given to

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composition to minimize substrate reflectivity and to the curing conditions to ensure no intermixing at the resist/ARC interface.

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