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Polymers for Microlithographic Applications: New Directions and Challenges

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SUMMARY: Advances in the fabrication technologies associated with electronic devices have placed increasing demands on microlithography, the technology used to generate today's integrated circuits. Within the next few years, a new form of lithography will be required that routinely produces features of less than 0.1 μm. As the exposing wavelength of light decreases to facilitate higher resolution imaging, the opacity of traditional materials precludes their use; and major research efforts to develop alternate materials are underway. As a current example, lithography tools utilizing 193 nm light are now being introduced into the manufacturing environment. Through understanding of materials structure and its relationship to device process requirements and performance, a new class of cyclo-olefin based polymers was designed for these applications. In particular, alicyclic monomers such as norbornene are readily copolymerized with maleic anhydride and substituted acrylates to afford a wide range of alternative matrices that exhibit transparency at the exposing wavelength and aqueous base solubility. Materials properties must be carefully tailored to maximize lithographic performance with minimal sacrifice of other performance attributes. Further reduction in exposing wavelength to 157 nm introduces new challenges in polymer materials design. Efforts to address those challenges will be discussed.

Introduction

Advanced silicon based semiconductor circuits are complex three-dimensional structures of alternating, patterned layers of conductors, dielectrics and semiconductor films. These structures are fabricated on ultrahigh purity wafer substrates of the silicon semiconductor, and their performance is to a large degree, governed by the size of the individual elements. As a general rule, the smaller the elements, the higher the device performance will be. The structure is produced by a series of steps used to precisely pattern each layer. The patterns are formed by lithographic processes that consist of two steps: i) delineation of the patterns in a radiation sensitive thin-polymer film called the resist, and ii) transfer of that pattern using an appropriate etching technique.[1]

The rapid evolution of VLSI electronic device manufacturing technologies has placed increasing demands on microlithography, the technology used to generate today's integrated circuits. Over the past three decades, device dimensions have decreased by approximately 2

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orders of magnitude: from a range of 10 to 12 μm in the 1970's, to <0.15 μm today.[1-3] Within the next few years, a new form of lithography will be required that routinely produces features of less than 0.1 μm.[4] As the exposing wavelength of light decreases to facilitate higher resolution imaging, the opacity of traditional materials based on novolac/diazonaphthoquinone chemistry[5] precludes their use, necessitating investigations related to the design of new materials. For device applications, resist materials properties must be carefully tailored to maximize lithographic imaging performance with minimal sacrifice of other performance attributes, e. g., adhesion, solubility, RF plasma etching stability.[3,6]

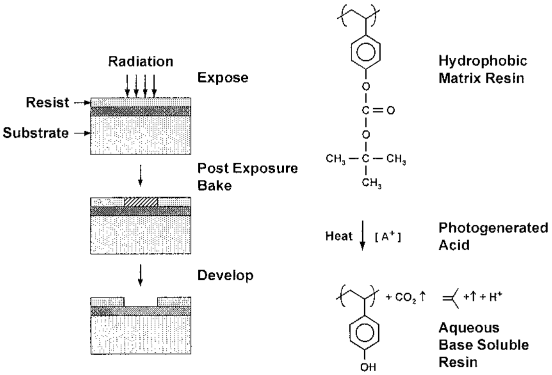


Figure 1. Acid-catalyzed deprotection of poly(tert-butoxycarbonyloxystyrene) affording the aqueous base soluble polyvinylphenol.

As noted above, the absorbance of conventional novolac/diazonaphthoquinone photoresists is too high to allow uniform imaging through a practical film thickness (0.5-1 μm) at exposing wavelengths less than about 300 nm. In addition, the available light at the exposure plane of commercial exposure tools using light sources below 300 nm is insufficient to provide adequate throughput when the quantum efficiency of a resist in less than 1.[2] The materials breakthrough that ultimately led to the adoption of 248 nm lithography as the technology of choice for advanced device fabrication was the announcement of what has been termed the "chemically amplified" resist mechanism. The pioneering work relating to the development of chemically amplified resists based on deprotection mechanisms was carried out by Ito, Willson and Frechet.[7] The initial studies dealt with the catalytic deprotection of poly(*tert*-butoxycarbonyloxystyrene) (TBS) in which the thermally stable, acid-labile *tert*-butoxycarbonyl group is used to mask the hydroxyl functionality of poly(vinylphenol). The mechanism of resist action is shown in Figure 1. Since these initial reports regarding chemically amplified resist mechanisms, significant research efforts have expanded on this revolutionary concept.[2,8]

193 nm Lithographic Materials

Clearly, materials structure plays the key role in defining performance. A partial list of key performance criteria coupled with relevant molecular characteristics that must be tuned to effect desired performance is presented in Table 1.[3,9]

Table 1. Key lithographic parameters vs. representative molecular characteristics.

Lithographic Parameter	**Molecular Characteristic**
Absorption	No olefinic or aromatic moiety
Etching Stability	*High levels of structural carbon, low oxygen content*
Aqueous base solubility	Base solubilizing groups such as OH, COOH, NH, etc.
Substrate adhesion	*Presence of polar moieties*
Sensitivity (photospeed)	Catalytic chain length for acidolysis, quantum yield for acid generation, acid strength, protective group chemistry
Post-exposure delay and substrate sensitivity	*Catalytic chain length for acidolysis, protective group chemistry, acid strength*
Outgassing	Protective group and photoacid generator chemistry
Aspect ratio of images	*Surface tension effects and mechanical strength of materials*
Low metal ion content	Synthesis and scale-up methodology
Manufacturability and cost	*Synthesis and materials scale-up methodology and lithographic process requirements*

As the drive to still smaller features to accommodate increased circuit densities continues, alternative lithographic exposure strategies are evolving. Advanced silicon device pilot lines today are using 193 nm exposure sources to produce 0.18-0.13 μm design rule devices.[3,10] The first experiments demonstrating the feasibility of UV light as an imaging source for lithography occurred at Bell Laboratories in 1975.[11] Bowden and Chandross demonstrated the concept using poly(butene-1-sulfone) which upon exposure to 185 nm light, exhibited a sensitivity of 5 mJ/cm^2. The drive towards increased integration, fueled by a desire to maintain the availability of optical lithography for device production, led to research and development efforts aimed at developing a production worthy 193 nm lithographic technology.

The intense absorption of aromatic molecules at 193 nm severely limits the use of conventional matrix resins such as novolacs and polyvinylphenols for 193 nm lithography and has necessitated yet another paradigm shift in materials design.[3,6] Processes under consideration for use with the 193 nm technology include the traditional solution developed methodologies in addition to dry-developed techniques, which span the range of bilevel approaches using thin silicon containing resists as the imaging layer, to silylation processes, to the more recently described all-dry, plasma deposit/plasma develop systems. Because of the large acquired knowledge base regarding solution developed resists, the preponderance of work related to 193 nm materials involves the design of new, single-layer chemistries that provide for aqueous base solubility, etching resistance, resolution, photospeed and process latitude. The fundamental design challenge that emerged was the necessary trade-off between plasma-etching resistance and requisite materials properties for lithographic performance.

Avenues that can lead to a transparent, etching resistant polymer include the incorporation of alicyclic and/or silicon bearing moieties.[12] Leading single layer resist candidates utilize the former approach. Early efforts focused on derivatized acrylate and methacrylate copolymers.[10,13-16] Approaches include both careful tailoring of polymer properties to maximize lithographic performance with minimal sacrifice in etching resistance, and development of three-component systems in which high carbon content alicyclic additives serve not only as dissolution inhibitors but also enhance the etching resistance of the matrix as a whole.[10b, 13, 17, 18]

While methacrylate-based resist platforms are attractive from an economic perspective, they possess a linear, oxygen rich scaffold whose poor plasma-etching stability can be offset only partially by functionalization with more stable pendant groups. Greater intrinsic plasma-etching stability has been demonstrated through incorporation of alicyclic moieties directly into the polymer backbone and by minimizing oxygen content. In such materials, oxygenated functionalities are incorporated into the design to serve in roles related to imaging, solubilization and adhesion rather than an incidental structural function.

In pursuing alternate 193 nm single-layer resist platforms, we developed a new class of matrix resins based on cycloolefin-maleic anhydride alternating copolymers. These polymers incorporate alicyclic structures directly in the polymer backbone,[19] and while some oxygenated functionalities are retained in structural roles, oxygen content is decreased relative to methacrylates. Compelling features of these copolymers include: i) facile synthesis via standard metal-ion free radical polymerization techniques, ii) a potentially large pool of cycloolefin feedstocks, iii) a generic structural motif that incorporates alicyclic structures directly into the polymer backbone yet provides a polar functionality that can promote adhesion between the resist and device substrate, and iv) allows incorporation of additional monomer units that can be chosen to address specific functional concerns.

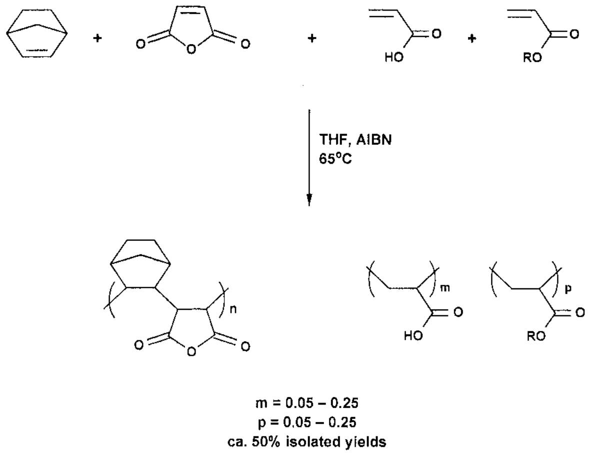


Figure 2. Schematic representation of the free radical initial polymerization of norbornene and maleic anhydride to afford the alicyclic based alternating copolymer.

A large number of cycloolefins are known to copolymerize with maleic anhydride (Figure 2). Alternating copolymerization of norbornene (NB) and maleic anhydride (MA) occurs readily at 65 °C in a variety of solvents including THF, dioxane, acetone, and cyclohexanone using 2,2'-azobisisobutyronitrile (AIBN) as an initiator.[19a] P(NB/MA) is thermally stable, has a T_g> 300°C, has excellent transparency at 248 and 193 nm, is soluble in several classes of solvents that are typically employed for casting films, and is hydrolytically robust. These characteristics provide a foundation that allows further structural elaboration to effect the desired lithographic performance. For example, terpolymerization with acrylic acid provides a controllable method of synthesizing aqueous base soluble resins: systematic variation of acrylic acid feed ratios from 5-20% yielded progressively more base-developable formulations. For acrylate loadings of up to about 20%, no significant deviation from a 1:1 ratio of the norbornene to maleic anhydride "repeat unit" was observed and the relationship between the feed of acrylate monomers and their incorporation into the final polymer appears linear. Compositions resulting from 15 and 17.5% acrylic acid feeds display the most useful aqueous base development behavior and were studied in some detail.[19,20] The acrylate terpolymers possess thermal and optical properties and organic solubilities indistinguishable from P(NB/MA). At 248 and 193 nm, the absorbance per micron of poly(norbornene-alt-maleic anhydride-coacrylic acid) where the acrylate unit constitutes 15 % was 0.05 and 0.27 respectively. Adhesion to silicon device substrates is improved upon incorporation of the acrylate unit.

In addition to its base solubilizing attributes, acrylic acid provides a template to further functionalize the polymer. Notably, t-butyl acrylate is readily incorporated into the polymer chain via free radical copolymerization.[19a] In the presence of acid and mild heating, the ester is cleaved to liberate isobutylene and the parent acid. This mechanism can be used in the design of sensitive, high-resolution, chemically-amplified resist formulations. In one example, a quaternary polymer of NB, MA, acrylic acid and t-butyl acrylate is formulated with a substituted cholate ester that acts as a dissolution inhibitor, and an onium salt-based photoacid generator. Structures of each of the resist components are shown in Figure 3. Exposure to 193 nm light generates an acid which then reacts with the ester appendages on both the polymer and inhibitor. Development of the resultant latent image in aqueous base affords high resolution patterns. Figure 4 depicts SEM micrographs of typical patterns obtained with these materials upon 193 nm exposure. Additionally, images as small as 60 nm have been demonstrated using advanced phase-shift mask techniques (Figure 5).

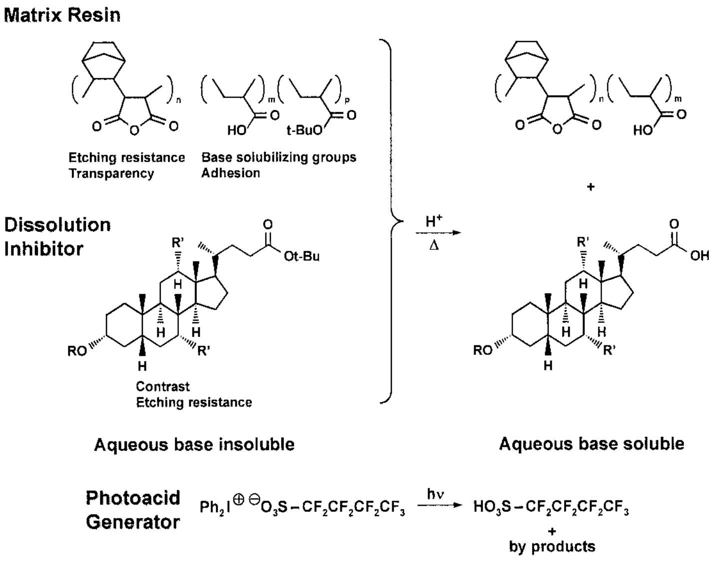
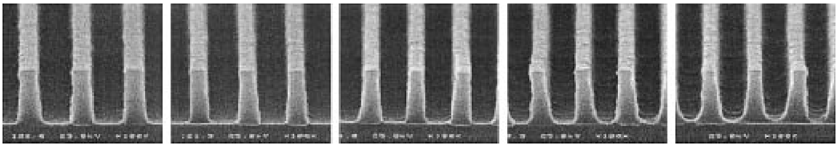


Figure 3. Structural representation of the polymer, dissolution inhibitor and photoacid generator components of a 193 nm resist formulation.



0.14 µm **0.13 µm** **0.12 µm** **0.115 µm** **0.1125 µm**

Figure 4. Scanning electron micrographs of nominal equal line/space features printed in a norbornene-maleic anhydride based photoresist using annular illumination.

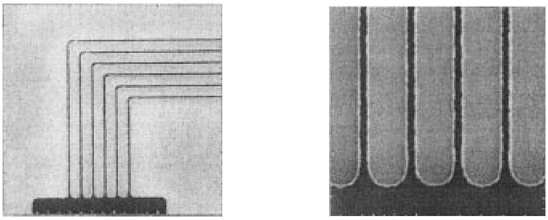


Figure 5. Scanning electron micrographs depicting 60 nm images printed in a 0.36 µm norbornene-maleic anhydride photoresist at 193 nm and a phase shift mask.

The inherent simplicity of the norbornene-maleic anhydride approach has led to the investigation of substituted norbornenes that incorporate the base solubilizing and imaging characteristics of the acrylate components present in the system described above into a single alicyclic monomer.[21] Advantages include the obvious decrease in synthetic complexity that arises from the four component matrix and improved plasma etching resistance that may obtain from elimination of the acrylate units. Representative examples of systems that have been investigated are shown in Figure 6.

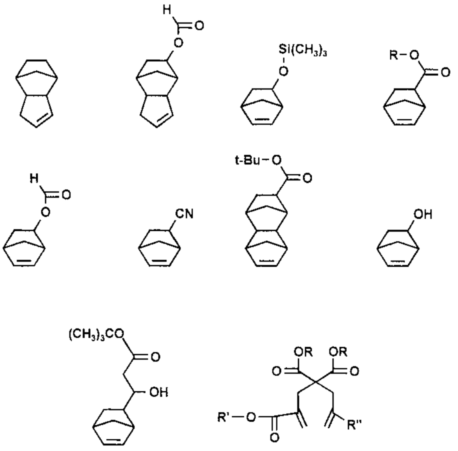


Figure 6. Examples of substituted norbornene analogs that have been incorporated into alicyclic/maleic anhydride alternating 193nm matrix resins.

157 nm Resist Design

As device design rules continue to shrink, research directions transition towards future lithographic alternatives. The next logical extension of optical lithography involves the continued progression to still shorter wavelengths. As optical lithography has evolved over the past few decades, first from 248 nm excimer laser (KrF) based tools, followed by 193 nm (ArF) based systems, the next frontier utilizes fluorine, 157 nm UV sources.[22] Where optical transparency and etching resistance were the key concerns for the development of 193 nm lithographic materials, the over-riding issue for 157 nm advanced optical lithography is resist materials transparency. Traditional resist materials platforms are too opaque to allow imaging in sufficiently thick films to address defect density concerns. For an optical density of 0.4, which is considered optimum for most resist applications, acrylic, phenolic and cycloolefin polymer platforms would require resist thicknesses less than 100nm (Table 2).[23] This value needs to be compared to the anticipated required film thickness for 50 to 100 nm imaging of 200 to ~400 nm, respectively.[23] Thus, avenues leading to decreased absorbance need to be identified and explored for this application.

Table 2. Resist thickness required to achieve an optical density of 0.4 for three classes of resist matrix polymers.[23]

Materials Platform	**Thickness to Achieve OD = 0.40**
Acrylic	46-87 nm
Phenolic	48 nm
Cycloolefin	77 nm

A list of representative polymeric alternatives along with their 157 nm absorbance characteristics was reported by Kunz, et al and is presented in Table 3.[23] While hydrocarbon platforms do not provide sufficient transparency, fluorinated analogs do show potential, as do siloxane based materials. Etching resistance and adhesion of the fluorinated materials to silicon substrates are concerns that will need to be addressed. Interestingly, the Kunz study demonstrated that the absorbance of many of the standard photoacid generator materials that have been used for both 248 and 193 nm chemically amplified resists have either similar or even lower absorbance at 157 nm than at the longer wavelengths,[23a] thus alleviating concerns regarding the PAG component. An aspect of materials design that will be increasingly important at 157 nm is that of resist outgassing, or rather, the level of volatile species evolving from the resist film during exposure. These may include residual solvent, volatile resist components, or by-products generated upon irradiation of the resist film. Clearly, the evolution of volatile species must be kept to a minimum at 157 nm so that these products do not deposit onto critical lens surfaces deleteriously affecting the lens transmission characteristics.

Table 3. Survey of 157 nm absorbance characteristics of selected polymeric platforms.

Polymer	**Absorbance** (μm^{-1})	**Film Thickness** (in nm for an OD = 0.4)
Si-O Backbone		
Poly(hydrosilsequioxane)	0.06	6667
Poly(dimethylsiloxane)	*1.61*	*248*
Poly(phenylsiloxane)	*2.68*	*149*
Carbon Backbone		
Fluorocarbon, 100% fluorinated	*0.70*	*571*
Hydrofluorocarbon, 30% fluorinated	*1.34*	*298*
Partially esterified hydrofluorocarbon, 28% fluorinated	*2.60*	*154*
Poly(vinyl alcohol)	4.16	96
Ethyl cellulose	5.03	80
Poly(methyl methacrylate)	5.69	70
Poly(norbornene)	6.10	66

Several groups are beginning to explore materials alternatives for 157 nm applications. Willson et al. [24], have utilized a "modular approach" in which chemical approaches to instilling necessary functionality into the 157 nm material is first tested in a model system. In this manner, they have identified the hexafluoroisopropyl group as an effective aqueous base solubilizing moiety and shown that it can be protected with acid labile alkyl acetal protecting groups while maintaining 157 nm transparency. Building from the 193 nm materials research that demonstrated the effectiveness of alicyclic backbone polymers for providing etching resistance, Willson also showed that substitution of norbornene with an electron withdrawing group such as fluorine or even a carbonyl group may afford resins with sufficient 157 nm transparency. For instance, the partially fluorinated poly(norbornene) shown in Figure 7a has an absorbance of only 1.7 AU/micron. Although this absorbance is still too high for practical applications, it demonstrates substantial improvement over non-fluorinated analogs which can have absorbances as high as 7 AU/micron and represents a promising starting point for the design of new materials.

Ober, et al have reported two design approaches to achieving 157 nm transparency.[25] One system is based upon a poly(trifluoromethylvinyl alcohol-co-vinyl alcohol) resin protected with acid labile THP protecting groups, while in another approach they investigated the introduction of hexafluoropropyl groups onto cyclized polyisoprene (Figure 7b). The material described to date does not have good transparency at 157 nm, but it does exhibit a high T_g (120-170°C) and good etching resistance. It is anticipated that hydrogenation of the olefinic moiety will address the absorbance issue. The Dupont research team[26] has shown some impressive initial results on at least one material that is developable in aqueous base, has good transparency at 157 nm (2.9 AU/micron) and good thermal characteristics (Tg, 171°C), and may have good etching resistance. Preliminary investigations of imaging performance also show promise: 0.35 micron features have been obtained upon 157 nm exposure. Unfortunately, few structural details are known beyond the general poly(norbornene-co-tetrafluoroethylene) motif (Figure 7c).

Conclusion

Materials structure plays the key role in defining lithographic materials performance. As shown in Table 1 each lithographic parameter that can be used to define materials performance has as its counterpart a definable molecular characteristic. It has been demonstrated that alicyclic units can be effectively incorporated into polymer architectures to afford manufacturable radiation sensitive resist compositions for 193 nm resist applications. Fundamental studies into the materials properties and interactions between resist components and materials radiation chemistry enables formulation of high-performance resists that display extremely reproducible lithographic properties. This requires careful manipulation of a large number of physical properties that govern solubility, sensitivity, image fidelity, etc. As future technologies continue to evolve, polymer materials will continue to play a critical role. With the advent of 157 nm options, it is fundamentally materials chemistry that will determine its future. Within the scope of new lithographic materials chemistry design, it is imperative to remember that the principles governing different properties may not always work in concert, and that in the end, a functional resist design will reflect multiple compromises and tradeoffs.

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