POLYMERS FOR ADVANCED TECHNOLOGIES
Polym. Adv. Technol. 2006; **17**: 94–103
Published online 17 January 2006 in Wiley InterScience (www.interscience.wiley.com). DOI: 10.1002/pat.662

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Review

Recent progress in high resolution lithography†

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The nanotechnology revolution of the past decade owes much to the science of lithography, an umbrella term which encompasses everything from conventional photolithography to ''unconventional'' soft lithography and the self-assembly of block polymers. In this review, some of the recent advances in lithography are summarized with special reference to the microelectronics industry. The next generation photolithography, two-photon lithography, step-and-flash imprint lithography and nanofabrication using block copolymers are covered, in an attempt to describe more recent work in this vibrant and active field of research. Copyright © 2006 John Wiley & Sons, Ltd.

KEYWORDS: lithography; extreme ultraviolet (EUV); molecular glass; block copolymers; nanotechnology

INTRODUCTION

Lithography and the processes associated with it are the backbone of the nanotechnology revolution. Several developments are occurring simultaneously: a drive to reduce minimum feature size leading to advances in microelectronics, the use of lithographically patterned structures to prepare devices for photonics, biotechnology and other forms of nanotechnology and finally the drive to create three-dimensional (3D) structures for the creation of new materials and devices. Thus the controlled formation of nanometer scale structures in two and three dimensions is of increasing interest in many applications. In this review, recent advances in high resolution lithography with special reference to the microelectronics industry are covered, concentrating on currant ''hot'' topics, including 193 nm immersion lithography, extreme ultraviolet (EUV) lithography, molecular glass photoresists, three-dimensional (3D) two photon lithography and nanofabrication using block copolymers. Current advances in soft lithography are not reviewed due to the recent publication of several reviews in this area.[1,2] However, step-and-flash imprint lithography is included as this technique is the method of ''unconventional'' lithography with prospects of adoption by the industry.

193 NM IMMERSION LITHOGRAPHY

The potential advantages of immersion lithography have been known for more than 10 years,[3] and since 2002, 193 nm immersion lithography has taken over as the front-runner technology for the 45 nm node on the International Technology Roadmap for Semiconductors (ITRS), displacing 157 nm technology.

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†Paper presented as part of a special issue on lithography.

Compared to all dry lithography, the key benefit is the possibility of constructing projection optics with numerical apertures (NAs) > 1 by introducing an immersion fluid with a refractive index > 1. A current proposed immersion medium is water which has low cost, low toxicity, and a refractive index of 1.44. It has been demonstrated that 90 nm patterns can be achieved by using a chemical amplified resist.[4]

As promising as immersion lithography is, there are several key problems which must be addressed before the technology gains widespread acceptance, most of which relate to the use of an immersion fluid. Issues include formation of micro- or nano-size bubbles which scatter the incoming radiation and affect the imaging process, contamination of the immersion media by the resist, damage to optical components from contaminated immersion media, as well as fluid heating changing the refractive index of the media during exposure.[4,5]

The development of resists materials for immersion lithography will have to deal with the obvious issues related to the exposure of the imaging stack to aqueous or other immersion media. Issues associated with the exposure of the imaging stack to an immersion medium can be generally classified in the areas of penetration of the immersion media into the imaging stack, effects of immersion media uptake on photoresist chemistry, and the leaching of photoresist components into the immersion media. One possible solution to these issues is the use of top barrier coatings.[6] These coatings isolate the resist layer from the immersion medium, and thus can make it possible to use existing resist formulations for dry lithography with minimal formulation optimization. Other challenges for the extension of 193 nm immersion lithography include pattern collapse at small feature size, as well as issues caused by ever rising requirements along the roadmap, e.g. specifications for line width roughness, post-exposure bake (PEB) sensitivity, etc. Although it is believed that the overall challenges facing 193 nm immersion technology are not insurmountable, there

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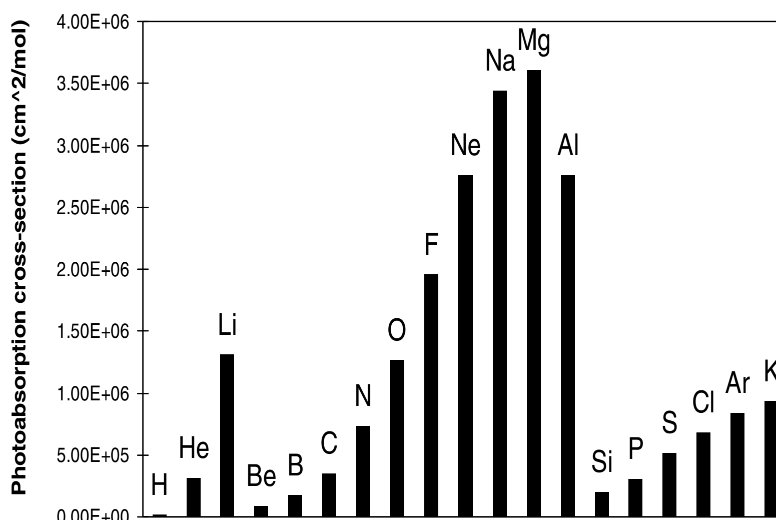

Figure 1. Photoabsorption cross-section of different atoms at 13.4 nm.

are still critical needs that must be addressed at this stage during its development.

EXTREME ULTRAVIOLET (EUV) LITHOGRAPHY

EUV lithography, in which high-energy photons with a wavelength of 13.4 nm (100 eV) are used to pattern resists, is one of the leading candidates for next generation lithography. Although termed EUV, this wavelength radiation actually falls in the range of soft X-rays. This technique is expected to produce feature sizes in the sub-50 nm regime due to the extremely short wavelength of the EUV radiation employed.[7–19] The primary events which occur upon absorption of these high-energy photons occur within the inner shells of atoms.[20] As a result, EUV absorption depends not on molecular composition but can be calculated based only on atomic composition. In the X-ray region, the absorption cross-section of resist materials is a function of wavelength and decreases substantially as wavelength decreases. Figure 1 shows the photoabsorption cross-sections of different atoms at 13.4 nm. Contrary to X-ray lithography (0.8–1.4 nm) in which most materials do not absorb, at 13.4 nm absorption is a very important issue, especially as currant EUV sources are rather weak and the imaging potential of every spare photon must be maximized.[21–25] To further complicate resist design, oxygen and fluorine are among the highest absorbing elements, which preclude many current designs for high resolution chemically amplified resists.

The critical features of a ''perfect'' EUV resist include high sensitivity, low absorption, high etch resistance (especially important if thinner films are used) and the ability to form high resolution images.[10] In particular, next generation resists for EUV must be capable of producing images with low line-edge roughness (LER).[7,9–12,26–34] LER is defined as 3σ of deviation from the predicted line edge, and since this parameter does not scale with line width, the effect of LER becomes more profound at smaller feature sizes. The target for LER is 1.5 nm for the 45 nm technology node.[10] The factors which affect LER are varied, and relate to chemical composition of the resist itself as well as the processing conditions. Both single and multi-layer resist systems have been studied for EUV. To date, no currently available resist formulation has proved capable of meeting these requirements.

Currently, most single layer positive tone EUV resists are based on poly(hydroxystyrene) (PHS), which has relatively low absorption compared with other chemically amplified resist platforms. Matsuzawa and coworker's have calculated the linear absorption coefficients of various polymers at 13.4 nm,[24] and found that aromatic substitution lowers the absorption coefficient by effectively diluting oxygen concentration. Additionally, polymers containing higher aromaticity exhibit higher etch resistance. Incorporation of these high etch resistant low absorbing aromatic units makes the application based on single layer resist possible for EUV lithography. Recently, Intel has initiated a joint program with photoresist suppliers to screen current commercial resists for use as a single layer EUV resist.[9,10]

In an alternative approach, fundamental research has also been carried out to design entirely new photoresists for EUV lithography. An oxygen-free negative tone resist, poly(trimethylsilylstyrene-co-chloromethylstyrene) was studied as an EUV resist.[35] Due to the absence of highly absorbing oxygen atoms, this polymer has excellent transparency with an absorption coefficient of around 1 μm^{-1}. However, pattern distortion caused by swelling limit the resolution attainable with this system.

Silicon and boron-containing functionalities can be added to resists to increase EUV transparency.[36–38] Norbornene-based structures have also been studied.[37] Excellent resolution, high sensitivity and good etch resistance have been demonstrated for PHS type of resists, although the absorption coefficients ($\sim 1.5\,\mu m^{-1}$) of these types of materials are higher than the aforementioned negative tone systems due to the presence of oxygen atoms.

Wood and coworkers reported both experimental and theoretical studies of pattern sidewall angles of poly(methyl methacrylate) (PMMA) and a novolac-based chemically amplified resist produced using EUV lithography.[25,39] It was found that high absorption resulted in a significant degradation in pattern profile at wavelengths of 37.5 and 13.9 nm.

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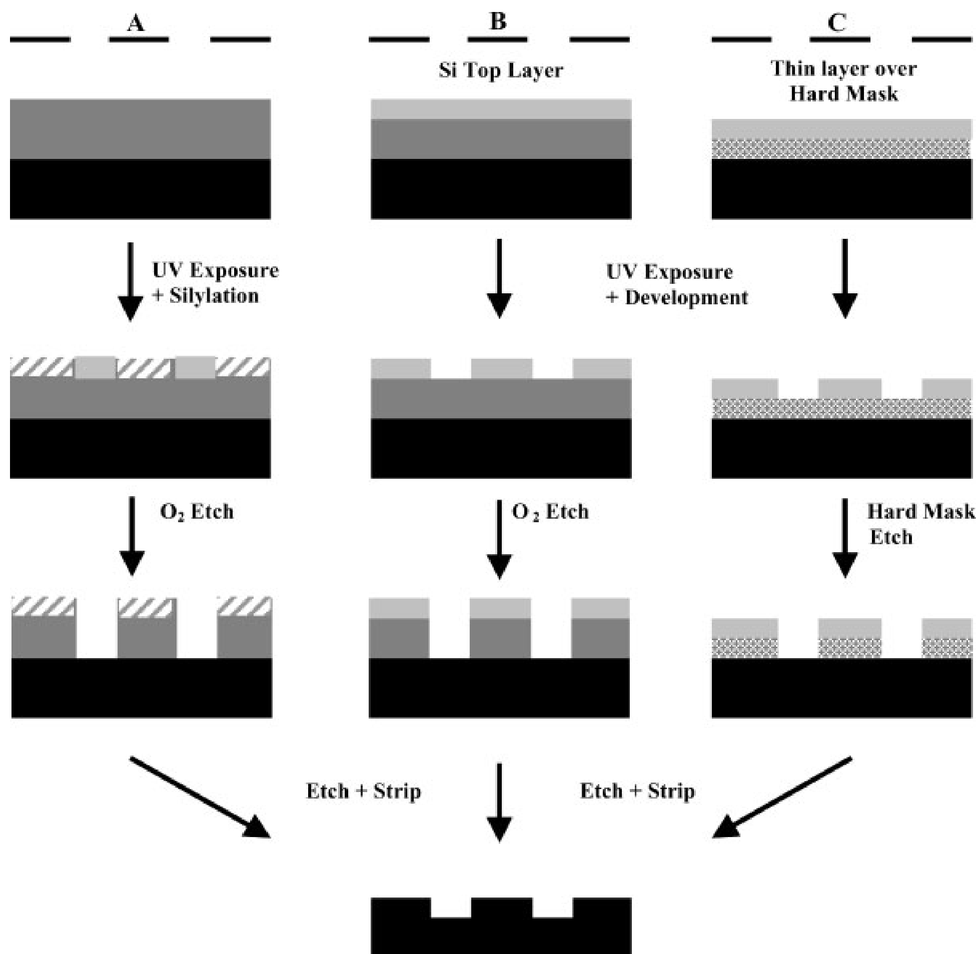

Figure 2. Imaging processes for EUV lithography. (A) Top surface imaging (TSI), (B) bilayer resists, (C) ultra thin layer resist over hardmask (UTR).

To address absorption issues, multi-layer resists which employ thin layer imaging (TLI) have been proposed.[27,32,34,40–46] Three main TLI approaches have been investigated for EUV lithography, namely top surface imaging (TSI)[28,31,32,34,40,44,47–49] bilayer resists[36,50] and ultra thin single layer resists over hardmask (UTR)[32,40–43] (Fig. 2).

In TSI, the uppermost layer of either the exposed or unexposed regions can be selectively silylated by gaseous or liquid silanes. In the bilayer process, a silicon-containing resist is used as the top-imaging layer. In both cases, the top silicon-containing layers serve as masks to transfer the patterns to an underlying organic planarizing layer under oxygen plasma etching. UTR involves the use of a thin resist layer on top of an etch resistant hardmask which provides sufficient etch resistance for the device layer patterning process. Besides the complexity introduced by the TLI techniques, problems such as swelling, low glass transition temperature (T_g) due to silylation, inability to form images with low LWR limit their applications in EUV lithography. Clearly, alternative resists are required in order for EUV lithography to fulfill its full potential.

MOLECULAR GLASS PHOTORESISTS

Molecular glass (MG) photoresists are a relatively recent advance for next generation lithography. In contrast to conventional resist materials which are usually polymeric and polydisperse, MG resists are small, monodisperse organic molecules that form stable amorphous glasses at room temperature.[51] The natural tendency of small molecules to crystallize can be avoided by incorporation of bulky groups, non-planer structures or a variety of conformers into the molecular design of new materials. If correctly designed, MGs are in a state of thermodynamic non-equilibrium (much like amorphous polymers) and hence show T_g values rather than crystalline melting points. MGs have been under investigation for some time for applications such as molecular electronics,[51] but chemically amplifiable groups and etch resistant moieties can readily be incorporated into MGs to enhance their utility as photoresists. In particular, the small size of these materials compared to amorphous polymeric resists is thought to lead to high resolution images with lower LER.[52]

Shirota and coworkers have reported the synthesis and lithographic characterization of a variety of MG photoresists for E-beam lithography, most of which are based around a tri-substituted aromatic core.[52–55] Some examples are shown in Fig. 3.

TsOTPB, a positive tone resist, and ASITPA, a negative tone resist, allowed the formation of 150 and 70 nm lines respectively.[53] The reported sensitivity of both was rather low, approximately 3 mC cm^{-2} at 50 KeV. The VCTPB class of

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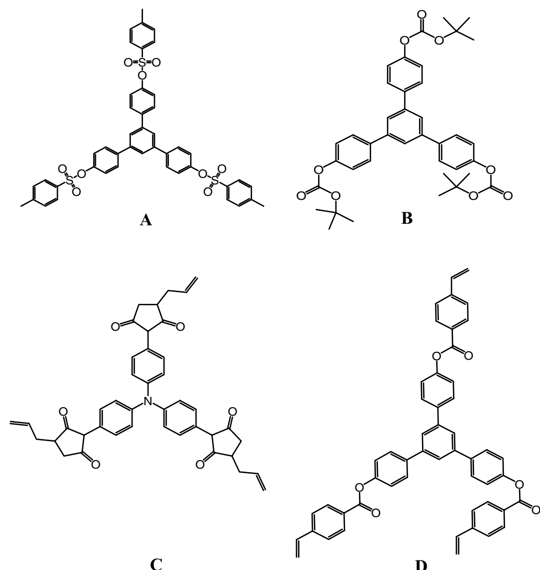


Figure 3. Examples of molecular glass resists for E-beam lithography. (A) TsOTPB,[53] positive tone chain scission resist, (B) BCMTPB, positive tone chemically amplifiable resist,[55] (C) ASITPA[53] and (D), *p*-VCTPB (*m*- and *o*-substituted analogs also reported) negative tone resists.[52]

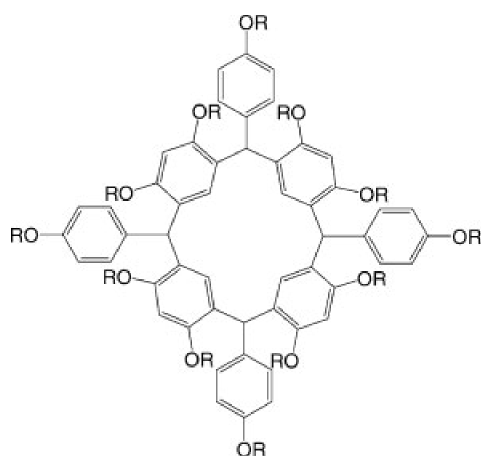
compounds which functioned as negative tone resists proved more sensitive (50 μC cm^{-1} at 50 KeV) giving comparable feature sizes.[52] Predictably, chemically amplifiable MG photoresists such as BCMTPB and analogs proved considerably more sensitive when exposed in the presence of a photoacid generator,[54,55] with sensitivities <5 μC cm^{-1}. After development, 40 nm feature sizes were obtained.

Calixarenes are a class of molecules with naturally irregular structures analogous to Novaloc resin, and have proved especially versatile cores for MG photoresists. Matsui and coworkers reported that *p*-alkylphenol calix[6]arenes function as negative tone resists for E-beam lithography, albeit with low sensitivity, requiring a dosage of 800 μC cm^{-2} to form an image.[56] Calix[4]resorcinarenes were employed as a negative tone resist for 365 nm lithography using a polyfunctional benzylic alcohol as a crosslinker in an acid catalyzed process.[57] *p*-Chloromethyl substituted calix[4]arenes also functioned as negative tone E-beam resists, in a similar mechanism to poly(chloromethyl styrene).[58] Calix[8]arenes mono-substituted with either acetic anhydride or toluene-*p*-sulphonyl chloride were blended with diazonapthoquinone (DNQ) dissolution inhibiters to produce a positive tone image using 248 nm radiation.[59] Ober and coworkers have focused on the design of chemically amplifiable MG photoresists for EUV and E-beam lithography.[60,61] Recently, the synthesis and lithographic characterization of chemically amplifiable calix[4]resorcinarene derivatives as positive tone resist for E-beam lithography was reported.[62] Images with feature sizes as low as 50 nm with low LER were obtained after exposure and development with an aqueous base (Fig. 4). It was also reported that small, commercially available crystalline molecules, could be mixed with an appropriate crosslinker such as Powderlink™ and spin-coated to form an amorphous film. An acid catalyzed crosslinking reaction resulted in negative tone images with feature sizes approximately 50 nm.[63]

Several MG photoresists designed with saturated aliphatic functionalities (rather than aromatic groups) designed for 193 nm lithography have been reported. Sooriyakumaran and coworkers described the synthesis and lithographic characterization of a resist based on polyhedral oligomeric silesquioxane (POSS).[64,65] POSS is best described as a cage of silicon and oxygen, and the reported molecule was functionalized with chemically amplifiable bulky side groups to raise the naturally low melting point of POSS. Using this resist, the authors reported the production of 120 nm feature sizes using 193 nm lithography with LER comparable to commercially available resists. This resist is also expected to show excellent dry etch resistance thanks to the silicon in the backbone. In addition, an adamantyl core functionalized with cholic acid was reported to have a T_g value of 90°C, and enabled the formation of 70 nm patterns.[66]

TWO-PHOTON 3D LITHOGRAPHY

Conventional optical lithography is often used for microfabrication. However, single-photon absorption as used in



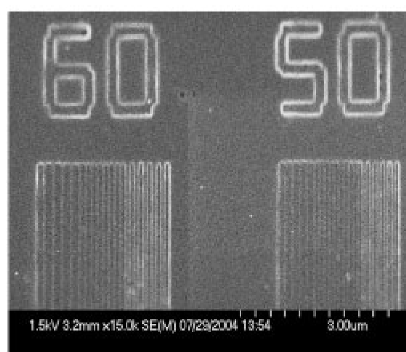


Figure 4. SEM image of 50 nm feature sizes obtained with *t*-Boc protected *C*-4-hydroxyphenyl-calix[4]resorcinarene using E-beam lithography.[63]

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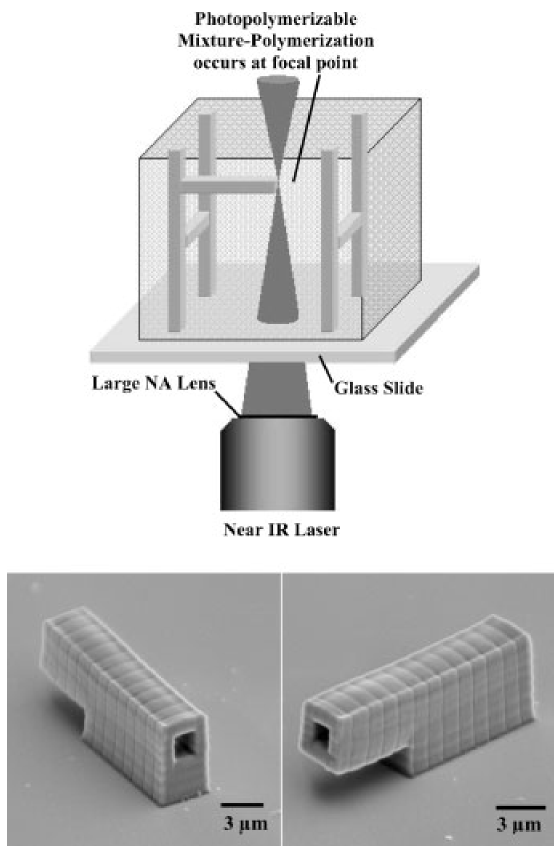

Figure 5. Two photon 3D lithography: schematic and example of 3D microfluidic channel fabricated using two photon lithography.

standard photolithographic techniques is limited to be a 2D process. The 3D structures required for more complex devices are currently built up by consecutive lithographic steps. Technological advances in areas such as micro- and nano-electromechanical systems[67] (MEMS, NEMS), microfluidic devices,[68] photonic crystals,[69] 3D optical data storage[70,71] require the development of fully 3D structuring capabilities at the nanometer scale.

Two-photon lithography is an intrinsic 3D lithography which has the highest potential for constructing arbitrarily shaped 3D devices in a one-step process. By tightly focusing a femtosecond near-IR laser beam into the resin, subsequent photo-induced reactions such as polymerization occur only in the vicinity of the focal point, allowing the fabrication of a 3D structure by directly writing 3D patterns by focus or sample scanning (Fig. 5). Two-photon lithography not only allows the fabrication of structures that are difficult to access by conventional single-photon processes, but also achieves greater spatial resolution than other 3D microfabrication techniques by far.[72,73]

The most commonly applied two-photon technique is two-photon polymerization. This is analogous to a negative-tone lithographic process in that, liquid starting materials are converted into solid structures upon exposure. The desired 3D structure is fabricated via two-photon absorption and subsequent photopolymerization, followed by removal of the unreacted liquid resin. Photoinitiators and photosensitizers are generally used to improve the efficiency of the polymerization. With this method, various micro-devices[74,75] and photonic crystals[76–78] have been readily produced with near-diffraction-limit 3D spatial resolutions. More recently, a chemically amplified positive-tone two-photon system using a two-photon photoacid generator which enabled the fabrication of buried 3D structures has been reported.[79,80] The ability to selectively remove material in exposed regions allows for efficient creation of small hollow features within a larger solid body. Such positive-tone material systems provide unique abilities in patterning complex 3D structures, such as waveguides, photonic lattices, and microfluidic structures.

Although two-photon lithography provides a unique ability to fabricate a very broad class of 3D micro- and nano-objects, one limiting factor is the low throughput due to the sequential nature of the laser scanning process. A possible solution is to use dynamic diffractive optics to generate and scan a distribution of independent focal points,[81] thereby resulting in a parallel rather than a serial process. Due to its versatile nature, it is generally believed that two-photon lithography could become a useful and powerful technique in future micro- and nanotechnology.

STEP-AND-FLASH IMPRINT LITHOGRAPHY (SFIL)

Step-and-flash imprint lithography (SFIL), a derivative of nano-imprint lithography, was first reported by Colbrun *et al*. in 1999.[82] The schematic procedure of step-and-flash lithography is illustrated in Fig. 6. First, an organic polymer transfer layer is spin-coated onto a silicon substrate. Then a

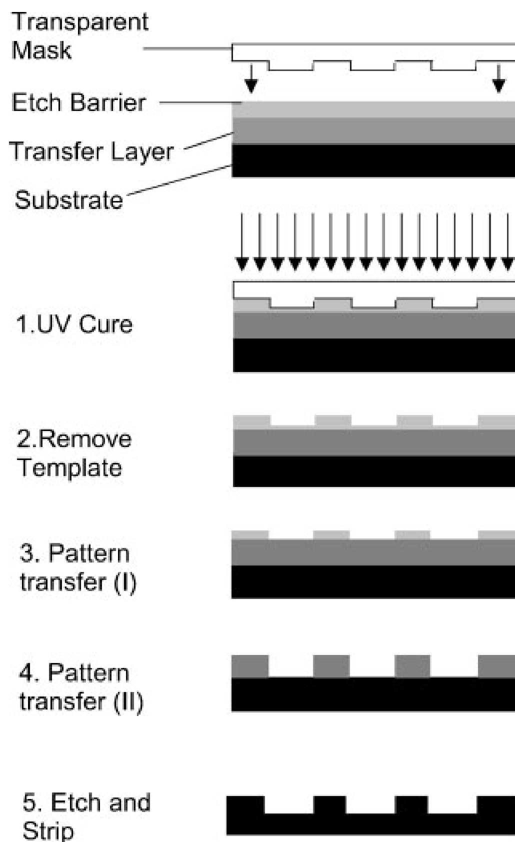

Figure 6. The process of step-and-flash imprint lithography (SFIL).

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low viscosity photopolymerizable mixture, consisting of an organosilicon monomer and a crosslinker and usually called the etch barrier, is dispersed in a single stepper field. A UV transparent mold with the desired patterns is aligned and compressed into the resist. The resist is then cured upon exposure by UV radiation. After the removal of the mold, a short fluorocarbon reactive ion etch step is performed to remove the residual organosilicon polymer ''scum layer''. Finally, an oxygen plasma etch is employed to etch the transfer layer which will serve as a mask to pattern the underlying substrate. Unlike nano-imprint lithography, this process is performed at low pressure and room temperature. By using a transparent template, the issue of alignment is addressed.[83–87] SFIL is considered to be the most suitable imprint technique for fulfilling the requirements of IC fabrication.

Several issues need to be considered in the design of the etch barrier, including adhesion, wettability, photopolymerization kinetics, shrinkage and etch selectivity.[84] A typical formulation contains a free radical initiator, an organic monomer, a silicon-containing monomer and a dimethylsiloxane (DMS) oligomeric crosslinker.[82,84] The free radical initiator generates radicals upon UV exposure in order to initiate polymerization. The organic monomer is chosen to provide adequate solubility for the initiator as well as good adhesion to the bottom transfer layer. The silicon-containing monomer and DMS are incorporated for high oxygen plasma etch resistance and low surface energy, which allows easy template release. The etch barrier contains more than 15 wt% silicon to achieve high oxygen plasma etch selectivity over the underlying organic transfer layer.[88] It has been reported that a typical SFIL photopolymer formulation undergoes a 9.3% (v/v) densification.[84] This increase in density during photopolymerization results in shrinkage of the resin in the direction normal to the substrate surface.

The transfer layer should be insoluble in the liquid etch barrier and remain intact after exposure. It should have good adhesion to the solidified etch barrier and low oxygen plasma etch resistance. Several antireflection layers and PMMA have been used for this purpose.[82,88,89]

NANOFABRICATION WITH BLOCK COPOLYMERS

Block copolymer nanofabrication can provide large-area periodic functional structures or objects with feature sizes of the order of tens of nanometers. However, in many applications, such as multifunctional on-chip bioseparations, simple periodic structure is insufficient and spatial control of the microdomains is necessary.

There are generally two types of strategies towards the spatial control of functional nanostructures using block copolymers: firstly, the substrates are patterned with lithographic techniques (a ''top down'' process) on top of which the block copolymer nanostructures subsequently take desired organizations (a ''bottom up'' process). Secondly, a suitably functionalized block copolymer can spontaneously organize themselves in a convergent ''top down'' and ''bottom up'' process.

A good example for the first case is the graphoepitaxy growth of block copolymers on lithographically defined substrates. By spin-coating onto photolithographically defined substrates, good long-range spherical microdomains were formed in the grooves,[90–92] thus the control of nanostructures were achieved in micro-sized dimensions. Furthermore, by applying the directional solidification process onto a topographically patterned substrate, Thomas and coworkers have also been able to induce two types of cylinder domain orientations in the same precise lateral patterns of poly styrene-block-polyimide (PS-b-PI) copolymer thin films.[93]

Besides photolithographically patterned substrates, other lithographic methods have also been used to form patterned substrates for the purpose of achieving spatial control over block copolymer nanostructures. For example, Manners and coworkers fabricated oriented nanoscopic ceramic lines from cylinder micelles of an organometallic polyferrocene block copolymer on E-beam defined resist grooves.[94] Thomas and coworkers applied soft-lithographically defined PDMS molds onto block copolymer solutions. After the solvent fully evaporated, PDMS molds were peeled off and submicron patterns of block copolymer spheres were generated on the substrates.[95] By introducing sol-gel block copolymer solutions into patterned PDMS microchannels, Stucky and coworkers have been able to fabricate mesostructured silica waveguide arrays and demonstrated their potential applications for integrated optical circuits.[96]

As examples of the second approach, the convergence of ''top down'' and ''bottom up'' fabrication in the same block copolymer architecture has been demonstrated using E-beam lithography. Through Scanning electron microscopy (SEM) E-beam exposure, Russell and coworkers[97] have been able to address non-porous PS-b-PMMA at defined locations on the substrate and thereby demonstrated its potential to generate integrated magnetoelectronic devices. By using metal precursor loaded PS-b-P2VP block copolymer micellar monolayers as negative E-beam resist, Möller and coworkers created metallic nanodots in microscopically defined locations.[98,99]

Although E-beam lithography can give excellent spatial control of functional microdomains, this direct-write patterning process is not time-efficient for large-area integration of functional devices. In the case of the soft lithographic approach, long processing time is an issue and the spatial control of nanostructures has been limited by the physical contact nature of the process. Techniques for rapid patterning of functional nanostructures are thus needed for real-time applications. Ober and coworkers have successfully developed a novel block copolymer system, poly(α-methylstyrene-block-4-hydroxystyrene) system,[100,101] to achieve spatial control through high-resolution deep UV lithographic processes. Through the incorporation of high-resolution PHS photoresist and thermodegradable poly(α-methylstyrene) in the block architecture, large-area uniform nanometer sized pores in submicron-sized patterns were generated through simple fabrication processes (Fig. 7). Additionally, this block copolymer was automatically aligned with vertical orientations during spin coating over a wide range of film thicknesses (40 nm–1 μm), thereby avoiding tedious alignment procedures. Nealey and coworkers recently reported that ternary blends (consisting of PS-b-PMMA/PS/PMMA) capable of self-assembling on chemically patterned

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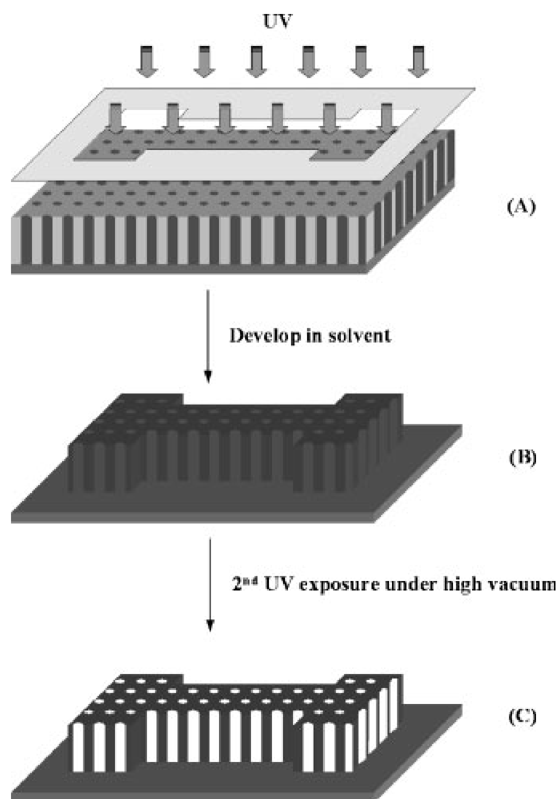

Figure 7. Novel patternable block copolymers to achieve spatially controlled nanostructures. (A) An asymmetric poly(α-methylstyrene-block-4-hydroxystyrene) copolymer/photoacid generator/crosslinker solution was spin-coated on a silicon substrate and formed vertical poly(α-methyl styrene) cylinders due to rapid solvent evaporation, (B) 248 nm stepper exposure and subsequent development to form micropatterns with features as small as 400 nm, (C) strong UV irradiation under high vacuum to remove poly(α-methylstyrene), thus generating patterned nanochannels.

substrates to form periodic arrays.[102] Although self-assembling building blocks for microelectronics have yet to be fully accepted by the industry, further research in this field will doubtless help stimulate development of the next generation of devices.

SUMMARY AND FUTURE OUTLOOK

Lithographic advances have been largely driven by perceived industrial needs currently described by the ITRS. Industry goals and need are constantly shifting and some technologies fail and others advance more rapidly than expected. While there remains debate over the costs of many of these methods and the ability to manufacture in large volume, all the methods described in this review currently have some real prospects for acceptance. The authors have no doubt that industry will achieve its goals of mastering true nanoscale lithography. Other reviews in this volume examine each of these areas in more detail. The authors hope the reader finds this special volume useful in understanding the current state-of-the-art in lithography.

Acknowledgments

The authors would like to acknowledge the long-term financial support for this work from the Semiconductor Research Corporation and the enjoyable interactions with Dr Dan Herr. Both Intel and International Sematech have supported the efforts in the study of molecular glass resists and EUV lithography. Finally, although not described, Intel, International Sematech and Air Products are acknowledged for support of the early efforts in 157 nm lithography. We would also like to thank Mr Shalin Jhavari for the schematic in Figure 5.

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