

157 nm Imaging Using Thick Single Layer Resists

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ABSTRACT

During the past year the probability that 157 nm lithography will precede next generation lithographies such as EUV or EPL has increased, partly due to encouraging advances in the design of polymeric materials, which have sufficient transparency at 157 nm to serve as platforms for single layer photoresists. We have identified several fluorinated resins which can be developed in aqueous 0.26 N TMAH, have reasonable etch resistances (comparable to poly-parahydroxystyrene), and can be formulated to yield photoresists with optical absorbancies at 157 nm which are low enough to be used at thicknesses of 150-200 nm. We have imaged a number of these formulated resists at 157 nm with the Exitech microstepper at International Sematech, and the results for formulated resists with optical absorption coefficients (base 10) as low as 2.1 per micron are described.

Keywords: 157 nm photoresists, fluoropolymers, etch resistance, transparency, tetrafluoroethylene

1. INTRODUCTION

Increasingly, 157 nm lithography appears to be a viable step in the continuous evolution of optical lithography. Originally planned for the 100 nm node, it now appears that 157 nm lithography will be introduced at 70 nm feature size and will be used down to the 50 nm node. At wavelengths shorter than 157 nm the difficulty of developing materials with suitable optical properties increases rapidly with decreasing wavelength, so it may be that 157 nm lithography will be the final step in the very successful history of optical lithography.

The primary challenge in the design of chemically-amplified resist resins for use at 157 nm is that of finding materials which simultaneously possess the following three properties: good optical transparency, suitable solubility in aqueous base after deprotection, and good etch resistance¹. One solution, suggested early in the development of 157 nm lithography, was to turn to fluoropolymers as the resist resins for single layer resists (SLR), since some examples of such materials were found to be surprisingly transparent at 157 nm². Our goal is to develop photoresists, utilizing DuPont's expertise in fluorine chemistry, which have optical absorbances in the range of 0.5-1.5 μm^{-1} , yielding optical densities of 0.1-0.3 for a resist thickness of 200 nm. Here we will describe our progress designing fluoropolymers to achieve this goal.

Copolymers based on TFE are one example of fluorinated resins that, when suitably functionalized and formulated, can serve as 157 nm resists³. These copolymers are typically of low molecular weight ($M_n \sim 2,000 - 6,000$) and approximately alternating. These and related fluoropolymers are readily soluble in organic solvents, have high glass transition temperatures, have good plasma etch resistance, and most importantly have good optical transparency at 157 nm. Introducing functionalities to impart developability in aqueous base generally decreases etch resistance and increases optical absorption at 157 nm. Here we show that fluoropolymers can be synthesized and formulated to be soluble in aqueous base after deprotection, while maintaining the optical absorption coefficient of the formulated resist at values as low as $\sim 2.1 \mu\text{m}^{-1}$. Furthermore, we believe that even lower optical absorption values for formulated resists are possible. Finally, we have imaged these resists at 157 nm, and the results strongly suggest that 200 nm SLR resists for 157 nm lithography are indeed achievable using fluoropolymer resins.



2. EXPERIMENTAL

2.1 Polymer synthesis

Caution! One of the monomers used to synthesize the materials described below is tetrafluoroethylene (TFE), a deflagrating explosive and an experimental carcinogen. All synthetic work with TFE described in this report was conducted within completely barricaded and ventilated facilities.

Two basic polymer platforms have been synthesized and studied. Both include the hexafluoroisopropanol functionality to impart aqueous base solubility. The first platform includes TFE as a comonomer, comonomers containing polycyclic entities to improve etch resistance as well as other comonomers to affect adhesion and imaging performance. The TFE copolymers are synthesized using standard free radical polymerization methods. The second platform is composed of copolymers containing protected and unprotected norbornene-fluoroalcohols. These polymers are synthesized by metal-catalyzed vinyl addition polymerization.

2.2 Resist formulation

Formulation solvents include 2-heptanone, PGMEA (propylene glycol methyl ether acetate), and cyclohexanone. Standard onium photoacid generators (PAGs) were used.

2.3 Optical properties

Vacuum ultraviolet (VUV) transmission measurements were made using a McPherson spectrometer equipped with a deuterium lamp. Each resist sample was spin-coated at several thicknesses on Si substrates to determine the resist spin-curve. CaF_2 substrates were then spin-coated at the appropriate speeds to achieve resist thicknesses between 50 and 200 nm. The VUV transmission spectra of these samples were then measured and are plotted in this paper as absorbance (base 10) normalized by the film thickness.

Spectral ellipsometry measurements were made at International Sematech using a Woollam VUV VASE. Resist samples were spin-coated on Si substrates for these measurements. Refractive indices and Cauchy coefficients were calculated from the ellipsometry data and used to determine resist film thicknesses.

The absorption coefficients measured by direct transmission and spectral ellipsometry were generally in good agreement (at least for the ranges of absorption coefficients and resist film thicknesses we describe here).

2.4 Dissolution and imaging

Dissolution (contrast) curves were measured using open frame exposures made with the Exitech microstepper at Sematech. Samples were spin-coated on Si wafers and post-apply baked (PAB) at 120 °C for 120 sec. An 11x11 matrix of exposure doses was then made, after which the wafer was post-exposure baked (PEB) at various temperatures, followed by puddle development using Shipley LDW-26[®]. The thickness of remaining resist was measured for each exposure dose using a Prometrix SM300 reflectometer. These data were used to generate contrast curves and development rates for the various resist formulations tested.

157 nm images were made using the Exitech/Tropel stepper at International Sematech. The stepper has a NA of 0.6 and a σ of 0.7 when used with the binary mask. Images using an alternating phase shift mask were obtained with a σ of 0.3. Focus-exposure matrices were generated for each resist, and the resulting images were observed using a JEOL tilt SEM, and KLA-Tencor top-down SEMs. Cross-sections were also obtained at Sematech for selected wafer regions. Standard 0.26 N tetramethylammonium hydroxide developer was used for resist processing.

2.5 Outgassing

Outgassing measurements⁴ for several prototypical fluoropolymer resin-based resists were measured at MIT Lincoln Labs by irradiating resist films with 157 nm excimer laser light and collecting in a cold trap any outgassed products for a time of 10 minutes during and after irradiation. The collected gases were then analyzed by mass spectroscopy.

3. RESULTS AND DISCUSSION

3.1 Optical properties

3.1.1 VUV absorption of resins

The VUV transmission of a resin used in a photoresist is important for several reasons. First, the resin is the major component in the formulated resist and therefore provides the largest contribution to total optical absorption. Second, optical absorption by the resin does not lead to useful photochemistry (excluding the possibility of energy transfer from the photoexcited resin to the PAG) and thus serves to decrease the resist sensitivity. Third, light absorption by the resin will contribute to the degradation of sidewall angles. Finally, 157 nm photons absorbed by the resin may generate unwanted photochemistry such as cross-linking reactions, or bond scissions leading to resist outgassing⁵.

We have reported that TFE copolymers can have excellent transparency at 157 nm³. Of course, such polymers must be suitably functionalized in order to serve as chemically amplified photoresists. For 193 nm resists, carboxylic acids and esters have generally been used for this purpose⁶. One would expect the incorporation of carboxylic acid groups to exert a negative impact upon the optical transmission at 157 nm since these groups contain carbon-oxygen double bonds which are known to absorb at wavelengths shorter than 200 nm due to the presence of $\pi \rightarrow \pi^*$ transitions (the lower energy oxygen lone pair transition, $n_p \rightarrow \pi^*$, typically appears at wavelengths between 200 and 300 nm), $\sigma \rightarrow \pi^*$ transitions, and Rydberg transitions for which a lone pair oxygen electron in a 2p orbital is promoted to higher oxygen atomic orbitals (such as 3s, 3p, 3d, 4s, 4p, 4d, etc.)^{7,8}. Other acid functionalities which have been reported in the literature, such as hexafluoroisopropanol groups⁹, are fully saturated and are therefore expected to absorb at shorter wavelengths than 157 nm since they do not have $\pi \rightarrow \pi^*$ transitions. Furthermore, the acid strengths of carboxylic acids ($pK_a \sim 5$) and fluoroalcohols ($pK_a \sim 9$) are also very different. These profound differences in optical properties and acidity are of obvious importance in the design of 157 nm resins.

In Figure 1 we show the optical absorbance spectra of two resist resins (i.e. resins that when formulated will image at 157 nm): one with TFE in backbone and the other a vinyl addition polymer containing no TFE. In both instances partially fluorinated unprotected and protected saturated alicyclic acid groups are included to impart etch resistance, solubility control and transparency. The significant decrease of optical absorbance at 157 nm due to incorporation of TFE is apparent. The origin of this decrease is at least partly due to the alternating nature of the TFE copolymers, which tends to interrupt conjugation and thereby shift the low-lying electronic states to higher energy compared, for example, to poly-norbornene or poly-tetrafluoroethylene¹⁰. *It thus is possible to design and synthesize functionalized resins with optical absorbances below $1.0 \mu\text{m}^{-1}$ at 157 nm, into the transparency regime where optimum imaging is expected with film thicknesses of 200 nm².*

A second interesting observation concerning optical properties is presented in Figure 2. The optical absorption spectrum of a vinyl addition copolymer of a norbornene-fluoroalcohol and tetracyclododecyl t-butyl carboxylate (TCDD) as the protected acid is shown there. This polymer has a remarkably large absorption coefficient ($4.5 \mu\text{m}^{-1}$) at 157 nm. Some of this absorption certainly originates in the carbonyl groups of the TCDD monomers. The total absorption, however, appears to be considerably greater than expected solely upon consideration of the carbonyl contribution³. This suggests that the saturated TCDD rings themselves contribute significantly to the absorption at 157 nm, and this effect might also be important in the cholate esters often used as dissolution inhibitors (see below).

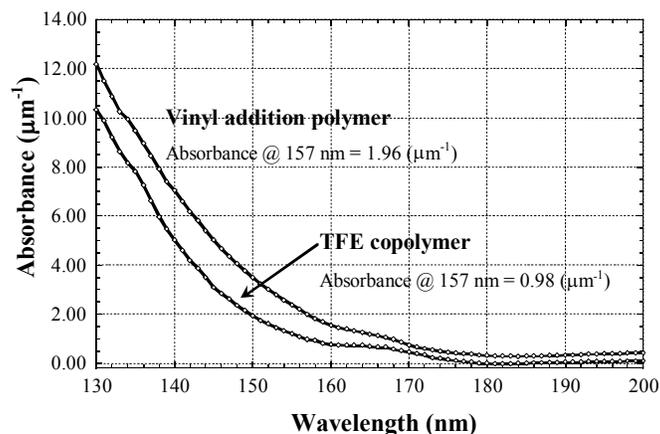


Figure 1. VUV absorbance spectra comparing fluoropolymers containing TFE in the background with those not containing TFE.

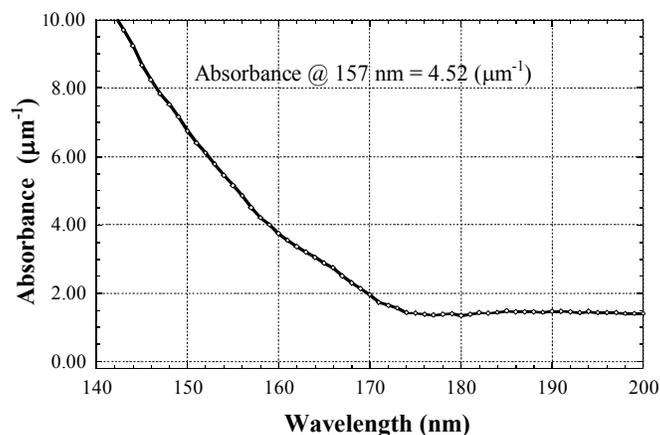


Figure 2. VUV absorption spectrum of a vinyl addition copolymer of norbornene-fluoroalcohol /tetracyclododecyl t-butyl carboxylate. Note the high absorption coefficient at 157 nm.

3.1.2 VUV absorption of dissolution inhibitors

Dissolution inhibitors are frequently used components of resist formulations due to their positive effects upon resist dissolution properties^{11, 12}. Most of the common dissolution inhibitors, however, contain protected carboxylic acid groups and are therefore expected to adversely affect the optical transparency at 157 nm, in the same manner as protected carboxylic groups affect the transparency of resins (see Section 3.1.1).

We have measured the optical absorbance of resins with and without a DI present (cholate ester), and we find the absorbance of this DI to be on the order of $5 \mu\text{m}^{-1}$. Since the DI effectively replaces some of the resin in a formulated resist (if the total weight per cent solids remains constant), then the impact of the DI on resist absorbance will depend upon the relative absorbances of each. Clearly, if the resin has an absorbance greater than $5 \mu\text{m}^{-1}$, then replacing some of the resin by DI can decrease the total resist absorbance. For the low absorbance resins that we are considering, however, the effect of DI is to significantly increase the optical absorbance at 157 nm. An example is shown in Figure 3, where

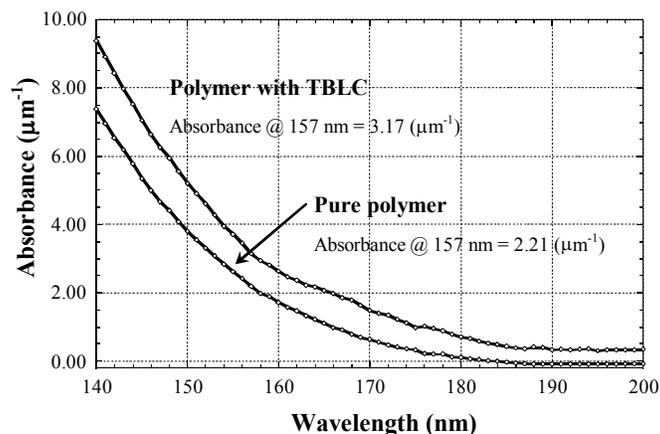


Figure 3. VUV optical absorbance spectra of a vinyl addition polymer with and without 20 weight % DI (TBLC = t-butyl lithocholate ester). Replacing 20 weight % resin by DI increases the total optical absorbance by about $1 \mu\text{m}^{-1}$ at 157 nm.

absorbance spectra for a partially protected norbornene copolymer with and without an added DI (in this case, t-butyl lithocholate ester) are presented. As can be seen, the DI adds nearly 50% to the optical absorption at 20 weight %. This large added absorbance is probably due to $\pi \rightarrow \pi^*$ transitions of the carbonyl group of the DI and additional transitions associated with the large saturated ring system. Thus it is clearly important to either eliminate the use of such DIs for 157 nm resists, or at least minimize the amount used. Of course, it may also be possible to develop new DIs that are more transparent at 157 nm than cholate esters.

3.1.3 VUV absorption of PAGs

Kunz et al.² have given cross sections for optical absorption at 157 nm for several common PAGs used at 193 and 248 nm. They generally find that the optical absorption cross sections for these PAGs at 157 nm are equal to or less than at 193 nm. As they describe, this is due to the fact that the strong $\pi \rightarrow \pi^*$ transitions in the aromatic PAGs are located near 193 nm, and the higher energy transitions near 157 nm generally have smaller oscillator strengths.

3.2 Resist dissolution and contrast curves

One of the important tradeoffs we described in the Introduction is that between optical transparency at 157 nm and aqueous base solubility. The use of fluorinated resins was, and still is, perceived as a serious impediment to achieving proper

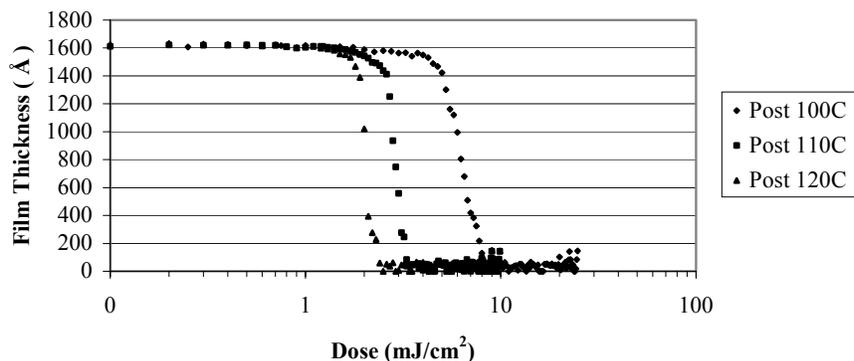


Figure 4. Contrast curves at three temperatures for exposure at 157 nm for a model polymer resin with TFE in the backbone formulated as a resist. The contrast curve shows a dramatic shift to lower exposure dose and an increased γ with higher PEB bake temperature.

dissolution characteristics due to the well-known hydrophobicity of many fluoropolymers. Nevertheless, we have found that it is possible to achieve reasonably good dissolution characteristics in resists which are highly fluorinated as long as sufficient amounts of hydrophilicity are incorporated through the judicious use of protected and unprotected acid groups.

In Figure 4 we show contrast curves measured at 157 nm for a model polymer resin with TFE in the backbone formulated as a resist and treated at three PEB temperatures. This resist shows almost no dark loss and good contrast at higher PEB temperatures. It is also worth noting that the resist photosensitivity is quite high, with a dose-to-clear of about 2.5 mJ/cm^2 at 120°C PEB. Thus TFE containing resists, in addition to exhibiting good optical transmission at 157 nm, can also be made soluble in standard aqueous base developers, and exhibit good photosensitivity.

In Figure 5 we show contrast curves for a vinyl addition copolymer of functionalized norbornenes formulated as a resist. This polymer exhibits very good photospeed, but also shows evidence of swelling at low exposures. Its optical absorption is slightly higher than the TFE-based systems (optical absorption coefficient of $2.6 \mu\text{m}^{-1}$).

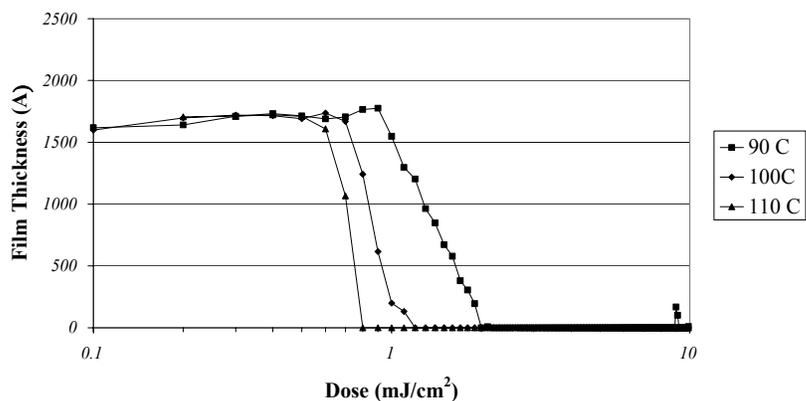


Figure 5. Contrast curves for a functionalized norbornene vinyl addition copolymer resist formulation at three PEB temperatures.

3.3 Etch resistance

Etch resistance is an important property for 157 nm resists since they will be used at thicknesses significantly less than for 193 or 248 nm resists¹. In that regard, highly transparent resists are clearly desirable since they can be used at greater thicknesses than highly absorbing resists. Nevertheless, if aspect ratios are limited to 3:1 then it is unlikely that resist thicknesses much in excess of 200 nm can be used, regardless of how transparent the resist may be. Thus the ability of a 200 nm thick resist to withstand etching requires etch resistance at least as high as poly-parahydroxystyrene (PHS), and preferably as good as Novolak resins.

In Table 1 we show etch rates relative to PHS for one formulated fluorinated resist and several fluorinated resins measured at Intel (Santa Clara) under both poly-Si and oxide etch conditions. The data demonstrate that etch rates comparable to PHS are achievable using fluoropolymers which have reasonably good transparencies at 157 nm. Furthermore, comparing protected and unprotected resins it is clear that there is no strong dependence of etch rates upon the presence of protecting groups for these systems. Finally, the TFE copolymers have quite good etch rates, indicating that incorporation of significant quantities of TFE in the polymer backbone does not cause the etch rate to increase, and in fact may have a slightly beneficial effect. The Ohnishi parameters¹⁰ for these resins ($R = N_T/N_C-N_O$) lie in the range 3-4 (assuming no contribution from the fluorine atoms).

Table 1. Etch rates, relative to a commercial PHS-based resist, of several resins and a resist similar to those discussed in this paper. Etch rates were measured at Intel (Santa Clara).

Resist/Resin	Polysilicon Etch Rate ^a	Silicon Oxide Etch Rate ^b
PHS control ^c	1.0	1.0
Partially protected vinyl addition (formulated resist)	1.6	1.5
Partially protected vinyl addition	1.3	1.2
Unprotected vinyl addition	1.2	1.3
Unprotected TFE polymer	0.9	1.2
Fully protected TFE polymer	0.9	1.1

^aCl₂/HBr chemistry on a TCP etcher

^bC₄F₈ chemistry on a Tel Oxide etcher

^c Commercial 248 nm resist based upon poly-parahydroxystyrene

3.4 Resist outgassing

The resists which have been imaged in this study were observed to exhibit outgassing rates which were comparable to, or at the lower end of the range, of standard 248 nm resists when exposed to 157 nm radiation. Most of the photofragments detected by mass spectroscopy were related to either PAGs or DIs, and there was no significant outgassing associated with the components of the fluorinated resins (such as TFE).

3.5 157 nm imaging

We have imaged a number of formulated resists using the 157 nm Exitech stepper at International Sematech. Here we will describe the results for fluoropolymers platforms previously described.

In Figure 6 we show top-down SEM images of 100 nm isolated and 1:5 lines and spaces for a TFE containing copolymer formulated as a resist and exposed using the 157 nm Exitech stepper at International Sematech. This resist is clearly capable of sub-wavelength imaging, although it does show significant isodense bias. The dose was approximately 20 mJ, and the lines are properly sized.

In Figure 7 we show cross-section SEM images for the 100 nm 1:3 and 1:2 lines and spaces for this resist. The cross-sections show wall profiles with some evidence of standing waves (also see images below). The optical density of this resist film is 0.36, which is low enough to expect reasonably good line profiles². It is worth noting, however, that the results

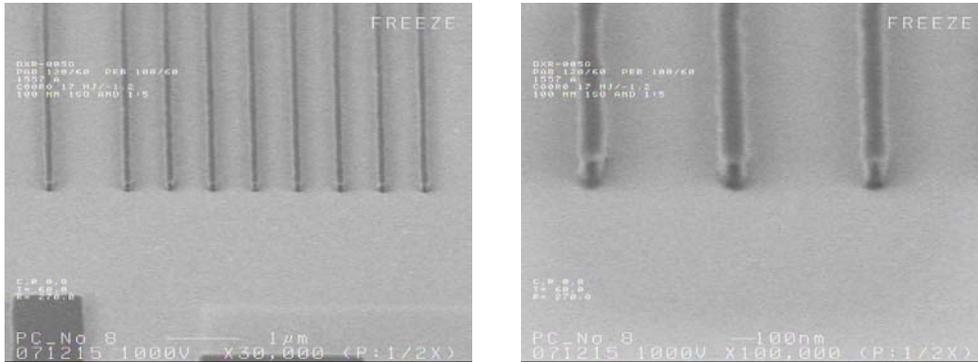


Figure 6. Top-down SEM images of 100 nm isolated and 1:5 lines and spaces of a 157 nm resist containing TFE as a comonomer. The resist thickness was 156 nm, the absorption coefficient was $2.3 \mu\text{m}^{-1}$, and the optical density of the film was 0.36.

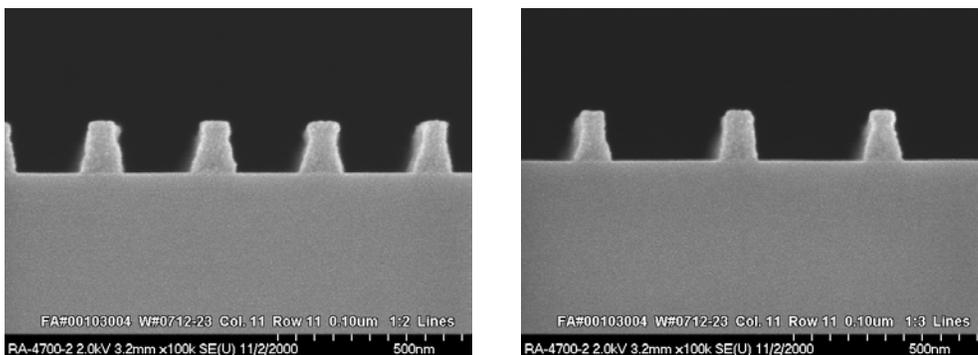


Figure 7. Cross-section SEMs of the 100 nm lines shown in Figure 6. The image on the left is of 1:3 lines and spaces, and that on the right is of 1:2 lines and spaces. The resist thickness is 156 nm.

of profile calculations¹⁴ as a function of photoresist absorption suggest that an optical absorption of $1.2 \mu\text{m}^{-1}$ will be required to achieve wall angles of 85° . *It is important to recognize that this conclusion is independent of resist thickness¹⁴.* Those calculations¹⁴ thus suggest that the resist images shown in Figure 7 may still suffer from too much optical absorption. This stringent absorption requirement illustrates the difficulty faced by the lithography community in developing resists that are sufficiently transparent for 157 nm lithography.

In Figure 8 we show top-down SEM images of a resist, again containing TFE as a comonomer, but this time having a slightly lower optical absorption coefficient of $2.1 \mu\text{m}^{-1}$. Here the presence of isodense bias can clearly be seen since the spaces between lines have opened down to 1:2 pitch, but the 1:1.5 and 1:1 lines and spaces have not opened. The 1:2 lines and spaces are open at 100 nm feature size, but not at 80 nm. Nevertheless, these data show that, using a resin based upon TFE, it is possible to image isolated features down to at least 80 nm using 157 nm radiation and conventional development processes.

In Figure 9 the SEM images of the cross-sections for the 100 nm features shown in Figure 8 are displayed. The wall profiles again show some evidence for standing waves, and the wall angles are not as large as desired. It is likely that the resist performance can be significantly improved through formulation variables and changes in the resin composition.

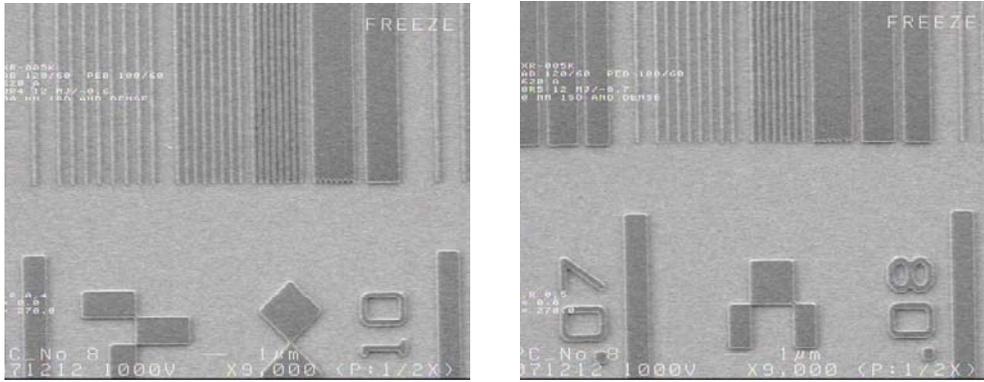


Figure 8. Top-down SEM images of 100 nm isolated and dense lines and spaces (left), and 80 nm isolated and dense lines and spaces (right), made with a 157 nm resist containing TFE as a comonomer in the resin. The resist thickness was 162 nm, the absorption coefficient was $2.1 \mu\text{m}^{-1}$, and the optical density of the film was 0.34.

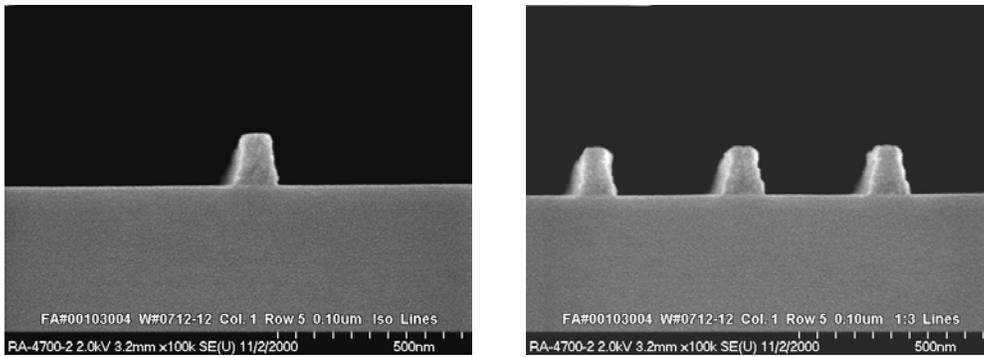


Figure 9. Cross-section SEMs of the 100 nm lines shown in Figure 8. The image on the left is of an isolated line, and that on the right is of 1:3 lines and spaces. The resist thickness is 162 nm.

In Figure 10 we show a top-down SEM image of a TFE-based resist made at 157 nm using an alternating phase shift mask. The 100 nm 1:1 lines and spaces have cleared, suggesting that some of the isodense bias seen using the standard mask may be a result of reduced aerial image quality at these dense feature sizes. The lines in figure 10 also show some variation in width, but little optimization of process conditions was attempted and perhaps improved results could be obtained with this mask configuration.

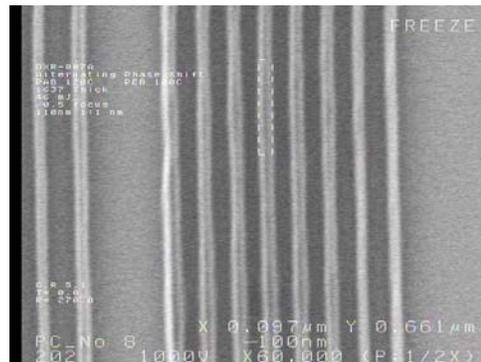


Figure 10. Top-down SEM image of 100 nm 1:1 lines and spaces for a TFE containing copolymer imaged at 157 nm using an alternating phase shift mask. The resist thickness is 164 nm.

The vinyl addition copolymers of functionalized norbornenes have substantially higher optical absorption at 157 nm than copolymers containing TFE. We have imaged the vinyl addition polymers at 157 nm, and an example is shown in Figure 11. Although these resists image at 157 nm, the lines are not properly sized in the exposures of the formulations we have made so far. This may be related to the swelling observed in Figure 5 and we are currently investigating the reasons for this behavior.



Figure 11. Tilt SEM image of 100 nm isolated and 1:5 lines and spaces in a 226 nm thick film of a vinyl addition copolymer of functionalized norbornenes exposed at 157 nm. The lines are oversized.

CONCLUSIONS

In conclusion, we have demonstrated that sub-wavelength imaging is possible at 157 nm using relatively thick (150-200 nm) single-layer resists based upon fluoropolymer resins. We have demonstrated sub wavelength imaging in both vinyl addition copolymers of functionalized norbornenes and in copolymers incorporating TFE in the polymer backbone. The incorporation of TFE in the resin backbone significantly decreases resin optical absorption. We believe that the TFE containing polymers represent a route to photoresists with optical absorption coefficients between 0.5-1.5 μm^{-1} . The TFE-based resins have been used to create lines, properly sized, as small as 80 nm in a 162 nm thick film. Both platforms represent promising single layer resist platforms for 157 nm lithography.

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