Fluorinated Dissolution Inhibitors for 157 nm Lithography

Alyssandrea H. Hamad, Young C. Bae, Xiang-Qian Liu, Christopher K. Ober[*]
Department of Materials Science and Engineering, Cornell University, Ithaca, NY 14853
Frank M. Houlihan[**], Gary Dabbagh, Anthony E. Novembre
Agere Systems, 600 Mountain Avenue, Murray Hill, NJ 07974

ABSTRACT

Fluorinated dissolution inhibitors (DIs) for 157 nm lithography were designed and synthesized as part of an ongoing study on the structure/property relationships of photoresist additives. The problem of volatilization of small DI candidates was observed from matrices such as poly(methyl methacrylate) (PMMA) and poly(hexafluorohydroxy-isopropyl styrene) (PHFHIPS) during post-apply bake cycles using Fourier Transform Infrared Spectroscopy (FT-IR). To avoid this problem, low volatility fluorinated inhibitors were designed and synthesized. Three fluorinated DIs, perfluorosuberic acid bis-(2,2,2,-trifluoro-1-phenyl-1-trifluoromethyl-ethyl) ester (**PFSE1**), perfluorosuberic acid bis-[1-(4-trifluoromethyl-phenyl)-ethyl] ester (**PFSE2**) and a fluorinated phenylmethanediol diester (**FPMD1**), largely remained in a PHFHIPS film during the post-apply bake. The dissolution behavior of the two fluorinated diesters was studied and found to slow down the dissolution rate of PHFHIPS with inhibition factors of 1.9 and 1.6, respectively. The absorbance of PHFHIPS films containing 10 wt% of the diester inhibitors is 3.6 AU/micron compared with an absorbance of 3.3 AU/micron for the polymer itself. The absorbance of 10% **FPMD1** in PHFHIPS was measured as 3.5 AU/micron compared with an absorbance of 3.4 AU/micron for the polymer itself. Thus, the non-volatility and transparency of the fluorinated inhibitors at 157 nm as well as their ability to reduce the development rate of fluorinated polymers make them suitable for use in a 157 nm resist system.

Keywords: dissolution inhibitors, 157 nm lithography, photoresist additives

1. INTRODUCTION

The design of photoresists for 157 nm lithography poses a significant challenge for the resist chemist. Studies have shown that classical deep-ultraviolet (DUV) photoresists absorb strongly at 157 nm. As a result, there has been a rush to design new resists from low absorbing materials. Fluorinated photoresists have emerged as strong candidates for 157 nm lithography due to their high transparency. To date, the goal of obtaining a polymer that can be used as a commercial 157 nm photoresist has not yet been achieved. In order to formulate such a photoresist, it may be necessary to employ photoresist additives that can be used to easily tune the properties of the resist material.

Dissolution inhibitors (DIs) are additives that can functionalize and improve the properties of a photoresist. For example, diazonapthoquinone (DNQ) additives are used to switch the solubility of Novolac resins[1]. Steroid based inhibitors have been used successfully in 193 nm lithography to reduce the dissolution rate of photoresists. Without use of dissolution inhibitors, such photoresists are impractical due to severe dark erosion[2,3]. Dissolution inhibitors may play an important role in improving the behavior of 157 nm photoresists by impeding their development and improving some properties such as contrast and etch resistance. The latter property has proven to be difficult to incorporate into a 157 nm resist. Fluorinated as opposed to non-fluorinated inhibitors have the advantage of being more transparent and having better miscibility with fluoropolymers. In this paper, we report on the investigation of several compounds for use as dissolution inhibitors, in particular, the design and synthesis, and dissolution behavior of several fluorinated dissolution inhibitors for 157 nm lithography.

2. EXPERIMENTAL

2.1 Materials

Poly(methyl methacrylate) (MW=120,000), trimethylsilylnonafluoro-1-butanesulfonate, trimethylsilyltrifluoromethane sulfonate, 2-hydroxy-2-(trifluoromethyl)propionitrile, and 2,2',3,3',4,4', 5,5', 6,6'-octafluoro1,6-hexanediol, and 2-methoxyethyl acetate, 1,3-bis(2-hydroxyhexafluoroisopropyl), hexafluoroacetone, *p*-chlorostyrene, hexamethyldisilazane (HMDS), anhydrous tetrahydrofuran (THF), 2,5-bis(trifluoromethyl)benzoic acid and 3,5-bis(trifluoromethyl)benzaldehyde were purchased from Sigma-Aldrich. Decafluorobenzophenone, perfluorosebaconitrile,

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[*] cober@ccmr.cornell.edu; phone (607)255-8417; fax (607)255-6575; http://ccmr.cornell.edu/~cober
[**] fmh@agere.com; phone (908) 582-4615; fax (908)582-2749

perfluoro(tetrahydrophenanthranene), dimethyl hexafluoroglutarate, dimethyl octafluoroadipate, tetrafluoroisophthalonitrile, hexafluoroglutaric acid, and perfluorosuberic acid were purchased from Synquest Labs, Inc. 1,4-bis(2-hydroxyhexafluoroisopropyl)benzene was purchased from Oakwood Chemicals. 1-[4-(trifluoromethyl)phenyl]ethanol and 1,1,1,3,3,3,-hexafluoro-2-phenyl-2-propanol were purchased from Lancaster Synthesis. Propyleneglycol methyl ether acetate (PGMEA) and OPD 262 developer was purchased from Arch Chemicals.

2.2 General Spectroscopic Procedures

Fourier Transform infrared (FT-IR) spectra were taken on a Thermo Nicolet 870, Nicolet Magna System 560, and Mattson Infinity Gold FT-IR spectrometers. Nuclear Magnetic Resonance (NMR) spectra were taken on a Bruker Avance 360, Varian Inova-400, and Varian VXR-400s spectrometers.

2.3 FT-IR Monitored Baking Studies

Polymer samples with and without 25 wt% of dissolution inhibitor were spincast onto HMDS solution primed silicon wafers. When using poly(methyl methacrylate) (PMMA) as a matrix, the samples were spun from 6 wt% solutions in methoxy ethyl acetate and baked at 130°C for 90s or otherwise noted time increments. Samples in a poly(hexafluorohydroxy isopropyl styrene) matrix were spincast from 10wt% solutions in propyleneglycol methyl ether acetate (PGMEA) and baked at 100°C for 60s or otherwise noted time increments.

2.4 Synthesis of di-tert-butyl carbonate of 1,4-bis(2-hydroxyhexafluoroisopropyl) cyclohexane (t-boc-BHHFIPC)

1,4-Bis(2-hydroxyhexafluoroisopropyl)cyclohexane was synthesized according to reported methods[4] and was protected with a *t*-butyl carbonate group also according to published methods[5].

2.5 Synthesis of perfluorosuberic acid bis-(2,2,2,-trifluoro-1-phenyl-1-trifluoromethyl-ethyl) ester (PFSE1)

Perfluorosuberic acid (15 g, 3.84 mmol) was added to 90 mL of thionyl chloride. Two drops of N,N-dimethyl formamide was added and the mixture was heated to reflux for more than four hours. Excess thionyl chloride was evaporated off and perfluorosuberic acid chloride was purified by vacuum distillation[6]. Hexafluoro-2-phenyl-2-propanol (3.4 mL, 22 mmol) in 4 mL of dry tetrahydrofuran was added to sodium hydride (0.53 g, 22 mmol) in 4 mL of dry tetrahydrofuran, which was stirred over dry ice. Perfluorosuberic acid chloride (4.1 g, 9.6 mmol) in 2 mL of tetrahydrofuran was dropped slowly into the reaction mixture through a dropping funnel. The reaction mixture was heated overnight at 75°C and monitored by FT-IR spectroscopy. The salt was filtered off and the reaction mixture was diluted with diethyl ether. The organic layer was washed three times with 5% sodium chloride solution, dried with magnesium sulfate, and the excess solvent was removed by evaporation. The diester was then purified using column chromatography on silica gel using a 4/1 mixture of hexanes and ethyl acetate as the elution solvent.

2.6 Synthesis of perfluorosuberic acid bis-[1-(4-trifluoromethyl-phenyl)-ethyl] ester (PFSE2)

PFSE2 was synthesized in the same manner as PFSE1, except 1-[4-(trifluoromethyl)phenyl]ethanol was used as the alcohol.

2.7 Synthesis of hexafluorohydroxyisopropyl styrene (HFHIPS)

Synthesis of HFHIPS was performed according to the procedure outlined by Pearce et al[7].

2.8 Synthesis of polyhexafluorohydroxyisopropyl styrene (PHFHIPS)

Polymerization of HFHIPS was performed according to the procedure outlined by Snow et al.[8] with the exception that THF was used as the polymerization solvent instead of benzene and the polymer was precipitated into pentane from chloroform and acetone.

2.9 Synthesis of Phenylmethanediol Bis(diphenyl acetate) (PMDBPA)

Synthesis of phenylmethanediol bis(diphenyl acetate) was performed according to the procedure outlined by Lee et al [9].

2.10 Synthesis of Fluorinated Phenylmethanediol Diester 1 (FPMD1)

An anhydride of 2,5-(trifluoromethyl)benzoic anhydride was synthesized according to the method described by Weintraub et al[10]. 2,5-(Trifluoromethyl)benzoic acid (7 g, 27.1 mmol) was dissolved in 50 mL of freshly distilled toluene. 1,3-Diisopropylcarbodiimide (2.11 mL, 13.6 mmol) was added to the reaction mixture and the solution was stirred overnight at room temperature under nitrogen. The reaction mixture was then cooled over ice and diisopropylurea was filtered off. Water (50 mL) was added and the reaction mixture was stirred for fifteen minutes. The organic layer was separated from the aqueous layer and dried over sodium sulfate. The solvent was removed by evaporation and the product was dried under vacuum.

The phenylmethanediol diester was synthesized as follows: 2,5-bis(trifluoromethyl)benzoic anhydride (3 g, 6.02 mmol) was dissolved in freshly distilled dichloromethane. 3,5-bis(trifluoromethyl)benzaldehyde (0.99 mL, 6.02 mmol) was added to the reaction mixture which was stirred overnight at room temperature and monitored by FT-IR. The reaction mixture was then diluted in 75 mL of methylene chloride and washed with 3 portions of 50 ml of 5% aqueous sodium chloride solution. The organic layer was dried with sodium sulfate. The organic layer was evaporated and the product was dried under high vacuum. The solid product was recrystallized in pentane to yield white crystals.

2.11 Absorption measurements

Optical spectra of polymers films with and without 10% DI were recorded on a VU-301 vacuum ultraviolet variable angle spectroscopic ellipsometer (VUV-VASE) instrument (J.A. Woollam Co.) at International SEMATECH.

2.12 Dissolution Rate Studies

Mixtures of dissolution inhibitor in PHFHIPS at weight ratios of 0-25wt% inhibitor were dissolved in PGMEA at 20wt% solid. The solutions were spincast onto HMDS primed silicon wafers. The wafers were baked at 100ºC for 60s. Film thicknesses were measured using a Nanometrics 210XP thickness gauge. Dissolution rate measurements were made in OPD 262 developer at 25ºC using a Perkin Elmer 5900 Development Rate monitored interfaced with the DREAMS application version 3.00 DRM software package.

3. RESULTS AND DISCUSSION

A series of candidate compounds considered for use in a 157 nm resist system were chosen to study the structure-property relationships of dissolution modifiers. The screened compounds are shown in **Figure 1**. Since fluoroalkyl groups, sulfonate groups, and nitrile groups have been shown to be low absorbing at 157 nm[11,12], many of the candidate materials include these functional groups. As strong interactions such as hydrogen bond have been shown to be crucial to the function of some dissolution inhibitors[13,14], most of the chosen compounds contain polar moieties capable of forming either strong dipolar interactions or hydrogen bonds.

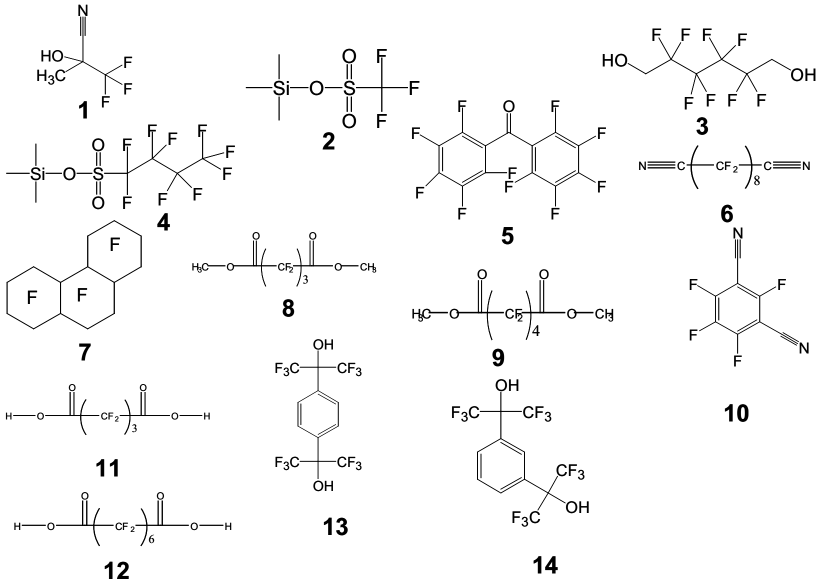


Figure 1. Compounds screened as possible dissolution modifiers. **1.** 2-hydroxy-2-(trifluoromethyl) propionitrile, **2.** trimethylsilyl trifluoromethanesulfonate, **3.** 2,2',3,3',4,4',5,5'-octafluoro-1,5-hexanediol, **4.** trimethylsilyl nonafluoro-1-butanesulfonate, **5.** decafluorobenzophenone, **6.** perfluorosebaconitrile **7.** perfluoro(tetradecahydrophenanthranene), **8.** dimethyl hexafluoroglutarate,

9. dimethyl octafluoroadipate, **10.** tetrafluoroisophthanonitrile, **11.** hexafluoroglutaric acid, **12.** perfluorosuberic acid, **13.** 1,4-bis(2-hydroxyhexafluoroisopropyl)benzene, **14.** 1,3-bis(2-hydroxyhexafluoroisopropyl)benzene

In order to address the possibility of volatilization of the additives, which have a wide range of boiling points, FT-IR was used to monitor the presence of the additives in a polymer matrix before and during a baking process. Films containing 25 wt% of the modifiers in poly(methyl methacrylate) were spin-coated onto silicon wafers. After baking the additives to remove residual solvent, the FT-IR spectra of most of the films were identical to the spectrum of the polymer by itself (**Figure 2**). Therefore, it was concluded that volatilization of the additives resulted in the complete removal of most of the additives under the baking conditions used. Consideration was given to the matrix of the polymer, and since poly(methyl methacrylate) is a matrix that is non-polar compared to the model matrices that would be used in a dissolution inhibition study, we chose to use a more polar polymer matrix to further investigate the volatility of the dissolution modifiers.

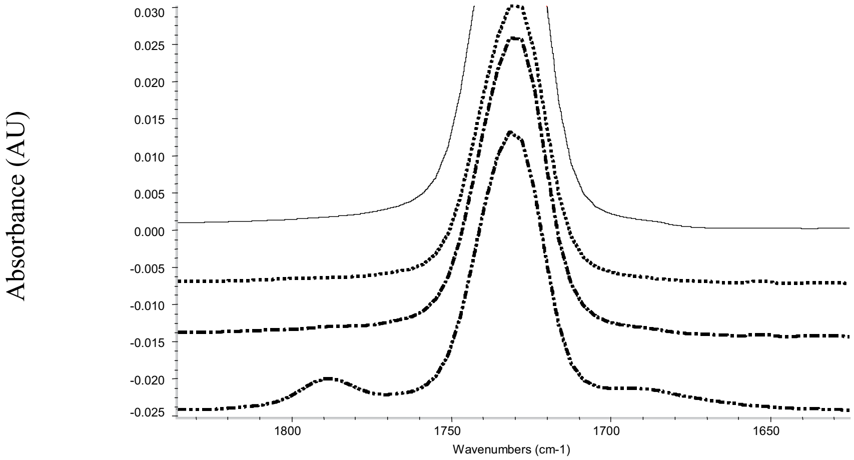


Figure 2. FT-IR spectra of PMMA with and without 25 wt% of dissolution modifiers. PMMA(—)25wt% decafluorobenzophenone (compound **5**) in PMMA (-------). Note absence of carbonyl peak from compound **5** at 1705 cm^{-1}. Spectra of 25 wt% dimethylhexafluoroglutarate (compound **8**) in PMMA (- —- - —- -). Note absence of carbonyl peak from compound **8** at 1782 cm^{-1}. Spectra of 25 wt% perfluorosuberic acid (compound **12**) in PMMA (— - - - — - - - — - -). Note peak from compound **12** at 1782 cm^{-1}). Samples were baked at 130° C for 90s.

A matrix polymer for our studies would need to satisfy two basic requirements. First, in order to perform the dissolution studies, the polymer must be soluble in the developer, which we chose to be 0.262 N aqueous tetramethyl ammonium hydroxide. Second, we believed that the polymer should mimic a 157 nm resist system, meaning that it should contain low absorbing functional groups such as fluorinated alkyl groups. The polymer that was chosen was poly(hexafluorohydroxy isopropyl styrene) (PHFHIPS) (**Figure 3**). This polymer has been investigated for use in 248 nm as well as 157 nm lithography[15, 12]. Copolymers containing the hexafluoroisopropyl styrene repeat unit can have low enough absorbance for use as a thin 157 nm resist.

One difficulty that we believed could arise from the use of this model matrix is that its quick dissolution would make the effects of the inhibitor undetectable. Because of similarities in their pK_as, the dissolution behavior of PHFHIPS was predicted to be comparable to that of poly(hydroxy styrene) (PHS). PHS is a polymer whose dissolution is difficult to impede, although there has been a report of dissolution inhibitors designed for it [9].

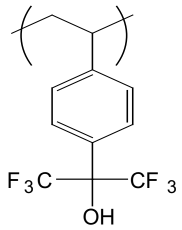


Figure 3. Poly(hexafluorohydoxyisopropyl styrene) (PHFHIPS) polymer used for baking and dissolution rate studies.

Baking studies performed on PHFHIPS containing 25 wt% of our dissolution modifiers showed that only the highest boiling point compounds remained in the films during the baking process (**Figure 4**). To overcome the problem of volatilization, larger dissolution inhibitors were designed to be less volatile. First, a simple inhibitor based on the protection of a fluorinated cyclohexyl diol was synthesized (**t-boc-BHHFIPC**, **Figure 5**). Still, this small inhibitor proved to be too volatile for practical use as can be seen by the reduction in intensity of the additive's the carbonyl peak during the baking process (**Figure 6**). Therefore, two large diester inhibitors perfluorosuberic acid esters 1 and 2 (**PFSE1 and PFSE2**) were designed and synthesized (**Figure 5**). Baking studies were performed on PHFHIPS films containing 25 wt% of these diesters. While some of the **PFSE1** volatilized from the polymer during the bake, most of the compound remains in the film, volatilization of **PFSE2** was not observed (**Figure 7**). This diester has the added advantage of breaking down by acidolysis,

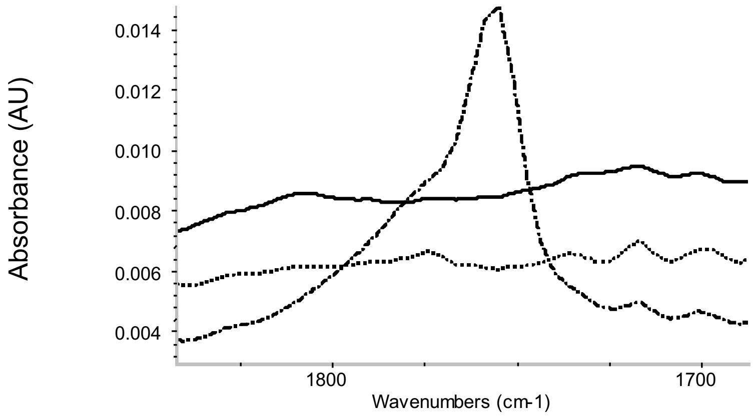
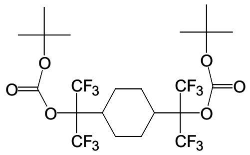


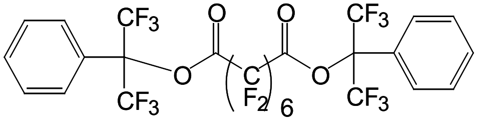
Figure 4. FT-IR spectra of PHFHIPS (——), 25 wt% dimethyloctafluoroadipate (compound 9) in PHFHIPS (--------). Note absence of carbonyl peak from compound **9** at 1782 cm^{-1}. Spectra of 25 wt% perfluorosuberic acid (compound 12) in PHFHIPS (–- — -— - —). Note presence of carbonyl peak at 1750 cm^{-1} from compound 12. Samples were baked at 100°C for 40s.

Dissolution inhibition studies on **PFSE1** and **PFSE2** showed that the fluorinated diesters inhibit the dissolution of PHFHIPS. A sample inhibition plot for **PFSE1** is shown in **Figure 8**. Inhibition factors for the two diesters were calculated to be 1.92 and 1.64 respectively. A saturation effect was observed in the dissolution inhibition plots of the diesters; at high weight percent loading of DI, the effect of adding more DI was negligible. This could be due to the phase segregation of non-polar inhibitors in a nonpolar matrix, behavior that could be aggravated by the presence of the long perfluorinated alkyl tail on the inhibitors. Dissolution inhibition studies on a compound known to inhibit the dissolution of PHS were performed for comparison. Phenylmethanediol bis(diphenyl acetate) (**PMDBPA**)[9] was found to inhibit the dissolution of PHFHIPS with a factor of 7.0. No saturation effect was observed in dissolution inhibition plots for this compound. However, it was predicted that it would absorb too highly absorbing to be useful for157 nm lithography (**Figure 9**).

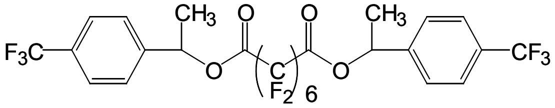
Absorption studies performed at International SEMATECH showed that while the absorption of both **PFSE1** and **PFSE2** do not drastically increase the absorption of the PHFHIPS matrix polymer, addition of 10 wt% of PMDBPA to the polymer results in a 75% increase in absorption (**Table 1**). Due to the high absorbance of PMDBPA, it is an unlikely additive for a 157 nm photoresist. Therefore, we synthesized a fluorinated phenylmethyldiol diester in an attempt to obtain a powerful inhibitor that is relatively transparent at 157 nm. To this end, **FPMD1** was synthesized. FT-IR baking studies showed that the compound largely remains in the film during the baking process and its presence does not significantly increase the absorbance of PHFHIPS at 157 nm (**Table 1**). Further investigations on this new DI are currently underway.



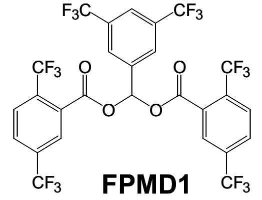
t-BOC-BHHFIPC



PFSE1



PFSE2



FPMD1

Figure 5. Dissolution inhibitors synthesized for this study.

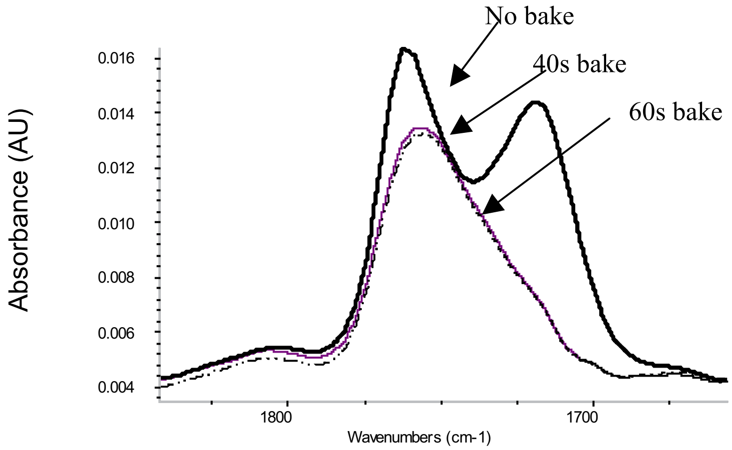


Figure 6. FT-IR spectra of films containing 25 wt% t-boc-BHHFIPC in PHFHIPS before baking (—), after 40s baking at 100°C (— — — —), after 60s baking at 100°C (— - — - —). Peak at 1760 cm^{-1} is from dissolution inhibitor, peak at 1718 cm^{-1} is from PGMEA spinning solvent.

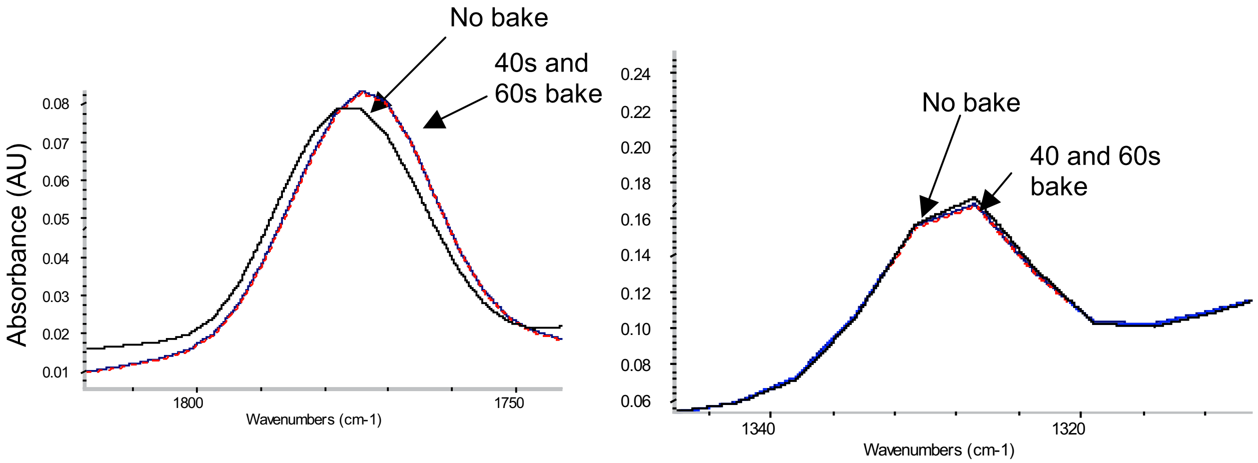


Figure 7. FT-IR spectra of 25 wt% PFSE2 in PHFHIPS before baking (—), after 40s bake at 100°C (-----), after 60s bake at 100°C (— — —).

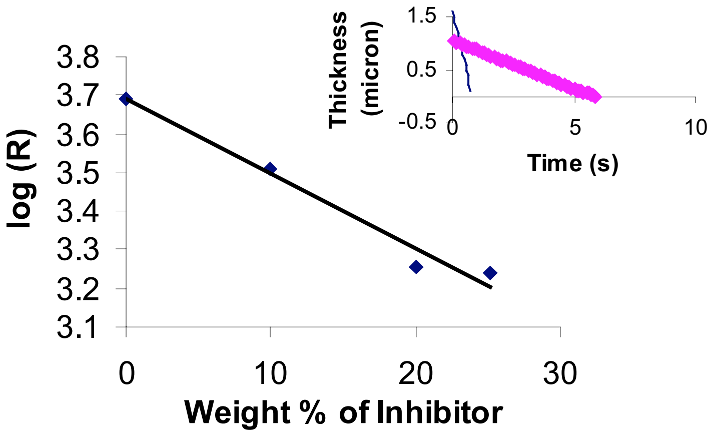


Figure 8. Inhibition curve for PFSE1. Inhibition factor (f) was calculated to be 1.92. Inset shows thickness vs. development time for films containing no inhibitor (light) and 20% inhibitor (heavy line).

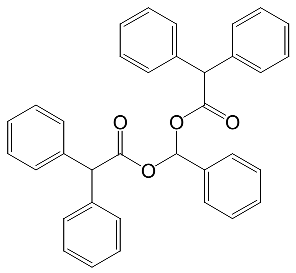


Figure 9 Phenylmethanediol bis(diphenyl acetate) **(**PMDBPA), a non-fluorinated inhibitor used in this study[9].

Sample	Absorbance (AU/μm)
PHFHIPS-1	3.3
PHFHIPS-1 with 10% PFSE-1	3.6
PHFHIPS-1 with 10% PFSE-2	3.6
PHFHIPS-2	3.4
PHFHIPS-2 with 10% PMDBPA	6.0
PHFHIPS-3	3.4
PHFHIPS-3 with 10% FPMDA	3.5

Table 1 Results from 157 nm absorption measurements.

4. CONCLUSIONS

We have synthesized several fluorinated DIs that successfully inhibit a model 157 nm polymer (PHFHIPS), are non-volatile, and only modestly increase absorbance at 157 nm. Volatilization was observed to be an issue when studying small photoresist additives. A saturation effect was observed for polymer films containing our fluorinated diester inhibitors (PFSE1 and PFSE2) possibly due to a low loading capacity of the non-polar compounds in the highly polar polymer.

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