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# Fluorinated Heteroaromatic Polyethers for Low Dielectric Constant / High Temperature Applications

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SUMMARY: Several series of poly(arylene ether)s with trifluoromethyl substituents were prepared and characterized. These materials are potential candidates for the use as low dielectric constant insulators (intermetal dielectrics, IMD, and interlayer dielectrics, ILD) on microchips. Thermal stability up to 450 °C and a dielectric constant below 3 preferably below 2.5) is required for this application. The thermal stability of the poly(arylene ether)s was increased from 320°C to more than 500 °C by optimization of the structure of the repeating unit. The dielectric constant of one of the most promising structures was determined to be 2.8. In addition, plasma polymerized thin films from hexafluorobenzene, tetrafluorobenzene, perfluorotoluene and perfluorodecaline were prepared and characterized with respect to solubility, dielectric constant, adhesion, and thermal stability.

## Introduction

The continuing trend to increase the capacity of memory chips in microelectronics and the resulting need to decrease of the size of the devices (transistors, capacitors etc.) on the chips has reached a stage where new materials are required for the conducting interconnection layers on the chips1,2,3,4). Driving force for the fabrication of chips with smaller structures are the decreasing cost per device, reduced power consumption, and decreasing intrinsic gate delay of smaller transistors. The major problems caused by this trend, however, are the decreasing speed of signal propagation, caused by increasing capacitance of the interconnecting metal lines, and increased cross talk as the distance between the metal lines shrinks. The signal delay time $\tau$ depends on the resistance and capacitance of the metal lines5):

$$\tau = RC = 2\rho\epsilon\epsilon_0\left[\frac{4L^2}{P^2} + \frac{L^2}{T^2}\right]$$

$\tau$: signal delay time
R: resistance
C: capacitance
$\rho$: specific resistance of the conductor
$\epsilon$: dielectric constant of the insulating material
$\epsilon_0$: dielectric constant of vacuum
L: length of the conductor
T: thickness of the conductor
P: distance between two conducting lines

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Figure 1 shows the signal delay time in dependence of the size of the smallest structures on a chip for a given layout of the interconnect lines.

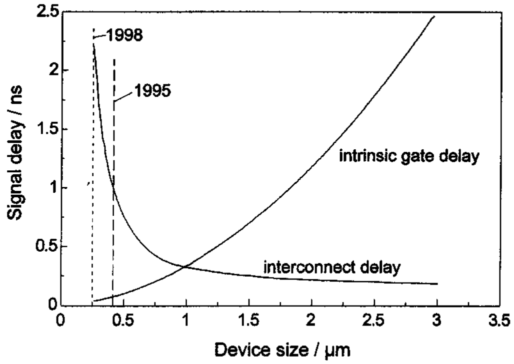


Figure 1: Signal delay time vs. device size for a given layout of the interconnect lines[1,6)

The graph in Figure 1 clearly shows that at feature sizes below 1 µm the signal delay time is dominated by the interconnect delay rather than the intrinsic gate delay. At sizes of 0.25 µm as used typically for DRAM memory chips and CPU's commercialized initially in 1998[3,6)], the signal delay is approximately 2 ns, limiting the maximum clock speed of these chips to 500 MHz. Further acceleration requires changes of the layout, use of an interconnect metal with higher conductivity than the presently used aluminum based alloy, or the use of an insulating material with a lower dielectric constant than the presently used[1,2,4,7,8)] $SiO_2$ with $\epsilon \geq 4$. Changes of the layout are generally strongly restricted by the desire to keep the chip size as small as possible and the limits of available photolithographic techniques. This can be compensated in part by extending the interconnects into the third dimension, that is by adding additional metallization layers. At present, typically 3-4 metallization layers are used, while the 4 GB RAM chips are expected to have up to 6-8 layers. In 1998, copper has been introduced as interconnect metal to replace the aluminum based alloy. This increases the signal speed by approximately 35% due to the higher specific conductivity of copper[7)]. However, copper metallization required a considerable change in processing, since dry etching and gas phase deposition of copper is difficult and slow. This new „copper damascene" process[2,7)] is too complex to be described here in detail. In principle, in this process the insulating material, e.g. a polymer, is deposited first, then trenches are formed in this layer by photolithographic methods, the trenches are then filled with copper by a combination of gas phase and galvanic deposition techniques, and then the excess copper is removed by chemical mechanical polishing (CMP). Etch stop layers, polish stop layers, and diffusion barriers for the copper as well as adhesion promoting layers are required to obtain reliable structures.

A quite promising concept to reduce the signal propagation time is the use of insulating materials with dielectric constants below that of $SiO_2$. Polymers can exhibit dielectric constants down to $\epsilon \approx 2$ for aliphatic perfluorinated Materials such as PTFE or amorphous TFE/Perfluorocycloolefin-co-polymers, the lowest value known for any dense, non-porous material⁹⁾.

## REQUIREMENTS FOR LOW-$\epsilon$-MATERIALS

The need for incorporation of the new low-$\epsilon$-materials into existing schemes for manufacturing of microchips results in strict requirements regarding various properties. The most important issues⁴⁾ are low dielectric constant, high temperature resistance, low moisture absorption, and good adhesion (Table 1).

Table 1: Requirements for low-$\epsilon$-materials⁴⁾

| **Property** | $\epsilon$ | $T_d$ | moisture uptake | adhesion to |
|---|---|---|---|---|
| **Value** | $\leq 2.5$ | >450°C | < 1% | Al, Si, $SiO_2$, Cu, ... |

At present, a dielectric constant of 2.5 or below is targeted, which corresponds to a decrease of the signal delay $\tau$ to 62% compared to a structure with $SiO_2$ as insulating layer. The processing temperature will be at least 400 °C in each metallization step, even in the copper metallization scheme⁷˒⁴⁾. In addition, there are many other parameters mostly concerning mechanical properties, coefficients of thermal expansion, compatibility with existing etching processes, compatibility with chemical mechanical polishing, planarization, etc⁴⁾.

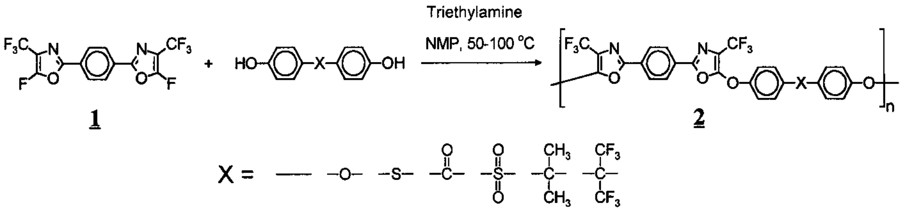
In order to keep the dielectric constant of a polymer low, polar and polarizable structures have to be avoided⁹⁾. This also helps to decrease moisture uptake. It has been found that the incorporation of fluorine also decreases the dielectric constant⁹⁾. Bulky, rigid structural elements increase the free volume of a polymer, which also results in a decreased value of $\epsilon$⁹⁾. A large number of different polymer classes has been suggested for the use as low-$\epsilon$-materials. The list⁴˒¹˒²˒⁸˒⁷⁾ includes polyimides, poly(benzoxazole)s, poly(quinoline)s, poly(aryl ethers) (each of these fluorinated or non-fluorinated), poly(silsesquioxane), poly(naphthalene), poly(perfluorocyclobutane), Parylene and Parylene F, PTFE and amorphous PTFE-copolymers. All of these polymers have their strengths and weaknesses, which have been discussed elsewhere¹⁰⁾ in detail. Some of these polymers (poly(naphthalene), poly(perfluorocyclobutane), and the Parylenes) are prepared by chemical vapor deposition (CVD), plasma polymerization or a related technique directly on the substrate. The others are applied from solution by spin coating[4,1,2,8,7].

We are pursuing both the development of polymers for spin coating on the basis of fluorinated poly(aryl ethers) as well as the application of plasma polymerization for the preparation of fluorinated dielectric films.

## Low-ε-poly(aryl ether)s for application from solution

### Poly(aryl ether oxazole)s and poly(aryl ether thiazole)s

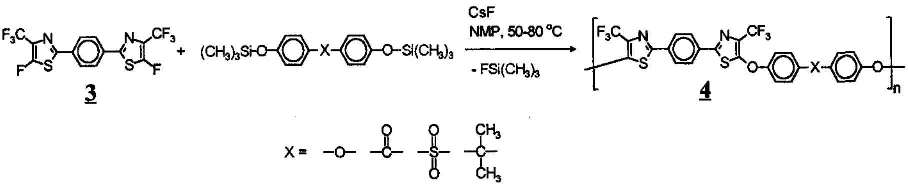
Poly(aryl ether)s are usually prepared by nucleophilic displacement of halogen from activated aryl bishalides by bisphenoxides[11,12,13]. The activation is achieved by electron withdrawing groups. Most of these groups are polar, such as carbonyl[13] and sulfone[12]. However, for the application discussed here the use of such functional groups is detrimental[9]. Activation can also be achieved by electron deficient heteroaromatic rings[11,14]. One of the least polar electron poor heteroaromatic groups is the oxazole ring[14,15]. Another ideally suited activating group is the perfluoroalkyl group[16,17], especially the trifluoromethyl group. Thus, polymers of the following type 2[18,19], derived from monomer 1 appeared to be promising candidates (Scheme 1):



Scheme 1: Bisoxazole monomer 1 and the corresponding poly(aryl ether)s 2

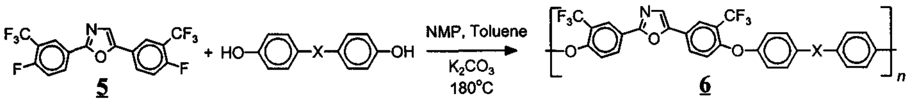
However, these polymers exhibit an unexpectedly low thermal stability with decomposition temperatures (initial weight loss) at 310 °C in air or nitrogen[18], independent of the exact structure of the repeating unit. The substitution pattern of the oxazole rings with a substituent in 4-position was identified to be the problem[18,19]. The $CF_3$-group stabilizes an intermediate formed in a thermal ring scission[18,19].

Consequently, similar polymers 4 with thiazole rings in place of the oxazoles were prepared (Scheme 2)[20]. Sulfur containing heteroaromatic rings are considered to be more stable due to their more pronounced aromatic character (higher number of mesomeric structures) than their oxygen analogs[21,22,23]. The thermal stability of the polymers 4 was indeed found to be higher with decomposition temperatures of 420°C[20].



Scheme 2: Bisthiazole polymers **4**

In addition, mono-oxazole polymers **6** (Scheme 3) were studied[15]. In these polymers, there is no substituent in 4-position of the oxazole rings. They exhibit decomposition temperatures of the order of 450-490°C (10% weight loss in air)[15], which is typical for polymer with five-membered heteroaromatic rings in the main chain[14].



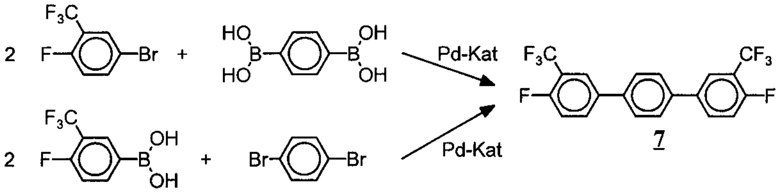
Scheme 3: Mono-oxazole polymers **6**

All polymers **2**, **4** and **6** are soluble in amidic solvents such as NMP, DMAc or DMF at room temperature[15,18,19,20]. Many of them are also soluble in chlorinated solvents and THF. Since the thermal stability of the polymers **6** appears to be sufficient, the dielectric constant of polymer **6a** with $X = -C(CF_3)_2-$ was determined. The presence of four trifluoromethyl groups per repeating unit results in a relatively low value of $\epsilon = 2.8$, which is a good basis for further developments.

The glass transition temperatures of the polymers **6** were found to be between 154 and 220°C (**6a**: 205°C)[15] and are lower than initially desired. The coefficient of thermal expansion increases above $T_g$. Thus, the $T_g$ of polymers used as low-$\epsilon$-dielectric material should be as close to the maximum processing temperature as possible, if not higher. The oxazole ring introduces a catenation angle[24] of approximately 132°, which increases the conformational flexibility of the polymer chain and hence decreases the glass transition temperature. Also, the oxazole ring is slightly polar with a dipole moment of $5 \cdot 10^{-30}$ Cm[25] across the polymer chain. The replacement of the oxazole wing with a *para*-substituted phenyl ring was expected to decrease the dielectric constant and to increase the glass transition temperature at the same time.

## Terphenyl-poly(aryl ether)s

Based on the considerations described above, we prepared terphenyl monomer 7, in which trifluoromethyl groups placed in *ortho*-position to fluorine atoms act as activating groups for the nucleophilic displacement reaction.

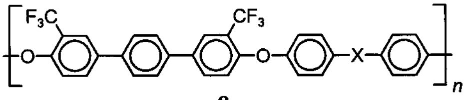


Scheme 4: Structure of terphenyl monomer 7

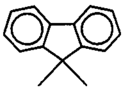
Monomer 7 was prepared by Suzuki-coupling[26,27] of either 2-fluoro-5-bromotrifluoromethyl benzene with benzene-1,4-bisboronic acid or 4-fluoro-3-trifluoromethyl benzene boronic acid with 1,4-dibromo benzene. The second approach proved to be much more successful with overall yields well above 80%, while yields were around 20% when the first pathway was followed. Possibly the trifluoromethyl group in the bromo-compound slows the coupling reaction to an extent were undesired side reactions become important.

The polycondensation of monomer 7 with various bisphenols under conventional reaction conditions proceeds quite successfully, and high molar mass polymers are obtained. Table 2 shows the structures of the resulting polymers 8 and their molar masses. The molar masses of the polymers 8 are quite high, with the exception of 8e. While all other polymers 8 form very tough, flexible films, the films prepared from 8e remain brittle. This can be attributed to the relatively low nucleophilicity of the phenoxide prepared from dihydroxydiphenyl sulfone, which is caused by the electron withdrawing effect of the sulfone group. Since the activation of the fluorine in monomer 7 is only due to the negative inductive effect of the trifluoromethyl groups in *ortho*-position rather than mesomeric stabilization of the intermediate Meisenheimer-Komplex, monomer 7 is not very reactive towards nucleophilic replacement of the fluorine atoms. Consequently, the substitution reaction is slow in the case of 8e, and side reactions may become important, which limit the molar mass of the resulting polymer.

Table 2: Structures and molar masses of the polymers 8

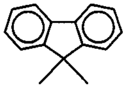


8

| Polymer No. | X | $\bar{M}_n$ g · mol$^{-1}$ | $\bar{M}_w$ g · mol$^{-1}$ |
|---|---|---|---|
| 8a | -$C(CH_3)_2$- | 43 200 | 83 400 |
| 8b | -$C(CF_3)_2$- | 30 000 | 74 500 |
| 8c | — | 21 200 | 62 200 |
| 8d |  | 44 600 | 71 600 |
| 8e | -$SO_2$- | 6 500 | 16 400 |

The decomposition temperatures of polymers 8c and 8d in air are well above 500 °C (expressed as 5% weight loss in dynamic thermogravimetry). However, the glass transition temperatures as determined by DSC and DMTA are lower than expected (Table 3).

Table 3: Thermal properties of the polymers 8

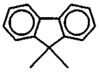
| Polymer | X | Polymers 8 | | | Polymers 6 |
|---|---|---|---|---|---|
| | | $T_d$[a)] °C | $T_g$ (DMTA)[c)] °C | $T_g$ (DSC)[b)] °C | $T_g$ (DSC) °C |
| 8a | -$C(CH_3)_2$- | 464 | 210 | 174 | 185 |
| 8b | -$C(CF_3)_2$- | 500 (520)[d)] | 218 | 197 | 205 |
| 8c | — | 514 | 198 | 209 | 200 |
| 8d |  | 530 | 240 | 234 | - |
| 8e | -$SO_2$- | 422 | not determined | 214 | 210 |

[a)] 5% weight loss, air, 10 K/min; [b)] 20K/min; [c)] 1 Hz, 2K/min; [d)] in nitrogen

The glass transition temperatures of the polymers 8 do not differ very much from those of the oxazole polymers 6 described above, despite the more rodlike terphenyl segment in the repeating unit of polymers 8. For 8a and 8b, there appears to be even a decrease in $T_g$ as the oxazole ring is replaced by a 1,4-phenyl ring. Possibly the small dipole moment of the oxazole ring contributes to chain interactions which increase $T_g$, an effect which is not present when a phenyl ring replaces the oxazole. The polarity and geometry of the oxazole ring push $T_g$ of the polymers 6 in opposite directions, and which of these influences dominates appears to depend on the rest of the repeating unit.

Some mechanical properties of the polymers 8 measured with thin film samples cast from THF solution are shown in Table 4.

Table 4: Mechanical properties of the polymers 8

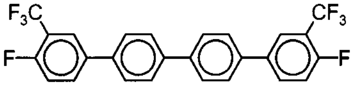
| Polymer | X | Young's Modulus[a] | Tensile strength[b] | Elongation at break[b] |
|---|---|---|---|---|
| | | GPa | MPa | % |
| **8a** | $-C(CH_3)_2-$ | 1.32 | 58 | 18 |
| **8b** | $-C(CF_3)_2-$ | 2.51 | 55 | 28 |
| **8c** | — | 1.77 | 70 | 10 |
| **8d** |  | 2.69 | 61 | 7 |

[a] DMTA, 1Hz, 2K/min; [b] stress-strain measurements, room temperature, stain rate 5%/min

As expected, the polymer with the bulky fluorene group (**8d**) proved to be the most brittle one, but even the best (**8b**) is not very ductile, despite the relatively high molar masses of these polymers. Measurements of the dielectric constants are in progress.

**Quadriphenyl-poly(aryl ether)s**

All in all, the performance of the polymers 8 was not as good as expected. Therefore, another monomer with even more extended rodlike structure was prepared (9). The same synthesis strategy as for 7 was applied, with the exchange of 4,4'-dibromo biphenyl in place of 1,4-dibromo benzene in order to achieve a quadriphenyl unit:

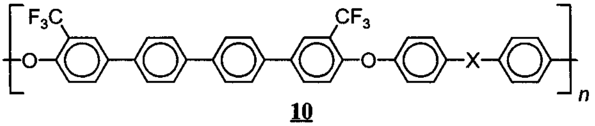


9

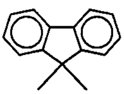
Scheme 5: Quadriphenyl monomer 9

The reactivity of monomer **9** is very similar to monomer **7**, and consequently under the same reaction conditions polymers **10** could be prepared with quite high molar masses (Table 5).

Table 5: Structures and molar masses of polymers **10**

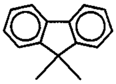


**10**

| Polymer No. | X | $\overline{M}_n$ | $\overline{M}_w$ |
|---|---|---|---|
| | | $g \cdot mol^{-1}$ | $g \cdot mol^{-1}$ |
| **10a** | $-C(CF_3)_2-$ | 32 000 | 88 000 |
| **10b** | — | 39 300 | 94 400 |
| **10c** |  | 42 000 | 106 000 |

The thermal stabilities exceed 500 °C for all of these polymers, and the glass transition temperatures are considerably higher than those of the terphenyl polymers **8** (Table 6). Polymer **10c** is especially promising with $T_g$ of 300 °C and its high thermal stability. The structure of this polymer contains no polar groups except for the ether links. Also, the bulkiness of the fluorene unit can be expected to result in an increase in free volume. As a consequence, we expect this polymer to exhibit its dielectric constant to be considerably lower than that of polymer **6a**, which was 2.8. The Measurements are in progress.

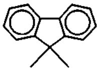
Table 6: Thermal stabilities and glass transition temperatures of polymers **10**

| Polymer | X | Polymers **10** | | | Polymers **8** |
|---|---|---|---|---|---|
| | | $T_d$[a] | $T_g$ (DMTA)[c] | $T_g$ (DSC)[b] | $T_g$ (DSC) |
| | | °C | °C | °C | °C |
| **10a** | $-C(CF_3)_2-$ | 510 | 225 | 210 | 197 |
| **10b** | — | 530 | 268 | 257 | 209 |
| **10c** |  | 534 | 315 | 300 | 234 |

[a] 5% weight loss, air, 10 K/min; [b] 20K/min; [c] 1 Hz, 2K/min

In addition to the excellent thermal properties the mechanical properties of the polymers 10 are also quite encouraging. Dynamic-mechanical testing and stress-strain measurements of films revealed high strength and high drawability of these polymers. The data are summed up in Table 7. For comparison, typical values for commercial PEEK and Polycarbonate are added.

Table 7: Mechanical properties of the polymers 10

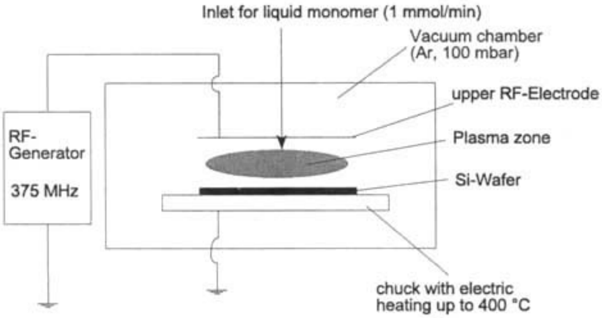
| Polymer | X | Young's Modulus a) | Tensile strength b) | Elongation at break b) |
|---|---|---|---|---|
| | | GPa | MPa | % |
| 10a | -$C(CF_3)_2$- | 2.59 | 65 | 78 |
| 10b | — | 2.04 | 75 | 76 |
| 10c |  | 2.18 | 115 | 120 |
| Victrex PEEK | - | 3.6 | 95-100 | >50 |
| PC | - | 2.4 | 72 | 120 |

a) DMTA, 1Hz, 2K/min; b) stress-strain measurements, room temperature, stain rate 5%/min

Tensile strength and elongation at break of polymer 10c are comparable to typical engineering and high performance thermoplastic materials. The tensile modulus of 10c is relatively low, but this may be due to the fact that we were using thin films cast from DMF solution with no optimization of solvent and casting conditions. The films were dried by heating and evacuation in several steps, with a final drying temperature of 170 °C under vacuum for 24 h and proved to be solvent free in dynamic thermogravimetry.

## Fluorinated low-ε-films by plasma polymerization

Plasma polymerization proceeds by free radicals and/or diradicals produced from various precursors in the gas phase under reduced pressure by collisions of the precursor molecules with the atoms, ions and electrons of a plasma. The radicals are deposited on the substrate and combine to larger structures, and finally a polymer is formed. Depending on the precursor, mechanism and reaction conditions, the structure of these polymers is more or less defined, and often crosslinking occurs, causing insolubility of the deposited films. A schematic drawing of the plasma deposition chamber used for our experiments is shown in Scheme 6.

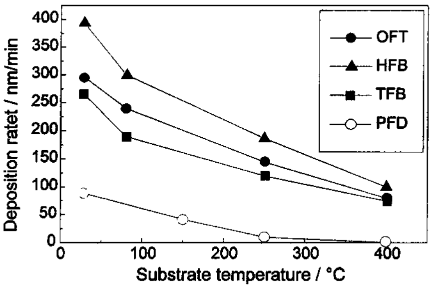


Scheme 6: Schematic drawing of the plasma deposition chamber

The need for high temperature stability of the deposited films caused us to use fluorinated aromatic compounds as precursors, because we expected higher thermal stability from aromatic or partially aromatic polymer films compared to purely aliphatic materials such as Teflon or Teflon AF. Therefore, we chose 1,2,4,5-tetrafluorobenzene, hexafluorobenzene$^{28)}$, and octafluoro toluene as precursors. Perfluorodecaline was also included in the studies, because it has a higher fluorine content than the aromatic compounds, which could result in a lower dielectric constant.

For the deposition experiments, argon at 100 mbar was used as carrier gas. The precursors were introduced at a constant rate of approximately 1 mmol/min, and the RF-generator was operating at 375 MHz at a power input of 20W. The substrate, a silicon wafer, was heated to various temperatures between 25 °C and 400 °C.

The first parameter studied in this series was the deposition rate in dependence of the substrate temperature. A minimum rate of 100 nm/min is desirable to sustain sufficient processing speed for chip production. Scheme 7 shows the results.



Scheme 7: Deposition rate at various substrate temperatures for:
octafluorotoluene (OFT), -●-; hexaflurobenzene (HFB), -▲-; tetrafluorobenzene (TFB), -■-; perfluorodecaline (PFD), -O-

All aromatic precursors tested here deposit sufficiently fast even at a substrate temperature of 400 °C. From perfluorodecaline no films are deposited at this high temperature. Even at 25 °C, film deposition from perfluorodecalin is slow under the conditions employed in our study. The thermal stability of the films from hexafluorobenzene and tetrafluorobenzene is acceptable. They show a weight loss of 3% at 400 °C within the first hour, but there is no further weight loss at this temperature after that. The film thickness remains constant during the initial weight loss, indicating that the weight loss is probably due to desorption of low molar mass absorbed oligomers rather than thermal decomposition of the films. Films deposited at lower substrate temperatures exhibit unsatisfactory thermal stability.

The adhesion of the films from hexafluorobenzene and tetrafluorobenzene also depends on the substrate temperature during deposition. While adhesion of films prepared as 25 °C is insufficient (tape test after scratching), adhesion to Si and Al of the films deposited at 400 °C is good. However, octafluorotoluene did not result in films with good adhesion under any conditions in the present study. All Films deposited at 400°C were insoluble in AZ thinner (a common solvent for photoresists, consisting of a mixture of aliphatic ethers), while some material could be extracted from those films prepared at 25°C from the aromatic precursors. Films deposited from perfluorodecaline were always insoluble. Internal stress in the films deposited at 400 °C is high. For hexafluorobenzene, it increases from 40 MPa for films deposited at 25°C to 190MPa for films deposited at 400°C. All films deposited in this study exhibited no detectable moisture absorption (« 1%).

The dielectric constants of the films deposited from the four precursors in this study are summarized in Table 8.

Table 8: Dielectric constants of the films prepared by plasma deposition

| Precursor | Deposition temperature °C | Dielectric constant at 100 kHz |
|---|---|---|
| **Perfluorodecalin** | 25 | 2.5-2.7 |
| | 400 | no film |
| **Octafluorotoluene** | 25 | 2.6-2.8 |
| | 400 | not determined |
| **Tetrafluorobenzene** | 25 | 2.7-2.9 |
| | 400 | 3.0-3.2 |
| **Hexafluorobenzene** | 25 | 2.8-3.0 |
| | 400 | 3.0-3.2 |

As can be seen from Table 8, the dielectric constants of the films prepared in this study are not as low as expected initially. IR spectra of the films reveal the presence of carbonyl functions in the deposited films. These are present even if the most stringent inert gas conditions are used during deposition. At present, in our opinion the presence of oxygen in the films is caused by reaction of oxygen with trapped free radicals during handling outside of the deposition chamber. So far, we have not succeeded in developing a deposition procedure which limits the number of trapped radicals to a value where this reaction with oxygen is no longer important.

## Conclusion

Polymers with potential use as high temperature low-ϵ-dielectric materials for spin coating as well as plasma deposition were studied. Starting from a series of heteroaromatic poly(aryl ether)s with oxazole rings and trifluoromethyl substituents, the thermal stability and glass transition temperatures could be increased by changing the chemical structure of the repeating unit. The introduction of terphenyl groups into the polymer backbone did result in the expected improvement of the decomposition temperature, but it did not result in the expected increase in glass transition temperature. Only when quadriphenyl groups were used instead of the terphenyl units high $T_g$'s of the order of 300 °C were achieved. These materials also exhibit excellent mechanical properties. Measurements of the dielectric constants are in progress. Based on the chemical structure, the prospects are promising because of the absence of polar groups (besides the ether bridges) and the presence of the trifluoromethyl side groups.

The conditions for plasma polymerization of hexafluorobenzene and tetrafluorobenzene could be adjusted in order to produce films at sufficient deposition rates with thermal stability in excess of 400°C and good adhesion to silicon and aluminum. The residual stress in these films is very high, and the dielectric constants are not as low as expected. The deposition conditions need to be improved further in order to reduce ϵ to values around 2.5. A deposition protocol has to be developed which avoids entrapment of large numbers of free radicals. The addition of hydrogen or hydrogen sources towards the end of the deposition process or the use of mixtures of argon and hydrogen containing gases (possibly hydrocarbons) for the deposition has to be checked in the future. This should result in elimination of at least a part of the entrapped radicals.

## Acknowledgement

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## References

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