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6. AUTHOR(S) Alex K-Y. Jen Current Address: Department of Materials Science & Engineering, Box 352120 University of Washington, Seattle, WA 98195-2120				5b. GRANT NUMBER F49620-97-1-0240	
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14. ABSTRACT
We have made significant breakthrough in the E-O polymers via the successful development of chromophores and polymer synthesis. A series of highly efficient, chemically and thermally stable (>310 °C) nonlinear optical chromophores were prepared through the replacement of the most reactive CN group in the tricyanovinylthiophene derivatives with perfluoroaryl units. In addition, we have demonstrated a new and efficient approach for synthesizing novel second-order NLO polymers by utilizing radical-mediated, stepwise cycloaddition polymerization reaction. The resulting NLO PFCB polymers exhibited excellent processability, small optical anisotropy, low optical loss, and very good thermal, nonlinear optical and mechanical properties. The extension of this method to other functional PFCB polymers for light-emitting diodes is currently in progress.

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High Performance Electro-optic Polymeric Material Systems for Device Applications

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May 1, 2000

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Program Objective:

The goal of our effort is to develop high performance Electro-optic (E-O) polymeric material systems with optimized nonlinearity, thermal, chemical and photochemical stability that are suitable for the design and fabrication of reliable integrated E-O devices.

Status of Effort:

Our work this year has focused on: 1) design and synthesis of highly efficient, nonlinear optical chromophores with optimized thermal, chemical, and photochemical stability, 2) development of new synthetic schemes to covalently attach chromophores onto high temperature polymers and effectively "harden" these materials to facilitate the processes of fabricating single-mode channel waveguides and efficient electric field poling.

Accomplishments and New Findings:

We have made significant breakthrough in the E-O polymers via the successful development of chromophores and polymer synthesis.

*** Chromophore Development**

A series of highly efficient, chemically and thermally stable ($>310\text{ }^{\circ}\text{C}$) nonlinear optical chromophores were prepared through the replacement of the most reactive CN group in the tricyanovinylthiophene derivatives with perfluoroaryl units.

In practice, polymer based electro-optic (E-O) devices require that nonlinear optical (NLO) polymers possess excellent E-O properties. The large E-O coefficient (r_{33}) of a polymer is generally realized by aligning the dipole moment (μ) of efficient NLO chromophores with a high external electric field at a temperature above the glass-transition temperature (T_g) of the polymer. Therefore, chromophores with large molecular nonlinearity (β) are crucial to the advancement of NLO polymers, and have drawn interests upon their theory-guided design and state-of-the-art synthesis. Among them, a series of highly polarizable chromophores based on the tricyanovinyl-substituted heteroaromatic chromophores have demonstrated very large $\beta\mu$ values. Although poled E-O polymers with these chromophores incorporated either as a guest in a host polymer or as a side-chain polymer have exhibited large electro-optic coefficients (r_{33}), there are several deficiencies associated with these materials. (Figure 1a) For instance, the α -position of the tricyanovinyl acceptor is very susceptible to attack from nucleophiles which may easily diminish the nonlinearity of the chromophores. On the other hand, the flat structure of these highly charge-transfer chromophores have a strong tendency to form aggregates in a polymer matrix due to intermolecular electrostatic interactions. This results in a lower poling efficiency and a higher light-scattering optical loss. To overcome these problems, a strategy was designed to replace the α -CN with a bulky and electron-deficient moiety which is also inherently not a good leaving group. In this paper, the tetrafluoropyridinyl-dicyanovinyl (TFPD) and the heptafluorotolyl-dicyanovinyl (HFTD) acceptors were adapted to improve the efficiency, chemical and thermal stability of NLO chromophores. As shown in the Figure 1b, the perfluoroaryl group blocks the α -position, prevents an attack from amine nucleophiles⁴ and

greatly improves the chemical stability of the derived chromophores. In addition, due to their electron-deficient characteristics, these perfluoroaryl groups will enhance the strength of the dicyanovinyl acceptor, and subsequently increase the β of the chromophores. Furthermore, from the simple MOPAC molecular modeling, the perfluoroaryl group is twisted out of the main conjugation plane due to steric hindrance. The 3-D structure may help to prevent intermolecular electrostatic interactions among the chromophores, which in turn, may enhance the poling efficiency and decrease the scattering-induced optical loss.

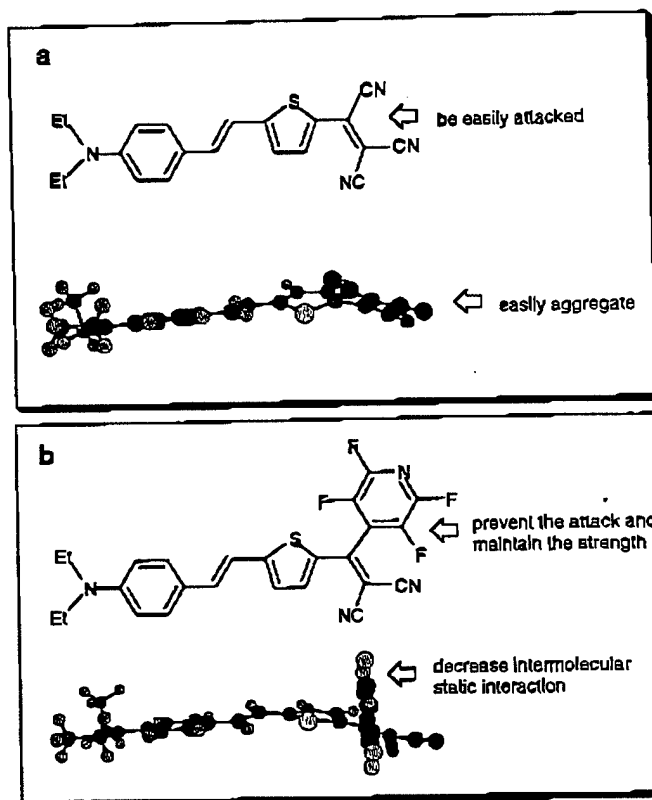
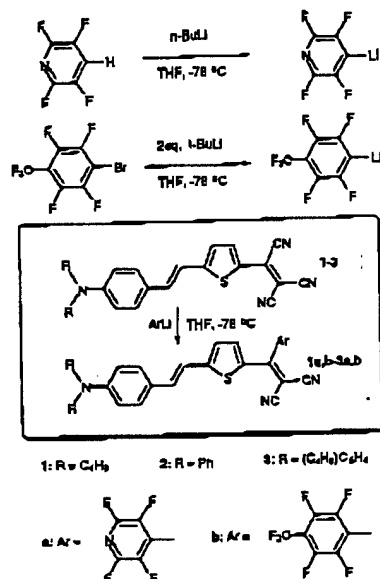


Figure 1

The general synthetic route for the α -perfluoroaryl-dicyanovinyl containing chromophores is shown in Scheme 1. The tetrafluoropyridinyl lithium was prepared by the lithiation of tetrafluoropyridine with *n*-BuLi at -70 °C. The heptafluorotolyl lithium was generated by the lithium-halogen exchange reaction of the *p*-heptafluorotolyl bromide with 2 eq. of *t*-BuLi at -78 °C. To the prepared perfluoroaryl lithium solutions in THF at -70 °C, tricyanovinyl chromophores were added neat in one portion and the reaction mixtures were stirred at -70 °C for 1 hour then warmed slowly to room temperature to give compounds **1a-3a** and **1b-3b** after purification by silica gel chromatography. All of the compounds were fully characterized by ¹H NMR and elemental analysis.

The chemical stability of the chromophores to a nucleophile was tested in a chloroform solution that was saturated with a large excess of diethylamine. The UV-vis absorption spectra of these perfluoroaryl-dicyanovinyl containing chromophores showed excellent chemical

stability in a nucleophilic environment (Figure 2), unlike their tricyanovinyl analog which was almost instantaneously decolorized.



Scheme 1

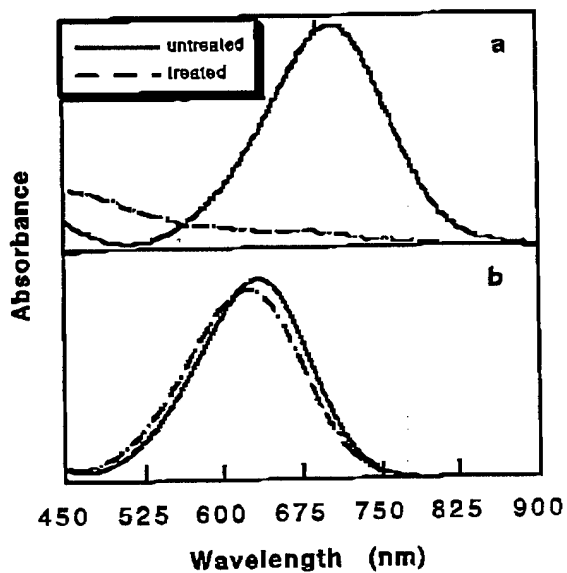


Figure 2

The charge-transfer (CT) properties, electrochemical properties, and intrinsic thermal stability of these chromophores are shown in Table 1. These perfluoroaryl-dicyanovinyl containing chromophores have a blue-shifted CT band when compared to their corresponding tricyanovinyl counterparts. For instance, the λ_{max} of **1a** and **1b** in dioxane are located at 595 and 590 nm, respectively while **1** is at 640 nm. The further deviation of the absorption band from the operating wavelengths of the lasers for telecommunication may help to decrease the absorption optical loss. However, the blue-shifted λ_{max} of both chromophores also indicated a decrease in strength of the new electron acceptors. This notion was supported by the electrochemical measurements using cyclic voltammetry (CV). The new chromophores **1a** and **1b** both possess higher reduction potentials at -1.02 V and -1.04 V, respectively when compared to -0.80 V for their parent compound **1**.

	λ_{max} (nm) ^a	E°_{Red} (V) ^b	E°_{Ox} (V) ^b	T_d (°C) ^c
1	640	-0.80	0.56	240
2	601	N/A	N/A	315
3	625	N/A	N/A	315
1a	595	-1.02	0.52	320
2a	554	-0.98	0.73	390
3a	577	-1.01	0.61	360
1b	591	-1.04	0.58	310
2b	563	-1.03	0.72	340
3b	573	-1.04	0.61	370

a) In a dilute dioxane solution

b) in a $\text{CICH}_2\text{CH}_2\text{Cl}$ solution of Bu_3NPF_6 (0.1 M), v.s. Ag^+/Ag

c) sealed-pan DSC measurement in N_2 , 20 °C/min

Table 1

Thermal stability measurements using the sealed pan differential scanning calorimetry (DSC) revealed that these chromophores possess excellent thermal stability (310-390 °C) which is much higher than that of **1** (240 °C).

The optical and E-O studies were performed on the polymers (guest/host) in which chromophores **1a** and **1b** (25 wt.%) were formulated into polyquinoline (PQ-100). Optical quality thin films were spin-coated onto glass slides and indium-tin-oxide (ITO) glass substrates using a 12 % w/w solution of the resin in cyclopentanone. These films were heated at 85 °C in vacuum for 24 h and then were briefly baked on a hot stage (160 °C) under nitrogen for 20 min to remove the residual solvent. The dipole alignment of the chromophores was achieved by poling with a high electric field of 1.0 MV/cm at 200 °C. The r_{33} values of NLO polyquinoline films of **1a** and **1b** were 32 pm/V and 28 pm/V at 1.3 μm . The slight decrease in the E-O coefficients when compared to **1** may be attributed to several factors, such as the lower number density of **1a** and **1b** because of the higher molecular weights of these compounds, and the weaker electron withdrawing ability of perfluoroaryl-dicyanovinyl.

The optical loss of the NLO polymer films (< 2 dB/cm measured at 1.3 μm) is much less than the 6.0 dB/cm for a similar film contains a tricyanovinyl substituted chromophore. This

supports our hypothesis that the 3-D structure of these perfluoroaryldicyanvinyl groups will prevent chromophores from forming aggregates.

•* Electro-optic polymers development

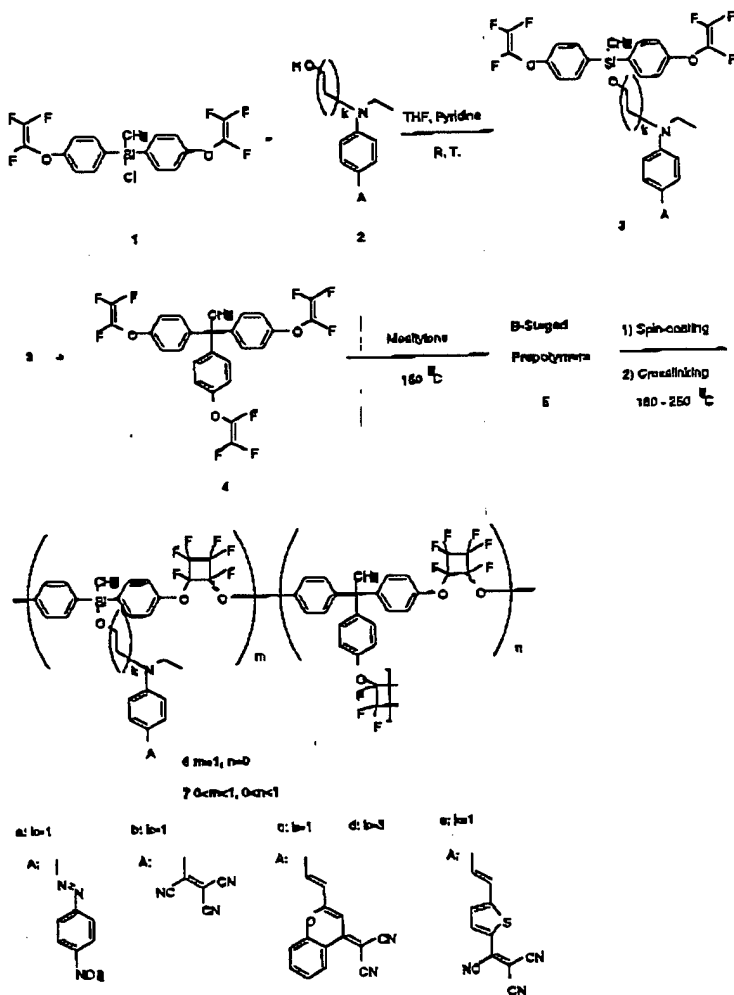
A series of novel second-order nonlinear optical (NLO) thermoset polymers containing silicon-perfluorocyclobutane (PFCB) have been developed. The synthesis was accomplished using the crosslinking reaction between the di(trifluorovinylether)-containing NLO chromophores and the tris(trifluorovinylether) monomer in a solid state at 180-250 °C. The radical-mediated, stepwise cycloaddition reaction offers great tolerance to very sensitive functional groups, such as the tricyanovinyl acceptor. A variety of NLO chromophores could be readily incorporated into these thermoset polymers. The preliminary results have indicated these polymers to possess excellent processability, low optical loss, and a combination of the highly desirable thermal, nonlinear optical, and mechanical properties.

In the past decade, a large number of high $\beta\mu$ chromophores has been synthesized and some of these exhibit very large macroscopic nonlinearities in guest/host poled polymers. However, in order to realize a stable dipole alignment, it is a critical prerequisite to utilize high glass-transition temperature (T_g) polymers with NLO chromophores as side-chain, or cross-linkable polymers with NLO chromophores that were locked in the polymer network. Typically, most of these materials are synthesized either by conventional radical polymerization of chromophore-containing monomers or by step-growth condensation reactions that yield polymers, such as polyacrylates, polyurethanes, polyimides or polyquinolines. Unfortunately, both of these strategies have major drawbacks that limit their use. In radical polymerization, the high charge-transfer chromophores often act as retarders or inhibitors to the reaction and thereby decrease the yield and molecular weight of the polymers. Besides, many of the NLO chromophores can not survive the conditions of radical polymerization. In the step-growth reactions, the monomers usually need to tolerate the harsh acidic/basic conditions at high temperatures.

Recently, perfluorocyclobutane(PFCB)-containing polymers have been studied as a unique class of fluoropolymers with a combination of excellent properties, such as low dielectric constant, good thermal stability, high glass-transition temperature, and optical transparency. PFCB polymers are prepared by the radical-mediated thermal cyclo-polymerization of trifluorovinyl ethers. The versatility of this methodology was demonstrated by developing microelectronics laminates, low dielectric coatings, and optical waveguides. In this study, we report a novel synthetic approach for incorporating second-order NLO chromophores into PFCB polymer for E-O applications, utilizing the well-defined nature and the unique combination of properties offered by PFCB polymers. A broad variety of NLO chromophores,¹² including those containing sensitive functional groups such as the tricyanovinyl acceptor, could be easily incorporated into PFCB polymers without decomposing chromophores or inhibiting polymerization (Scheme 1).

NLO chromophore-containing monomers 3a-e were prepared in high yields (>95%) by the condensation reaction between di(trifluorovinylether)silylchloride and hydroxy-containing chromophores at ambient temperature under mild conditions¹⁴ as described in Scheme 1. All of the monomers were purified by column chromatography and fully characterized using ¹H NMR and elemental analysis. Exothermic cyclodimerization of trifluorovinylether groups in these NLO chromophores are typically detected just above 140 °C by DSC (10 °C/min) with reaction

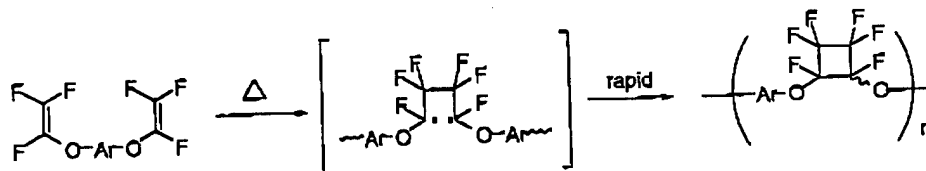
profiles similar to that of general di(trifluorovinylether) compounds. For example, the reaction of **3c** showed a peak polymerization temperature at 243 °C and gave an enthalpy of -16 kcal/mol per perfluoroalkene linkage. Consequently, polymerization was accomplished by simply heating the neat monomers or the solution of monomers under an inert atmosphere at 150-250 °C. The NLO chromophores remained intact since the polymerization process is a radical-mediated stepwise addition that is followed by a rapid ring cyclization (Scheme 2). This is very different from the conventional radical and condensation polymerization.



Scheme 1

The linear NLO polymers **6a-e** obtained by bulk polymerization of **3** at 180-250 °C had molecular weights ranging from 30,000 to 60,000 but relatively low T_g s in the range of 90-140 °C due to high chromophore concentrations (38-51 wt %). In order to adjust the chromophore loading density to a suitable level for poling studies, monomers **3** and **4** were copolymerized at 150 °C in mesitylene at a solution concentration of 35-45 wt% to yield reactive oligomer/monomer mixtures with controlled molecular weights (Scheme 1). The B-staged prepolymers **5** possess very good solubility in the mixture of mesitylene and cyclopentanone (30-

40 %, w/w). Optical quality thin films can be easily prepared from these solutions. The prepolymers have low T_g s or melting points (T_m s) that allow them to be melted, reflowed and efficiently crosslinked in thin films at 180-250 °C. All of the resulting NLO PFCB thermosets have high T_g (>180 °C) and excellent thermal stability (Table 1). Weight losses of <3% up to 350 °C were observed by thermogravimetric analysis (TGA) for all of the NLO polymers. Most importantly, these NLO PFCB thermosets possessed excellent solvent resistance, which is highly desirable for multilayer process in the fabrication of E-O devices.



Scheme 2

Polymers	Chromophore density (w/w%)	T_g (°C) ^a	Weight loss at 350 °C ^b (%)	λ_{max} (nm) ^c	n^d		Loss (dB/cm) ^d
					TE	TM	
7b	30	185	0.9	506	1.5855	1.5840	6.0
7c	25	230	0.2	502	1.5670	1.5634	2.7
7d	20	195	0.8	511	1.5454	1.5413	2.4
7e	25	192	2.4	636	1.5752	1.5711	2.9

^a DSC (10 °C/min) in nitrogen. ^b TGA (10 °C/min) in nitrogen. ^c In film. ^d At 830 nm for polymer 7b-7d and at 1300 nm for polymer 7e.

Table 1

In order to measure the stability of NLO chromophore during the crosslinking reaction, thin film samples of the NLO prepolymers were isothermally heated on a hot stage at temperatures between 180 and 275 °C under N_2 at 25 °C intervals for 20 min. The π - π^* charge transfer absorption band of the chromophores was used to monitor the extent of decomposition. NLO prepolymer 7c possessed a high stability similar to that of the side-chain polyquinoline with the same NLO chromophore (Figure 1), indicating that the chromophore can tolerate the radical intermediate during the polymerization. It is noteworthy that highly efficient tricyanovinyl-styrylthiophene-based chromophore can also be incorporated into the thermoset NLO PFCB polymer 7e.

The NLO polymers have refractive indices ranging from 1.541 to 1.586 with low birefringence (0.002-0.004) and optical loss (2-3 dB/cm) for polymer 7c-d at 0.83 μ m and polymer 7e at 1.3 μ m. (Table 1) They were measured by the prism coupling and streak method in a Metricon system using 2-3 μ m thin films on glass substrates. The low optical anisotropy may be attributed to the isotropic structures of these polymers originating from the equal amount of *cis* and *trans* linkages in the 1,2-perfluorocyclobutyl ether moieties. For E-O measurements, the B-staged prepolymer solutions in mesitylene/cyclopentanone (30-40%, w/w solution, filtered through a 0.2 μ m syringe filter) were spin-coated onto an indium tin oxide (ITO) glass substrate.

The films were heated in a vacuum oven at 180 °C for 0.5 h to ensure removal of the residual solvent. Thin gold electrodes (~20 nm) were sputtered on top of the resulting 1-2 μm polymer thin films. The reactive prepolymers were advanced by heating at 190-250 °C under nitrogen. The dipole alignments in the NLO polymers were achieved by contact poling. Preliminary poling results showed that these thermoset NLO PFCB polymers possessed large r_{33} s and good temporal stability. Polymer 7e, for example, exhibited an r_{33} value of 21 pm/V at 1.3 μm under a poling field of 1.0 MV/cm. The r_{33} value of polymer 7e retained approximately 80% of its original value at 85 °C for more than 1000 hours. Further optimizations of temporal stability and E-O activity of these materials are currently under investigation.

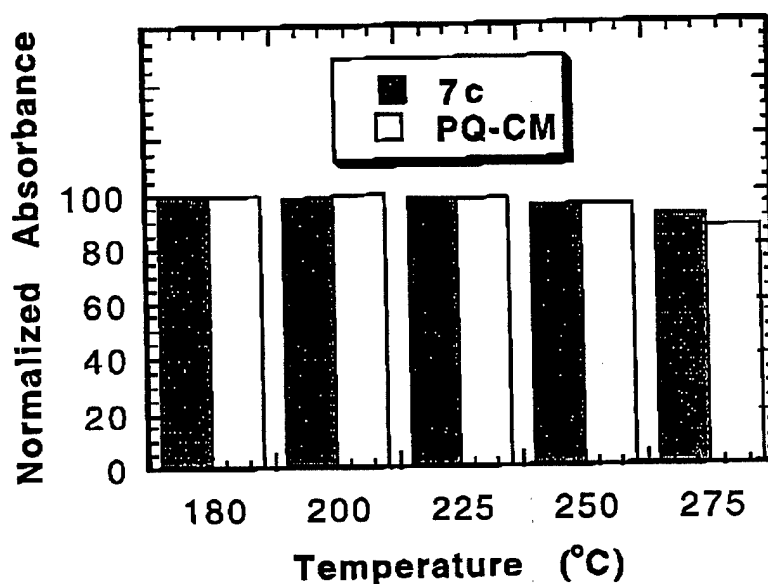


Figure 1

In conclusion, we have demonstrated a new and efficient approach for synthesizing novel second-order NLO polymers by utilizing radical-mediated, stepwise cycloaddition polymerization reaction. The resulting NLO PFCB polymers exhibited excellent processability, small optical anisotropy, low optical loss, and very good thermal, nonlinear optical and mechanical properties. The extension of this method to other functional PFCB polymers for light-emitting diodes is currently in progress.

Personnel

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- Total number of Full-Time equivalent Graduate Students and Post-doctoral Associates supported during this period under F49620-97-1-0240

Graduate Students: 1
 Postdoctoral Associate: 1
 including number of
 Female graduate students: 0
 Female Post-doctoral Associates: 0
 Minority* graduate students: 0
 Minority* Postdoctoral Associates: 0
 Asian Graduate Students: 1
 Asian Postdoctoral Associates: 1

Professor: Alex Jen (1 month); Postdoc: Hong Ma (12 months); Graduate Student: Sen Liu (12 months).

• Publications

- a. Number of papers submitted to referred Journals, but not published: 5
- b. +Numbers of papers published in refereed journals (for each, please provide a complete citation): 2
- c. +Number of books or chapters submitted, but not yet published: 0
- d. +Number of books or chapters published (for each, provide a complete citation): 0
- e. +Number of printed technical reports/non-refereed papers (for each, provide a complete citation): 2
- f. +Number of patents filed: 0
- g. +Number of patents granted (for each, provide a complete citation): 0
- h. +Number of invited presentations (for each, provide a complete citation): 4
- i. +Number of submitted presentations (for each, provide a complete citation): 3
- j. +Honors/Awards/Prizes for contract/grant employees: Alex Jen received the Boeing-Johnson Professorship

2. Papers submitted to refereed journals:

1. "A Novel Class of High Performance Perfluorocyclobutane-Containing Polymers for Second-Order Nonlinear Optics", H. Ma, J. Wu, P. Herguth, B. Chen and A. K-Y. Jen, *Chem. Mater.* (in press)
2. "Polyquinolines: Multifunctional Polymers for Electro-optic and Light-emitting Applications", A. K-Y. Jen and H. Ma, *Mat. Res. Soc. Proc.* (in press).
3. "Realization of Polymeric Electro-optic Modulators with Less Than one Volt Drive Voltage Requirement", C. Zhang, M. Lee, A. Winkleman, H. Northcroft, C. Lindsey, A. K-Y. Jen, T. Londergan, W. H. Steier, and L. R. Dalton, *Mat. Res. Soc. Proc.*, (in press).
4. "High Performance Side-Chain Polyquinolines and Perfluorocyclobutane-Containing Thermoset Polymers for Electro-optic Applications", H. Ma, J. Wu, X. Wu, P. Herguth, B. Chen, and A. K-Y. Jen, *Mat. Res. Soc. Proc.*, 1999, (in press)

5. "Design, Synthesis and Characterization of Tetracyanobutadienyl-Containing Nonlinear Optical Chromophores", X. M. Wu, J. Wu, and A. K-Y. Jen, *Mat. Res. Soc. Proc.*, 1999, (in press).

b) **Papers published in refereed journals**

1. "Design and Synthesis of Highly Efficient Chromophores and Polymers for Electro-optic Applications", A. K-Y. Jen, H. Ma, X. Wu, J. Wu, S. Liu, P. Herguth, S. R. Marder, C. F. Shu and L. R. Dalton, *Nonlinear Optics*, 1999, **22**, 9.
2. "Synthesis and Characterization of Highly Efficient, Chemically and Thermally Stable Chromophores with Chromone-containing Electron Acceptors for Nonlinear Optical Applications", A. K-Y. Jen, Y. Liu, L. Zheng, S. Liu, K. J. Drost, and Y. Zhang, *Adv. Mater.*, 1999, **11(6)**, 452.
3. "Highly Efficient, Thermally and Chemically Stable Second-order Nonlinear Optical Chromophores with a 2-Phenyl-tetracyanobutadienyl Acceptor", X. Wu, J. Wu, and A. K-Y. Jen, *J. Am. Chem. Soc.*, 1999, **121(2)**, 472.
4. "A Modular Approach of Functionalizing Aromatic Polyquinolines for Electro-optic Devices", H. Ma, A. K-Y. Jen, J. Wu, X. Wu, S. Liu, C-F. Shu, L. R. Dalton, S. Thayumanavan and S. R. Marder, *Chem. Mater.* 1999, **11**, 2218.
5. "The Molecular and Supramolecular Engineering of Polymeric Electro-optic Materials", B. H. Robinson, L. R. Dalton, A. W. Harper, A. Ren, F. Wang, C. Zhang, G. Todrovac, M. Lee, R. Aniszfeld, S. Garner, A. Chen, W. H. Steier, S. Houbrechte, A. Persoon, I. Ledoux, J. Zyss, and A. K-Y. Jen, *Chem. Phys.*, 1999, **245**, 35.
6. "Synthesis and Characterization of Nonlinear Optical Chromophores with Conformation-locked Polyenes Possessing Enhanced Thermal Stability", Y. Shu, Z-H. Gong, C-F. Shu, E. M. Breitung, R. J. McMahon, G-H. Lee and A. K-Y. Jen, *Chem. Mater.*, 1999, **11**, 1628.
7. "From Molecules to Opto-Chips: Organic Electro-Optic Materials," L. R. Dalton, L. Irwin, B. Carlson, F. Fiffield, G. Phelan, C. Kincaid, J. Amend, C. Zhang, A. Ren, M. Lee, G. Todorov, S. M. Garner, A. Chen, W. H. Steier, H. Fetterman, and A. K. Y. Jen, *J. Mater. Chem.*, 1999, **9**, 190.
8. "Highly Efficient, Thermally and Chemically Stable Nonlinear Optical Chromophores Based on The α -Perfluoroaryl-dicyanovinyl Electron Acceptors", X. Wu, J. Wu, Y. Liu and A. K-Y. Jen, *Chem. Commun.*, 1999, **23**, 2391.
9. "Thermal Poling of Soda-Lime Glass for hybrid Glass/Polymer Electro-optic Modulators", Y. Enami, P. Poyhonen, D. Mathine, A. Bashar, M. Paratheepan, S. R. Marder, S. Honkanen, B. Kippelen, N. Peyghambarian, A. K-Y. Jen, J. Wu, *Appl. Phys. Lett.*, 2000, **76(9)**, 1086.

c) **Book chapters published**

N/A

d) Printed technical reports

1. "Recent Progress of Highly Efficient Chromophores and Polymers for Electro-optic Devices", A. K-Y. Jen, H. Ma, X-M. Wu, J-Y. Wu, S. Liu, S. R. Marder, L. R. Dalton and C-F. Shu, *Proc.SPIE*, 1999, **3623**, 112.
2. "High Performance Perfluorocyclobutane-Containing Polymers for Electro-Optic and Light-Emitting Applications", A. K-Y. Jen, H. Ma, J. Wu, S. Liu, P. Herguth, X. Jiang, M. Liu, B. Chen, and L. Zheng, *Polymer Print*, 2000, **41(1)**, 774.

Interactions-Transition**a) Invited presentations**

1. Alex Jen, "Binaphthyl-Based Conjugated Polymers for Light-Emitting Diodes", Society of Information and Displays, San Diego, CA., November 7, 1999.
2. Alex Jen, "High Performance Perfluorocyclobutane-Containing Polymers for Electro-Optic and Light-Emitting Applications", American Chemical Society National Meeting, San Francisco, CA., March 28, 2000.
3. Alex Jen, "High-Performance Electro-Optic Materials: From Molecules to Device Applications", Bay Area Golden Gate Polymer Forum, CA, May 11, 1999.
4. Alex Jen, "Multifunctional Polyquinolines for Electro-Optic and Light-Emitting Applications", Materials Research Society Meeting, San Francisco, CA, April 26, 1999.

b) Contributed presentations

1. Alex Jen, "High-Performance Side-Chain Polyquinolines and Perfluorocyclobutane-Containing Thermoset Polymers for Electro-Optic Applications", Materials Research Society Meeting, Boston, MA, November 29, 1999.
2. Alex Jen, "Poly(binaphthylenevinylene-alt-phenylene vinylene) Derivatives: Novel Luminescent Polymers for Light-Emitting Devices", Materials Research Society Meeting, Boston, MA, November 29, 1999.
3. Alex Jen, "Multifunctional Polyquinolines for Electro-Optic and Light-Emitting Applications", Santa Clara, CA, October 18, 1999.

c) Transition

We have been working with Lockheed-Martin (Susan Ermer), U. of Arizona (Dave Methine) to transfer the high performance E-O polymers for testing and fabrication of devices. In particular, Lockheed-Martin is interested in using these materials for the fabrication of high speed E-O modulators. University of Arizona's Optical Science Center is interested in using the highly efficient polymers that we developed for the fabrication of high speed glass/polymer hybrid modulators.