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POLYMERIC MATERIALS

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**Polymers Branch
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Final Report**

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14. ABSTRACT This report summarizes the technical progress of the in-house projects supporting the Polymeric Materials Effort, Materials and Manufacturing Directorate, Air Force Research Laboratory. These include structurally and morphologically tailored materials for improved photovoltaic and molecular electronic responses; bio-derived photonic assemblies; advanced polymeric materials; polymer nanostructured materials; and polymer nanostructured materials (PNMs).					
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Table of Contents

Section	Page
1.0 Structurally and Morphologically Tailored Materials for Improved Photovoltaic and Molecular Electronic Responses.....	1
1.1 Research Objectives	1
1.2 Summary of Progress	1
2.0 Bio-Derived Photonic Assemblies.....	3
2.1 Research Objectives	3
2.2 Summary of Progress	3
3.0 Advanced Polymeric Materials.....	4
3.1 Research Objectives	4
3.2 Summary of Progress	4
4.0 Polymer Nanostructured Materials (PNMs)	7
4.1 Research Objectives	7
4.2 Summary of Progress	7
5.0 Publications (Refereed)	11
5.1 Publications (Unrefereed).....	22
5.2 Publications (Books, Articles, Other)	35
5.3 Invention Disclosures and Patents.....	37
List of Acronyms, Abbreviations, and Symbols	38

1.0 Structurally and Morphologically Tailored Materials for Improved Photovoltaic and Molecular Electronic Responses

1.1 Research Objectives:

1. Model electron-sponges, materials capable of multiple reversible electrochemical reductions, at theoretical levels adequate to treat the complex electron-electron and electron-lattice interactions intrinsic to such redox processes.
2. Prepare and characterize electronic-accepting molecules and n-dopable polymers based on thiazolothiazole and thieno[3,4-b]thiophene materials expected to have good electron-accepting properties and enhanced charge mobilities.
3. Evaluate materials and structures for improved charge transport characteristics within photovoltaic devices.
4. Evaluate novel processing techniques for control of the nanoscale architecture of thin films for photovoltaic device applications.

1.2 Summary of Progress:

FY04 saw a number of important developments in meeting all four objectives. The level of theory required to accurately map geometric distortions in fullerenes was better defined. This enables the electronic-lattice coupling to be modeled, which is of central importance in electron transfer processes. Advances in the synthesis of thiazolo[5,4-b]thiazoles paved the way for developing monomers with good polymer-forming reactions. The electron-accepting capability of thiazolo[5,4-b]thiazoles was modeled by high level calculations and found to exceed that of C60 even for a short (i.e. pentamer) oligomer. Photovoltaic cells were fabricated and evaluated using thienylvinylene polymers with bulk heterojunction cells. Nanotubules of TiO₂ were grown via templating within nanoporous Al₂O₃ films. For FY04 there were refereed publications, unrefereed publications, patents, book chapters, and presentations (of which seven were invited).

Progress was made for all four objectives in FY05. The electron attachment energy for the C₁₂₂ dianion was found to be significantly endothermic which is counter intuitive. Modeling reorganization energies of electron-accepting materials has led to a number of promising structures for enhanced mobility. Novel PBZT-thienylthiophene polymers were prepared and characterized for enhanced spectral response. Previously developed quaternization chemistry was applied to BBL and BBB materials with surprising solubility. Thermal history was found to be significant in determining performance of devices made with alternative conjugated polymers. By optimizing thermal conditioning and combining these new conjugated polymer systems with current state of the art (SOA) devices, the next step is to fabricate 'multi-junction' devices to harvest energy in multiple solar spectral regions. Two new, alternative processing techniques were examined for fabrication and interfacial modification of active devices. Initial efforts demonstrated the utility of holographic photopolymerization for patterning electronically active materials. A proof of concept demonstration will be pursued to show its use in the fabrication of PVs. Layer-by-layer assembly was utilized to deposit ultra-thin, interfacial modification layers of the

conducting polymer, PEDOT, which is routinely used as the buffer layer in PVs, and of the gate dielectric in organic thin film transistors. Further work will involve demonstrating this technique for transistors based on small molecules, such as pentacene. For FY05 there were refereed publications, unrefereed publication, patent, book chapters, and presentations (invited).

2.0 Bio-Derived Photonic Assemblies

2.1 Research Objectives:

The goal of this task is to develop techniques to manipulate naturally occurring periodic materials into mechanically robust, photonic crystal systems possessing 3D band structures that cannot be obtained through traditional methods. Two general approaches will be examined: 1) top-down fabrication based on size reduction of micron-scale periodic structures, such as found in sea urchin skeletons, and 2) bottoms-up fabrication based on assembly of viral particles such as the iridiovirus. In addition, the investigated processing and fabrication techniques will be integrated with conventional microfabrication technologies to optimize potential for integrated optic devices.

2.3 Summary of Progress:

Photonic band gap structures provide enticing platforms to combine large-scale processability (classical motivation driving polymer use) with enhanced photonic properties via exploitation of spatially-defined and locally enhanced optical fields. Current studies have barely scratched the surface of the multitude of possible structures and material properties that might be exploited, but have been limited by the complexity associated with fabrication of these more complex structures. Naturally occurring biomaterials can be used as scaffolds to construct photonic crystals with structures that are difficult/impossible to fabricate by artificial means. Two general areas are being explored within this program: biotemplates and biocolloids, essentially being top-down and bottom-up approaches to photonic crystals. With regard to biotemplates, simulations predict a complete 3D bandgap, given sufficient dielectric contrast (9:1), for the sea urchin *cidaris cidaris* skeleton ("Plumber's Nightmare" surface (space group Pm3m), 50 micron lattice. A novel cyclic size-reduction scheme enabled reduction and replication of the structure by approximately 50% per cycle allowing a tailorable photonic crystal structure in the IR-spectral region. With regard to biocolloids, forced assembly of iridiovirus (200 nm) using ultra-centrifugation and a glutaraldehyde cross-link scheme provided colloidal crystals with reflectivities that depend on pellet thickness and water content. Development of surface patterning techniques to enable directed assembly of the biocolloids have lead to the development of a new AFM-patterning technique based on local resistive heating of polymer film above its glass transition and subsequent electrostatic attraction toward the tip. Since polymer is not degraded in the process, the patterns are reworkable through application of an external thermal field. The program is jointly executed with Prof. Edwin Thomas at MIT. In addition, the program leverages an AOARD grant to Prof. Vernon Ward, Department of Microbiology, and University of Otago, New Zealand.

3.0 Advanced Polymeric Materials

3.1 Research Objectives:

The goal of this task is to provide the fundamental knowledge base to develop high performance polymers for diverse Air Force applications. The research effort focuses on two primary objectives: (1) Unusually high performance and environmental properties in polymeric materials for Air Force structural applications; (2) Desirable photonic properties in organic and polymeric materials having environmental resistance, structural performance, or multi-functional capability.

3.2 Summary of Progress:

FY02 research activities have covered the area of high performance structural polymers (rigid-rod polymers with improved compressive strength, functionalized single-wall carbon nanotubes and hyperbranched polymers) and organic two-photon materials (optical limiting applications and microfabrication). Among several new aromatic and heterocyclic-aromatic hyperbranched polymers prepared, an allyl-terminated hyperbranched poly(ether-ketone-imide) was found to be very effective in reducing the melt viscosity (65x less) when blended with a BMI resin at 4 wt%. Initial mechanical testing results indicated 80-100% improvement in toughness with a two-fold increase in modulus. Also, linear-hyperbranched random copolymerization was found to be effective in suppressing the crystallinity of semicrystalline linear polyetherketone. Unusual solution viscosity behaviors were also noted for a number of hyperbranched benzoxazole-quinoxaline polymers. Research on third-order nonlinear optical materials continues to focus on improving AFX-based two-photon resonant (TPA) materials for the optical power limiting applications and microfabrication. In addition, efforts have been initiated to explore research and application opportunities in nanophotonics and biophotonics in conjunction with an AFOSR-DURINT program on the same theme. During FY02, journal articles (refereed), presentations, and patent actions with task personnel as authors were recorded.

FY03 research activities have covered the areas of high performance structural polymers (block copolymers based on rigid-rod polymers, functionalized carbon nanofiber (CNF) and hyperbranched polymers) and organic two-photon materials (optical limiting applications and microfabrication). The ABA triblock copolymers were prepared from the copolymerization of carboxylic acid-terminated PBZT and an appropriate AB₂ monomer to generate the hyperbranched poly(ether-ketone) A-blocks in polyphosphoric acid. The resulted ABA triblock copolymer with phenoxy-terminated hyperbranched A-blocks were soluble in NMP and that with carboxylic acid end-groups was soluble in alkaline aqueous solution, thus providing the spectroscopic properties of rigid-rod PBZT in non-protonated state in solution for the first time. A new method was developed to covalently attach arylcarbonyl groups (3 in every 100 carbons of the CNF). Preliminary results also indicated that the grafting of a poly(ether-ketone) onto the surfaces of CNF is possible, thus opening the door to a variety of designed blends and composite materials. Research on third-order nonlinear optical materials continues to focus on improving AFX-based two-photon

resonant (TPA) materials for the optical power limiting applications, microfabrication, and novel energy-transfer systems. Specifically, AF-455 has been identified as a very promising material for sensor protection as evaluated by MLPJ. During FY03, 27 journal articles (12 refereed), 17 presentations (6 invited), and 14 patent actions with task personnel as authors were recorded.

FY04 research activities have covered the areas of high performance structural polymers, *viz.* chain-end-functionalized hyperbranched polymers (PAEKI), and *in-situ* nanocomposites based on vapor-grown carbon nanofibers (VGCNF) as well as organic two-photon materials (optical limiting applications and microfabrication). The hyperbranched PAEKI were functionalized with reactive end-groups such as phthalonitrile (PN), ethynyl (ET) and phenylethynyl (PE) that are chemically compatible with the respective high-temperature thermoset resins. PE-PAEKI is being evaluated at MLBC for applicability in the polymer matrix composite program as a high-temperature processing additive for AFR-PE-RTM resins. From the standpoint of affordability, an “ $A_2 + B_3$ ” synthetic methodology was explored with commercially available A_2 -monomers and B_3 cross-linkers. An *in-situ* nanocomposite was successfully generated from the grafting of a *meta*-poly(ether-ketone) onto VGCNF via a Friedel-Crafts acylation in poly(phosphoric acid) with up to 30 wt% VGCNF. Research on third-order nonlinear optical materials continues to focus on improving AFX-based two-photon resonant (TPA) materials for AF needs in sensor protection area with the new emphasis on enhanced excited state absorption and emission quenching under laser irradiation. In addition, some efforts are also directed toward incorporating AFX into optically clear polymer matrices that can lend themselves easily to fibers or windows. Finally, large-scale coarse-grained molecular dynamics simulations of nanomaterials are currently ongoing to better understand what inherent properties are most important to the development of well dispersed nanoparticles. During FY04, publications (refereed), presentations (invited), and patent actions with task personnel as authors were recorded.

FY05 research activities have covered the areas of high performance structural polymers, *viz.* chain-end-functionalized hyperbranched polymers (PAEKI), and *in-situ* nanocomposites based on vapor-grown carbon nanofibers (VGCNF) as well as organic two-photon materials (optical limiting applications and microfabrication). The hyperbranched PAEKI were functionalized with phenylethynyl (PE) group with ortho-, para- and meta-substitution pattern. The thermal analysis results indicated the para-PE has wider processing window than the other two systems. 50 g scale-up synthesis of para-PE-PAEKI was also successful. It is being evaluated at MLBC for applicability in the polymer matrix composite program as a high-temperature processing additive for AFR-PE-RTM resins. Further demonstration of functionalization of VGCNF with the Friedel-Crafts/PPA method developed previously was successful to provide surface-modified VGCNF with an aromatic amine function. *In-situ* polymerization of 6FDA and an aromatic diamine in the presence of amine-grafted VGCNF has afforded the corresponding polyimide composite films with VGCNF content ranging from 0.1-5 wt%. Research on third-order nonlinear optical materials continues to focus on improving AFX-based two-photon resonant (TPA) materials for AF needs in sensor protection area with the new emphasis on enhanced excited state absorption and emission

quenching under laser irradiation. In addition, some efforts are also directed toward incorporating AFX into optically clear polymer matrices that can lend themselves easily to fibers or windows. The possibility of extending the application of TPA materials to CCD camera protection is being examined together with RXPJ. Finally, a molecular dynamics simulation study incorporating the effect of solvent and temperature on the conformation and dynamics of the nanoclay sheets was completed. The result suggests their swelling on reducing the temperature in a poor solvent, based on the preliminary analysis of the equilibrium radius of gyration of the sheet. During FY05, publications (refereed), presentations (invited), and patent actions with task personnel as authors were recorded.

FY06 research activities have covered the areas of high performance structural polymers, *viz.* thermally cross-linkable hyperbranched polymers (PAEKI) functional additive for high temperature thermosets, and *in-situ* nanocomposites based carbon nanofibers and nanotubes as well as two-photon materials (sensor protection and microfabrication). Improvement in the synthesis of the hyperbranched PAEKI functionalized with para-phenylethynyl (PE) has reduced the number of steps from 5 to 3; greatly facilitate the materials availability for further processing optimization studies and fabrication of carbon-fiber composite panels based on AFR-PE-RTM. At 3 additive wt%, PE-PAEKI was able to stabilize the melt viscosity of an AFR-PE-RTM resin at isothermal condition (280 °C). Further demonstration of functionalization of VGCNF with the Friedel-Crafts/PPA method developed previously was successful to provide surface-modified VGCNF with hyperbranched poly(ether-ketone), which drastically improved the solubility/dispersibility of the resulting nanocomposites in common organic and aqueous solvents. Promising extension of this synthetic methodology to MWNT & SWNT is currently underway. Research on third-order nonlinear optical materials continues to focus on improving AFX-based two-photon resonant (TPA) materials for AF needs in sensor protection area with the new emphasis on enhanced excited state absorption and emission quenching under laser irradiation. In addition, some efforts are also directed toward incorporating AFX into optically clear polymer matrices that can lend themselves easily to fibers or windows. The possibility of extending the application of TPA materials to CCD camera protection and space sensor protection is being examined together with MLPJ. During FY06, publications (refereed), presentations (invited), and patent actions with task personnel as authors were recorded.

4.0 Polymer Nanostructured Materials (PNMs)

4.1 Research Objectives:

The Goal of this task is to provide the knowledge base to *develop* and *evaluate* polymer-based nanostructured materials for diverse Air Force applications that require multi-functionality and/or responsivity from the material system. New properties derived from nanoscopic dimensions enable the creation of such materials and the circumvention of classic property-processing trade-offs. The laboratory task focuses on two primary objectives: (1) Creating the fundamental understanding necessary to establish predictable structure-processing-property (SPP) relationships for polymers containing nanoparticle dispersions and (2) demonstrating the feasibility of these polymer nanocomposites to address unique military needs. The establishment of SPP relationships is enabled by innovations ranging from morphological techniques that quantitatively identify a “critical volume element representative of the bulk characteristics,” to novel synthetic methodologies to create nanoparticle assemblies that exhibit liquid-characteristics at room temperature. These fundamental investigations provide the basis to evaluate polymer nanocomposites (PNCs) for Air Force needs including RF/Microwave systems (antennas, negative index materials, RF MEMs switches), “morphing” aero-structures, and self-sensing / self-responsive structures.

4.2 Summary of Progress:

Polymer-based nanostructured materials (PNMs) provide many opportunities to address current technological shortfalls, as well as providing for future military systems. New properties derived from nanoscopic dimensions enable the creation of multi-functional materials, which circumvent classic property-processing trade-offs. To establish feasibility for military needs, current opportunities being examined include:

1. Electrically conductive elastomers for electromagnetic management and shielding,
2. Active materials (stress recovery and dynamic stiffness) for deployables and morphing aerostructures,
3. Piezo- and pyro-resistive PNMs for health monitoring via strain and temperature sensing.
4. Magnetic nanoparticle/polymer nanocomposites for microwave/RF applications,
5. High-temperature thermoset resins,
6. Photonic band gap materials with tailorable susceptibility for light amplification (lasing), optical limiting and nonlinear-optics, and
7. Polymer nanofabrication approaches for device and data storage concepts on flexible substrates.

To enable complete evaluation of the potential of PMNs, pervasive fundamental issues common to these opportunities are addressed, and when required unique modeling and experimental approaches, such as NMR, scattering or nano-probe techniques are developed.

Example efforts during FY03 with noteworthy findings include: 1) emulsion-based PNM synthesis routes, 2) template synthesis of carbon nanotubes with prescribed length and diameter, 3) models, theory and supporting experiment to quantify morphological features of PNMs by x-ray scattering, 4) establishment of NMR facility and application to organic-inorganic interfacial behavior, 5) quantification of nanoparticle response to external fields, such as electrical and shear, 6) utilization of holographic photopolymerization to create switchable organic lasers and 7) detailed development of structure-deformation-property models for remotely actuated stress recovery of nanotube-elastomer nanocomposites. During FY03, journal articles (refereed), presentations (invited), and patent action with task personnel as authors were recorded.

Example efforts during FY04 with noteworthy findings include: 1) development of initial magnetic nanoparticle nanocomposites for RF/microwave evaluation, 2) adhesive approach for creating multicomponent interposed carbon nanotube micro-patterns by region-specific contact transfer and self-assembling on soft-matrices, 3) development of a spin-on-catalyst system for single-wall carbon nanotubes models, 4) demonstration of single-step fabrication of electrically switchable, optically pumped lasing in an organic PBG, 5) impact of deformation on polymer crystallinity within semicrystalline polymer nanocomposites, and 6) detailed development of structure-deformation correlation for conductive nanotube-elastomer nanocomposites. During FY04, 31 journal articles (24 refereed), 47 presentations (18 invited), and 3 patent actions with task personnel as authors were recorded. During FY05, funding for NMR of Inorganic-Organic Interfaces will be transferred to other sources and emphasis will be on the elucidation of structure-property correlations (and the necessary approaches) to facilitate development of mechanically adaptive (actuators), optically adaptive (optically limiting PBGs) and RF/microwave systems.

Example efforts during FY05 with noteworthy findings include: 1) demonstration of tri-axial control of nanoparticle orientation distribution through a uniaxial external processing field (magnetic); 2) development of models to predict electric field impact on alignment, exfoliation and buckling of nanoplates in electric fields; 3) development of HREM techniques for lattice imaging, defect evaluation and observation of local failure modes of layered silicates; 4) demonstration of single-step fabrication of all organic two-dimensional, PBGs with electrically tunable, optically pumped lasing; and 5) synthesis of complex core-shell hard-soft magnetic nanoparticles exhibiting spin coupling. During FY05, 46 journal articles (23 refereed/submitted) and 73 presentations (32 invited) with task personnel as authors were recorded. Additionally, task personnel were interviewed by Popular Mechanics, Aviation Week, High Performance Composites and C&E News. Also, the current task has enabled the successful leveraging of additional external funding from the CIA and ONR as well as the continual growth of complementary computer simulations (PI: Barry Farmer). To enable future growth, developments on soft-matter patterning have been concluded. During FY06, optically adaptive materials for optical limiting applications will be phased-out of this task and solely supported by 96ML03COR (PI: L-S Tan) and AFRL/MLPJ. Emphasis will continue on the elucidation of structure-property correlations

(and the necessary approaches) to facilitate development of optically-triggered, mechanically adaptive and RF/microwave systems as well as to begin to explore molecular and nano-based approaches to tailoring interface adaptivity and to tailor ϵ and μ (e.g. negative n materials).

Example efforts during FY06 with noteworthy findings include: 1) demonstration of utility of cryogenic compounding to enhance dispersion of nanoparticles in B-staged epoxy resins; 2) calculation of the magnitude and decay length of the interaction potential between two sheets of montmorillonite; 3) development of processing techniques to create optically active viral assemblies (highlighted on cover of Advanced Functional Materials); 4) extension of durability of RF MEMs switching by 1 order of magnitude using Au nanoparticle liquids and 5) completion of scale-up 13g of Fe₃O₄ core for preparation of a 5 inch x 5 inch x 40 mil antenna substrate based on Irogran containing a Fe₃O₄/CoFe₂O₄ core/shell nanoparticle. During FY06, 22 journal articles (17 refereed/submitted), 2 patent applications and 49 presentations (25 invited) with task personnel as authors were recorded. Additionally, Dr. Vaia spent 6 months at UCSB Materials Research Laboratory developing concepts for polymer nanocomposite dielectrics, predicting and quantifying nanoparticle-polymer phase behavior, and surface chemistries for interfacial adaptivity. To complement these efforts, collaborative programs with external funding have been established, including membership at the BNLS X27C Beam Line, two visiting ASEE Summer faculty, two EOARD programs, two CINT (Sandia)-funded programs and two approved proposals for neutron scattering time at NIST. To enable future growth, optically adaptive materials for optical limiting applications was phased-out of this task and solely supported by 96ML03COR (PI: L-S Tan) and AFRL/MLPJ. Emphasis will continue on the elucidation of structure-property correlations (and the necessary approaches) to facilitate development of remotely-triggered, mechanically adaptive and RF/microwave systems as well as to begin to explore molecular and nano-based approaches to tailoring interface adaptivity, to tailor ϵ and μ (e.g. negative n materials) and to explore possibilities for polymer nanocomposites dielectrics.

Example efforts during FY07 with noteworthy findings include: 1) determination that electric fields can enhance nanoparticle dispersion (exfoliation) process; 2) development of electron tomography techniques to quantitatively evaluate nanoparticle dispersion in three-dimensions; 3) robust synthesis protocol for controlled purity nanoparticle liquids and subsequent further extension of durability of RF MEMs switching to 3 orders of magnitude over current Au coated SOA surfaces; 4) demonstration of hybrid shape memory foam concept to enable separation of energy storage and shape recovery processes, and 5) fabrication of monodisperse MFe₂O₄ (M= Mn, Y, Co) and Fe₃O₄ spherical nanoparticles, as well as SmFe₂O₄ nanocubes via thermal decomposition and seed-mediated growth methods. To complement these efforts, collaborative programs with external funding have been established, including membership at the BNLS X27C Beam Line, one visiting ASEE Summer faculty, one EOARD program, two CINT (Sandia)-funded programs and six approved proposals from AFRL/ML's FY07 Laboratory Directors Funds. During FY07, 11 journal articles, 5 patent actions and 72 presentations (33 invited) with task personnel as

authors were recorded. FY07 was the last year for this task. The fundamental investigations from this task has provided numerous opportunities for AF development programs (e.g. Shape Memory Polymer Nanocomposites for Morphing Aircraft) as well as revolutionary directions for future fundamental projects focused on single-phase, structured nanoparticle assemblies (e.g. Nanoparticle Liquids, NanoDielectrics, Large Verdet Materials).

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LIST OF ACRONYMS, ABBREVIATIONS, AND SYMBOLS

ACRONYM	DESCRIPTION
AOARD	Asian Office of Aerospace Research and Development
CNF	Carbon Nanofiber
ABA	A polymer with similar ends and a different middle
AFOSR-DURINT	Air Force Office of Scientific Research-Defense University Research Initiative and Nano Technology
NMP	N-Methyl-2-pyrrolidone (a chemical compound)
AFX	Air Force Nonlinear Optical Chromophore
PAEKI	Chain-end-functinalized hyperbranched polymers
VGCNF	Vapor Grown Carbon Nano Fibers
CCD	Charge Coupled Detector
MWNT	Multi Walled Nano Tubes
SWNT	Single Walled Nano Tubes
PNM	Polymer Nanostructured Materials
PNC	Polymer Nanocomposites
NMR	Nuclear Magnetic Resonance
PBG	Photonic Band-gap
CIA	Central Intelligence Agency
ONR	Office of Naval Research
UCSB	University of California-Santa Barbara
ASEE	American Society for Engineering Education
CINT	Center for Intergrated Technologies
NIST	National Institute of Standards and Technology
RF MEM	Radio Frequency Microelectromechanical Systems
SOA	State of the Art
TPA	Two-photon Resonant