PROJECT I.1

FUNCTIONAL MOLECULAR MATERIALS FOR LITHOGRAPHY AND ORGANIC/MOLECULAR ELECTRONICS

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Research orientation

a. Development of Resists for low LER ithography - Optimization of Resist materials and Patterning Processes
Development of resists with potential low Line Edge Roughness (LER) capable for sub-32 nm patterning. Current activities include "molecular" resists, resist based on polymer back-bone breaking and simulation studies for supporting material and process optimization.

b. Lithographic materials for new (unconventional) micro-nano structure fabrication processes
Investigation of material issues for novel radiation-assisted patterning processes, including formation of 3D structures and patterning of biological systems.

c. Materials for organic/molecular electronics
Materials research and device architecture studies for molecular/organic electronics. Current activities include materials for molecular memories, organic light emitting diodes (OLEDs), photovoltaics and new optoelectronic devices.

Funding

- **More Moore**, EU FP6 Integrated Project (IST), 2004-2007
- **Nano2Life**, EU FP6 Network of Excellence (NMP), 2004-2008
- **GSRT-PENED 03ED276**, "Critical sub-100nm Industrial scale Patterns for CMOS - NANO Architectures, (CMOS-NANO), 2005-2008
- **GSRT-NON-EU 467**, "Proton Beam NANOlithography for high aspect ratio structures of optical COMPonents" (PB.NANOCOMP), 2006-2008

MAIN RESULTS

A. Development of Resists for low LER ithography - Optimization of Resist materials and Patterning Processes

Low LER lithography with polycarbocycle – based molecular resist

The new resist family, based on functionalized anthracenes and other polycarbocycles, which has been introduced by our group and Prof. E. Couladouros group at the Inst. of Physical Chemistry, has been proved to be capable for low LER, sub 32 nm lithography, as shown in Fig.1. Evaluation has been carried out in collaboration with LETI - France and Paul Sherrer Institute-Switzerland). The research continues towards performance mprovement through both material and process optimization. The emphasis is given on sensitivity enhancement.

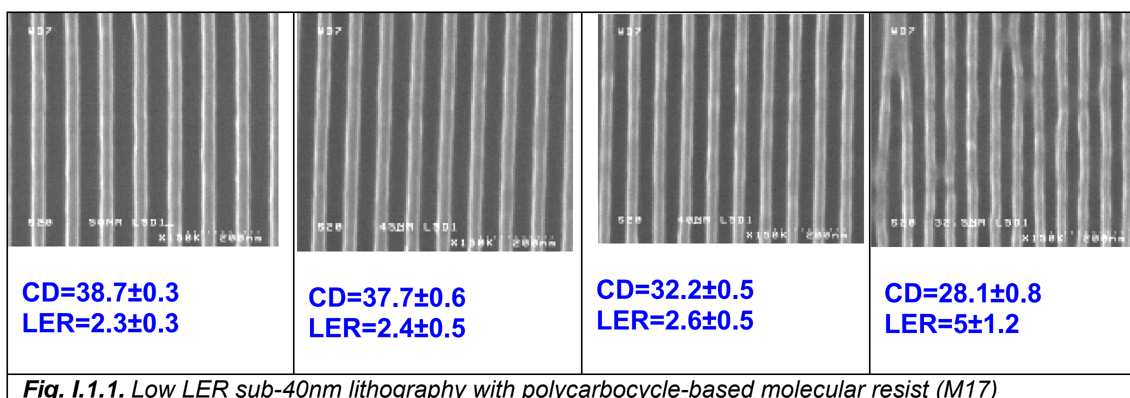

Fig. I.1.1. *Low LER sub-40nm lithography with polycarbocycle-based molecular resist (M17)*

Stochastic simulation studies of molecular resists

The influence of resist material and its architecture becomes very high in the sub-45nm patterning scales in parameters like critical dimension and line-edge roughness (LER). Molecular resist exhibited very low LER compared with polymer chains of the same overall radius of gyration. Two positive molecular resist architecture, (their digital representation is shown in Fig.2), are modeled with our stochastic lithography simulator in order to predict their LER behavior. The architecture and conformations of the overall molecule followed the modeling of a randomly grafted chain and each part of it could be a different chemical group, modeling the different properties of the molecular resist. This algorithmic representation improvement resulted in very compact molecular resist film lattices with very low free volume (5 – 10% depending on the molecular architecture in 2D and even less than 5% in 3D lattices. For M21 molecules with 0.2nm/cell, 20%PAG concentration and 0.6nm diffusion length, the resulting values for a 50nm trench were LER (3σ) ≈ 0.51 nm and LWR (3σ) ≈ 0.69 nm. Another molecule, M17 of approximately the same size with M21, under the same condition of simulated processing lithography resulted in LER (3σ) ≈ 0.60 nm and LWR (3σ) ≈ 0.75 nm. The values are very promising and recent experimental data verified the low LER/LWR values of these materials.F ig.3, shows a 32nm design rule CMOS inverter gate fabrication-modeling using M17.

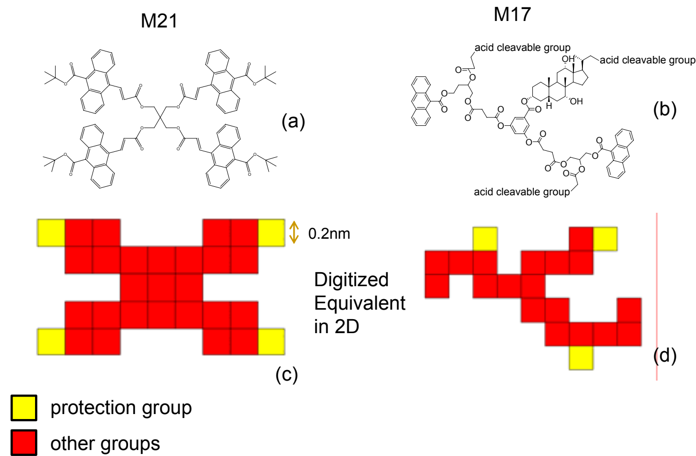

Fig. I.1.2. The digital representation of the molecular resists was based on decomposing the material in terms of its molecular groups and their functionality and then digitizing it geometrically as tiles of size equal to the minimum chemical group size.

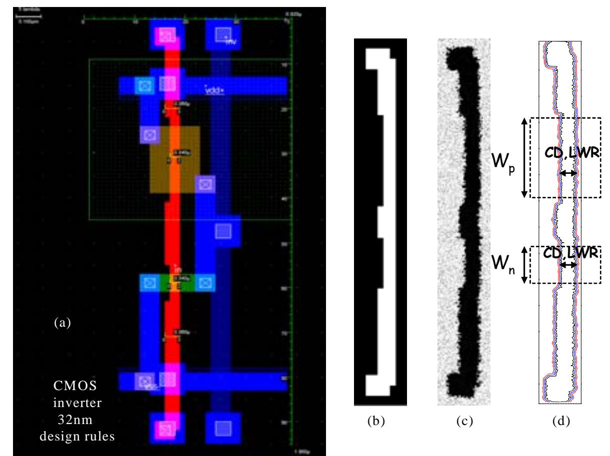

Fig. I.1.3. Application of stochastic simulation on the design of 32nm CMOS inverter. (a) Layout design, (b) gate mask, (c) resist profile after lithography simulation, (e) etching of gate and metrology.

Electron-beam simulation over multilayer substrates

Extreme ultra-violet lithography (EUVL) mask is a complex multilayer stack, fabricated with electron-beam lithography. Detailed understanding of the scattering events and energy loss mechanism of the electron beam within this stack is mandatory due to the high accuracy requirements of the fabrication process. Simulation of electron-beam lithography is performed incorporating the details of the mask material-stack and the metrological information of the final layout is quantified. Three-dimensional modeling of the electron beam interactions inside the material stack is considered. The deposited energy calculation takes into account the location in the multilayer stack (fig. 4) where scatterings took place, the material properties (e.g., density, atomic number), and the type of scattering (elastic or inelastic). The energy deposition follows the Monte Carlo procedure of Salvat and Parellada. Hiroguchi's et al. method was incorporated in order to correctly determine the mean free path of electron track due to multi-layer presence in the Mo/Si structure. The stack model consists of 150nm PMMA resist over a film stack of 40 Mo/Si layers (thicknesses: 4 nm Si, 3 nm Mo) on top of bulk Si. Initial electron energies of 100keV is assumed. In Fig. 5 experimental results from high resolution patterns exposed on EUVL mask blanks are compared with the simulation ones and the agreement is good.

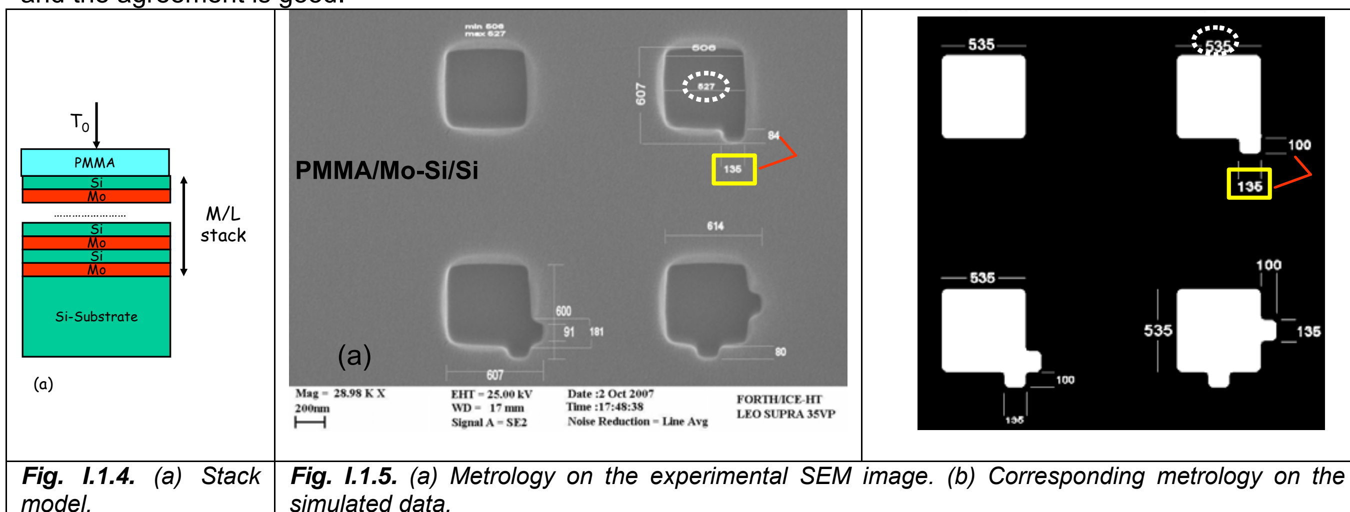

Fig. I.1.4. (a) Stack model.

Fig. I.1.5. (a) Metrology on the experimental SEM image. (b) Corresponding metrology on the simulated data.

Resists based on backbone breaking with acceptable etch resistance

Backbone breaking could ultimately lead to both low LER and improved sensitivity in sub50 nm lithography. Unfortunately the typical resists working by backbone breaking chemistry (e.g. PMMA) typically suffer from poor etch resistance, since they decompose easily in the reactive plasmas used in etching. A recent direction of our research targets to overcome this problem by using radiation-stable polyacetals that can only break through acid-catalysed chain scission. A polymer that has shown promising results is depicted in Fig. 6, where the acid catayzed back bone chain scission is depicted. In Fig 7, monitoring with FTIR spectroscopy provides clear evidence that the chain scission takes place under heating in presence of H^+ (Posrt Exposure Bake step) but not during exposure.

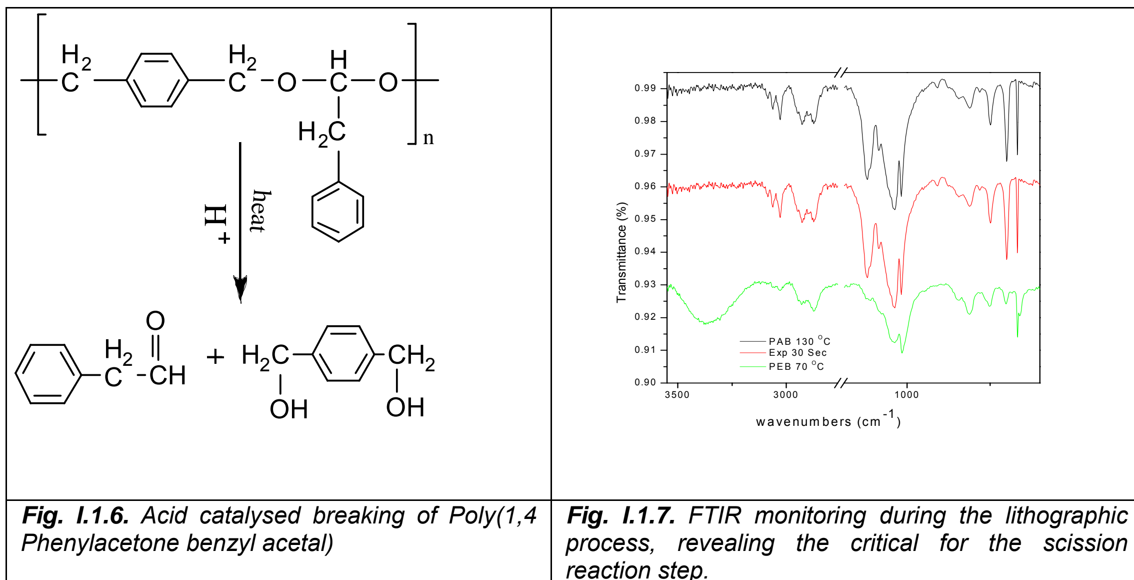

Fig. I.1.6. *Acid catalysed breaking of Poly(1,4 Phenylacetone benzyl acetal)*

Fig. I.1.7. *FTIR monitoring during the lithographic process, revealing the critical for the scission reaction step.*

B. Lithographic materials for micro-nano structure fabrication processes to be applied in MEMs and Nano-biotechnology

High aspect ratio proton beam writing

Proton Beam Writing (PBW) is a valuable tool for maskless patterning of high aspect ratio structures due to the unique ability of protons to maintain a straight path over long distances. TADEP (Thick Aqueous Developable Epoxy) resist is a new promising high-aspect-ratio chemically-amplified resist, developed by our group, which can be developed in aqueous base developer and has the capability of stripping by using conventional stripping schemes. From experimental point of view, the performance of PBW was explored and proved through the patterning of an aqueous base developable negative chemically amplified resist (TADEP). By employing PBW on TADEP resist a 280nm linewidth with a thickness of 12 μm has been resolved showing an aspect ratio of 42 which is the highest aspect ratio even fabricated on strippable resists with deep sub-micron resolution.

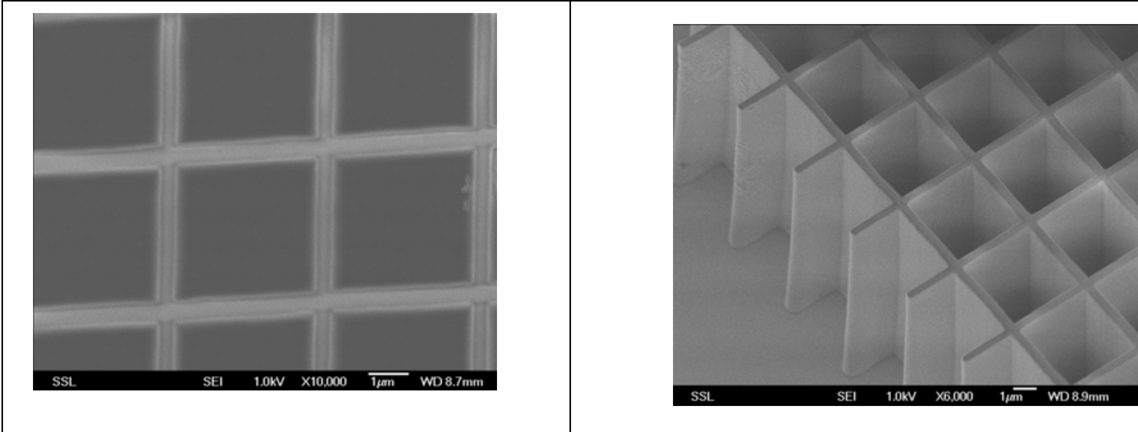

Fig. I.1.8. *SEM images of PBW double line irradiation on TADEP resist. Film thickness 12μm, line width 280nm in X-direction, aspect ratio: 42. a) Top view and b) side view (tilt 20°).*

Surface functionalization for biological patterning

Towards the further development of resist – based biocompatible processes for the patterning of biological substances a research effort aiming at the investigations of the interactions of organic film coated substrates with biological substances has been launched. Emphasis has been given on the chemical binding of small biomolecules, like biotin, on patterned polymeric films. Epoxy resist (EPR) surfaces have been proved suitable for the binding of biotin if properly functionalized with either amine or NHS ester groups. The binding capability on unexposed and exposed at different doses EPR films is depicted in Fig 9, whereas the correspondence of the different doses at a photoresist contrast curve is presented in Fig 10.

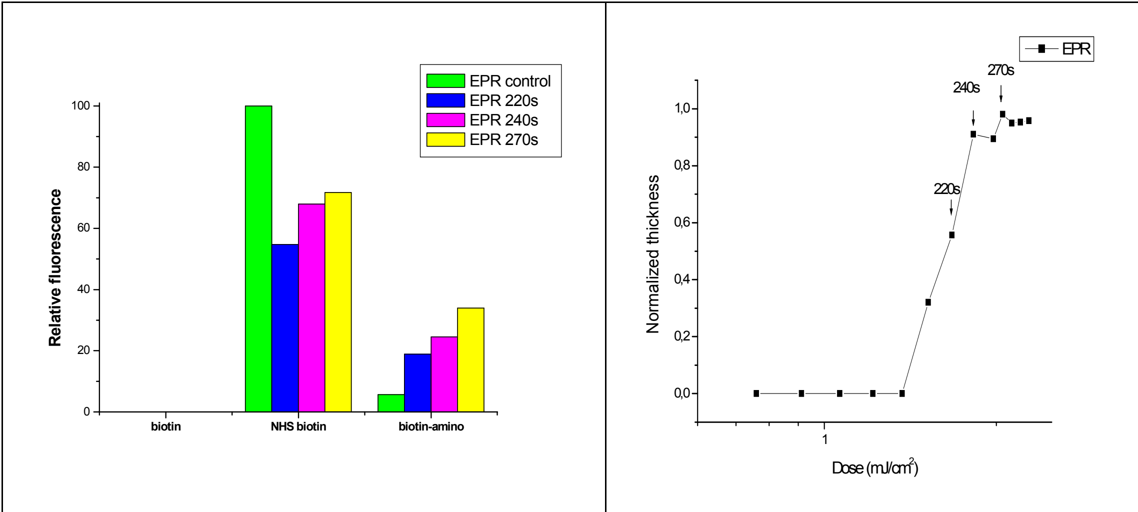

Fig. I.1.9. *Fluoresence measurements for the assessment of the chemical binding capability of epoxy resist surfaces. Unfunctionalized biotin cannot be bound but functionalization, especially with NHS ester, allows effective binding.*

Fig. I.1.10. *Contrast curve of EPR resist showing that the exposure doses used in the biotin binding experiments are adequate for lithographic patterning.*

C. Materials for organic/molecular electronics

Polyoxometallates as potential molecular components of electronic devices

Polyoxometallates of Mo and W are investigated as potential molecular components of electrtronic devices and in particular memory devices. The relevant activity is carried out in collaboration with Project II.3 (leader N. Glezos) and the results are reported in the corresponding section.

Tuning the emitting colour of OLEDs

Using the proposed by our group patterning scheme of acid induced spectral changes in the active layer of OLEDs three colour (blue-red-green) pixels can be defined in the same polymer layer (see figure 11). The optimization of device structure (mainly the active layer thickness) is necessary for the enhancement of external efficiency and for the electroluminescence spectral distribution to follow a Lambertian pattern. Towards this end simulation (Fig.12) and experimental work is being carried out.

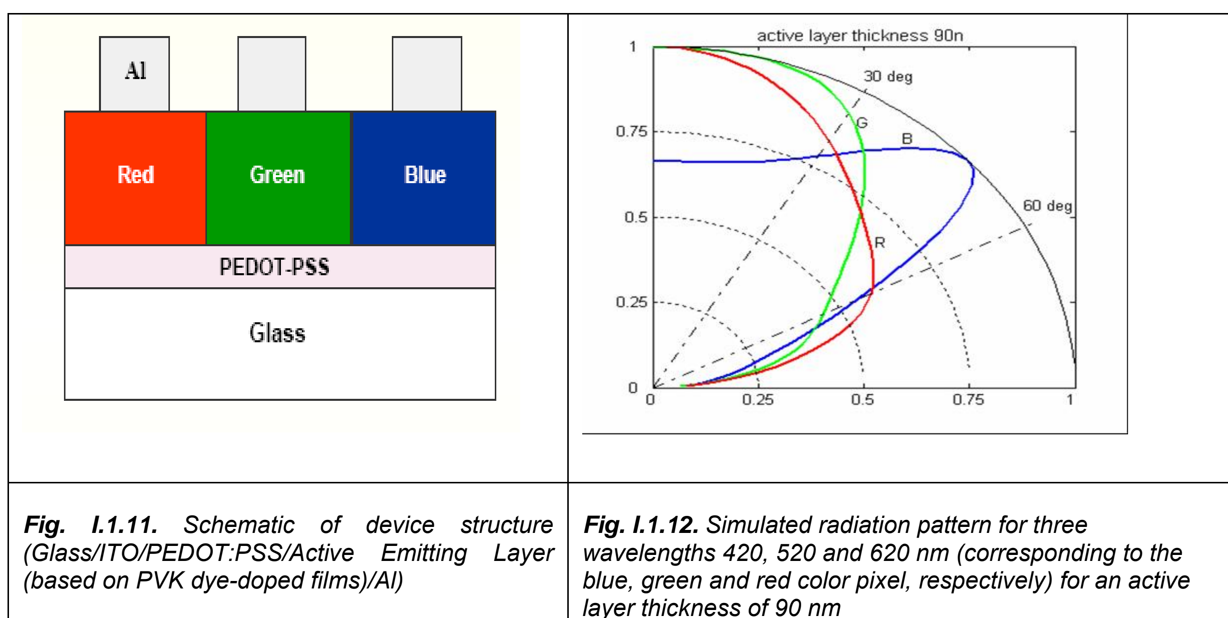

Fig. I.1.11. Schematic of device structure (Glass/ITO/PEDOT:PSS/Active Emitting Layer (based on PVK dye-doped films)/Al)

Fig. I.1.12. Simulated radiation pattern for three wavelengths 420, 520 and 620 nm (corresponding to the blue, green and red color pixel, respectively) for an active layer thickness of 90 nm

Photovoltaics – new organic optoelectronic devices

Research on organic/hybrid photovoltaics has been launched during 2007. Polythiophene/functionalized fullerene bulk heterojunction photovoltaic cells have been fabricated and characterized. Molecular redox active layers, like polyoxometalate-based layers, have been investigated as potential optical spacers and oxygen/water protective layers. In addition new organic optolectronic devices, such as a polymer optocoupler (Fig. 13), have been designed, fabricated and characterized as a first step towards all-plastic electronic devices.

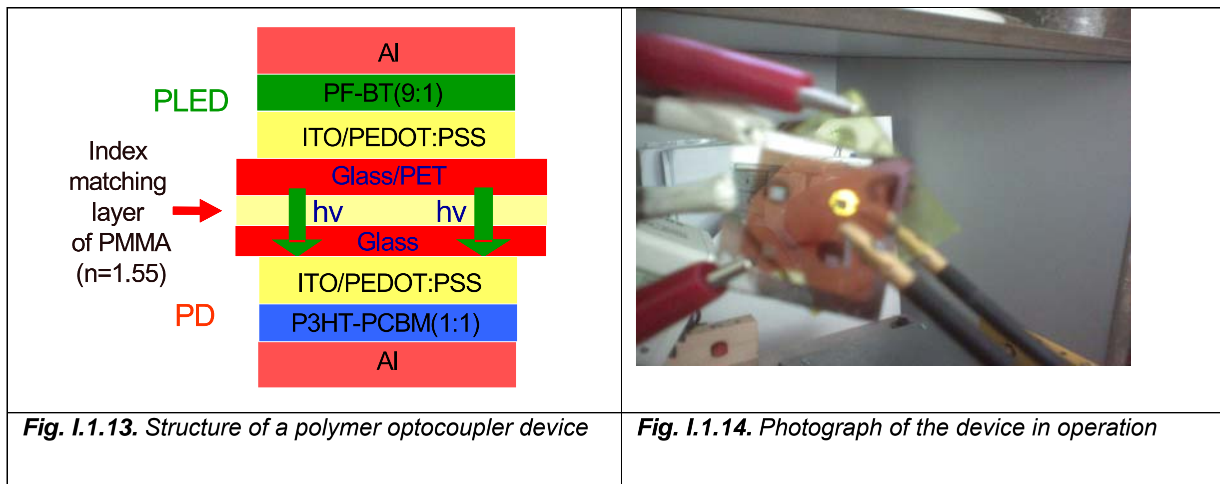

Fig. I.1.13. *Structure of a polymer optocoupler device*

Fig. I.1.14. *Photograph of the device in operation*

PROJECT OUTPUT IN 2007

Publications in international Journals

1. "A biomolecule friendly photolithographic process for fabrication of protein microarrays on polymeric films coated on silicon chips", P.S. Petrou, M. Chatzichristidi, A. M. Douvas, P. Argitis, K. Misiakos and S.E. Kakabakos, **Biosensors and Bioelectronics, 22, 1994-2002, 2007**.
2. "Stochastic simulation studies of molecular resists", D.Drygiannakis, G.P.Patsis, I.Raptis, D.Niakoula, V.Vidali, E.Couladouros, P.Argitis, E.Gogolides, **Microelectron. Eng. 84 1062, 2007**
3. "Effect of magnetic-field on metal-insulator transitions in Bi wire structures", M.Chatzichristidi, Th.Speliotis, I.Raptis, I.Haritantis, D.Niarchos, C.Christides, **Microelectron. Eng. 84, 1528, 2007**
4. "Proton beam micromachined buried microchannels in negative tone resist materials"**,** I.Rajta, M.Chatzichristidi, E.Baradács, C.Cserháti, I.Raptis, K.Manoli, E.S.Valamontes, **Nucl. Instrum. Meth. B 260, 414, 2007**
5. "Process simulation at electron beam lithography on different substrates", K.Vutova, G.Mladenov, I.Raptis, A.Olziersky, **J. Mater. Process. Tech. 184 305, 2007**
6. "Pattern matching, simulation and metrology of complex layouts fabricated by electron beam lithography", N.Tsikrikas, D.Drygiannakis, G.P.Patsis, I.Raptis, A.Gerardino, S.Stavroulakis, E.Voyiatzis, **J. Vac. Sci. Technol. B 25, 2307, 2007**
7. "Stochastic simulation of material and process effects on the patterning of complex layouts"**,** N.Tsikrikas, D.Drygiannakis, G.P.Patsis, I.Raptis, S.Stavroulakis, E.Voyiatzis, **Jpn. J. Appl. Phys. B 46, 6191, 2007**
8. "Nano-scale spatial control over surface morphology of biocompatible fluoropolymers at 157 nm", E. Sarantopoulou, Z. Kollia, A. C. Cefalas, A.M. Douvas, M. Chatzichristidi, P. Argitis, S. Kobe, **Materials Science and Engineering: C, Vol. 27, 1191-1196, 2007**.
9. "Polymer self-assembled nano-structures and surface relief gratings induced with laser at 157 nm", E. Sarantopoulou, Z. Kollia, A.C. Cefalas, A.M. Douvas, M. Chatzichristidi, P. Argitis and S. Kobe, **Appl. Surf. Science, 253, 7884-7889, 2007**.
10. "Tuning the emitting colour of Organic Light Emitting Diodes through photochemically induced transformations: towards single layer, patterned, full colour displays and white lighting applications", M. Vasilopoulou, G. Pistolis, D. Georgiadou, P. Argitis, **Adv. Funct. Mater., 17 (17), 3477-3485, 2007**

Publications in international Conference Proceedings

1. "Exposure of molecular glass resist by e-beam and EUVIL", Cyril Vannuffel, Damien Djian, Serge Tedesco, Dimitra Niakoula, Panagiotis Argitis, Veroniki P. Vidali, Elias Couladouros, and Harun Solak, **Proc. SPIE 6519, 651949, 2007.**

2. “Stochastic simulation of material and process effects on the patterning of complex layouts with e-beam and EUV lithography”**,** D.Drygiannakis, N.Tsikrikas, G.P.Patsis, G.Kokkoris, I.Raptis, E.Gogolides, **SPIE Advanced Lithography conf. (Jan Jose, USA, 02/2007)**
3. “Simulation of the combined effects of polymer size, acid diffusion length and EUV secondary electron blur on resist line-edge roughness”**,** D.Drygianakis, M.D.Nijkerk, G.P.Patsis, G.Kokkoris, I.Raptis, L.H.A.Leunissen, E.Gogolides, **SPIE Advanced Lithography conf. (Jan Jose, USA, 02/2007)**
4. Patterning scheme based on photoacid induced spectral changes for single layer, patterned full color light emitting diodes, *in Organic Electronics — Materials, Devices and Applications,* Maria Vasilopoulou, Giorgos Pistolis, Athanasios Botsialas, Nikos Stathopoulos, Maria Rangoussi, Panagiotis Argitis, *edited by F. So, G.B. Blanchet, Y. Ohmori*, **Mater. Res. Soc. Symp. Proc. 965E, Warrendale, PA, 2007, paper no 0965-S03-24**.
5. Photolithographic Process Based on High Contrast Acrylate Photoresist for Multi-Protein Patterning, *in Biosurfaces and Biointerfaces,* Margarita Chatzichristidi, Panagiota S. Petrou, Antonios M. Douvas, Constantinos D. Diakoumakos, Ioannis Raptis, Konstantinos Misiakos, Sotirios S. Kakabakos, Panagiotis Argitis, *edited by M. Firestone, J. Schmidt, N. Malmstad*t, **Mater. Res. Soc. Symp. Proc. 950E, Warrendale, PA, 2007, paper no 0950-D15-15.**

Invited talks in international Conferences

1. "High-aspect-ratio micro/nanomachining with proton beam writing on aqueous developable - easily stripped negative chemically amplified resists", M. Chatzichristidi, E.Valamontes, N.Tsikrikas, P. Argitis, I. Raptis , J.A. Van Kan, F. Watt, **33rd International Conference on Micro- and Nano-Engineering (MNE), Copenhagen, Denmark, September 23-26, 2007**.
2. *“Polymeric and molecular glass resist models for stochastic lithography simulation”***,** D.Drygiannakis, G.P.Patsis, I.Raptis, E.Gogolides, **5th IISB Litho workshop, Hersbruck, Germany 09/2007.**

Presentations in international Conferences

1. “Electrochemical detection of biomolecules patterned on electrode arrays by biocompatible photolithography”, Mònica Mir, Srujan Kumar Dondapati, Maria Viviana Duarte, Ioanis Katakis, Margarita Chatzichristidi, Konstantinos Misiakos, P.S. Petrou, S.E. Kakabakos, Panagiotis Argitis, **Materials Research Society (MRS) Fall Meeting, Boston, MA, USA, November 27-30 2007**.
2. “Charging Effects in Hybrid Structures Based on Polyoxometalate Layers for Molecular Memory Applications”, E. Makarona, A. M. Douvas, E. Kapatanakis, D. Velessiotis, P. Argitis, P. Normand, N. Glezos, J. Mielczarski, E. Mielczarski, T. Gotszalk, W. Miroslav, **Materials Research Society (MRS) Fall Meeting, Boston, MA, USA, November 27-30 2007**.
3. “Magnetotransport properties of $[Co/Bi]_n$ wire structures”, C.Christides, Th.Speliotis, M.Chatzichristidi, I.Raptis, **18th Soft Magnetic Materials Conf., Cardiff, UK, 09/2007**
4. “Electron Beam Lithography Simulation for the Patterning of EUV Masks”, N.Tsikrikas, G.P.Patsis, E.Valamontes, I.Raptis, A.Gerardino, **Microprocess & Nanotechnology Conf. (MNC 07), Kyoto, Japan, 11/2007**
5. “Realization and simulation of high aspect ratio micro/nano structures by proton beam writing”**,** M.Chatzichristidi, E.Valamontes, I.Raptis, J.A.vanKan, F.Watt, **Microprocess & Nanotechnology Conf. (MNC 07), Kyoto, Japan, 11/2007**
6. “Vertical architectures of self-assembled hybrid organic/inorganic monolayers based on tungsten polyoxometalates: a step towards molecular electronic devices”, E. Makarona, E. Kapetanakis, D. Velessiotis, A. Douvas, P. Argitis, P. Normand, T. Gotszalk, M. Woszczyna, N. Glezos, **33rd International Conference on Micro- and Nano-Engineering (MNE), Copenhagen, Denmark, September 23-26, 2007**.
7. “Evaluation of polymers containing ketal or acetal groups in the backbone as candidate photoresist components”, T. Manouras, A. M. Douvas, V.P. Vidali, M. Chatzichristidi, N. Vourdas, E. Gogolides, E.A. Couladouros, P. Argitis, **3rd International Conference on Micro-Nanoelectronics, Nanotechnology & MEMs (Micro&Nano), Athens, Greece, November 18-21, 2007**.
8. “Photoresist material and process optimization for the patterning of biomolecules on functionalized surfaces”, P. Pavli, M. Chatzichristidi, A. M. Douvas, P. S. Petrou, S. E. Kakabakos, D. Dimotikali, P. Argitis, **3rd International Conference on Micro-Nanoelectronics, Nanotechnology & MEMs (Micro&Nano), Athens, Greece, November 18-21, 2007**.
9. “Polyoxometalate-based multilayers: fabrication and electrical characterization”, A. M. Douvas, E. Makarona, D. Velassiotis, J. A. Mielczarski, E. Mielczarski, N. Glezos, P. Argitis, **3rd International**

Conference on Micro-Nanoelectronics, Nanotechnology & MEMs (Micro&Nano), Athens, Greece, November 18-21, 2007.

10. "VUV laser circular microstructured surface relief gratings induced on PTFEMA surface", E. Sarantopoulou, Z. Kollia, A. C. Cefalas, A. M. Douvas, M. Chatzichristidi, P. Argitis, **3rd International Conference on Micro-Nanoelectronics, Nanotechnology & MEMs (Micro&Nano), Athens, Greece, November 18-21, 2007**.
11. "Hybrid polymer-inorganic solar cells based on polythiophene and phthalocyanine/polyoxometalate blends", L. C. Palilis, A. M. Douvas, G. Chaidigiannos, M. Vasilopoulou, N. Glezos, S. Nespurek, P. Falaras, P. Argitis, **3rd International Conference on Micro-Nanoelectronics, Nanotechnology & MEMs (Micro&Nano), Athens, Greece, November 18-21, 2007**.
12. "Flexible WO3 based electrochromic displays using proton conducting solid electrolytes", M. Vasilopoulou, P. Argitis, G. Aspiotis, G. Papadimitropoulos and D. Davazoglou, **3rd International Conference on Micro-Nanoelectronics, Nanotechnology & MEMs (Micro&Nano), Athens, Greece, November 18-21, 2007.**M. Vasilopoulou,
13. "Flexible Organic Light Emitting Diodes (OLEDs) based on blue emitting polymers", L. C. Palilis, A. Botsialas, D. Georgiadou, P. Bayiati, N. Vourdas, P. S. Petrou, G. Pistolis, N. Stathopoulos, and P. Argitis, **3rd International Conference on Micro-Nanoelectronics, Nanotechnology & MEMs (Micro&Nano), Athens, Greece, November 18-21, 2007.**
14. "Energy transfer processes among emitters dispersed in a single polymer layer for colour tuning in OLEDs", D. Georgiadou, M. Vasilopoulou, G. Pistolis, D. Dimotikali and P. Argitis, **3rd International Conference on Micro-Nanoelectronics, Nanotechnology & MEMs (Micro&Nano), Athens, Greece, November 18-21, 2007.**
15. "All-organic optocouplers based on polymer light-emitting diodes and photodetectors", N. Stathopoulos, L. C. Palilis, M. Vasilopoulou, A. Botsialas, P. Falaras, and P. Argitis, **3rd International Conference on Micro-Nanoelectronics, Nanotechnology & MEMs (Micro&Nano), Athens, Greece, November 18-21, 2007.**
16. "An all-polymeric optocoupler based on polymer light-emitting diodes and photodetectors", L. C. Palilis, M. Vasilopoulou, A. Botsialas, N. Stathopoulos, P. Falaras, P. Argitis, **5th European Conference on Organic Electronics (ECOER07), Varenna, Italy, 01-04 October 2007**.
17. "Single layer white organic light-emitting diodes for lighting applications", M. Vasilopoulou, D. Georgiadou, L. C. Palilis, G. Pistolis, P. Argitis, **5th European Conference on Organic Electronics (ECOER07), Varenna, Italy, 01-04 October 2007**.
18. "Full colour single layer organic light emitting diodes (OLEDs) based on poly(9-vinylcarbazole) via photochemically induced emission tuning", M. Vasilopoulou, A. Botsialas, P. Bayiati, N. Vourdas, N. Stathopoulos, M. Rangoussi and P. Argitis, **4th International Workshop on "Nanosciences & Nanotechnologies - NN07" Thessaloniki, Greece, 16-18 July 2007**.
19. "Flexible all-solid state electrochromic displays based on polymeric electrolytes", M. Vasilopoulou, P. Argitis, G. Aspiotis, G. Papadimitropoulos and D. Davazoglou, **4th International Workshop on "Nanosciences & Nanotechnologies - NN07" Thessaloniki, Greece, 16-18 July 2007**.
20. "Optimization of the external efficiency of single layer full color light emitting diodes based on blue emitting polymers", N. Stathopoulos, M. Vasilopoulou and P. Argitis, **Interrnational Conference on Organic Electronics, Eindhoven, 4-7 June 2007**.

Presentations in National Conferences

1. "Βελτιστοποίηση της Εξωτερικής Απόδοσης Οργανικών Διόδων Εκπομπής Φωτός (OLEDs) Με Χρήση Θεωρητικού Μοντέλου", Α. Κορρές, Ν. Σταθόπουλος, Π. Αργείτης και Μ. Βασιλοπούλου, **Πανελλήνιο Συνέδριο Φυσικής Στερεάς Κατάστασης, ΕΚΕΦΕ «Δημόκριτος, Σεπτέμβριος 2007**.
2. "Σύστημα Ηλεκτροπτικού Χαρακτηρισμού Οργανικών Διόδων Εκπομπής Φωτός (OLEDs)", Α. Μποτσιαλάς, Ν. Σταθόπουλος, Π. Αργείτης και Μ. Βασιλοπούλου, **'Πανελλήνιο Συνέδριο Φυσικής Στερεάς Κατάστασης, ΕΚΕΦΕ «Δημόκριτος», Σεπτέμβριος 2007**.

Technical Reports

Final Report on Polycarbocycle-based Molecular Resists for EUV Lithography, More Moore-EU-FP6-Intergrated Project

Master theses

1. "Photochemically-induced polymer back-bone breaking for applications in lithography", Th. Manouras, Department of Chemistry", University of Athens, November 2007

2. “Photoresist material and process optimization for the patterning of biomolecules on functionalized surfaces”, P. Pavli, School of Applied Mathematical and Physical Sciences, National Technical University of Athens, December 2007

Patent applications

1. “Tuning the emitting color of single layer, patterned full color Organic Light Emitting Diodes”, P. Argitis, G. Pistolis, M. Vasilopoulou, **Greek Patent (OBI) appl. No 20060100359, 19 June 2006.** Greek Patent (OBI) appl. No 20060100359, 19 June 2006, PCT Application June 19, 2007.