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SMR: 1643/21

WINTER COLLEGE ON OPTICS ON OPTICS AND PHOTONICS IN NANOSCIENCE AND NANOTECHNOLOGY

(7 - 18 February 2005)

"Organic Light-Emitting Nano-Patterned Devices"

presented by:

D. Pisignano
Università di Lecce
Italy

ORGANIC LIGHT-EMITTING NANOPATTERNED DEVICES

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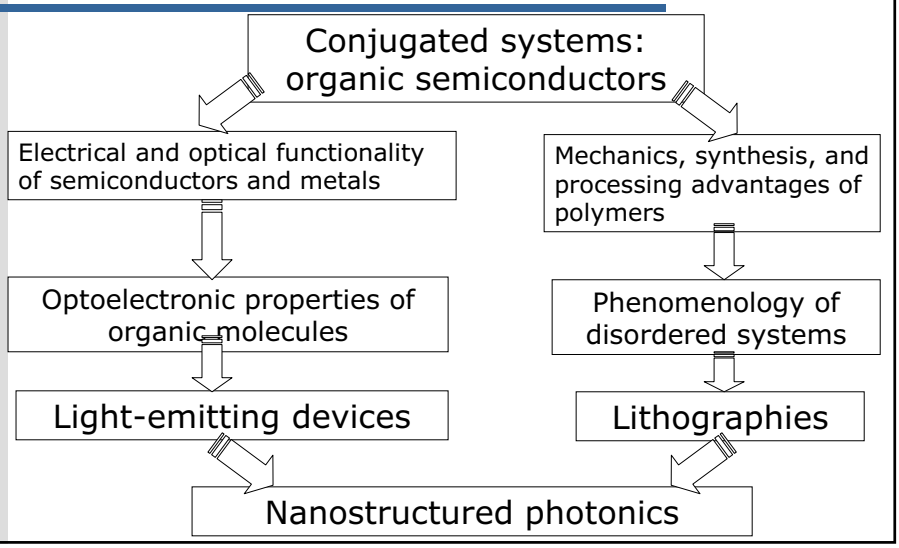
RATIONALE:



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OUTLINE:

- PHYSICS AND PHENOMENOLOGY OF DISORDERED SYSTEMS

- OPTOELECTRONIC PROPERTIES OF ORGANIC MOLECULES

- ORGANIC-BASED PHOTONIC DEVICES

- CONVENTIONAL AND SOFT LITHOGRAPHY FOR ORGANIC MATERIALS

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OUTLINE:

- PHYSICS AND PHENOMENOLOGY OF DISORDERED SYSTEMS

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PHYSICS AND PHENOMENOLOGY OF DISORDERED SYSTEMS

- CONVENTIONAL CLASSIFICATION SCHEME OF THE STATES OF MATTER: *SOLID (CRYSTALLINE), LIQUID, GAS*

GLASSY STATE OF MATTER

GLASS TRANSITION [J. Jäckle, *Rep. Prog. Phys*. **49**, 171 (1986)]
IMPORTANCE OF KYNETICS:
COOLING RATE VS. CHARACTERISTIC RELAXATION TIME

GLASS-FORMERS COMPOUNDS:

-ORGANIC COMPOUNDS: ABLE TO GLASSIFY AT LOW COOLING RATE (10^{-2} Ks^{-1})
–METALS ALLOYS (10^{5} Ks^{-1})
– Ar (10^{12} Ks^{-1}) –SIMULATIONS-
[J. R. Fox, H. C. Andersen, *J. Chem. Phys.* **88**, 4019, (1984)]

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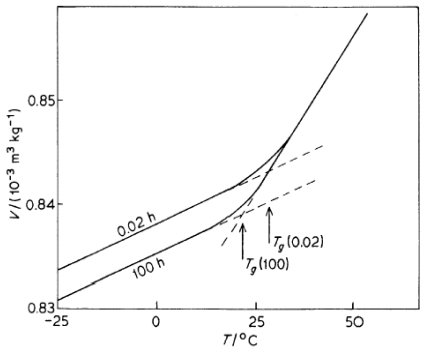
PHYSICS AND PHENOMENOLOGY OF DISORDERED SYSTEMS



v_1/v_2=5000 → ΔT_g=8K

Specific volume dependence on temperature for polyvinylacetate. The two employed cooling rates differ by a factor 5000, which shifts the glass-transition temperature of about 8 °C. [A.J.Kovacs, J.M.Mutchinson, J.J.Aklonis, *The Structure of Noncrystalline Materials*, P.H. Gaskell Ed., London (1977)]

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PHYSICS AND PHENOMENOLOGY OF DISORDERED SYSTEMS

EXAMPLE OF DISORDERED SYSTEMS:

-ORGANIC MATERIALS (POLYMERS, OLIGOMERS) OPTOELECTRONICS, AERONAUTICS, CARS, BUILDINGS

-BIO-SYSTEMS: PROTEINS, PEPTYDES, LIPIDES (GLYCEROL IN ARTICS INSECTS)

[K.B.Storey, J.M.Store, *Scientific American*, **62**, December 1990]

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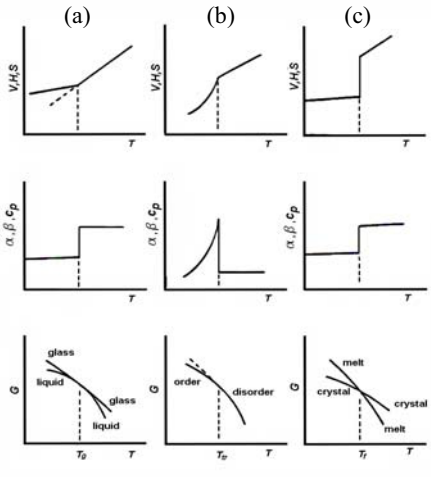
PHYSICS AND PHENOMENOLOGY OF DISORDERED SYSTEMS

GLASS TRANSITION

V, *H*, *S* (extensive thermodinamic quantities) → 2nd order according to Ehrenfest's classification?



Scheme comparing three different transitions: (a) glass transition; (b) second-order phase transition; (c) first-order phase transition. *V*: volume, *S*: entropy; *H*: enthalpy; α: volume thermal expansion coefficient, β: isothermal compressibility; c_p: isobaric specific heat; *G*: free energy.

PHYSICS AND PHENOMENOLOGY OF DISORDERED SYSTEMS

2nd ORDER TRANSITION (CONTINUITY OF ENTROPY AND VOLUME AT T_g [P.Ehrenfest, *Commun. Kemmerling Onnes Lab.*, University of London, **756** (1933)]):

$$\frac{dT_g}{dp} = T_g V \frac{\Delta\alpha_p(T_g)}{\Delta c_p(T_g)}$$ OK

$$\frac{dT_g}{dp} = -\frac{\Delta k_T(T_g)}{\Delta\alpha_p(T_g)}$$ <!

IMPORTANCE OF KINETICS: DEFINITION OF T_g ON KINETIC BASIS

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PHYSICS AND PHENOMENOLOGY OF DISORDERED SYSTEMS

CLASSIFICATION OF SYSTEMS IN GLASSY STATE: FRAGILITY INDEX [C.A.Angell, *J. Non-Cryst. Solids*, **131-133**, 13 (1991)]

$$m = \left.\frac{dLog(\eta)}{d(T_g/T)}\right|_{T=T_g}$$

EXPERIMENTALLY: $16 \le m \le 200$

HIGH FRAGILITY=LARGE NUMBER OF ACCESSIBLE CONFIGURATIONS

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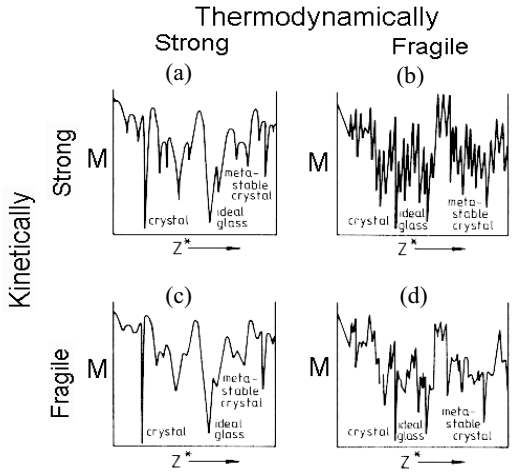
PHYSICS AND PHENOMENOLOGY OF DISORDERED SYSTEMS



Z^*: GENERALIZED COORDINATE IN THE SPACE OF THE CONFIGURATIONS

M: POTENTIAL ENERGY

[R.Böhmer, C.A.Angell, *Local and Global Relaxation in Glass Forming Materials*, in: R.Richert, A.Blumen, *Disorder Effects on Relaxational Processes*, Springer-Verlag, Berlin, (1994)]

Scheme of the behaviour of the potential energy (M) versus a generalised coordinate, Z^*, in the space of configurations for a fragile or strong liquid, in a thermodynamic or kinetic sense. [42] The thermodynamic fragility is determined by the density of M minima -higher in (b) and (d)-, whereas the kinetic fragility decreases upon increasing the M barriers –(a) and (b)-.

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PHYSICS AND PHENOMENOLOGY OF DISORDERED SYSTEMS

FRAGILE COMPOUNDS → LARGE VARIATIONS OF MEASURABLE QUANTITIES AT T_g

FRAGILE COMPOUNDS: NO OR SMALL STERIC HINDRANCE (AND LOWER T_g)

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PHYSICS AND PHENOMENOLOGY OF DISORDERED SYSTEMS: RELAXATION PROCESSES

GENERAL ASPECTS OF RELAXATION PROCESSES FOR CAUSAL, TIME-INDEPENDENT AND LINEAR SYSTEMS:

$$x(t) = \chi_0 \int_{-\infty}^{t} -\frac{d\Psi(t-t')}{dt} F(t')dt'$$

x: GENERALIZED MOVEMENT
F: GENERALIZED FORCE
χ_0: GENERALIZED STATIC SUSCEPTIVITY
Ψ: TIME-DOMAIN RELAXATION FUNCTION (RESPONSE TO $F(t)=\Theta(-t)$)

Ψ: THE SYSTEM GOES BACK TO THERMODYNAMIC EQUILIBRIUM

$$\tau_c = \int_0^{+\infty} \Psi(t)dt$$

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PHYSICS AND PHENOMENOLOGY OF DISORDERED SYSTEMS: RELAXATION PROCESSES

RELAXATION FUNCTIONS

$$\Phi(t) = \exp(-t/\tau_c)$$

MONOEXPONENTIAL RELAXATION
[P.Debye, *Polar Molecules*, Dover Publications, Inc., London (1945)]

$$\Phi(t) = \exp\left[-(t/\tau_c)^{\gamma}\right]$$

STRETCHED EXPONENTIAL
[G.Williams, D.C.Watts, *Trans. Faraday Soc.* **66**, 80 (1970)]

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PHYSICS AND PHENOMENOLOGY OF DISORDERED SYSTEMS: RELAXATION PROCESSES

DEPENDENCE ON TEMPERATURE:

$$\left.\begin{aligned} k_A &= \kappa \exp\left[-\frac{\Delta E}{k_B T}\right] \\ -\frac{dC_A}{dt} &= k_A C_A \end{aligned}\right\} \tau = \tau_0 \exp\left[\frac{\Delta E}{k_B T}\right]$$

ARRHENIUS LAW
[A.R.Blythe, *Electrical Properties of Polymers*, Cambridge University Press, Cambridge (1979)]

$$\tau_c = \tau_{c0} \exp\left[\frac{DT_0}{T - T_0}\right]$$

VOGEL-FULCHER LAW
[H.Vogel, *Physic Z*, **22**, 645 (1921); G.A.Fulcher, *J.Am.Ceram.Soc.*, **8**, 339 (1925)]

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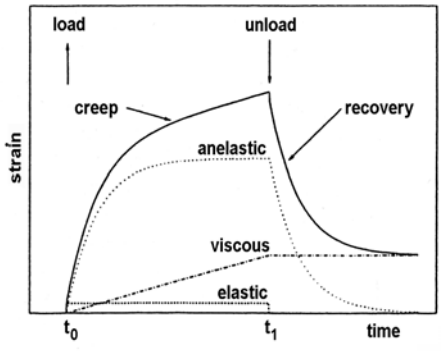
PHYSICS AND PHENOMENOLOGY OF DISORDERED SYSTEMS: POLYMER TRANSPORT

$$e_x = J_0 \sigma_x^0$$

$$e_x = \frac{\sigma_x^0}{\eta} t$$



Deformation dynamics of a polymer. An external stress is applied at t_0 and removed at t_1 [G. Strobl, *The Physics of Polymers*, Springer, Berlin (1997)]

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OPTOELECTRONIC PROPERTIES OF ORGANIC MOLECULES

20s H. Staundiger: Concept of Macromolecule proposed (Nobel Prize in 1953)

1935 W. Carothers: Invention of Nylon

1963 K. Ziegler and G. Natta: Nobel Prize for Polymerisation Catalysis

1974 P. Flory: Nobel Prize for Physical Chemistry

SATURATED POLYMERS:

all the four valence electrons of carbon are occupied in covalent bonds

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OPTOELECTRONIC PROPERTIES OF ORGANIC MOLECULES

1976: University of Pennsylvania

conjugated polymers -one unpaired (π) electron per carbon-:

from insulating ($\sigma \cong 10^{-5}$ S cm^{-1}) to metallic ($\sigma > 10^{2}$ S cm^{-1}) behaviour by chemical doping

So far: σ from 10^{-12} to 10^{5} S cm^{-1}

2000 A. J. Heeger, A. G. MacDiarmid and H. Shirakawa: Nobel Prize for "The discovery and the development of conductive polymers"

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OPTOELECTRONIC PROPERTIES OF ORGANIC MOLECULES

π-bonded conjugated systems: sp^2p_z electronic configuration

overlap of the adjacent carbon orbitals along the molecular backbone.

The π electrons are therefore available for delocalisation into a band [C. K. Chiang, C. R. Fincher, Jr., Y. W. Park, A. J. Heeger, H. Shirakawa, E. J. Louis, S. C. Gau, and A. G. MacDiarmid, Phys. Rev. Lett. **39**, 1098 (1977)]

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OPTOELECTRONIC PROPERTIES OF ORGANIC MOLECULES

Molecules and crystals: Many interacting bodies systems

$$H = \sum_i \frac{p_i^2}{2m} + \sum_I \frac{P_i^2}{2M} + \sum_i V(\mathbf{r_i}) + \frac{1}{2}\sum_{i,j} \frac{e^2}{|\mathbf{r_i} - \mathbf{r_j}|} + \frac{1}{2}\sum_{I,J} \frac{Z_I Z_J e^2}{|\mathbf{R_i} - \mathbf{R_j}|}$$

$$V(\mathbf{r_i}) = -\sum_I \frac{Z_I e^2}{|\mathbf{r_i} - \mathbf{R_I}|}$$

Conventional (inorganic) semiconductors (Si, GaAs etc.): crystalline materials, covalent bonds between neighbour atoms → long-range structural order & extended delocalisation into bands

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OPTOELECTRONIC PROPERTIES OF ORGANIC MOLECULES

Organic semiconductors:

i) intermolecular interactions ruled by weaker non-covalent (Van der Waals and hydrogen) bonds

ii) small overlap between orbitals belonging to different molecules → optoelectronic properties still related to single-molecule features (such as functionalisation groups and steric hindrance)

iii) small dielectric constant, namely strong excitonic interactions (several hundred meV) → localised Frenkel excitons

Transport:

Weak intermolecular coupling giving rise to an energy bandstructure (around 0.1 eV)
$\mu = \sigma/ne$ (n=density of free carriers) from < 10^{-7} to 10^{1} $cm^2V^{-1}s^{-1}$ for holes in conjugated compounds

intermolecular hopping transport < 1 $cm^2V^{-1}s^{-1}$ < band-like conduction

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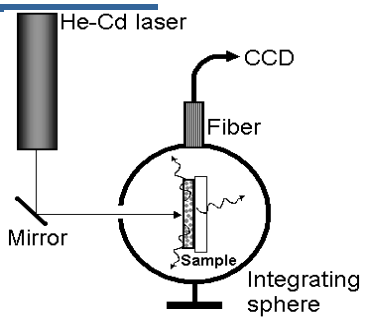
OPTOELECTRONIC PROPERTIES OF ORGANIC MOLECULES

EXCITON DECAY

$$\frac{d\xi}{dt} = \frac{\xi}{\tau_r} + \frac{\xi}{\tau_{nr}}$$

$$\xi = \xi_0 \exp\left[-\frac{t}{\left(\tau_r^{-1} + \tau_{nr}^{-1}\right)^{-1}}\right] = \xi_0 \exp\left(-\frac{t}{\tau}\right)$$

$$\eta = \frac{\text{photons generated}}{\text{excitons generated}} = \frac{\tau_{nr}}{\tau}$$



Schematic diagram of the experimental set-up employed for the measurements of the PL absolute quantum efficiency.

U. Lemmer, R. F. Mahrt, Y. Wada, A. Greibner, H. Bässler, and E. O. Göbel, *Appl. Phys. Lett*. **62**, 2827 (1993). M. Yan, L. J. Rothberg, F. Papadimitrakopoulos, M. E. Galvin, and T. M. Miller, *Phys. Rev. Lett*. **73**, 744 (1994). I. D. W. Samuel, B.Crystall, G. Rumbles, P. L. Burn, A. B. Holmes, and R. H. Friend, *Synth. Met*. 54, 281 (1993).

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ORGANIC-BASED PHOTONIC DEVICES

ELECTROLUMINESCENCE

1963 First EL from anthracene single-crystals [M. Pope, H.P. Kallmann, and P. Magnate, *J. Chem. Phys*. **38**, 2042 (1963)]

1987 First device by evaporated low-molar-mass molecules at Kodak [C. W. Tang and S. A. VanSlyke, *Appl. Phys. Lett*. **51**, 913 (1987)]

1990 First observation from an amorphous film of poly(*p*-phenylenevinylene) (PPV) at University of Cambridge [J. H. Burroughes, D. D. C. Bradley, A. R. Brown, R. N. Marks, K. Mackay, R. H. Friend, P. L. Burn, and A. B. Holmes, *Nature* **347**, 539 (1990)]

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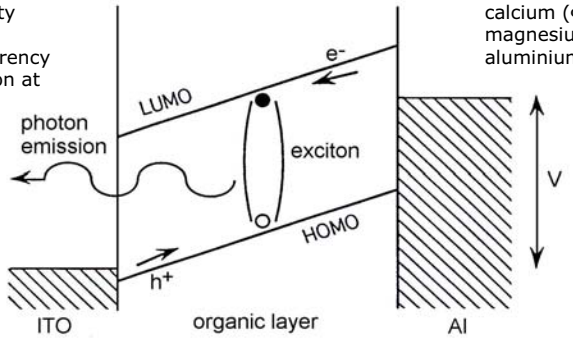
ORGANIC-BASED PHOTONIC DEVICES

ITO:
low electric resistivity ($2\text{-}4\times10^{-4}\ \Omega\ cm^{-1}$)
high optical transparency (90% of transmission at 550 nm)
Φ_A=4-5 eV

calcium (Φ_C=2.9 eV)
magnesium (Φ_C=3.7 eV)
aluminium (Φ_C=4.3 eV)



Working principle of an organic EL diode. *V*: applied bias.

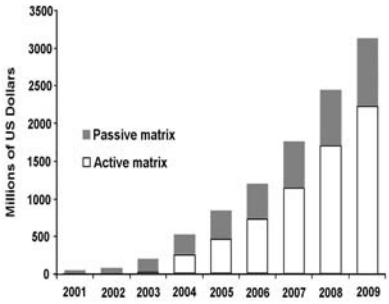
ORGANIC-BASED PHOTONIC DEVICES

OLEDs today:
high luminescence efficiency
wide tunability of the emission wavelength
low cost and simple deposition techniques

Performances:

external quantum yields larger than 4%, and luminances as high as 70000 cd/m^2

[T. Tsutsui, N. Takada, and S. Saito, *Appl. Phys. Lett.* **65**, 1868 (1994). M. T. Bernius, M. Inbasekaran, J. O'Brien, and W. Wu, *Adv. Mater.* **12**, 1737 (2000)]



Expected growth path for the global OLED market in the next years (www.stanfordresources.com).

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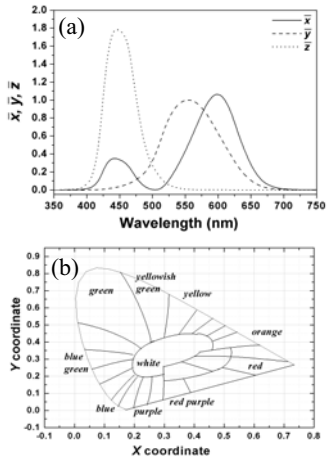
ORGANIC-BASED PHOTONIC DEVICES



CIE Color Matching Functions. (b): CIE diagram of the colour coordinates *X* and *Y*.

$$X = \frac{\int_0^{+\infty} S(\lambda)\bar{x}(\lambda)d\lambda}{\sum}$$

$$\sum = \sum_{t=x,y,z} \int_0^{+\infty} S(\lambda)\bar{t}(\lambda)d\lambda$$

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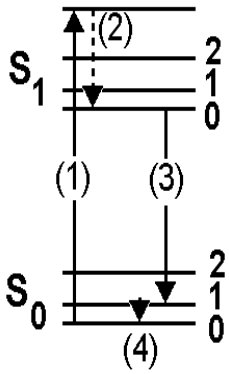
ORGANIC-BASED PHOTONIC DEVICES



Microrings [S. V. Frolov et al. *Appl. Phys. Lett*. **72**, 2811 (1998). S. X. Dou et al. *Appl. Phys. Lett*. **80**, 165 (2002)]
Microdroplets [M. Berggren et al. *Adv. Mater*. **9**, 968 (1997)]
Planar and circular distributed Bragg reflectors, [M. Bergreen, et al., *Appl. Phys. Lett*. **71**, 2230 (1997). A. Shaw et al., *Appl. Phys. Lett.* **75**, 3051 (1999)]
Distributed feedback (DFB) resonators

Four-levels scheme of lasers based on conjugated optically gaining compound. (1): photoexcitation, (3): stimulated emission, (2) and (4): ground-state relaxations. [N. Tessler, *Adv. Mater*. **11**, 364 (1999). G. Kranzelbinder, G. Leising, *Rep. Prog. Phys*. **63**, 729 (2000)]

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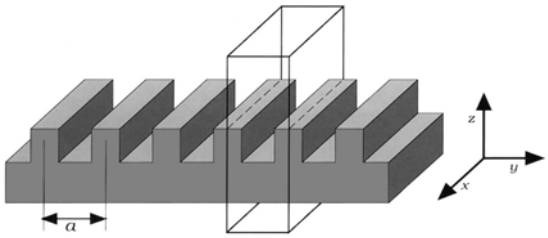
ORGANIC-BASED PHOTONIC DEVICES



Dielectric structure with discrete translational symmetry along the *y* direction, of periodicity *a*. This is exactly the imprinted geometry used for the distributed feedback lasers ($a=\Lambda$).
[J. D. Joannopoulos, R. D. Meade, and J. N. Winn, *Photonic Crystals – Molding the Flow of Light*, Princeton University Press, Princeton (1995)]

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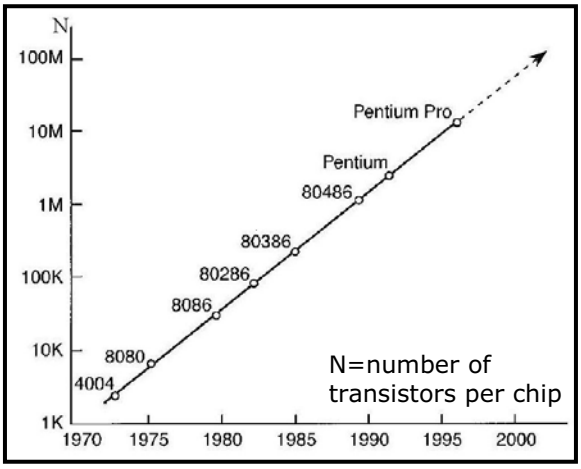
MOORE'S LAW (1965):

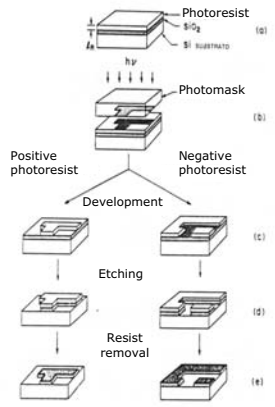
THE NUMBER OF TRANSISTORS IN AN INTEGRATED CIRCUIT WILL DOUBLE EVERY 18 MONTHS (THE SIZE OF THE FEATURES WILL DECREASE BY A FACTOR TWO EVERY 3 YEARS)



→ 22 nm in 2010!

[G. E. Moore, *Electronics* **38** (1965)]

PHOTOLITHOGRAPHY:



PROCESS STEPS:

- (a) SUBSTRATE CLEANING AND RESIST DEPOSITION
- (b) PHOTOMASK, UV EXPOSURE
- (c) RESIST DEVELOPMENT, METAL EVAPORATION AND LIFT-OFF (OPTIONAL)
- (d) WET ETCHING OR REACTIVE ION ETCHING
- (e) RESIST REMOVAL

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PHOTOLITHOGRAPHY:

ADVANTAGES

-WELL OPTIMIZED TECHNOLOGY
-PARALLEL AND FAST OPERATION (<1h)
-LARGE AREA LITHOGRAPHY

DRAWBACKS

- SIZE OF THE FEATURES LIMITED BY OPTICAL DIFFRACTION $R=k\lambda/\text{N.A.}$
- HIGH-ENERGY RADIATION REQUIRES COMPLEX FACILITIES
- NOT SUITABLE FOR NON-PLANAR SURFACES
- NO FLEXIBILITY TOWARDS THE EMPLOYABLE MATERIALS
- NO CONTROL OVER THE CHEMISTRY OF PATTERNED SURFACES

SOFT LITHOGRAPHY:

"SOFT" BECAUSE AN ELASTOMERIC MOLD IS THE KEY ELEMENT TO TRANSFER THE PATTERN AND, MORE BROADLY, AS IT USES FLEXIBLE ORGANIC MATERIALS

BASED ON THE REPLICATION OF AN ALREADY EXISTING NANO-STRUCTURE (MASTER)

ADVANTAGES:

-HIGH RESOLUTION (<100nm)
-PARALLEL OPERATION, HIGH THROUGHPUT
-LARGE AREA LITHOGRAPHY
-VERY LOW COST
-EXTREME FLEXIBILITY TOWARDS THE EMPLOYABLE MATERIAL

SOFT LITHOGRAPHY VS. PHOTOLITHOGRAPHY:

	PHOTO LITHOGRAPHY	SOFT LITHOGRAPHY
DEFINITION OF PATTERNS	Rigid photomasks (patterned Cr on quartz substrates)	Elastomeric elements with relief structures
MATERIALS THAT CAN BE PATTERNED DIRECTLY	Photoresists	Photoresists, EBL resists, conjugated polymers, curable polymers (epoxies, PU), colloidal materials, biomolecules
CURRENT LIMIT TO RESOLUTION	200 nm	10 nm (Nanoimprinting)
MINIMUM FEATURE SIZE	100 nm (?)	<10 nm (?)

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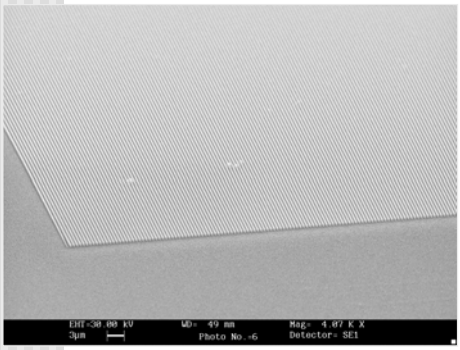
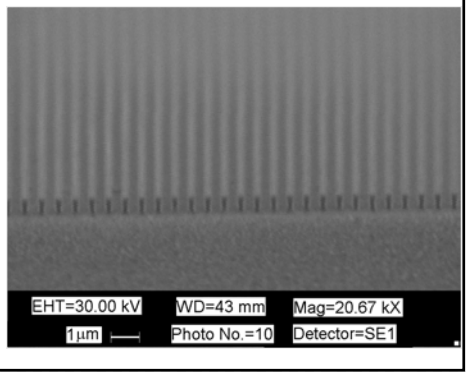
FABRICATION OF MASTER STRUCTURES FOR VISIBLE AND NIR PHOTONICS



Area≥20 mm²

200nm ≤ Λ ≤ 600nm

A.R. ≥ 1.5



[D.Pisignano, L. Persano, et al. *Appl. Phys. Lett.* **83**, 2545 (2003)]

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OPTIMIZATION OF THE REPLICA MOLDING PROCEDURE:

SEMICONDUCTOR MASTERS:	*POLYMERIC MASTERS:*
Possibility of curing PDMS at high temperature and to carry out a faster process	Necessity of curing PDMS at $T<T_g$ of the polymer of the master to not compromise it
PDMS POLYMERIZATION AT 140°C FOR 15'	*PDMS POLYMERIZATION AT 60°C FOR 2hs*

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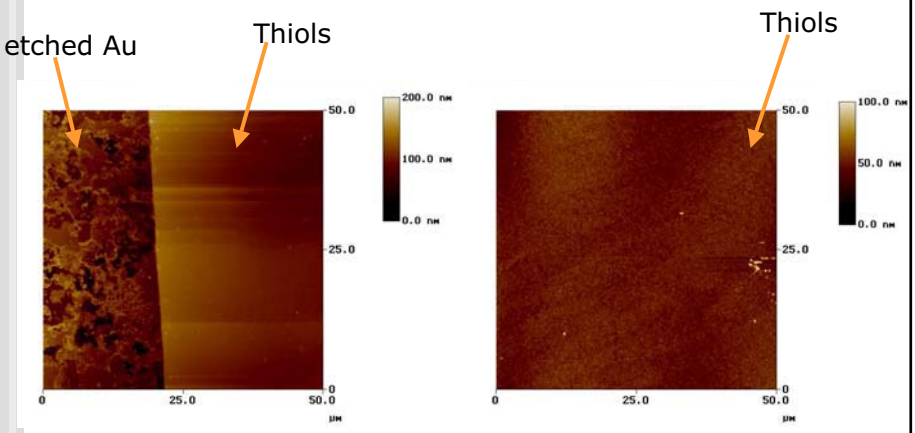
MICROCONTACT PRINTING, EXAMPLES



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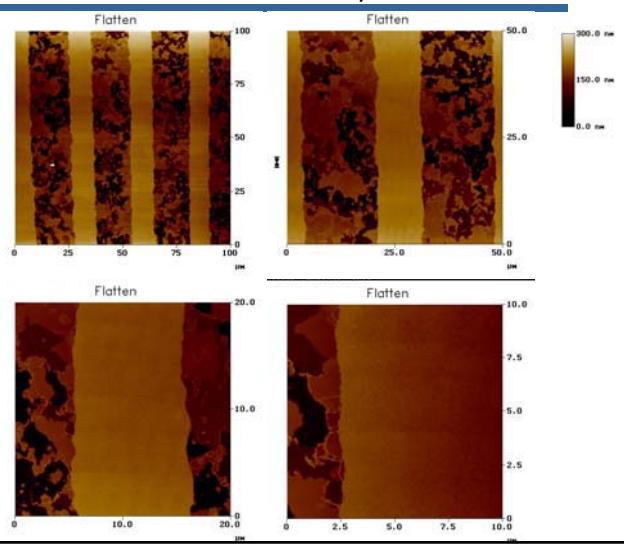
MICROCONTACT PRINTING, EXAMPLES



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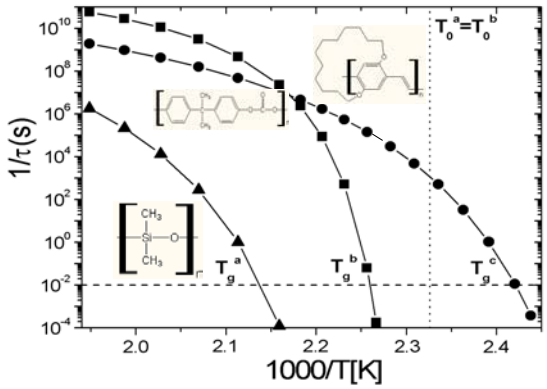
SOFT MOLDING, BASIC PRINCIPLES



$$\eta(T)=\eta_0\exp\left(\frac{DT_0}{T-T_0}\right) \qquad \tau_\alpha(T)=\tau_{\alpha 0}\exp\left(\frac{DT_0}{T-T_0}\right)$$

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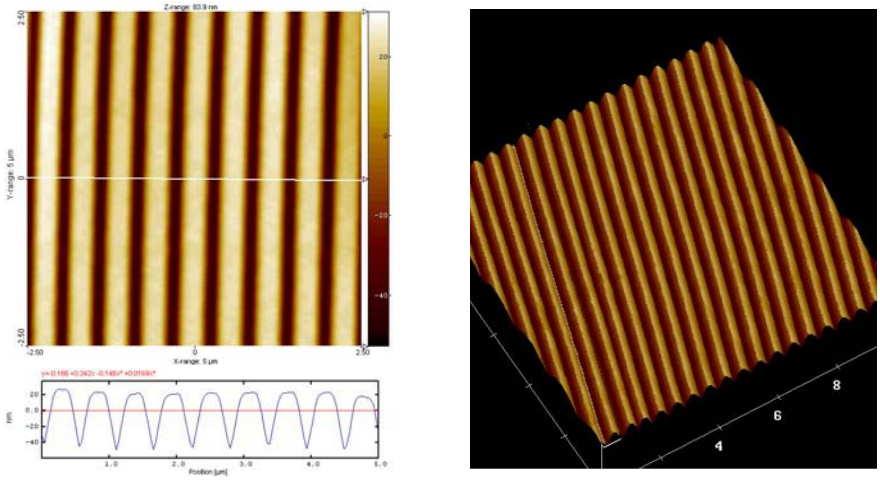
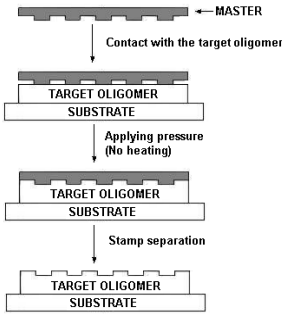
SOFT MOLDING, EXAMPLES



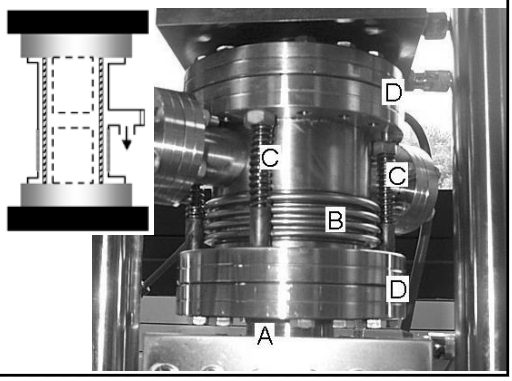
500-nm GRATINGS (250-nm FEATURES) IMAGED BY AFM

NANOIMPRINTING LITHOGRAPHY (NIL)

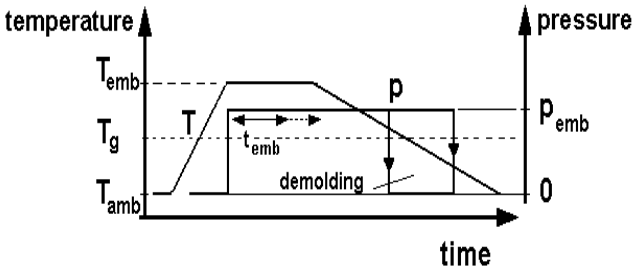


[S.Y.Chou et al. *Science* **272**, 85 (1996)]

[D. Pisignano et al. *J. Vac. Sci. Technol. B* **22**, 185 (2004)]



NANOIMPRINTING LITHOGRAPHY (NIL)



Typical cycle of pressure and temperature in a NIL experiment. The polymer film is driven from T_{amb} (room temperature), to T_{emb}, well above T_g. Then a pressure (p_{emb}) is applied for a time interval, t_{emb}, depending on the thermoplastic properties of the organics, i.e. on the dependence of the structural properties (viscosity and structural relaxation time) on temperature. Finally, there is the demolding step.

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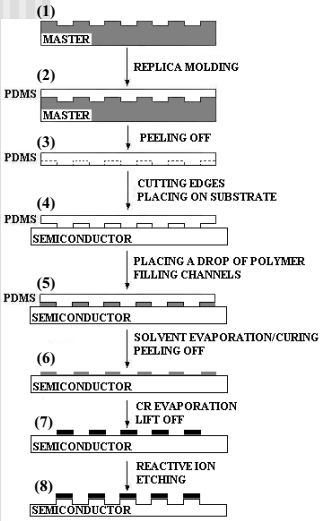
MICROFLUIDIC LITHOGRAPHY



STEP 1:

REPLICA MOLDING (20');
CUTTING μ-CHANNELS TO EXPOSE THEM;
PLACEMENT OF REPLICA ON SEMICONDUCTOR;
PLACEMENT OF A DROP OF DILUTED RESIST;
EVAPORATION OF SOLVENT-UV/THERMAL CURING (1h-24hs);
PEELING OFF REPLICA (1'').
[Kim, Xia and Whitesides Nature (1995)]

STEP 2:

Cr EVAPORATION (10-25 nm);
LIFT OFF (ACETONE-CH_2Cl_2);
REACTIVE ION ETCHING OF SEMICONDUCTOR.

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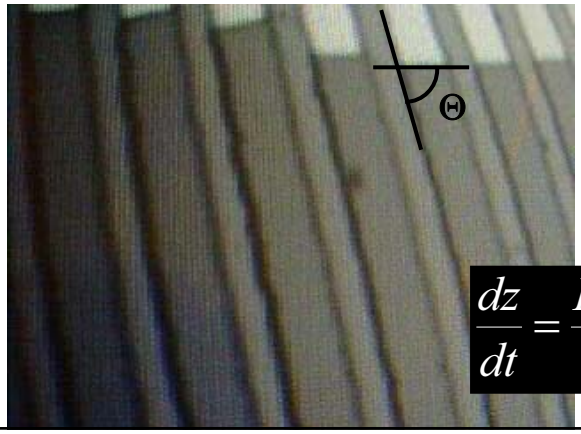
MICROFLUIDIC LITHOGRAPHY

REAL-TIME FORMATION OF 30 μm GRATINGS (15 μm FEATURES)

FILMED BY OPTICAL MICROSCOPY



$$\frac{dz}{dt} = \frac{R\gamma \cos\Theta}{4\eta z}$$

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CONCLUSIONS

Organic semiconductors: Electrical and optical functionality of semiconductors and metals + Mechanics, synthesis, and processing advantages of polymers

Mechanical patterning: Set of complementary technologies (REM, NIL, soft molding, MIMIC, mCP) for lithography on organics –resolutions: tens nm to mm-

Devices: Lasers, leds, diffractive elements, photonic crystals, waveguides, biomolecular patterns

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