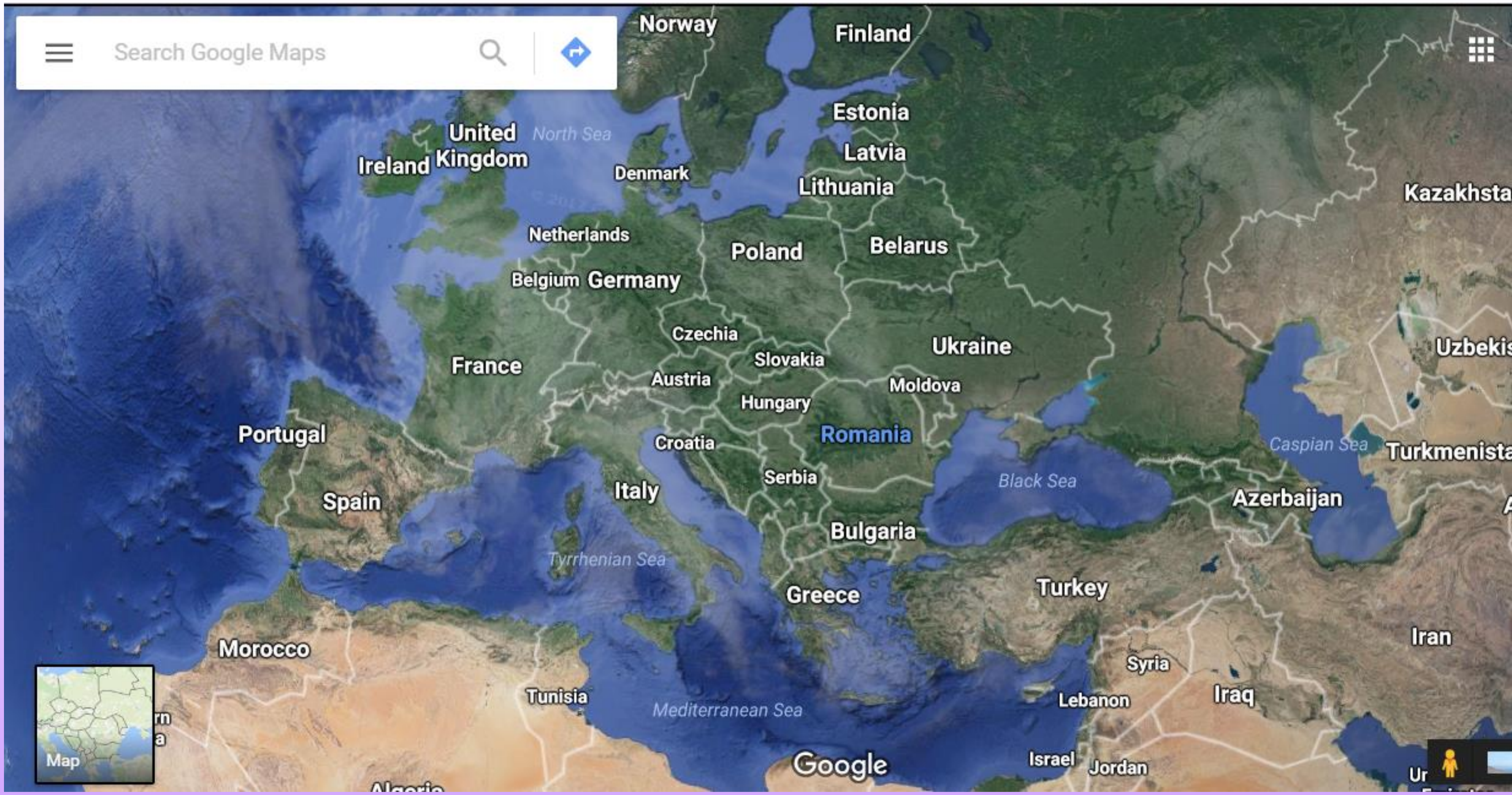
INCDTIM

Optical Lithography: basics and practice

Dr. Nicoleta Tosa

National institute for Research and Development of Isotopic and Molecular Technologies

Winter College on Optics, 13-24 February, ICTP, Trieste, Italy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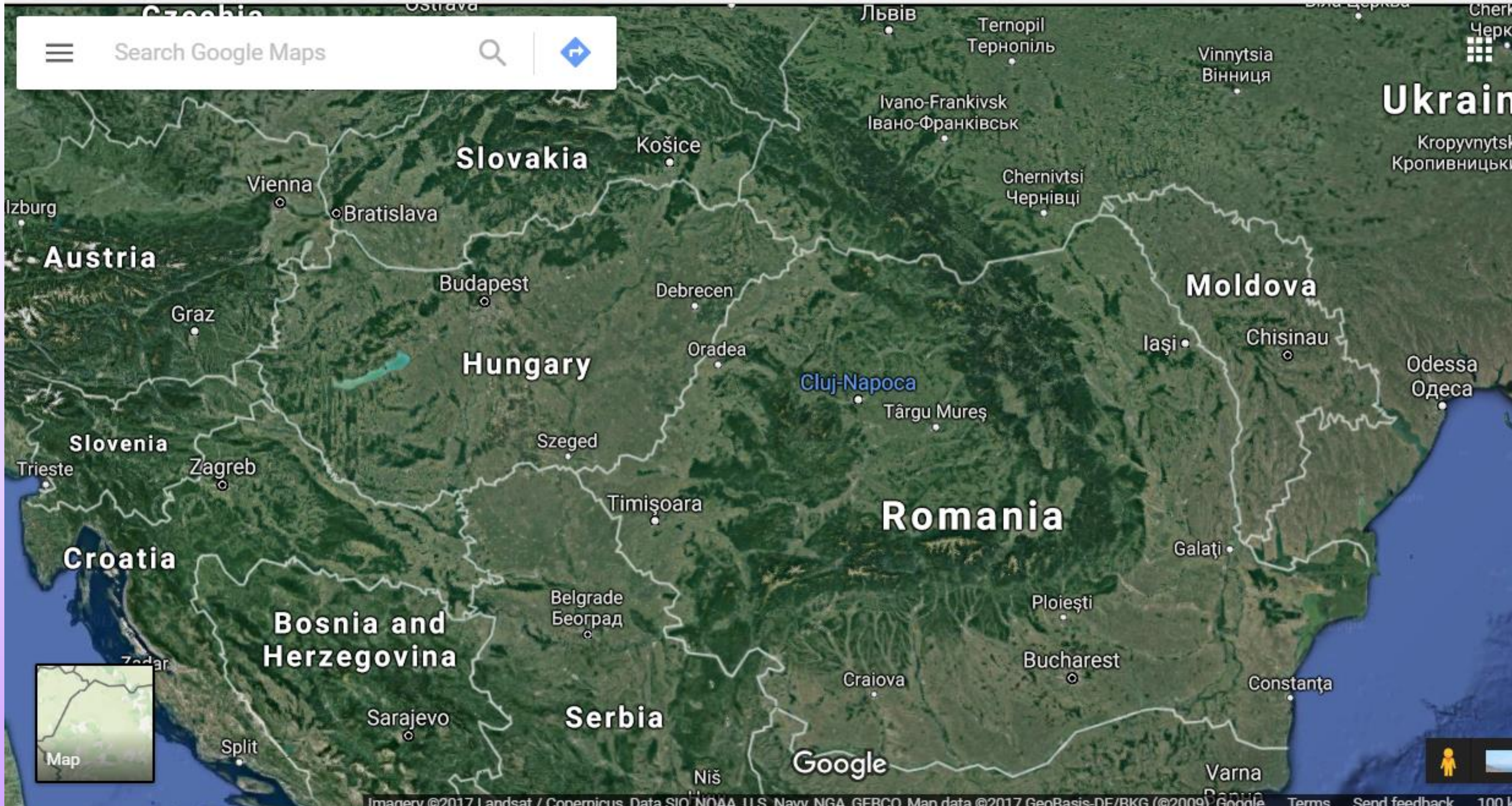

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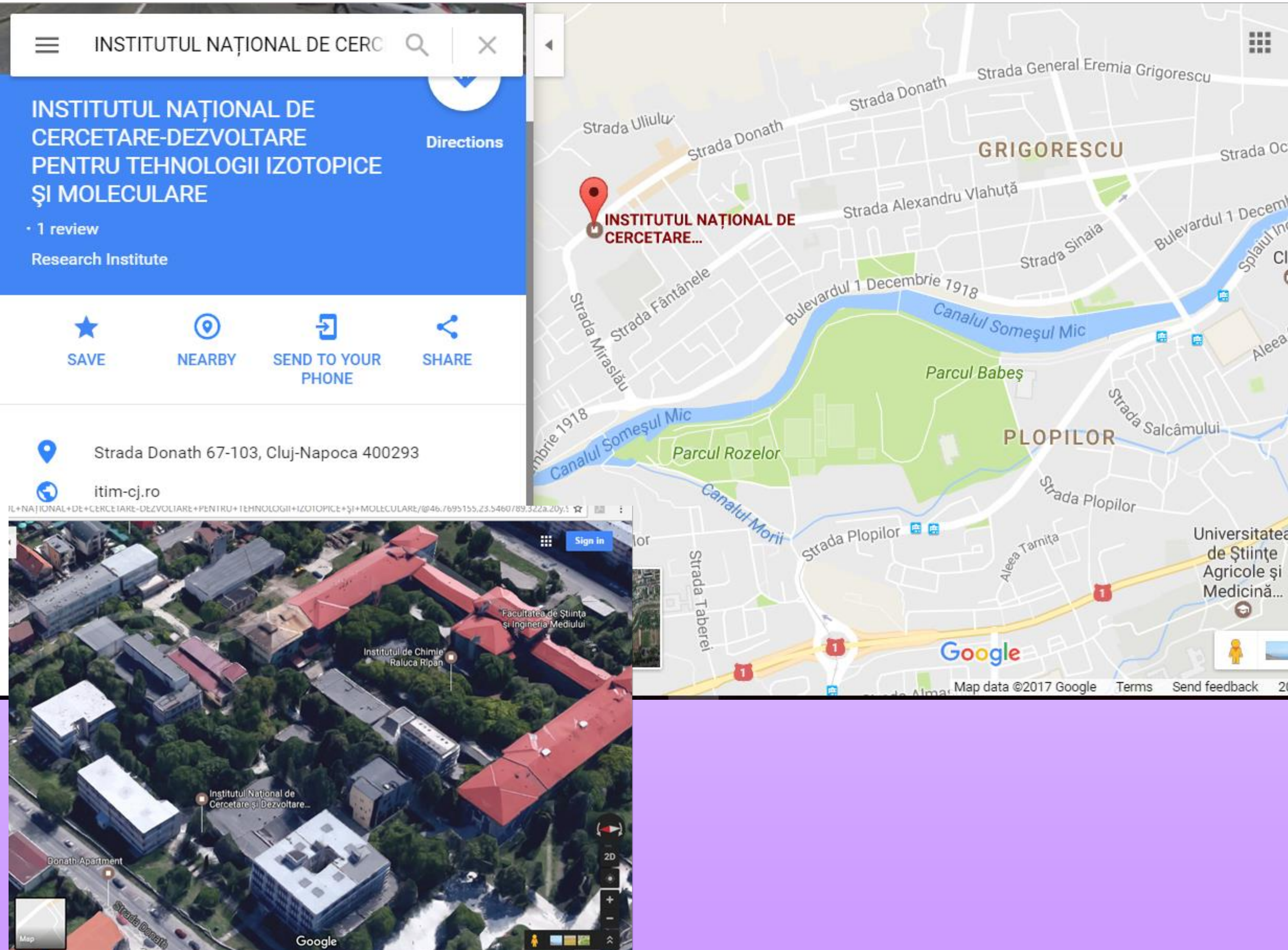
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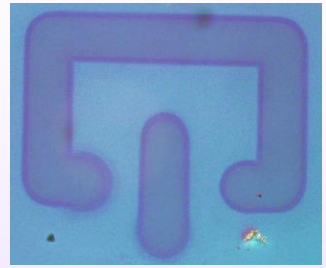
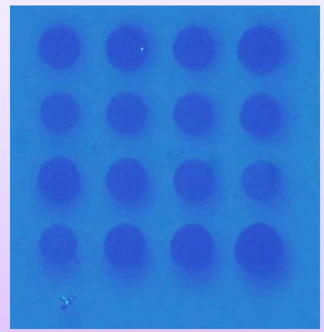
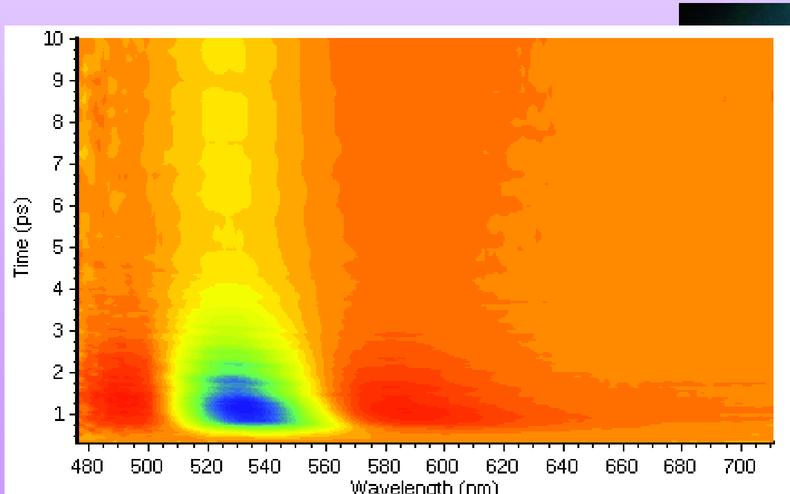
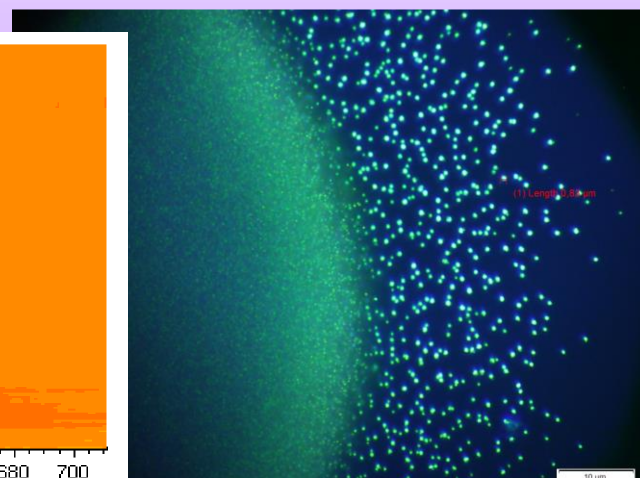








Motivation

Why optical lithography?

- Large number of applications such as optical limiting and 3D fluorescence imaging
- 3D microfabrication for industry (electronics) and 3D data storage.

Why metallic micro/nanostructured materials?

- Larger interaction surfaces than a flat surface
- Localized Surface Plasmon Resonance (LSPR)

Why controlled metallic micro/nanostructured patterns of noble metals?

- Stability of the patterned areas (oxidation proof for gold)
- Tunable sizes and geometries
- Compatibility with biomolecules
- Metallic electrodes for electrochemistry- Interdigitated electrodes with increased sensitivity
- Solid support for SERS detection

Outline

- Optical lithography: origin and key stages
- Direct laser writing
  - TP-induced polymerization
  - Metallic structuring induced in thin films
- Optical microscopy imaging
- SEM and AFM investigations
- Conclusions

Origin of Lithography

Lithography: from Ancient Greek

λίθος, lithos, meaning "stone"

&

γράφειν, graphein, meaning "to write"

“to write on a stone”

Lithography in Art

lithos,
White surface
Limestone

&

black surface

graphein, to write

"to write on a stone"

without carving but etching based on

immiscibility of oil and water

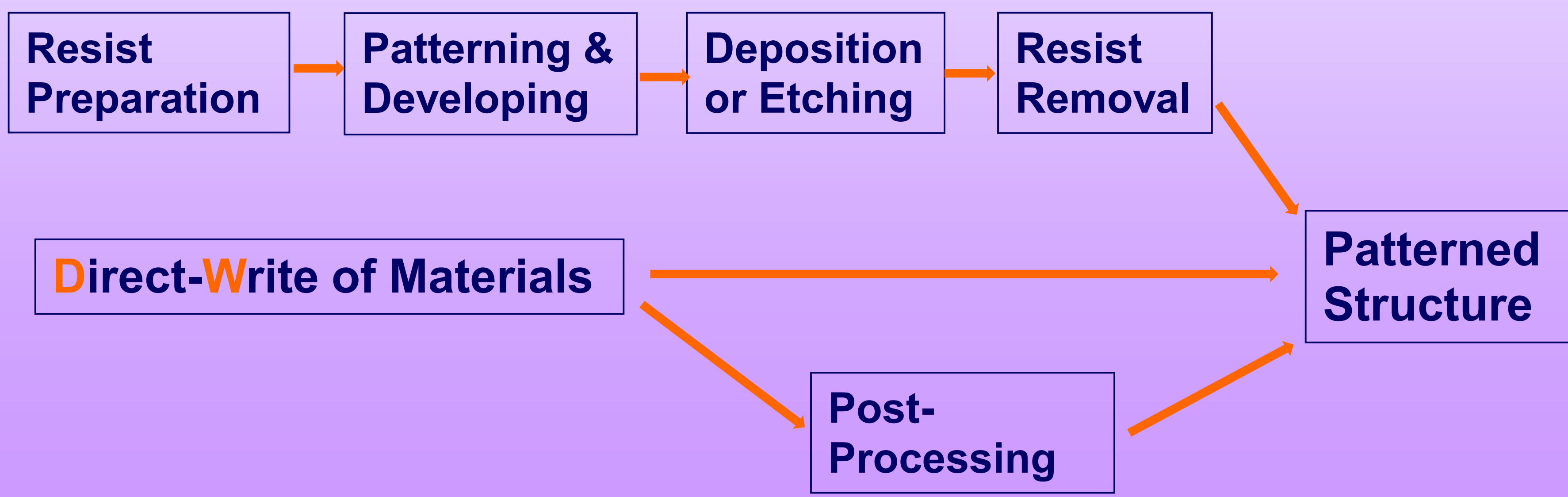
Inventor: 1796, Alois Senefelder, german author and actor

Optical Lithography

"*chisel*" = carving tool

etching process → acid&water system as "chemical *chisel*"

Optical Lithography ← light or laser beam as "optical *chisel*"



Optical Lithography Stages

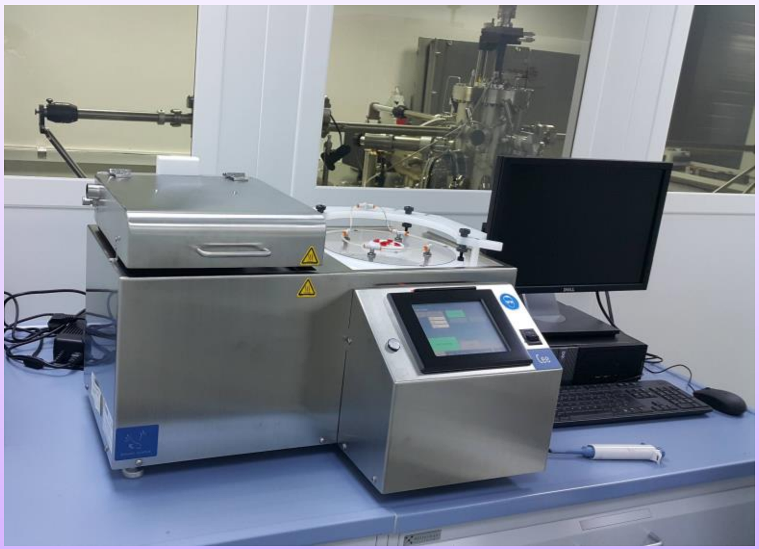
1. Substrate preparation – droplet onto the cover plate or thin films

2. Optical lithographic process itself

3. Developing and characterization of patterned structures

Thin films preparation by “spin-coating”

Parameters setting : speed, acceleration and time



Spin-coater – general view

Parameters setting recipe

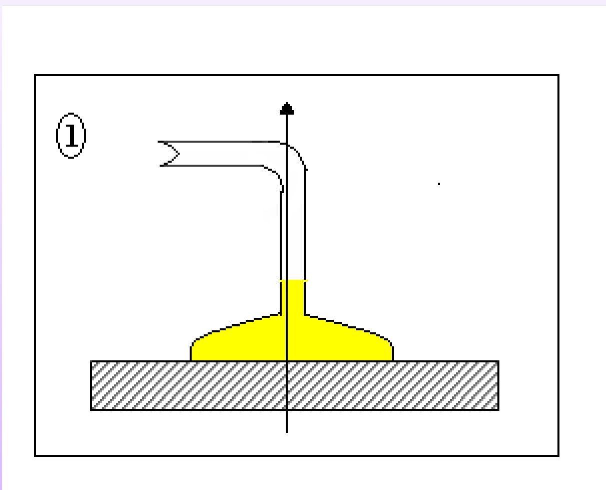
Steps	Speed (rpm)	Acceleration (rpm/s)	Time (s)
1	500	500	5
2	1000	1000	5
3	1500	2000	20
4	500	500	5
5	100	100	5

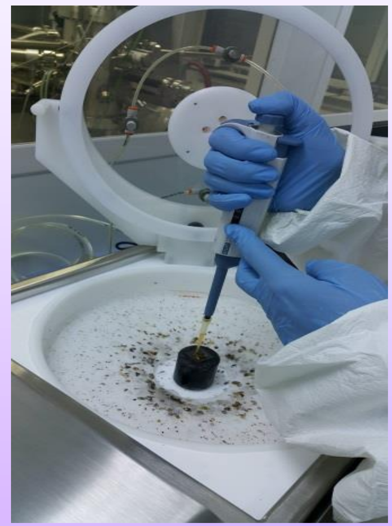
Thin films preparation by “spin-coating”

Substrate placing on the spin-coater holder & holding the vacuum to fix the substrate

The Key Stages in Spin-Coating

Stage 1: The deposition of the coating solution onto the substrate

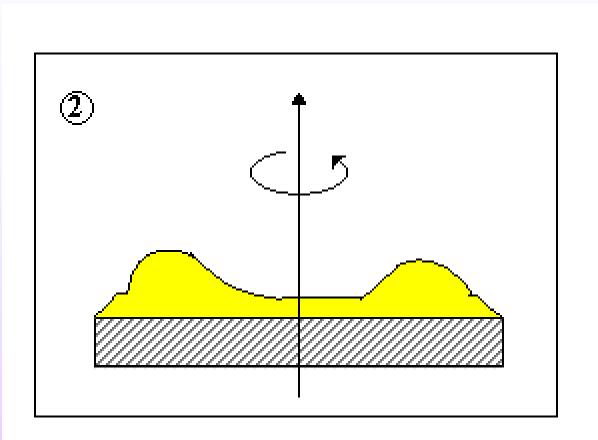


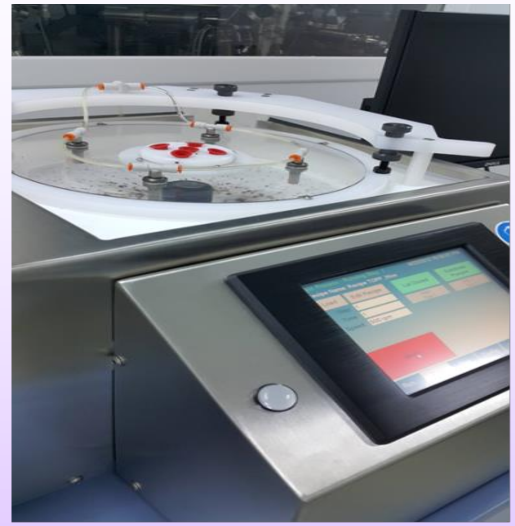


- pouring out or spraying the coating fluid onto the surface
- homogeneous coating fluids for uniform films
- wettability of the coating fluid related to the surface – complete vs partial covering

D. Bornside, C. Macosko, L. Scriven, "On the modelling of spin coating", *J. Imaging Tech.*, 1987, 13, 122-130.

Stage 2: The solution flowing out under the centrifugal forces action





- massif fluid expulsion from the plate surface by the centrifugal forces during the rotation motion

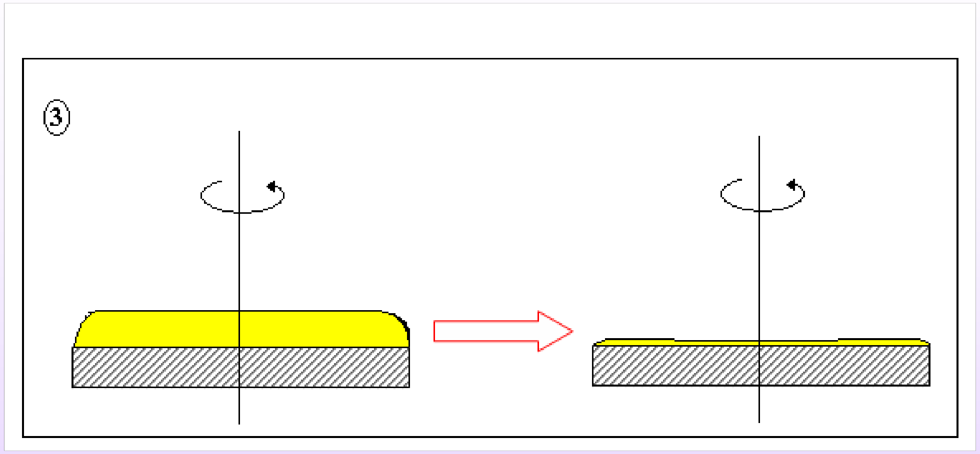
- appearance of vortexes shortly during the process due to the twisting motion, generated by the top of the layer inertia at faster and faster cover plate rotation

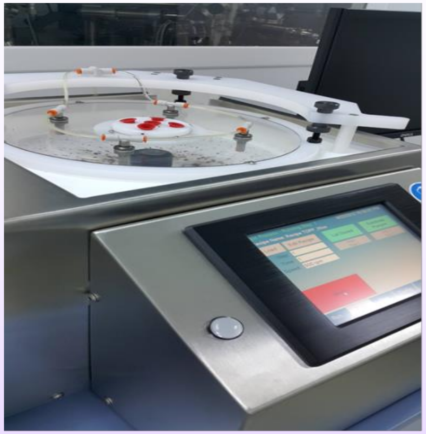
- thin enough fluid layer completely co-rotates with the wafer & no evidence of fluid thickness differences is observed

- the support reaches its desired speed and the fluid is thin enough that the viscous shear drag balances exactly the rotational accelerations.

D. Bornside, C. Macosko, L. Scriven, "On the modelling of spin coating", *J. Imaging Tech.,* 1987, 13, 122-130.

Stage 3: The layer spinning at a constant rate and the fluid thinning behaviour induced by fluid viscous forces





- uniform process using solutions containing volatile solvents which require a lower centrifugal action speed

- the thickness of the layer is reduced more by the solvent evaporation

- the solvent evaporation increases the viscosity and reduces the solvent diffusion through the film

- equilibrium between the centrifugal forces, which push back the liquid outward, and the opposed viscosity forces.

- appearance of the "edge" effect at the margins of glass cover plate

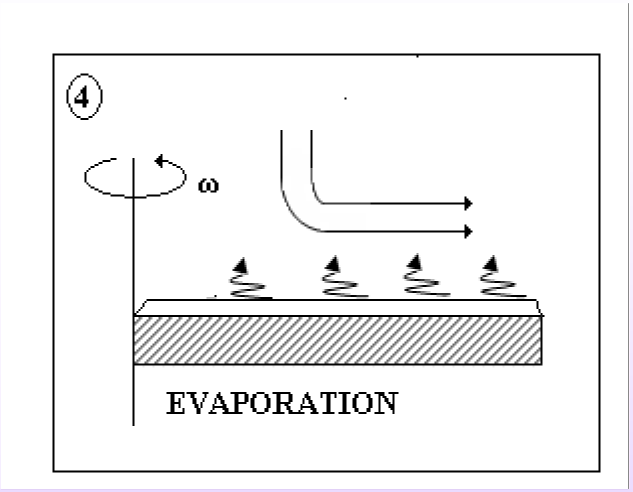
- the deposition solutions may be considered as being newtonian liquids with the viscosity independent on the shearing constraints

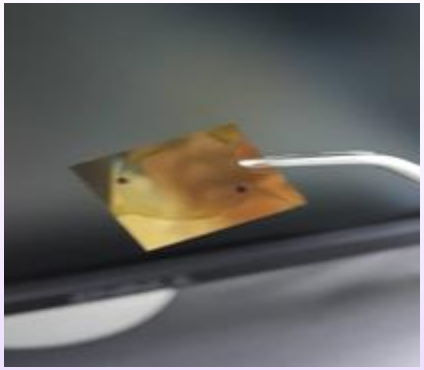
thickness of the layer at the end of the process: $e = e_0/(1 + 4\rho\omega^2 e_0^{\ 2} t/3\eta)^{1/2}$

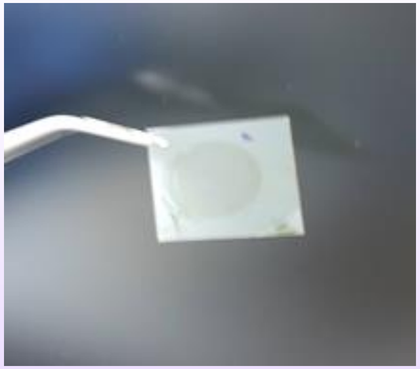
viscosity (η), the rotation rate(ω), the liquid density(ρ), rotation time(t) and the initial thickness(e_0)

A. Emslie, F. Bonner, L. Peck, "Flow of the viscous liquid on a rotating disk", *J. Appl. Phys.,* 1958, 29, 858-862.
D. Meyerhofer, "Characteristics of resist films produced by spinning", *J. Appl. Phys.,* 1978, 49, 3993-3997.
D. Bornside, C. Macosko, L. Scriven, "Spin coating: one-dimensional model", *J. Appl. Phys.,* 1989, 66, 5185-5193.

Stage 4: The layer spinning at a constant rate and the coating thinning behaviour dominated by solvent evaporation







Thin films

- the coating effectively "gels" on the substrate

- the viscosity of the remaining solution will rise likely freezing the coating in place

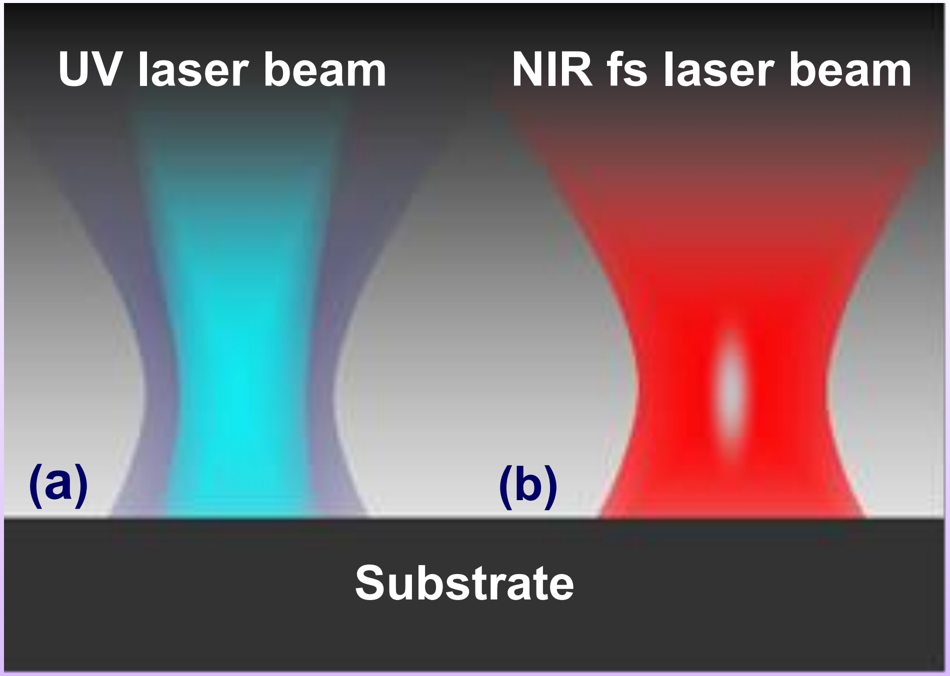
- *viscous flow and evaporation* must undergo simultaneously throughout the spinning (Stages 3&4)

- *viscous flow effects early dominate on as time* must undergo simultaneously throughout the spinning

- *evaporation processes* dominate later

D. Bornside, C. Macosko, L. Scriven, "On the modelling of spin coating", *J. Imaging Tech.,* 1987, 13, 122-130.

Laser Regime for Direct Writing (DLW)



1-photon absorption (**OPA**) **vs** 2-photon absorption (**TPA**)

Materials (monomers or oligomers) doped with specific molecules capable to absorb at 1-photon and 2-photon

Irradiation and **photopolymerization** of photosensitive substrates by **UV (a)** and **NIR fs (b)** laser radiation, respectively.

2-Photon Absorption (**TPA**)

In 1931, M. Göppert-Mayer has theoretically predicted that all non absorbing materials become absorbing by the simultaneous absorption of two photons when they are irradiated by a large density of photons.

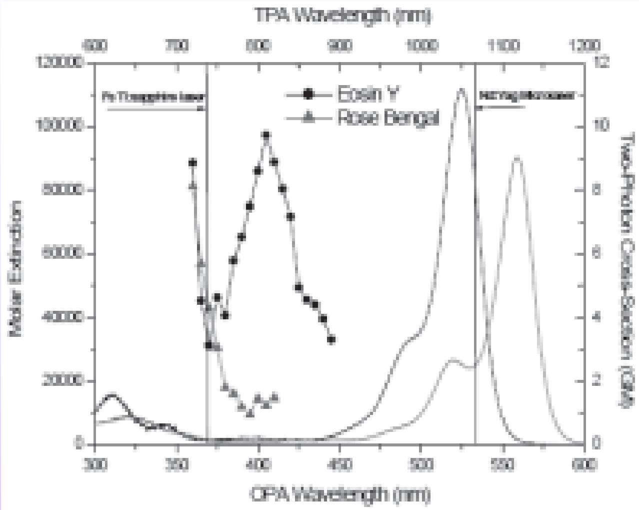
At present, this nonlinear absorption can easily be obtained at the focal point of lasers with conjugated organic compounds exhibiting large optical nonlinearities.

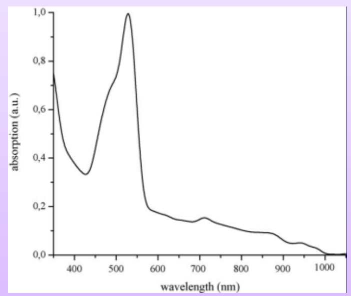
Push-pull molecules of type A-*π*-D. D-*π*-D or D-*π*-A-*π*-D type
A acceptor group and D – donor group
π - charge transfer system,

M. Göppert-Mayer, Über Elementarakte mit zwei Quantensprüngen. *Ann. Phys. 1931,* 9, 273–294.

TPA Optical Lithography

2-photon absorption (**TPA**)

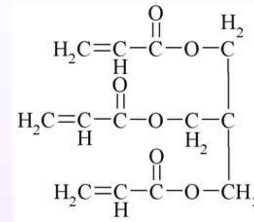




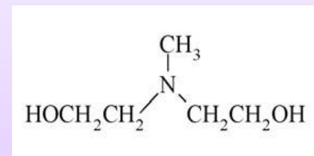
1-photon absorption (**OPA**)
in **450-550 mn**

situated in the TPA window for laser

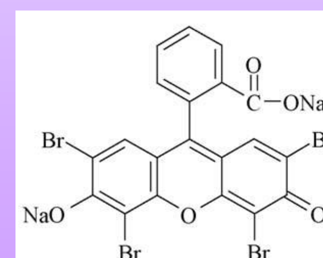
Absorption in the green range due to Eosin Y



Pentaerythritol Triacrylate (PETIA)
multifunctional ligand (monomer)



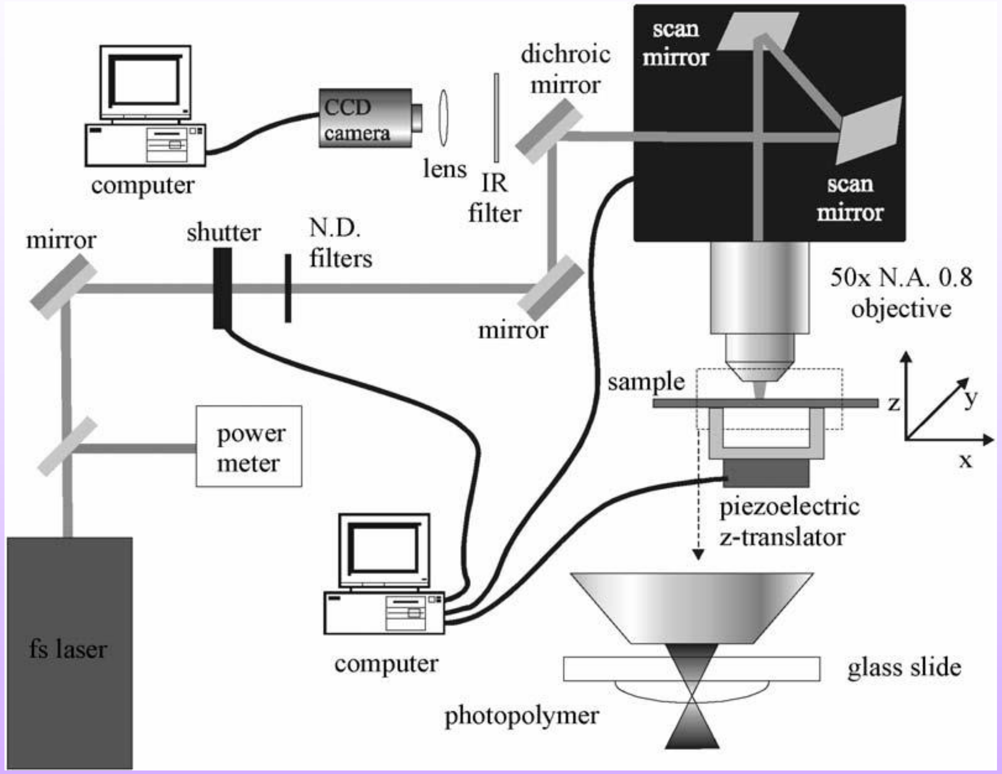
N-methyl Diethanolamine (MDEA)
co-initiator amine, oxidized by a triplet state of the dye



Eosin Y - iniitietor
(2, 4, 5, 7-tetrabromo fluoresceindisodium salt
Sensitizer dye – TPA absorption in IR

M. Farsari, G. Filippidis, K. Sambani, T. S. Drakakis, C. Fotakis, J. Photochem. Photobiol. A: Chemistry, 2006, 181, 132–135..

TPA Optical Lithography



Experimental set-up

1028 nm fs laser

200 fs pulse duration

50 MHz repetition rate

1 W average power

50x objective, NA = 0.8

Lateral resolution

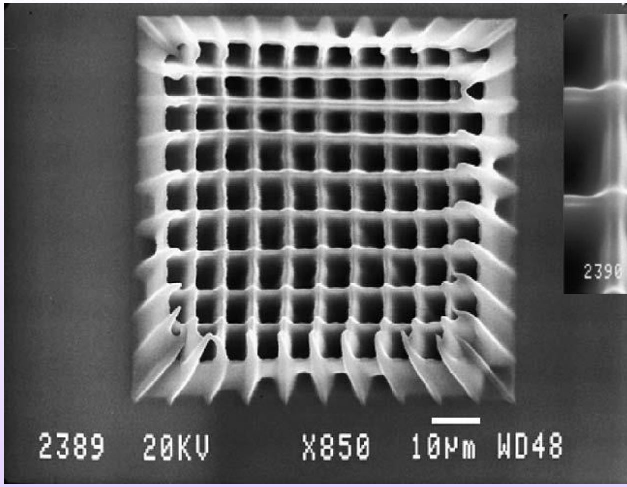
$r_l = 0.61\lambda/NA$ **785 nm**

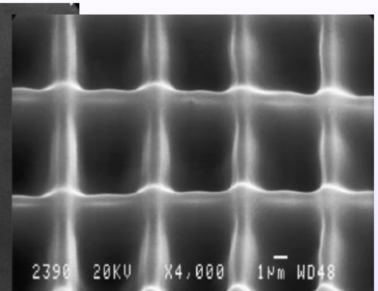
Axial resolution

$r_a = 2\lambda n/NA^2$ **3.2 µm**

M. Farsari, G. Filippidis, K. Sambani, T. S. Drakakis, C. Fotakis, J. Photochem. Photobiol. A: Chemistry, 2006, 181, 132–135..

TPA Optical Lithography





Detail feature

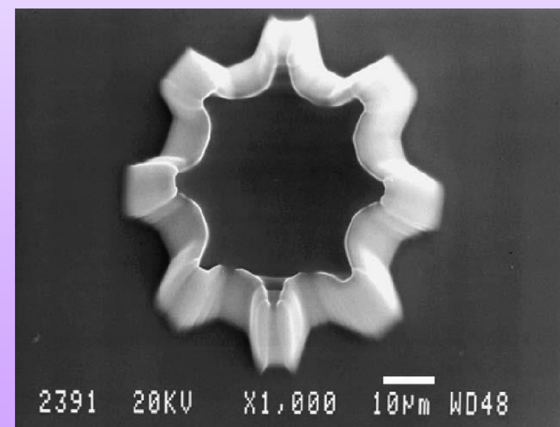
Resolution = 1 µm

Distortion effect due to polymer shrinkage

Error source: use of monomer instead of olygomer

Structure obtained by microstereolithography – SEM image

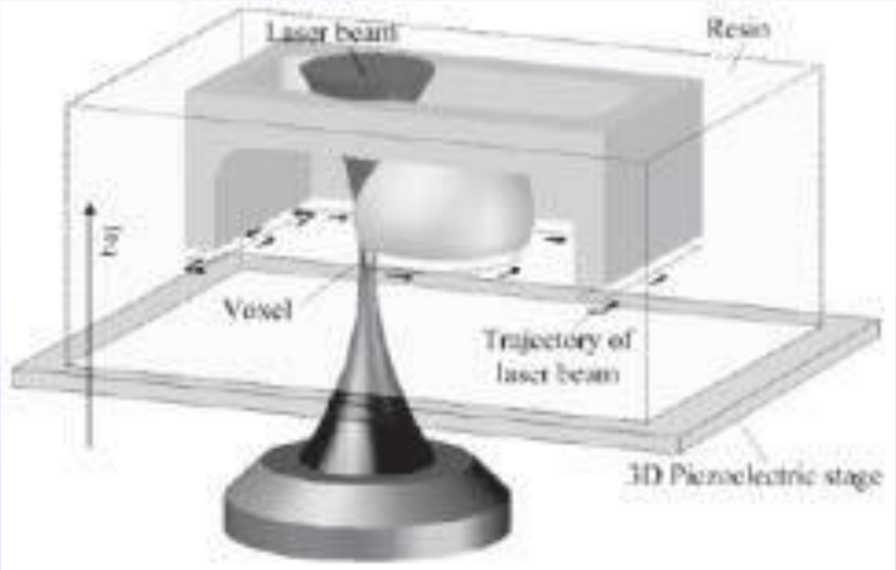
Layer spacing = 1 µm



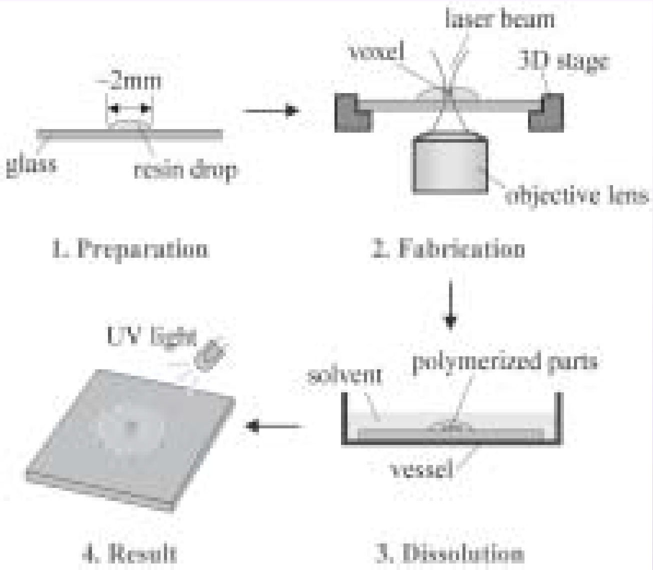
A hollow micro-gear - SEM image

M. Farsari, G. Filippidis, K. Sambani, T. S. Drakakis, C. Fotakis, J. Photochem. Photobiol. A: Chemistry, 2006, 181, 132–135..

Two-Photon Induced Polymerization



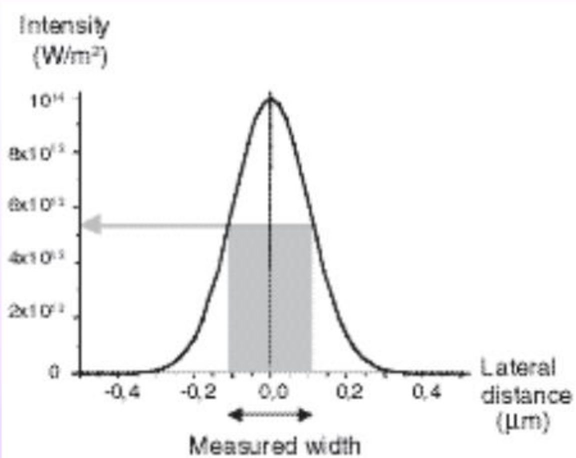
Schematic 3D microfabrication
by TP polymerization (**TPA**)

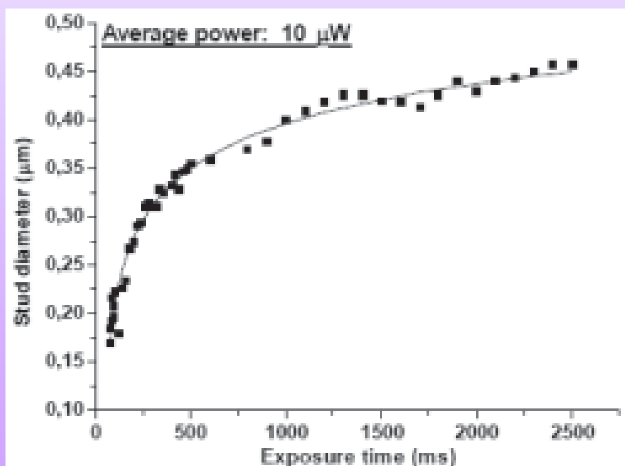


Microfabrication steps of
TP - induced polymerization

(a) T. W. Lim, S. H. Park, D.-Y Yang, *Microelectron. Eng. , 2005,* 77, 382–388; (b) K. Takada, H.-B. Sun, S. Kawata, *Appl. Phys. Lett.,* 2002, 86, 071122/1–071122/3.

Two-Photon Induced Polymerization





Voxel lateral size on the exposure time dependence

Voxel lateral size

$$v_a = w_0 \cdot \left[\left(\frac{4 \cdot P^2 \cdot t}{\pi^2 \cdot w_0^4 \cdot E_{th}}\right)\right]^{1/2}$$

Voxel vertical size

$$v_b = \frac{2\pi \cdot w_0^2}{\lambda} \cdot \left[\left(\frac{4p^2 \cdot t}{\pi^2 \cdot w_0^4 \cdot E_{th}}\right)^{1/2} - 1\right]^{1/2}$$

λ - wavelength,
w_0 - beam waist, P - laser power,,
E_{th} - threshold energy for photo polymerization
t - exposure time, respectively.

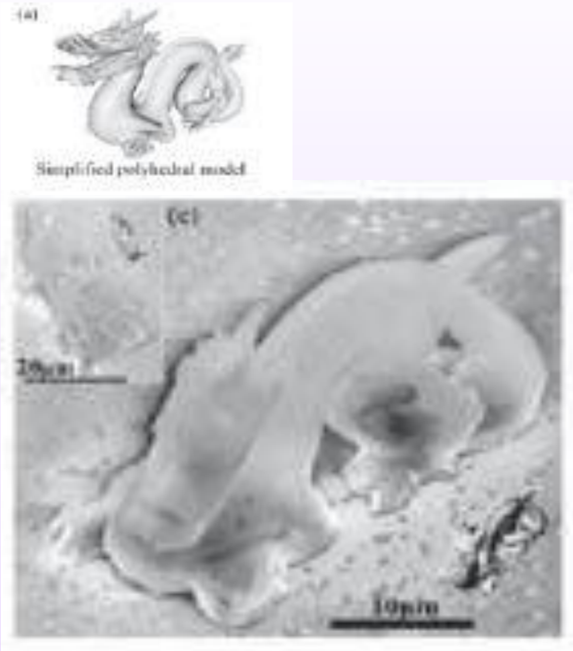
P.L. Baldeck, O. Stephan and C. Andraud, *Nonlinear Optics and Quantum Optics,* 2010, 40, 199–222.

Fabrication Time

Fabrication time = f(elementary time)

time needs to polymerize a voxel 1-10 ms/voxel

scanning speed (voxels connection) tens of μm/s



Dragon with large near flat surfaces
by 3D generalized layer by layer strategy

Fabrication time = 19 minutes

z-translation
axis

3D micro-objects that mixes 1D, 2D and 3D features by path planning strategy

Fabrication time = 12 hours

Time Optimization: microlens array(a) or holografic multiple spots (b)

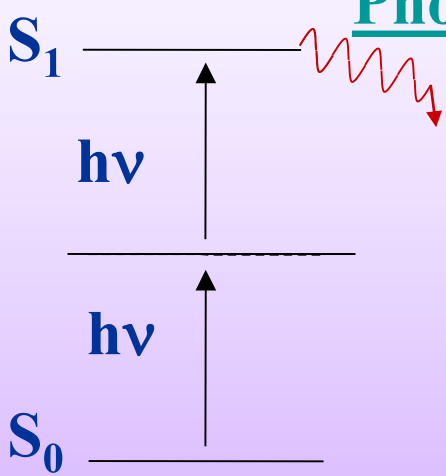
C.Y. Liao, M. Bouriau, P.L. Baldeck, J.C. Leon, C. Masclet, T-T. Chung, *Appl. Phys. Lett.* 2007, *91*, 033108, 1-3.

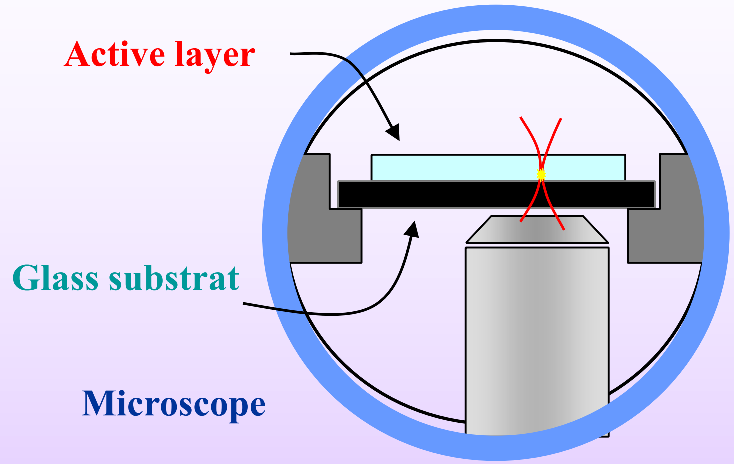
·(a) J. Kato, N. Takeyasu, Y. Adachi, H. B Sun. and S. J.. Kawata, *Appl. Phys. Lett.* 2005, 861, 044102; (b) S. H. Park, T. W. Lim, S. H. Lee, D. Y. Yang, H. J. Kong and K. S. Lee, *Polymer-Korea*, 2005, 29 , 146–150.

Two-photon Induced Metal Chemistry

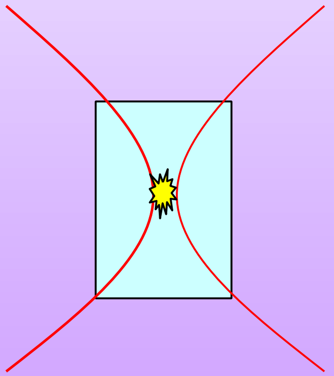
Two-photon absorption

Photo-chemistry



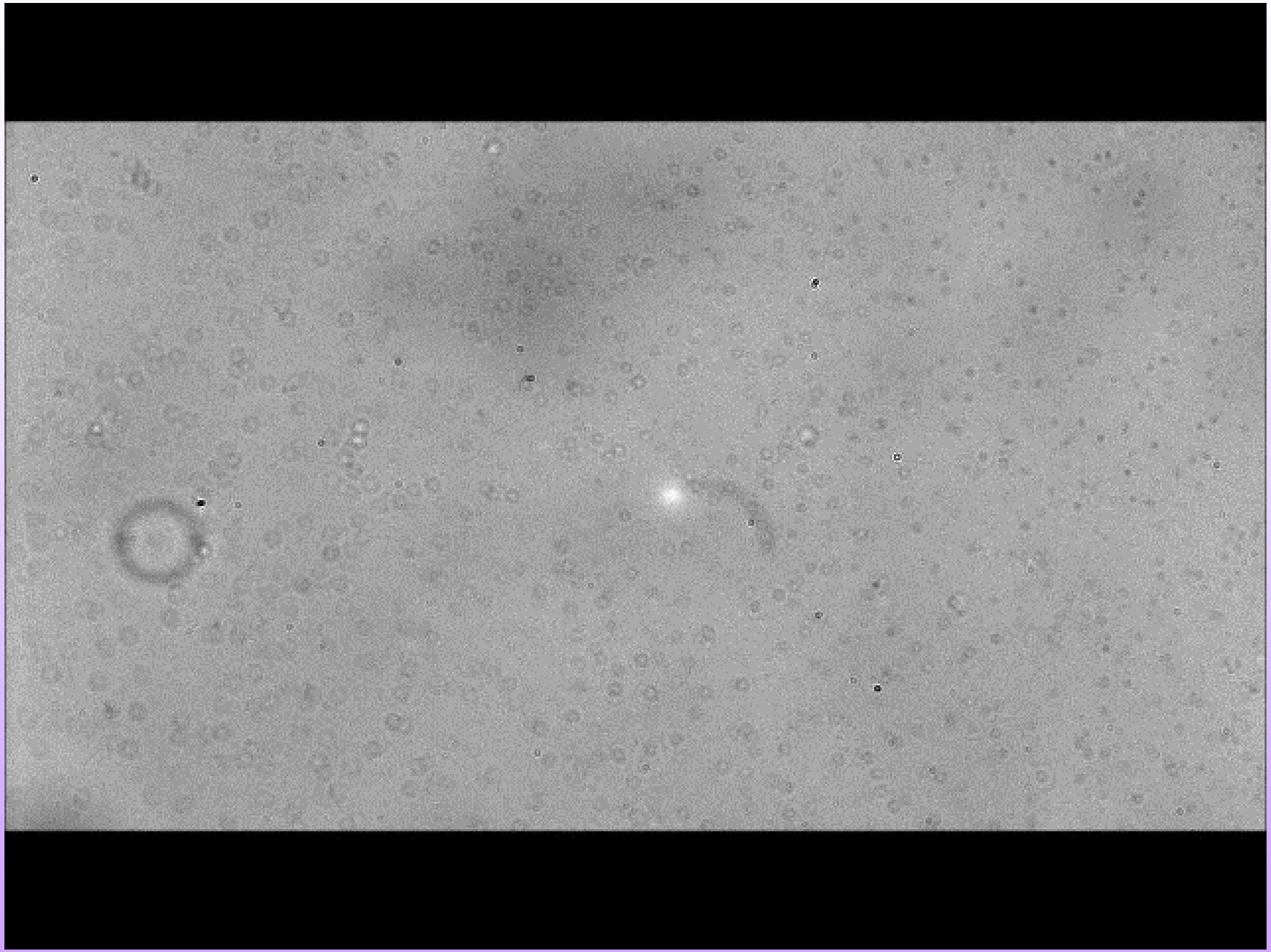


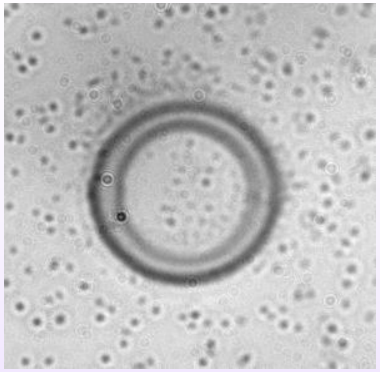
Nonlinear absorption = αI^2
confined at the focal point



Metallic Cations Photo-reduction → ***Neutral Metal Deposition***

MOVIE…





Metallic nanowire fabrication… LIVE

The Photo-reduction Process

- Gold/metallic salt very soluble in water: **$HAuCl_4$**

- Photo-sensitizer
  Sodium citrate & Polyvinylpyrolidone (PVP)

- Reduction reaction involving Metallic cation in Polymer matrix :

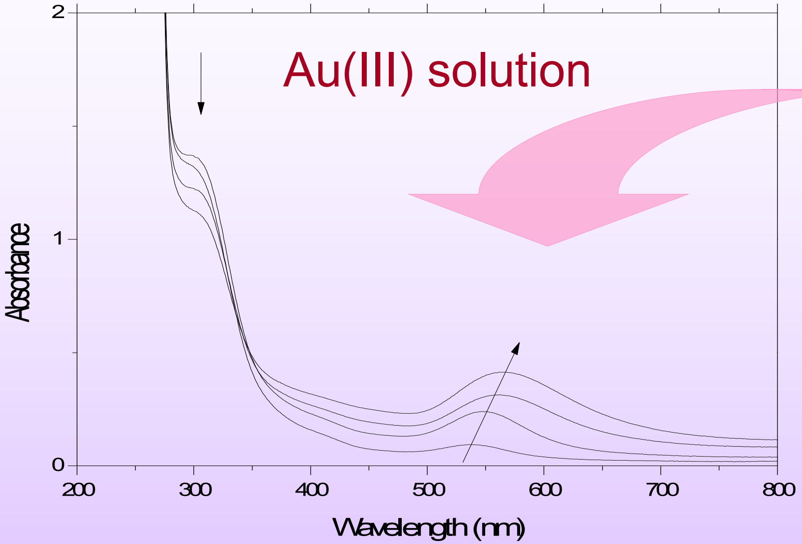
Au (III) → Au (0)

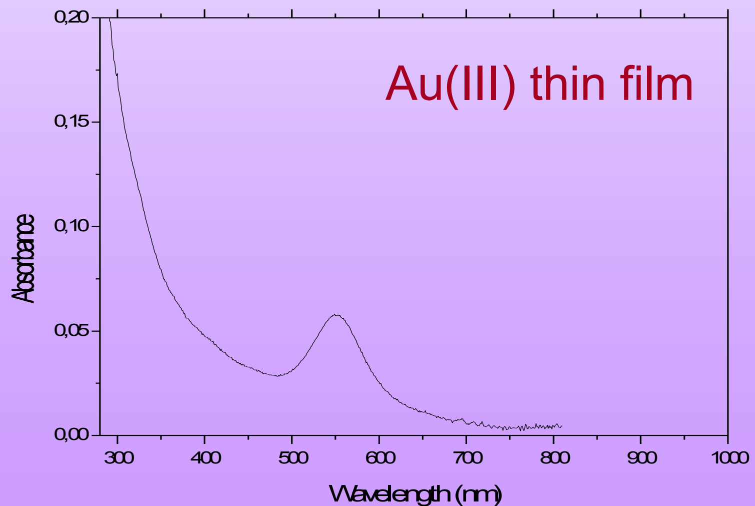
($HAuCl_4$)

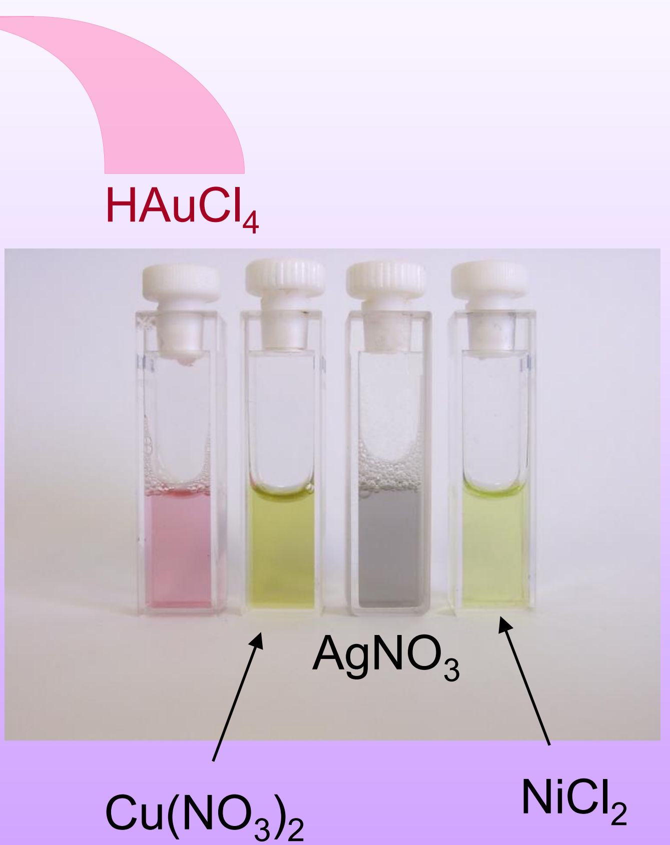
soluble in water

insoluble in water

UV Photo-reduction of Metallic Cations

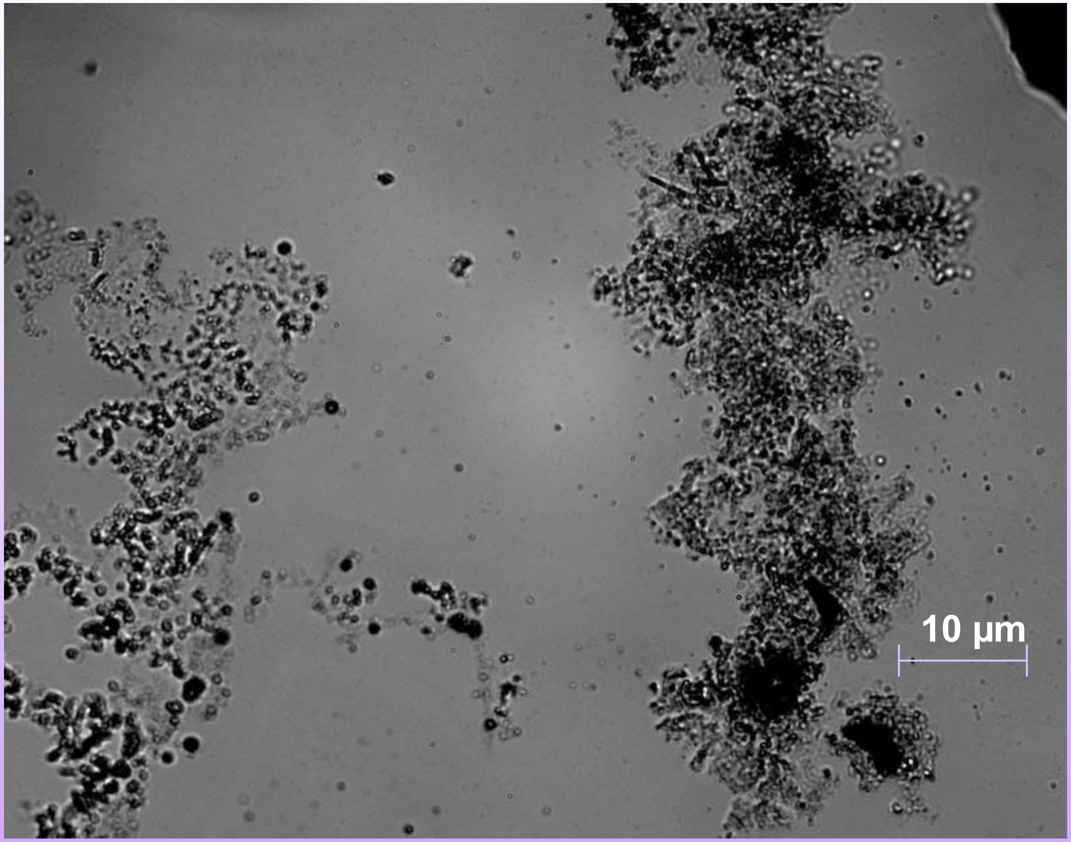






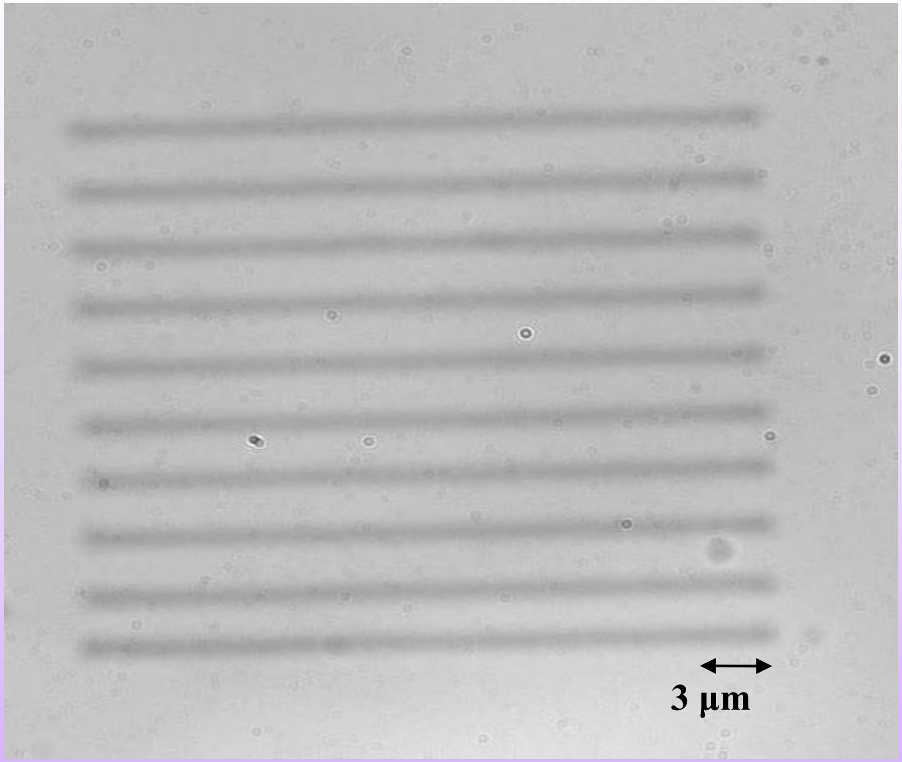
J. Bosson-Ehoomann *et al*, *Nonlin. Opt. and Quant. Opt.*, **2006**, X(19), 1-6.

Two-Photon Generation of Nanoparticles in Water



Gold colloid in water
(optical image in transmission)

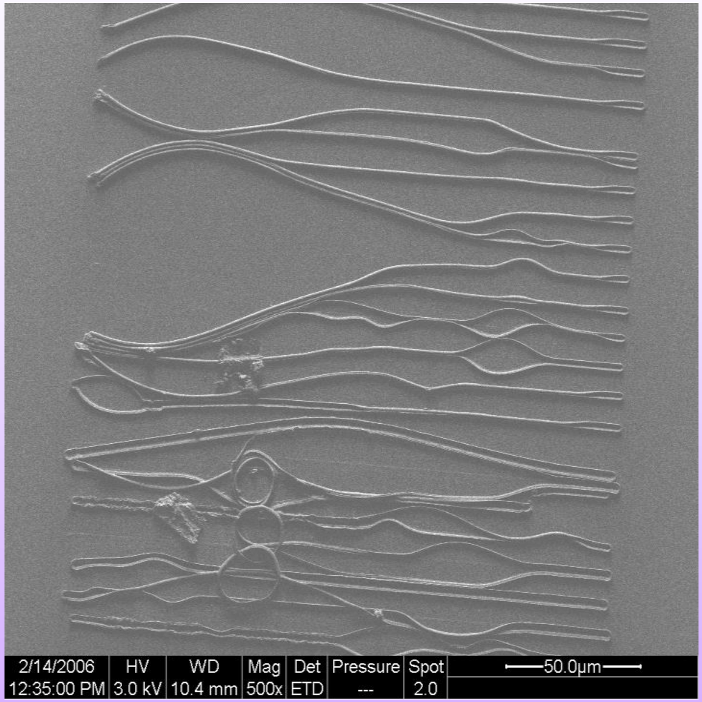
Photo-precipitation in Viscous Medium

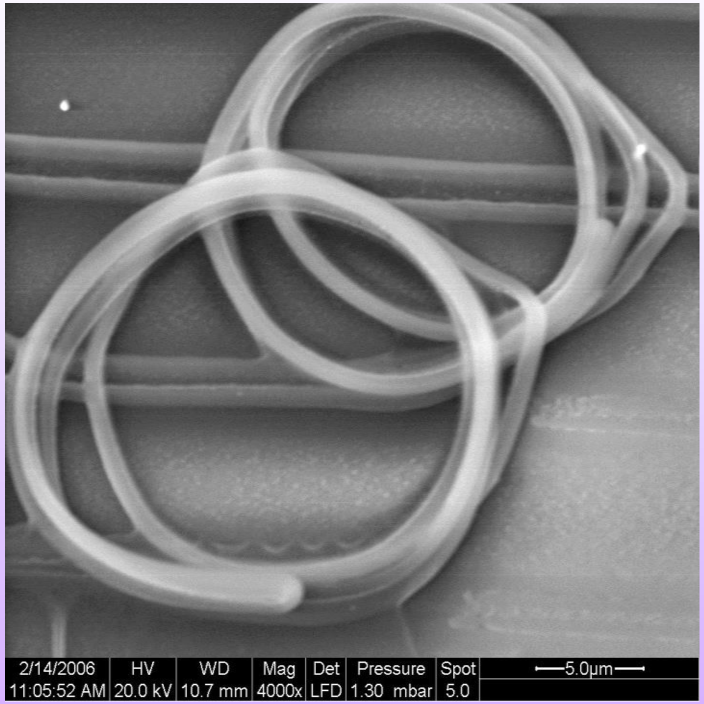


Optical image of horizontal gold wires (in transmission)

Hydrosoluble polymer: Polyvinylpyrolidone (PVP)

Gold Nanowires Fabrication

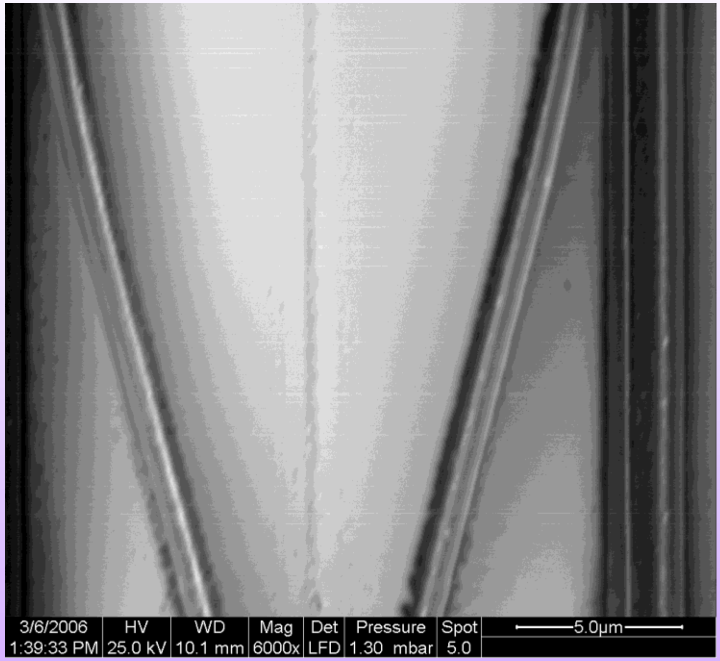


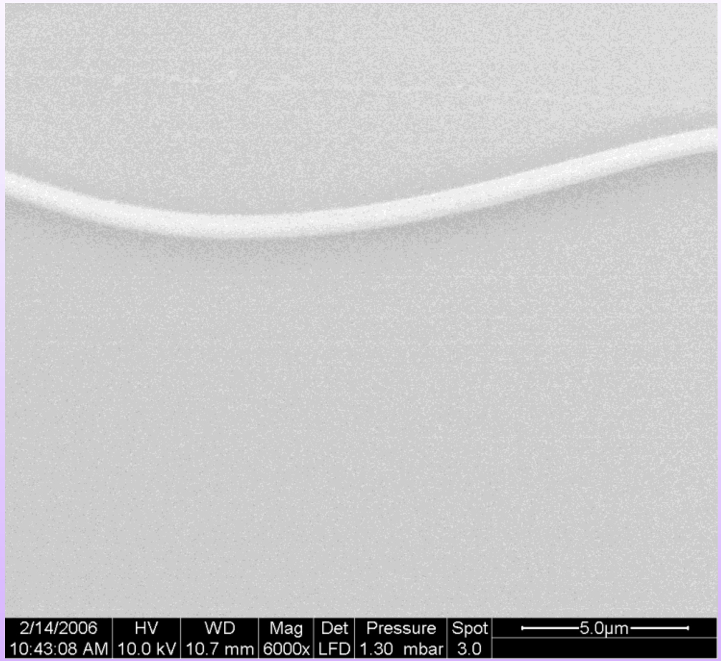


Gold wires on untreated glass - disconnected from the substrate

Continuous wires are obtained

Gold Nanowires Fabrication

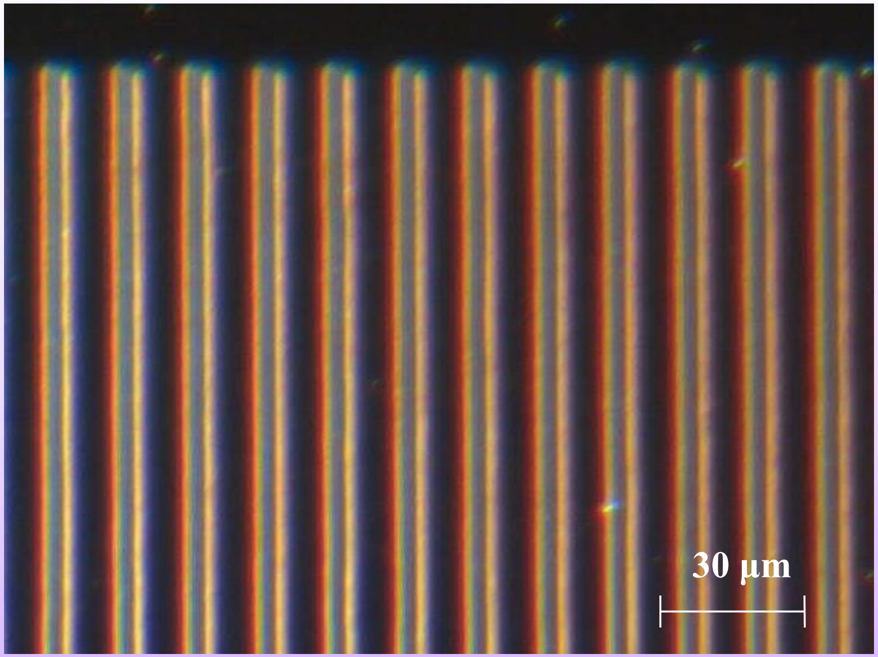




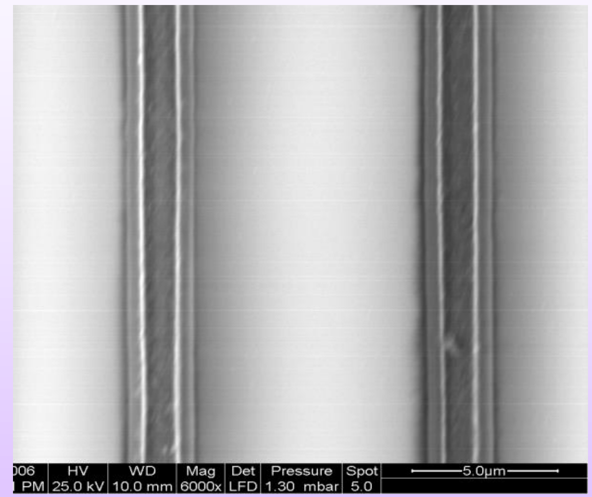
Gold wires on untreated glass - disconnected from the substrate

Regular width wires are obtained

Gold Wires on Polyimide Underlayered Glass



Optical image of a gold wires array (in dark-field scattering)

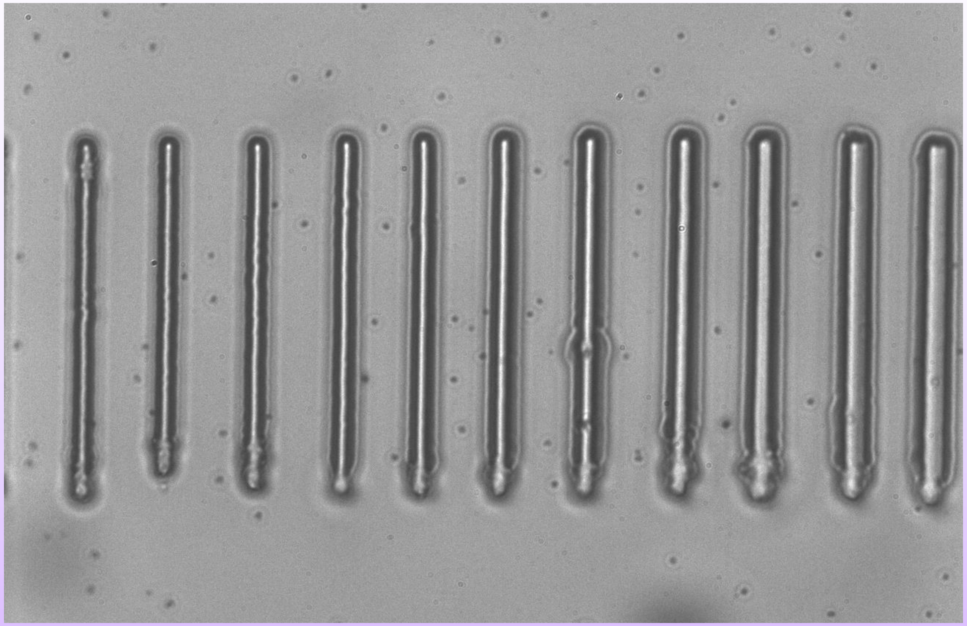


SEM image of two gold double wires

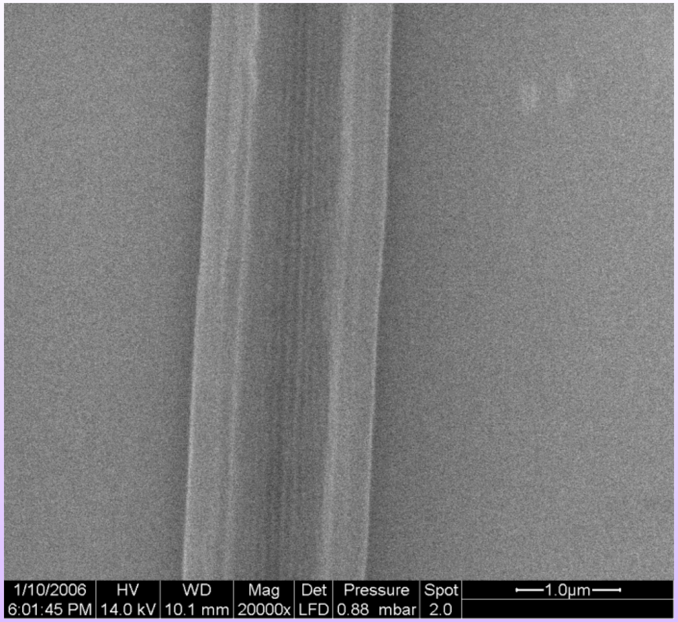
Strong gold-substrate adhesion
due to the coordinative bonds between gold and polyimide

N. Tosa *et al.*, *Proc. of SPIE,* 2006, 6195, 1-8.

Influence of the power



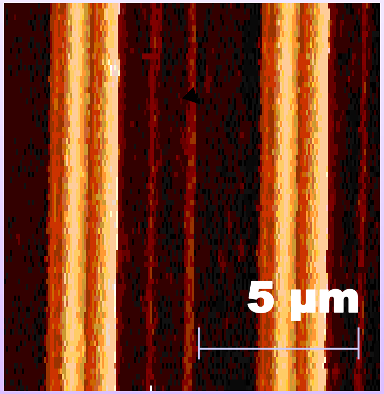
Optical image of a gold wires array (phase contrast)



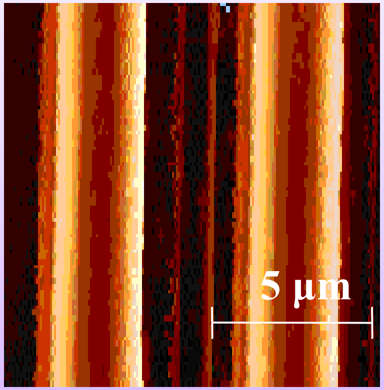
SEM image of a gold double wire

Regular aspect wires with larger width at increased powers (20-80 mW)

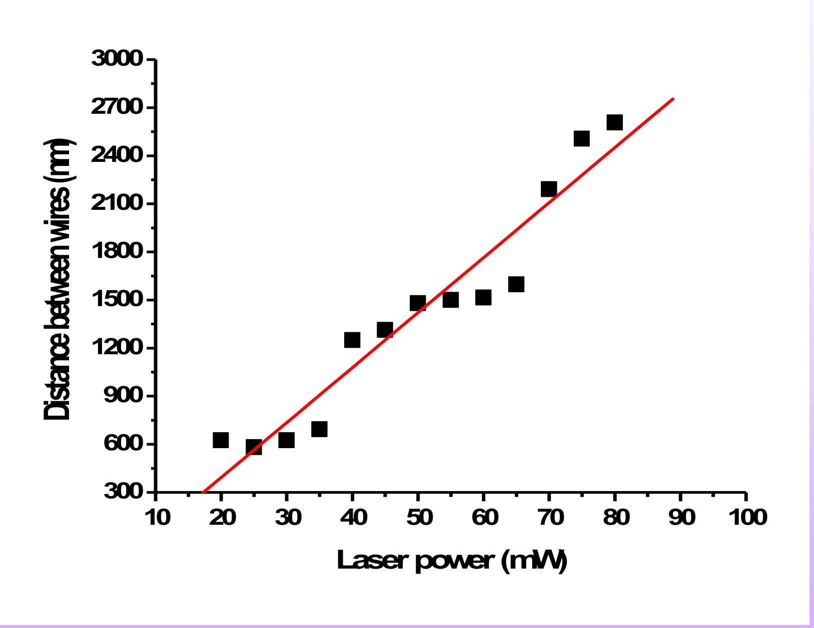
Distance between Wires vs Laser Power



35 mW

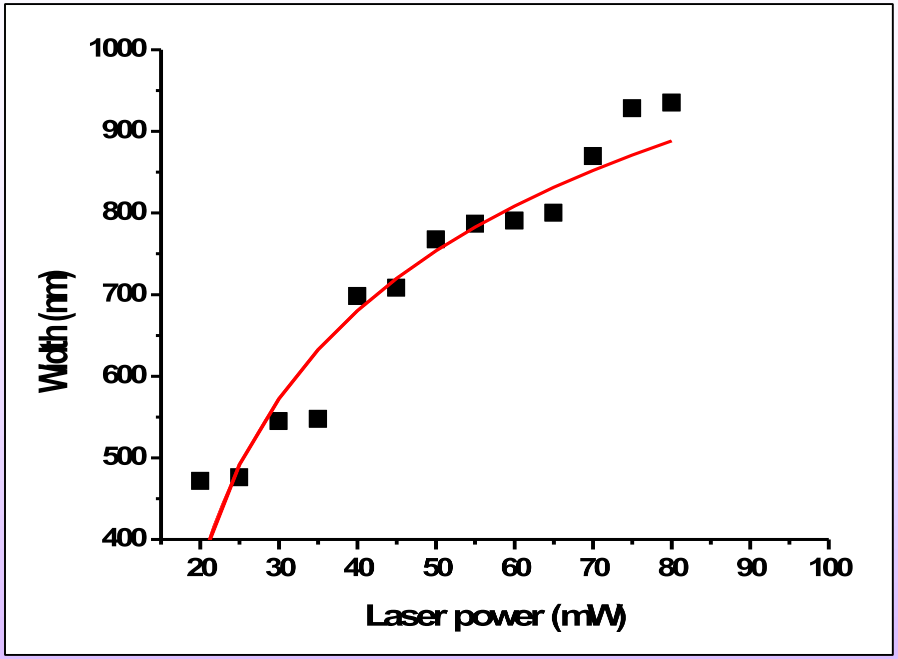


80 mW



AFM top view images of two double-wires at different laser powers

Width of the Wire vs Laser Power



$$I = I_0 \exp(-2r^2 / w_0^2),$$

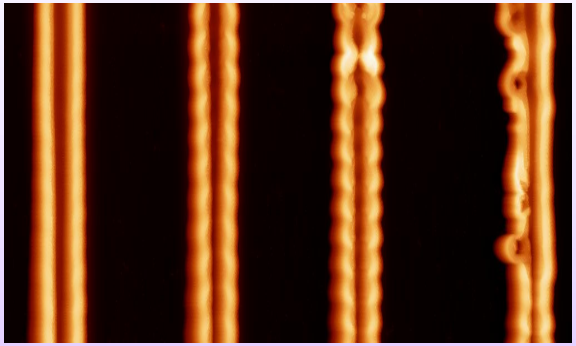
(Gaussian beam)

w_0 - the beam waist

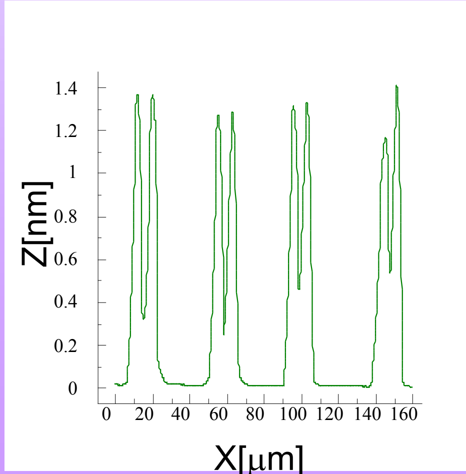
r - the beam radius

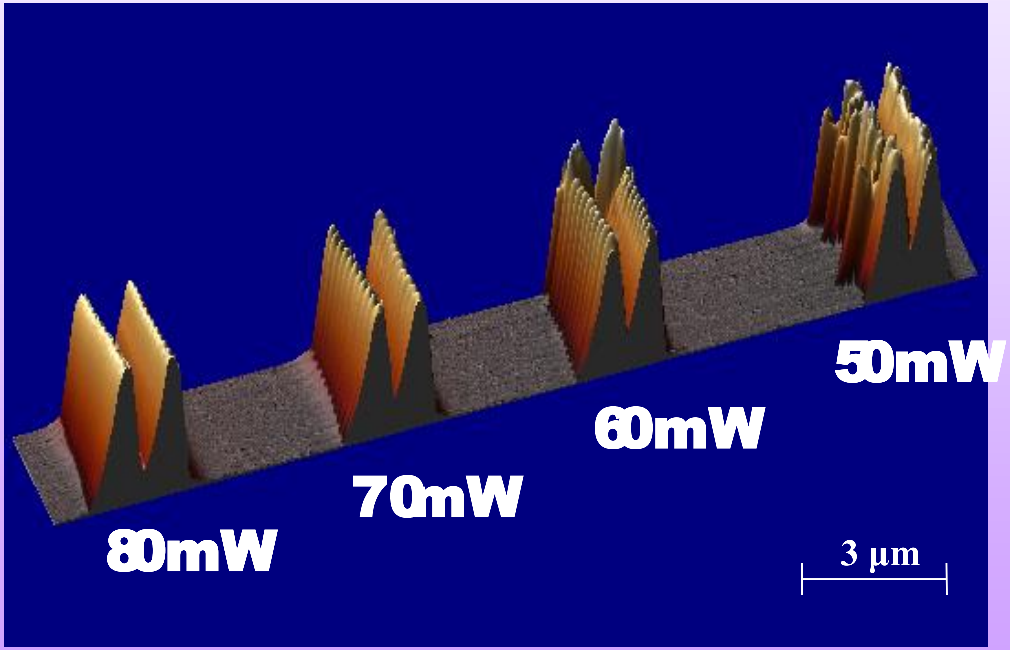
Smoothness of the Wires vs Power

AFM top view, cross-section and 3D images of gold wires at various laser power

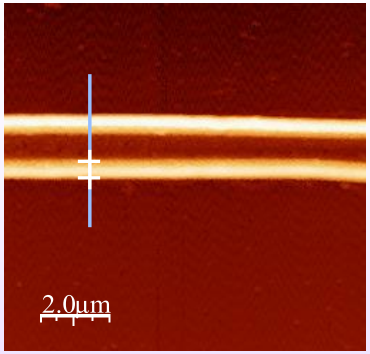


80 mW 70 mW 60 mW 50 mW



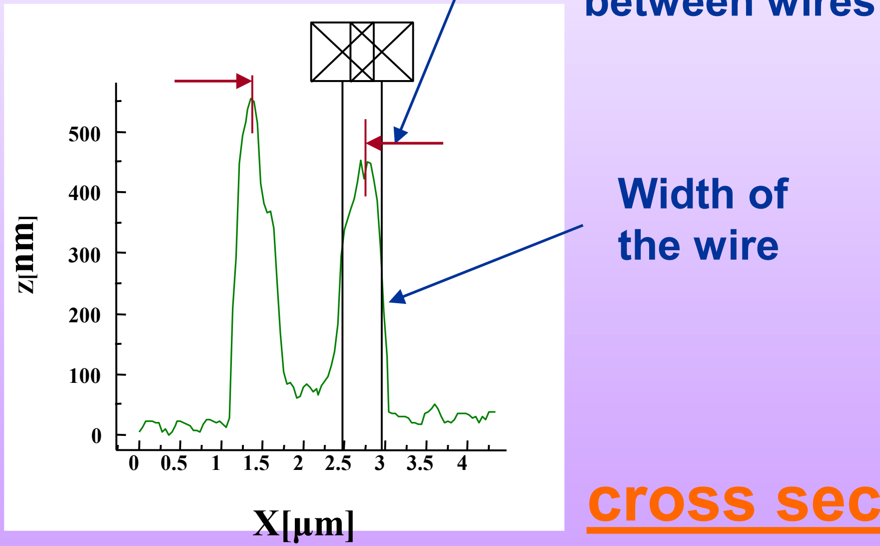


Double Wire

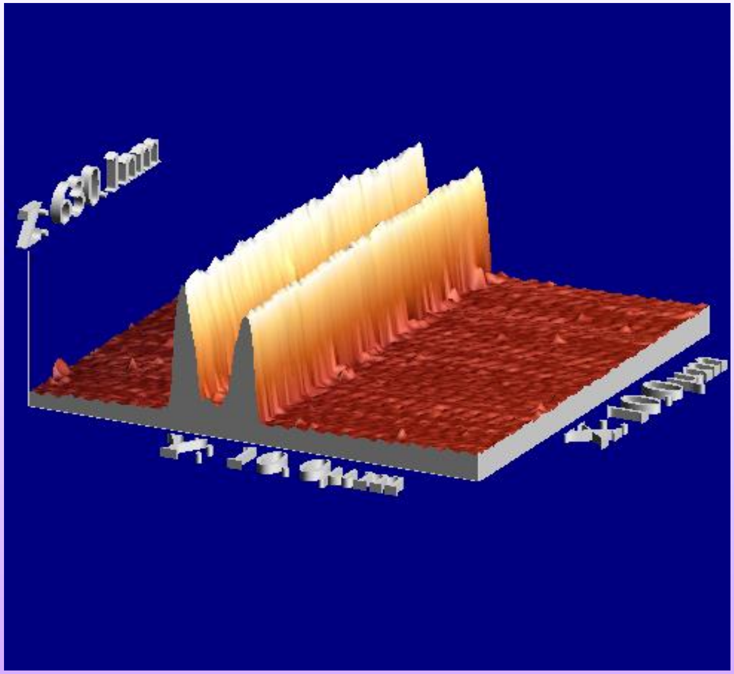


top view

AFM measurements of typical gold wire



cross section

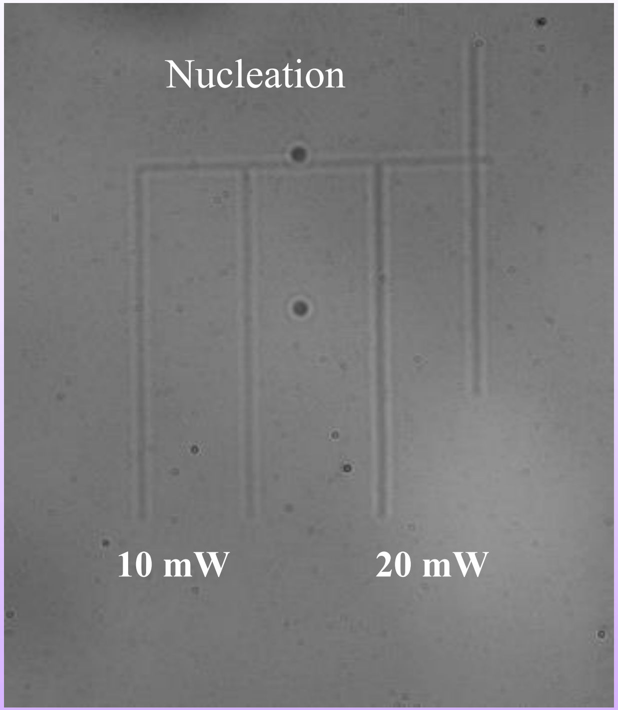


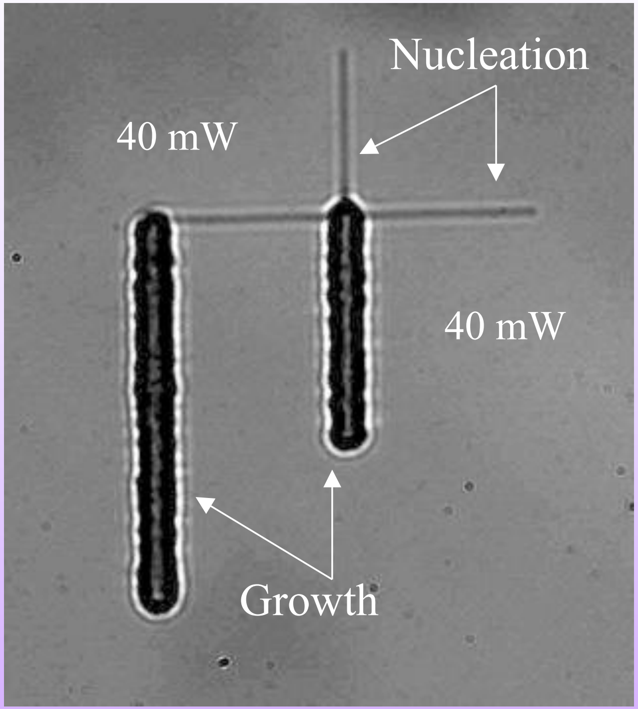
3D view

Double wire due to the thermal effect induced by the colloids during the laser irradiation of the sample

N. Tosa *et al.*, *J. Optoelectron. Adv. Mater*, 2007, 9(3), 641-645

The origin of the double-wire

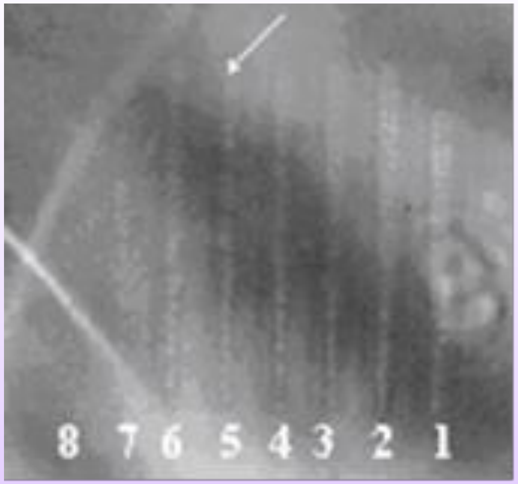


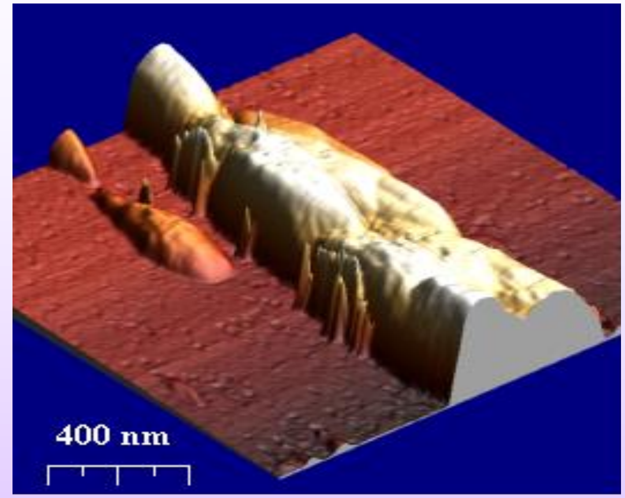


The structures are very sensitive to the "writing" conditions

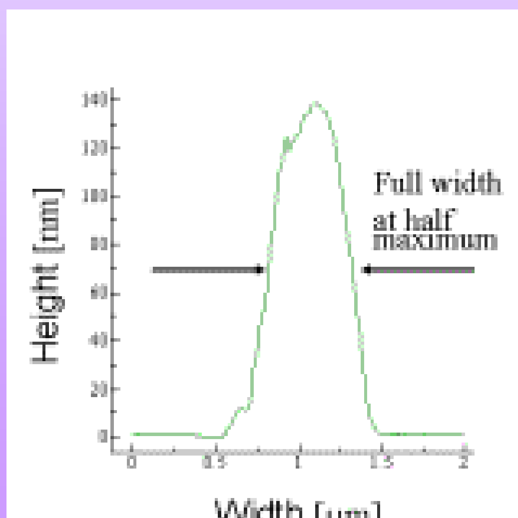
N. Tosa, G. Vitrant, P. L. Baldeck, O. Stephan, I. Grosu, *J Optoel.Adv.Mater.* 2008, *10*, 2199-2204.

Single wire





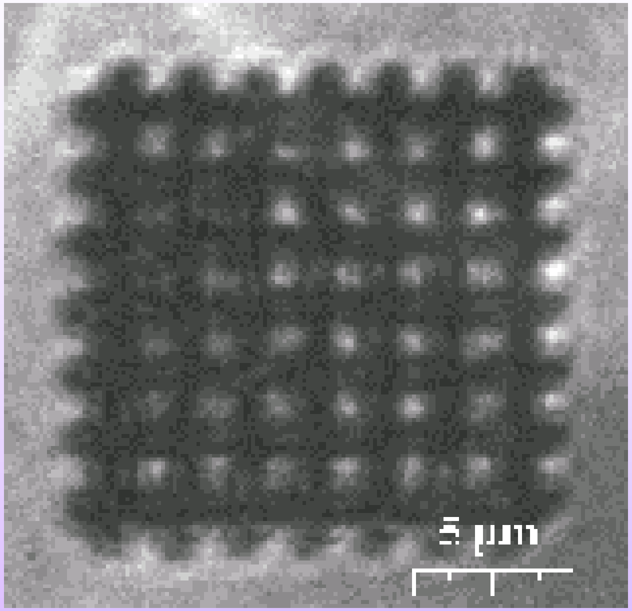
a) 3D view



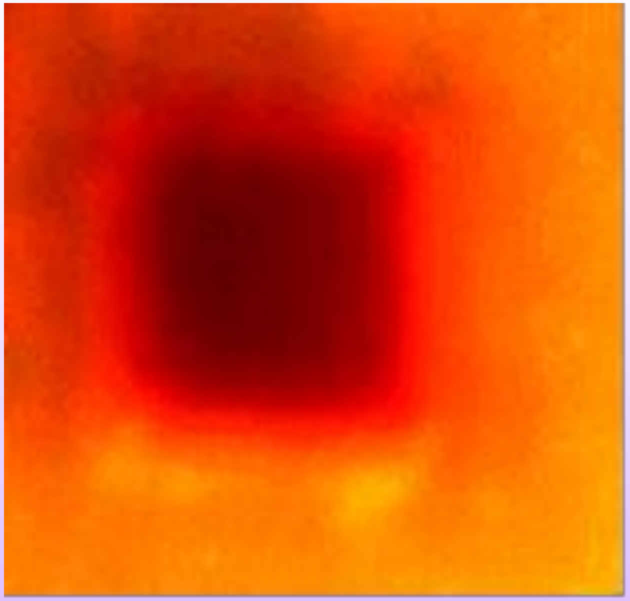
b) Cross-section

N. Tosa, G. Vitrant, P. L. Baldeck,
O. Stephan, I. Grosu,
J. Optoelectr. and Adv.Mater.
2008, *10*, 2199-2204.

Gold 3D structures



a

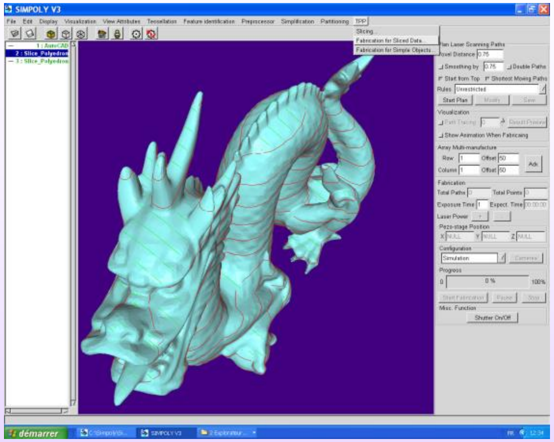


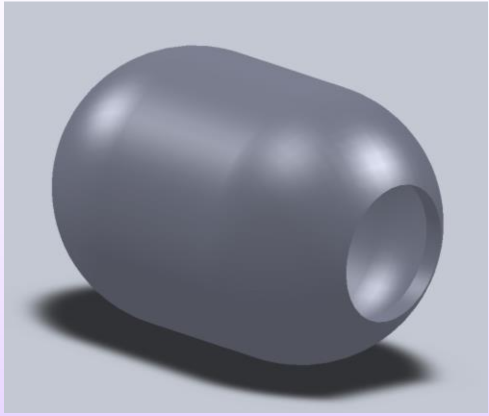
b

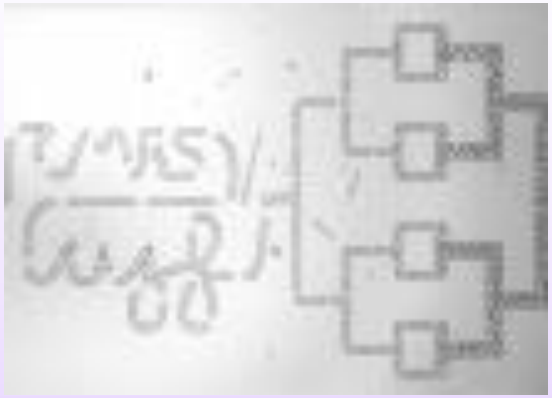
Optical images of a 3D woodpile with a period of 2.5 µm, 7x7 lines in a layer and 20 µm height: (a) in transmitted light with x100 oil-immersion objective; (b) in dark-field with x20 objective.

N. Tosa, G. Vitrant, P. L. Baldeck, O. Stephan, I. Grosu, *J. Optoel. Adv. Mater.* 2008, *10*, 2199-2204

Polymer 2D /3D structures

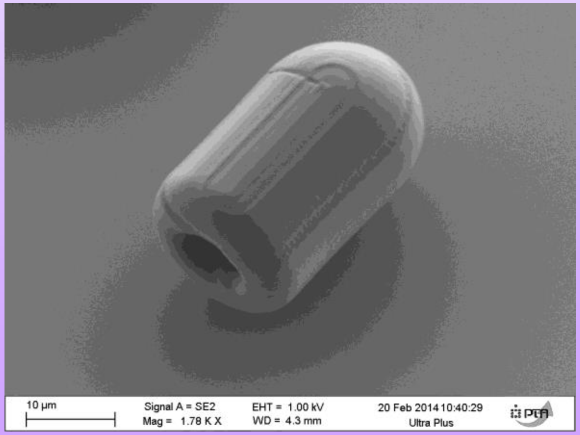




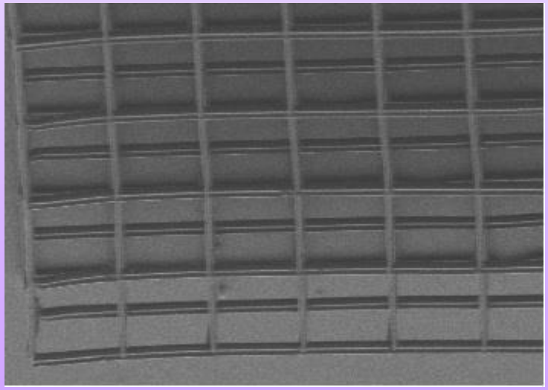


2D elemnt of circuit

Micro – dragon



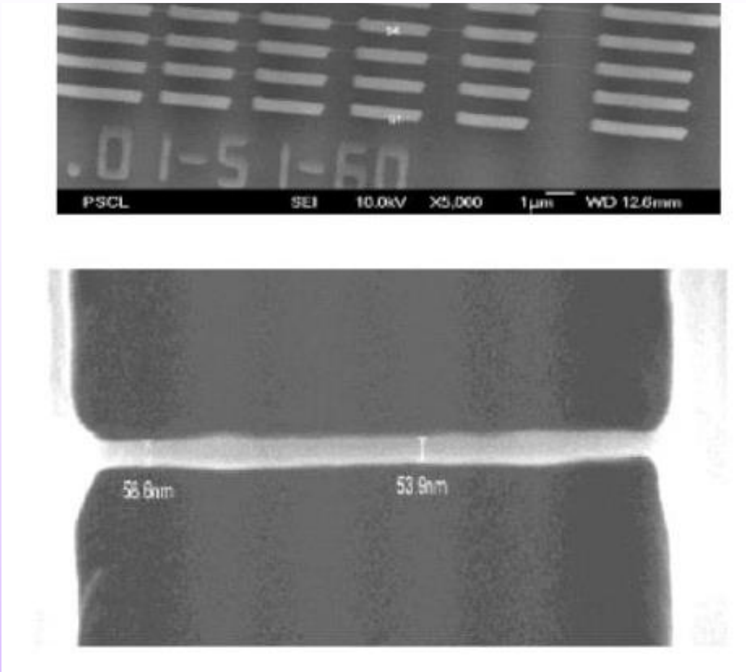
Micro-capsule ORMOCOMP

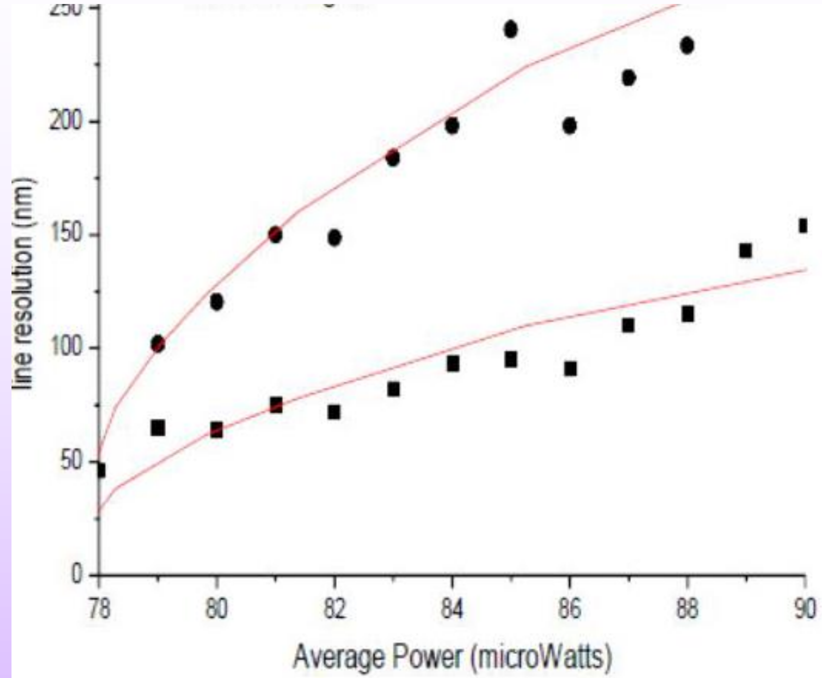


3D scaffold for biological applications

C.Y. Liao, M. Bouriau, P.L. Baldeck, J.C. Leon, C. Masclet, T-T. Chung, *Appl. Phys. Lett.* 2007, *91*, 033108, 1-3..

Polymer 2D structures





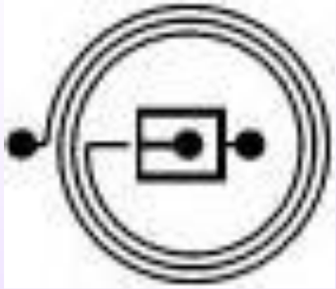
Repetition rate: 6 kHz
Pulse duration: sub-nanosecond
Q-switched microchip laser: Nd:YAG 532 nm

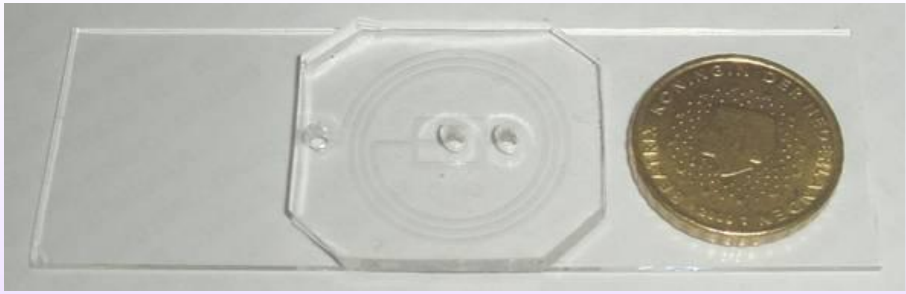
voxel distance of 60 nm
exposure time 1 ms
decreasing laser power steps of 50 μW

P.L. Baldeck, P. Prabakharan, C. Y. Liu, M. Bouriau, L. Gredy, O.Stephan, T. Vergote, H. Chaumeil, J-P. Malval, Y-H. Lee, C-L. Lin, C-T. Li, Y. H. Hsueh, T-T. Chung, *Proc. of SPIE,* 2013, *8827*, 88270E-6.

Polymer 2D /3D structures

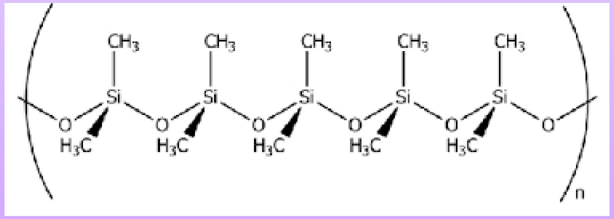
Optical lithography by mask



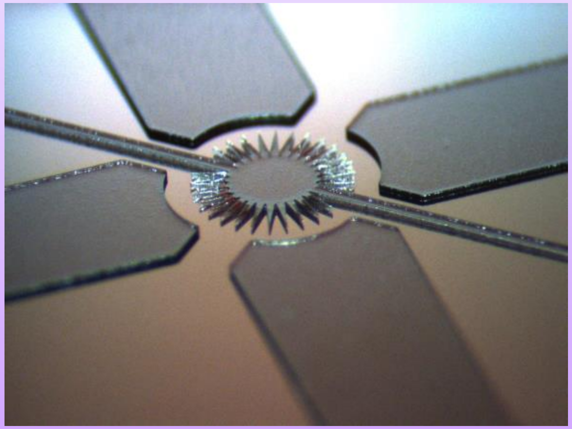


Microfluidic circuit

PDMS
Poly(dimethylsiloxane) or
Dimethicone



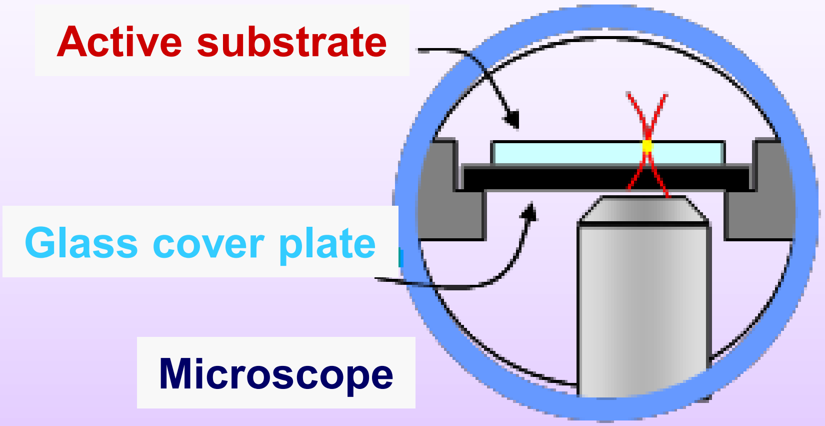
transparent material widely used for fabrication and prototyping of microfluidic chips

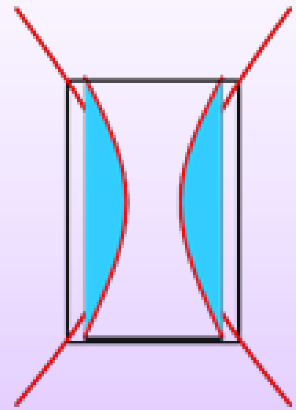


140 µm size microfluidic circuit
With 2 channels

C.Y. Liao, M. Bouriau, P.L. Baldeck, J.C. Leon, C. Masclet, T-T. Chung, *Appl. Phys. Lett.* 2007, *91*, 033108.

Direct Laser Writing(DLW) photo-reduction in polymer doped thin films



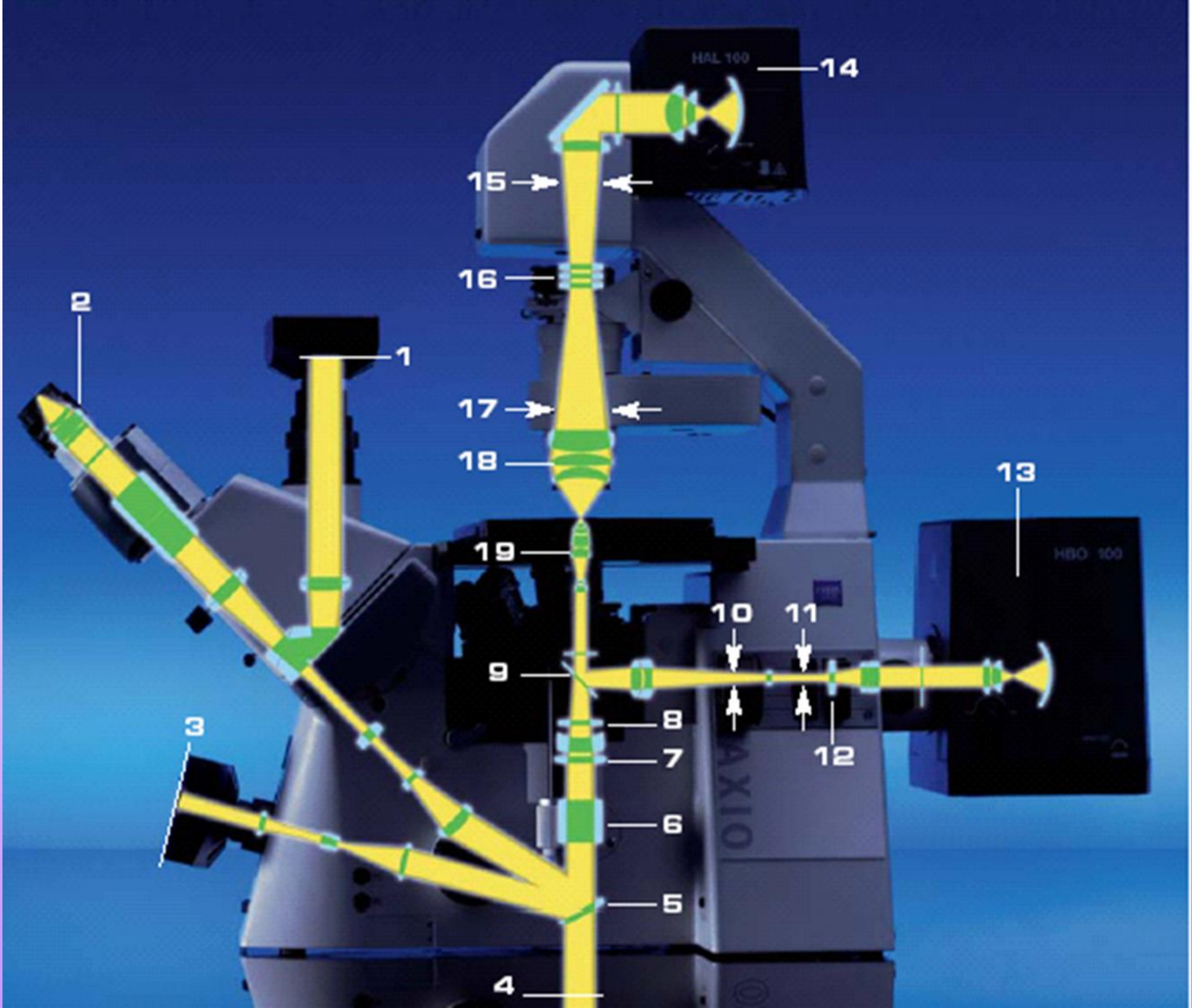


1-photon absorption

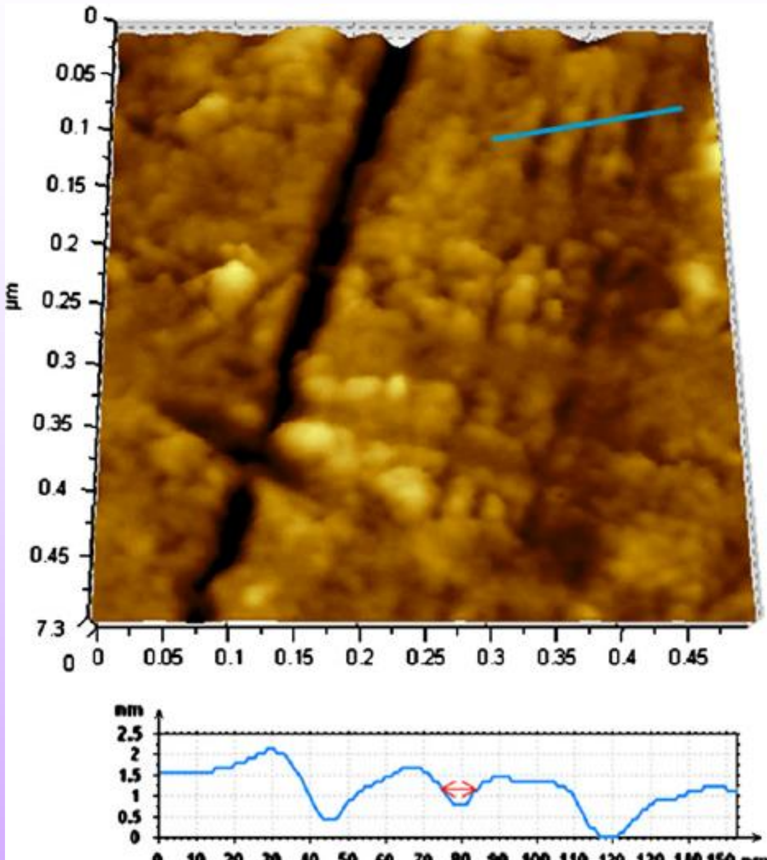
λ = 380 nm

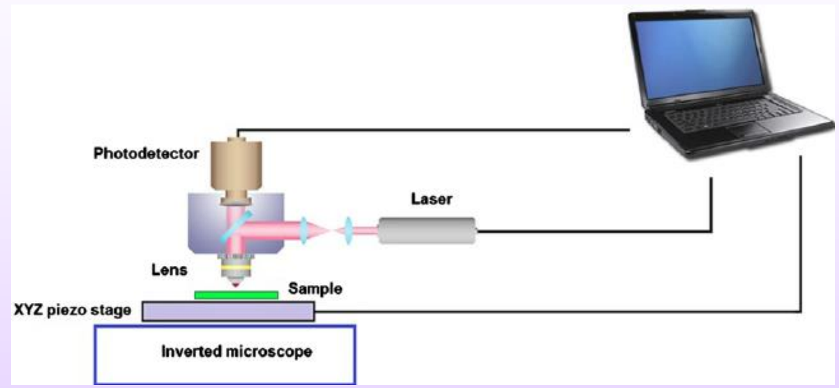
Photo-reduction

entire irradiated volume


HAL 100
14
15
16
2
1
17
18
13
19
HBO 100
10
11
9
8
3
7
12
AXIO
6
5
4

2 nm Optical Lithography





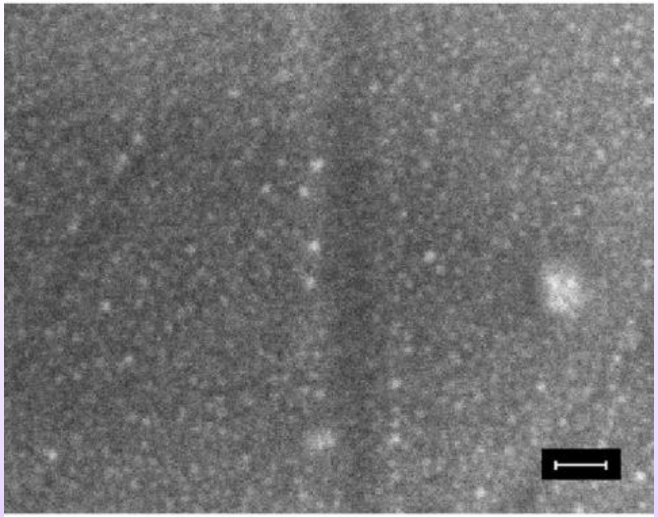
Rare-earth based fluorescent photosensitive glass-ceramics

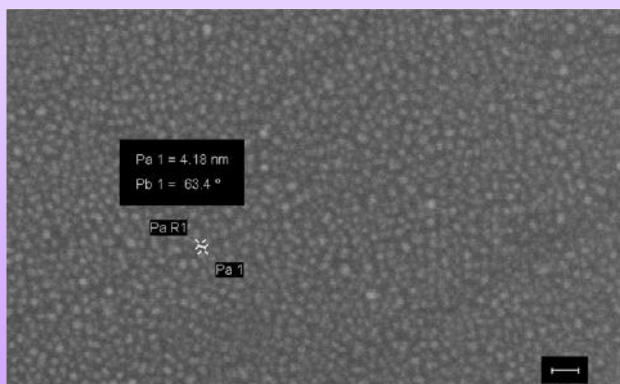
$$Ce^{3+} - 1e^{-} \longrightarrow Ce^{4+}$$

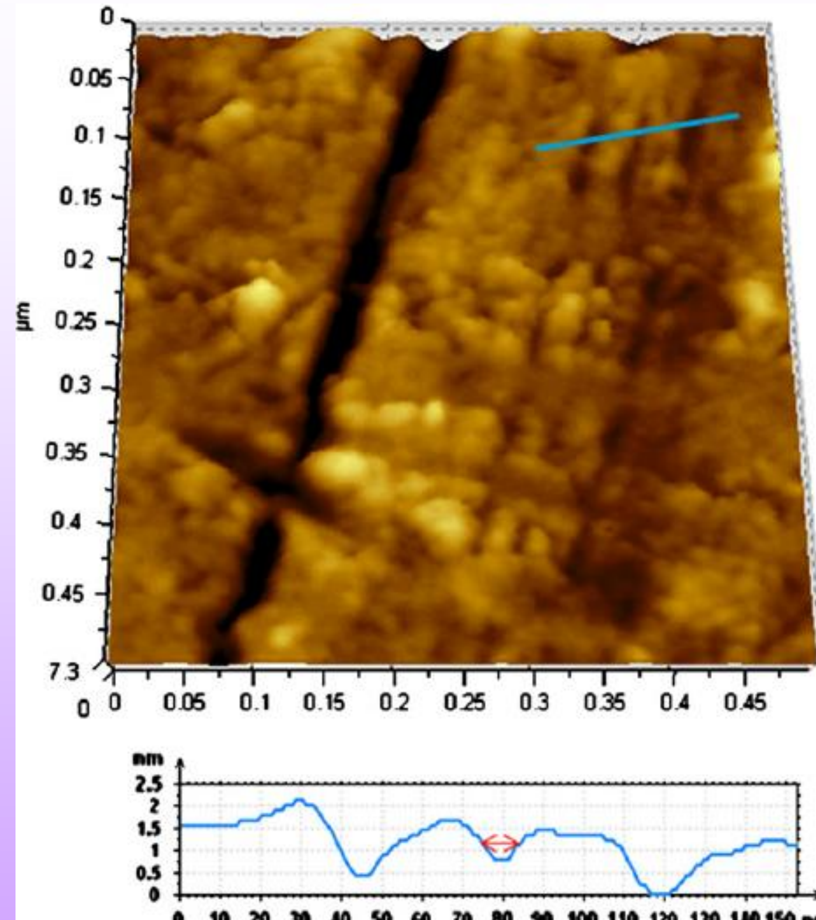
$$Ag^{+} + 1e^{-} \longrightarrow Ag^{0}$$

E. Pavel, S.Jinga, E.Andronescu, B.S.Vasile, G.Kada, A.Sasahara, **N.Tosa**, A.Matei, M. Dinescu, A.Dinescu, O.R.Vasile, “2 nm Quantum Optical Lithography”, Optics Communications 291 (2013) 259–263

2 nm Optical Lithography

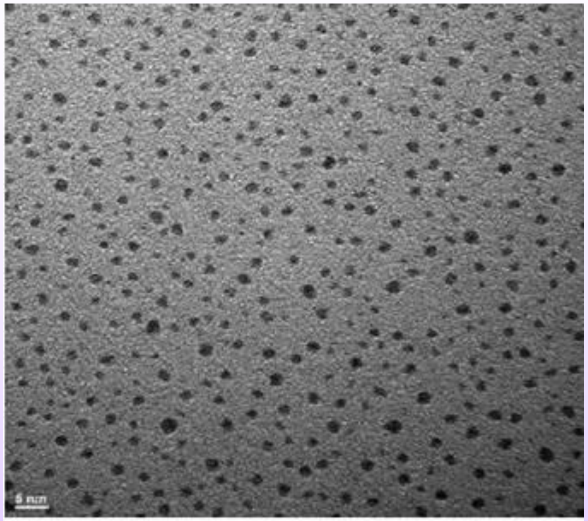


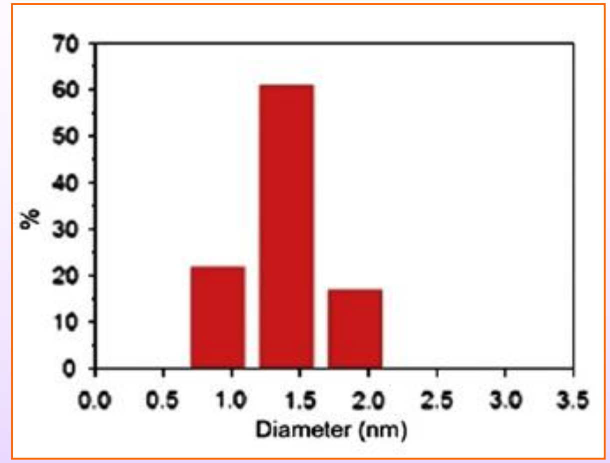




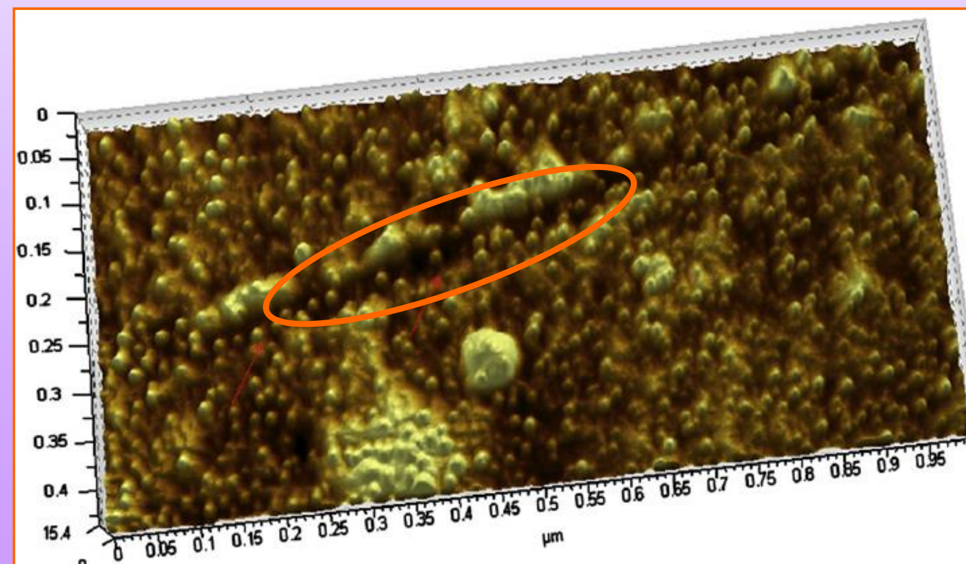
E. Pavel, S.Jinga, E.Andronescu, B.S.Vasile, G.Kada, A.Sasahara, **N.Tosa**, A.Matei, M. Dinescu, A.Dinescu, O.R.Vasile, "2 nm Quantum Optical Lithography", Optics Communications 291 (2013) 259–263

How to measure a 2 nm line width





Distribution of gold nanoparticles:
(a) TEM image of AuNps,
(b) histogram &
(c) AFM top view of a 2 nm line covered by AuNPs



E. Pavel, S.Jinga, E.Andronescu, B.S.Vasile, G.Kada, A.Sasahara, **N.Tosa**, A.Matei, M. Dinescu, A.Dinescu, O.R.Vasile, "2 nm Quantum Optical Lithography", Optics Communications 291 (2013) 259–263

Advantages vs disadvantages

The chief advantage of the laser-controlled synthesis (deposition) is demonstrated here by :

- **First, the photoeffect is of a general chemical nature. No limitation in the choise of reducing agent was observed in both metallic Ag and Au synthesis.**

- **Second, the growth is terminated either by turning off the laser or removing the sample (ionic solution).**

- **Third, the use of microscope objectives for illumination allows the fabrication of micrometer-sizes metallic structures in devices with limited accesibility. Ex. Ag photodeposition inside of a glass capillary**

Conclusions

- Optical lithography by DLW is a maskless procedure with spatial control of the process, confined at the focal point
- Metallic structures are generated selectively in thin films displaying well defined patterns and tunable sizes function on the process parameters
- Metallic microstructures contain long range arrays of lines/nanoparticles with size and shape uniformly distributed along the pattern
- The size of structures decrease with velocity increasing due to the laser exposure time diminishing
- Metallic microstructures can be anchored on active surfaces proving that they are eligible as substrates for metallic electrodes

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Dr. Alexandra Falamas INCDTIM
Dr. Cristian Tudoran INCDTIM
Dr. Lucian Barbu Tudoran INCDTIM SEM measurements

Dr. Patrice Baldeck   MOTIV group
Dr. Olivier Stephan
Dr. Michel Bouriau
Eng. Jean Francois Motte for SEM measurements
Dr. Guy Vitrant   MINATEC Grenoble, France

Laboratoire de Spectrometrie Physique, Grenoble, France
(Laboratoire Interdisciplinaire de Physique LIPhy)

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Prof. Dr. Alberto Diaspro

Mrs. Frederica Delconte

Prof. Joe Niemela
Prof. Mitco Danailov

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Thank you for your attention!