

Micromachining with fs-pulses

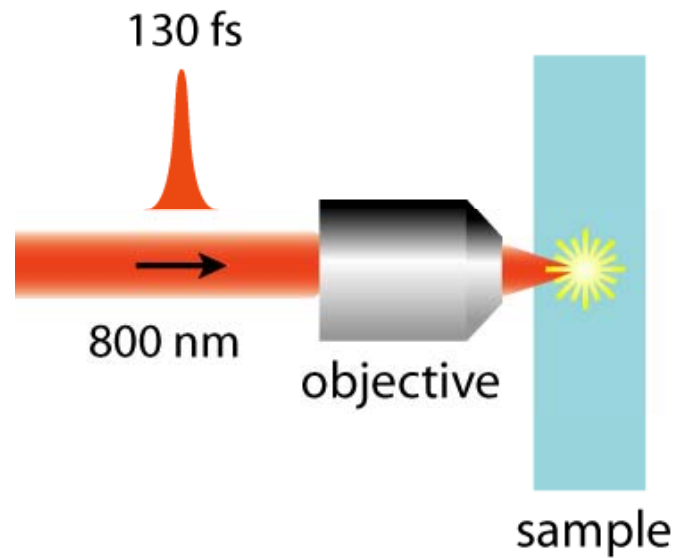


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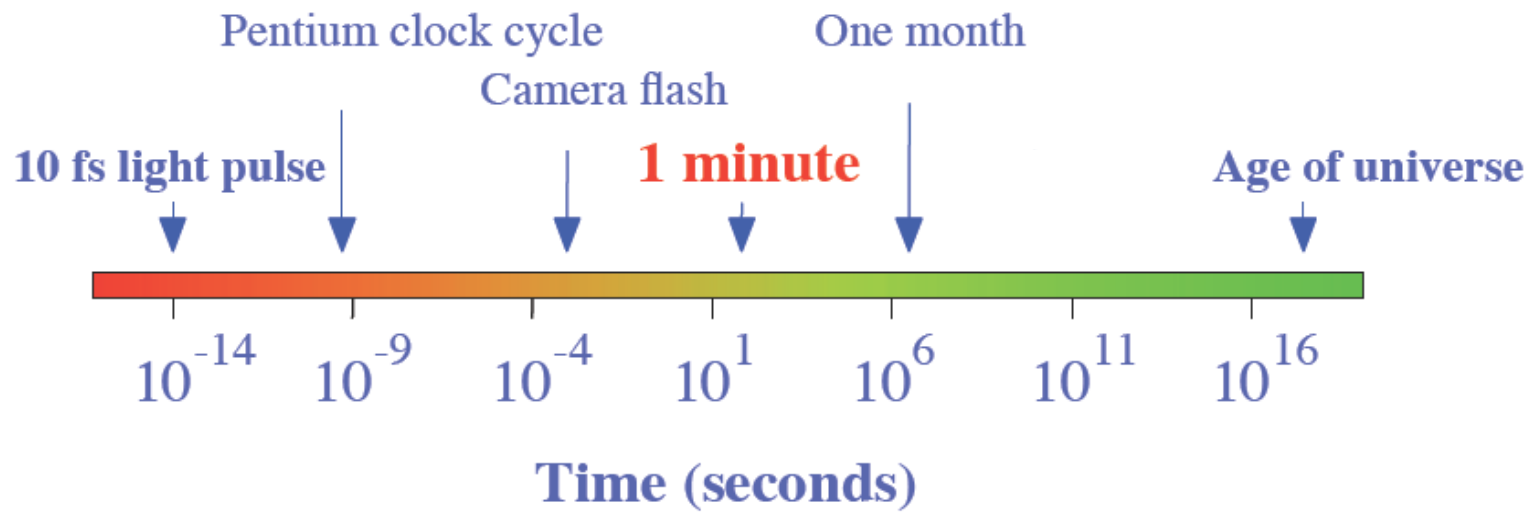
Microfabrication

Microfabricate and microstructure materials using fs-laser and nonlinear optical processes



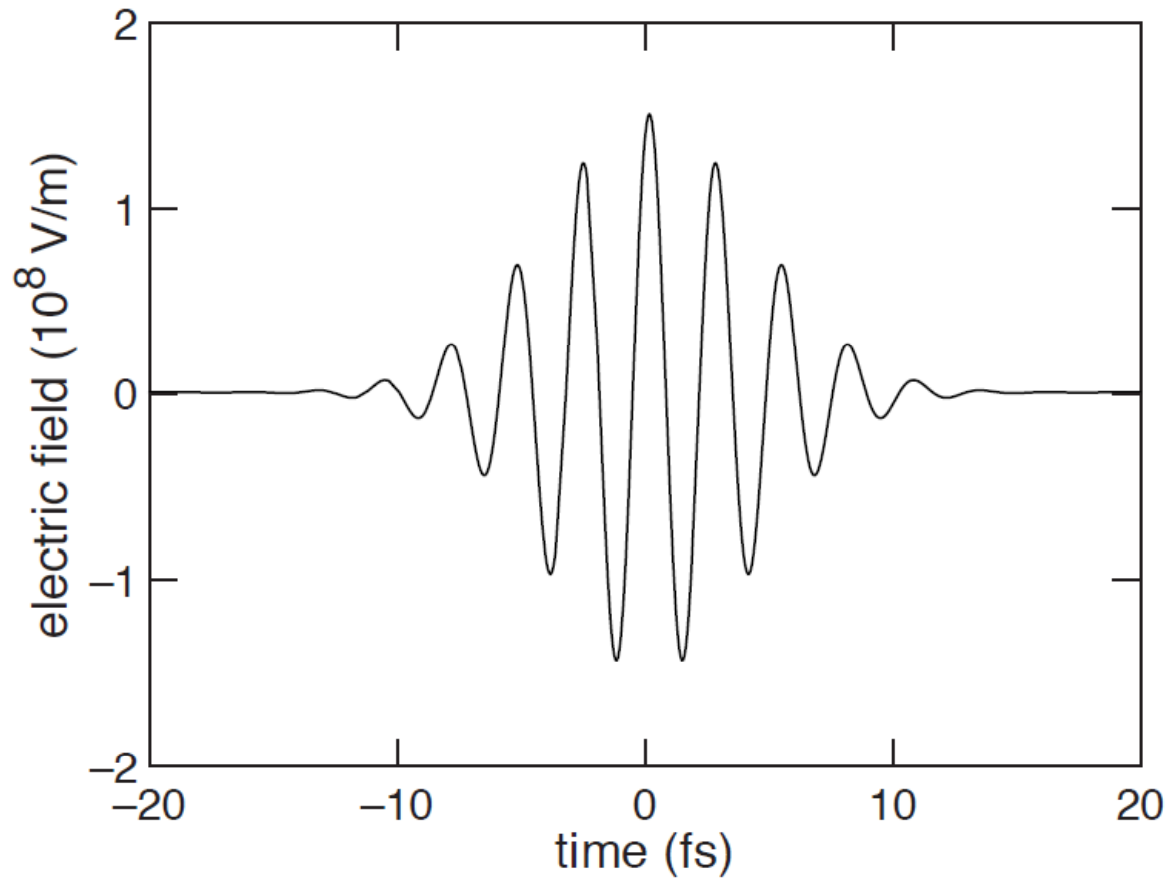
Microfabrication

$$1 \text{ fs} = 10^{-15} \text{ s}$$



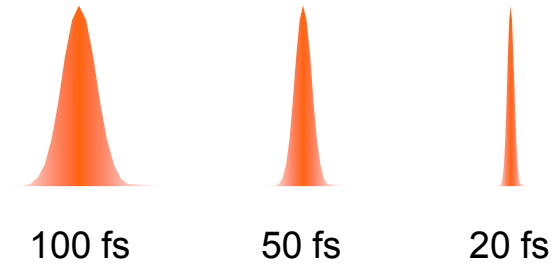
introduction

how short is a femtosecond pulse ?



Microfabrication

Ti:Sapphire lasers



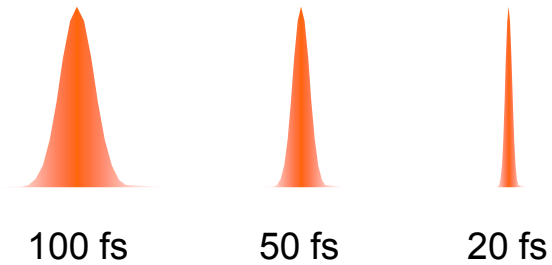
Very intense light

Laser intensities ~ 100 GW/cm²
1 x 10¹¹W/cm²

Laser pointer: 1 mW/cm² (1 x10⁻³W/ cm²)

fs-laser micromachining

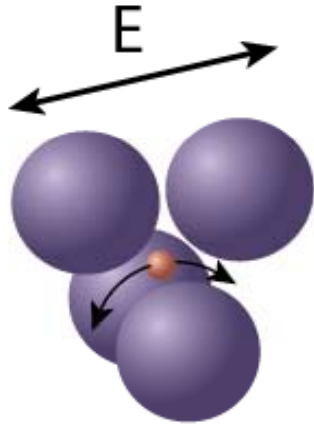
Ti:Sapphire lasers



Very intense light

Nonlinear Optical Phenomena

Nonlinear Optics



anharmonic oscillator

high light intensity

$$E_{\text{rad.}} \sim E_{\text{inter.}}$$

nonlinear polarization response

$$P = \varepsilon_0 \left(\chi^{(1)} E + \chi^{(2)} E^2 + \chi^{(3)} E^3 + \dots \right)$$

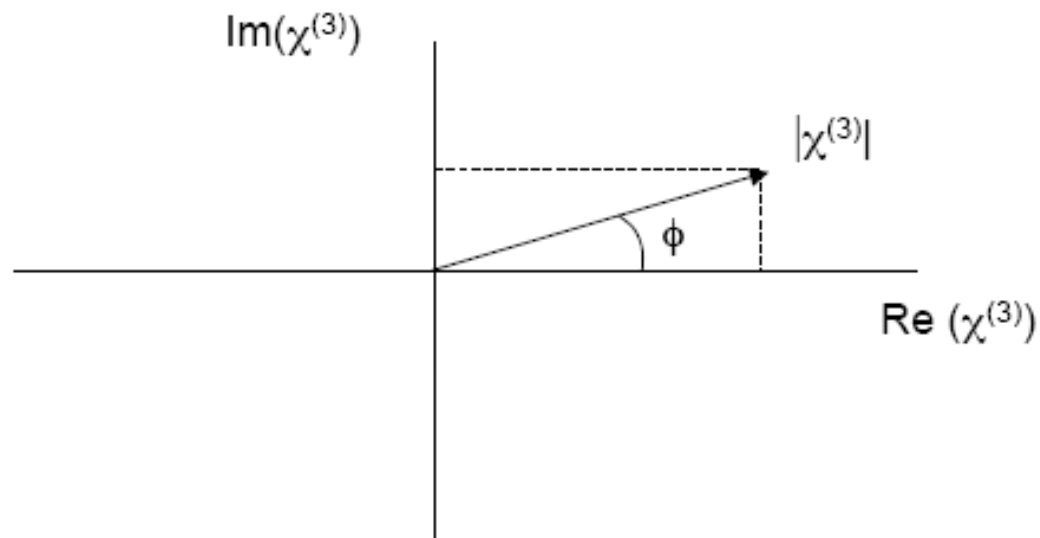
Nonlinear Optics

$\chi^{(3)}$ is a complex quantity

$$\chi^{(3)} = \text{Re}(\chi^{(3)}) + i \text{Im}(\chi^{(3)})$$

Related to intensity
dependent refractive index

Related to two-photon
absorption

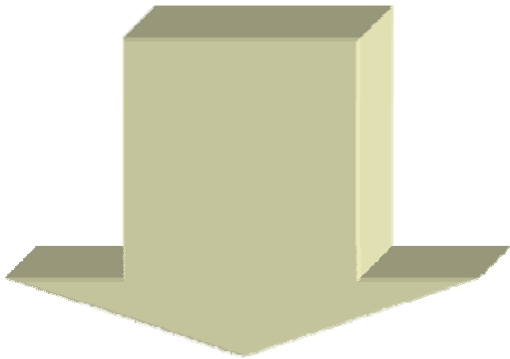


Nonlinear Optics

Third order processes: $\chi^{(3)}$

Refractive process:

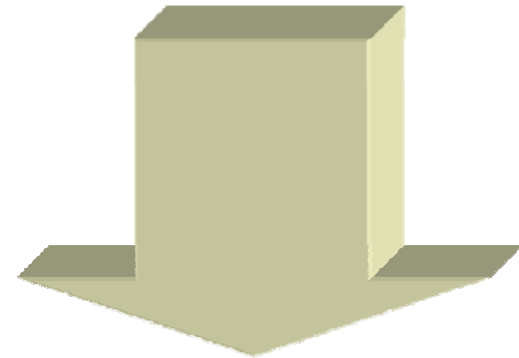
$$n = n_0 + n_2 I$$



- self-phase modulation
- lens-like effect

Absorptive process:

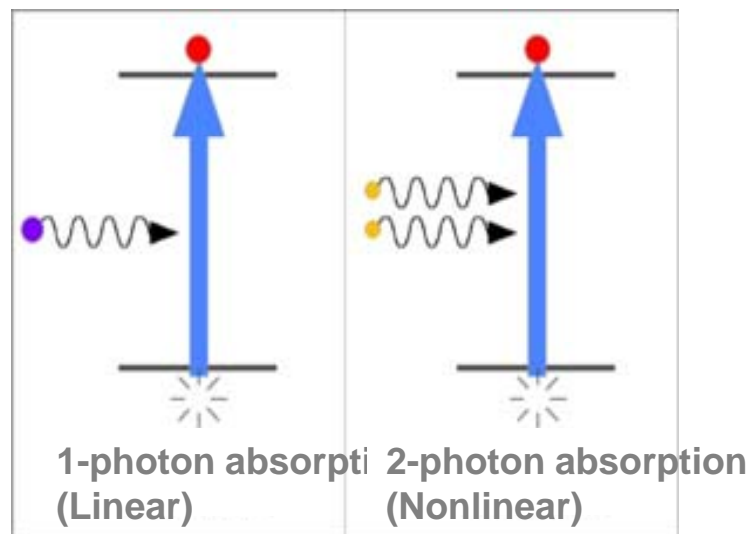
$$\alpha = \alpha_0 + \beta I$$



- nonlinear absorption
- two-photon absorption

Two-photon absorption

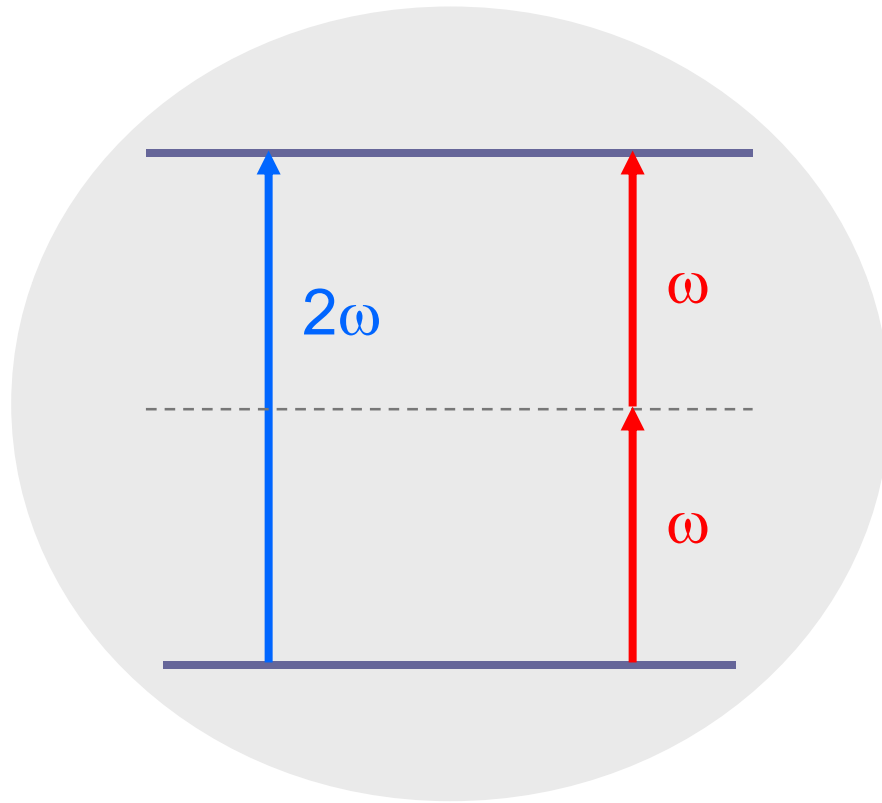
Phenomenon does not described for the Classical Physics and **does not observed until the development of the Laser.**



Theoretical model: Maria Göppert-Mayer, 1931

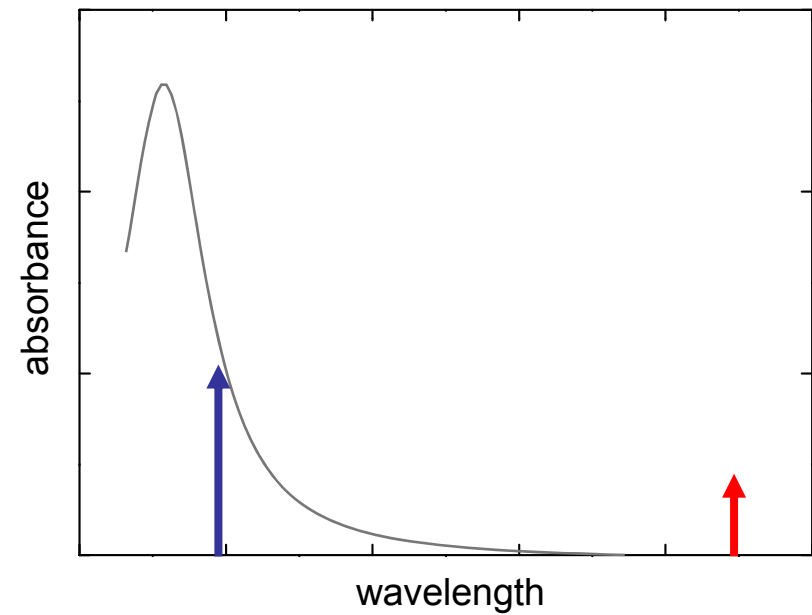
Two photons from an intense laser light beam are simultaneously absorbed in the same “quantum act”, leading the molecule to some excited state with energy equivalent to the absorbed two photons.

Two-photon absorption



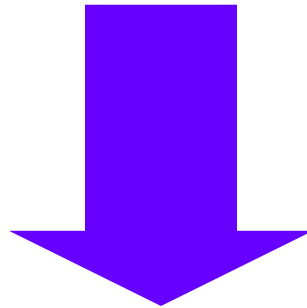
$$\alpha = \alpha_0 + \beta I$$

Third order processes $\chi^{(3)}$



fs-laser microfabrication

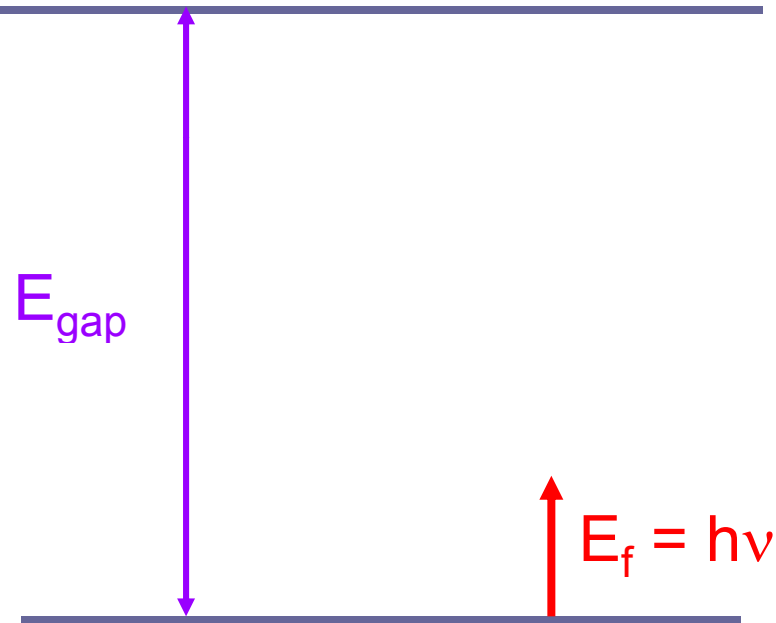
photon energy $<$ bandgap



nonlinear interaction

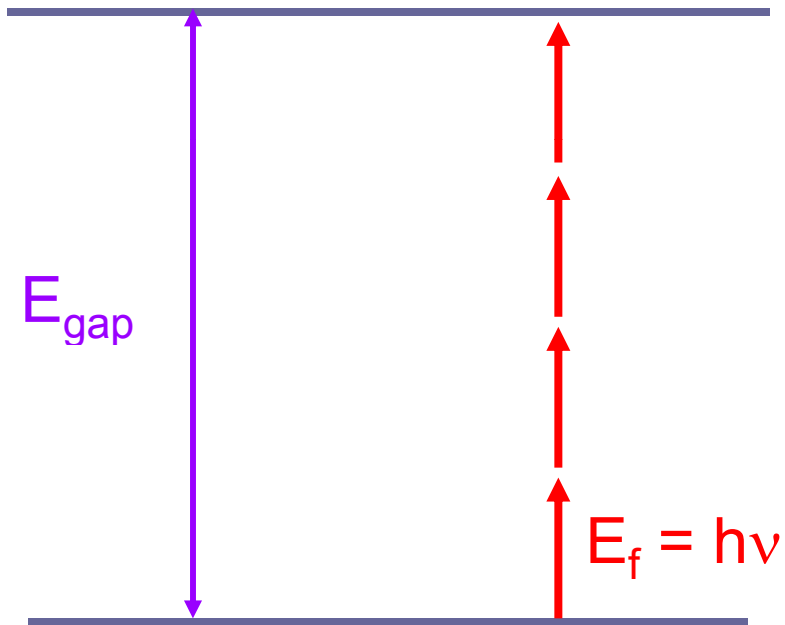
fs-laser microfabrication

nonlinear interaction



fs-laser microfabrication

nonlinear interaction



multiphoton absorption

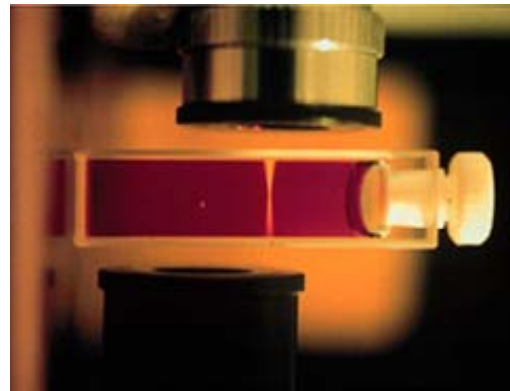
multiphoton absorption

nonlinear interaction



spatial confinement of excitation

two-photon absorption

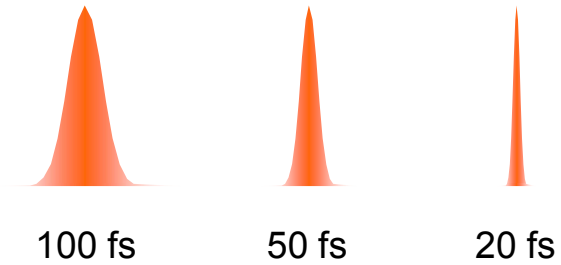


$$\alpha = \alpha_0 + \beta I$$
$$R \propto I^2$$

feature exploited for microfabrication

femtosecond pulses

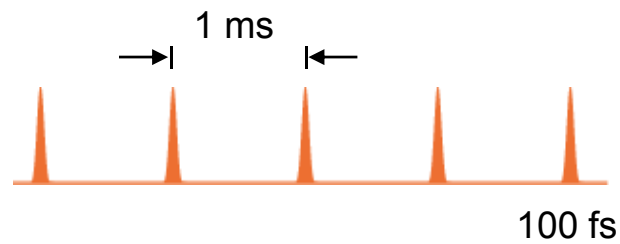
Ti:Sapphire lasers



$$1 \text{ fs} = 10^{-15} \text{ s}$$

Repetition rate

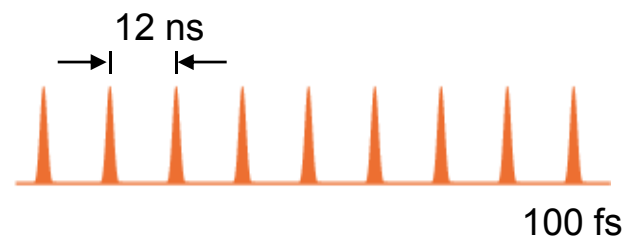
1 KHz



Energy

mJ

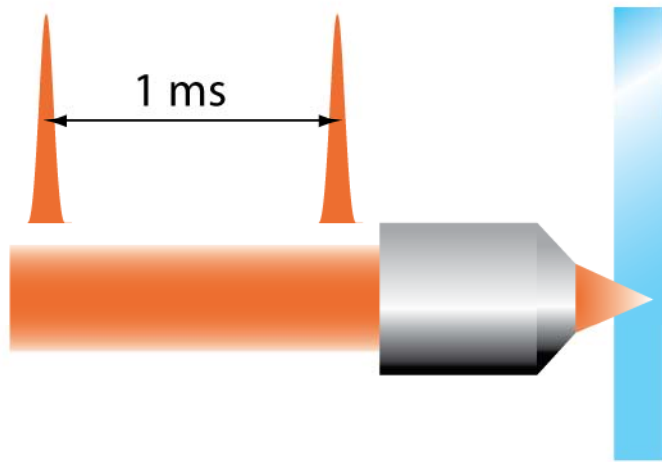
86 MHz



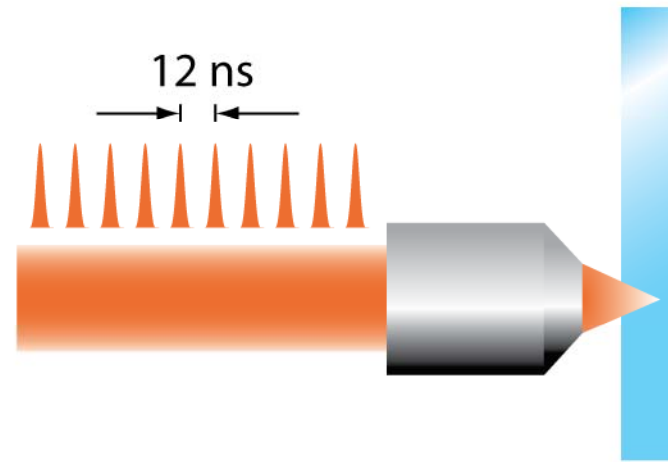
nJ

fs-micromachining

amplified laser



oscillator



heat diffusion time: $t_{\text{diff}} \sim 1 \mu\text{s}$

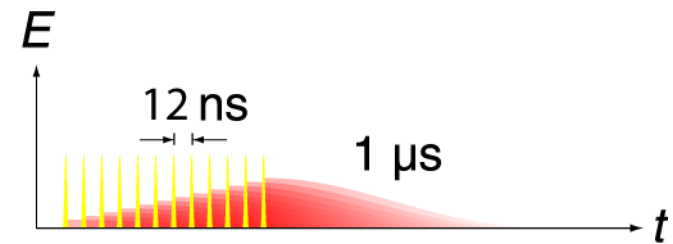
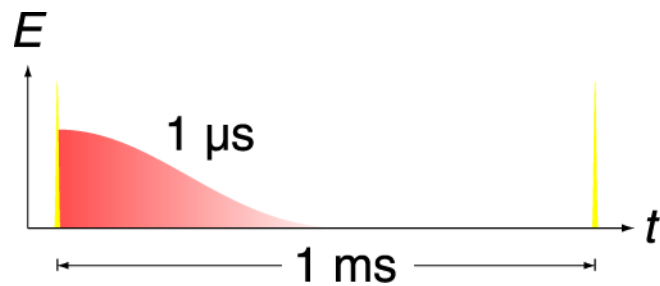
fs-micromachining

amplified laser

oscillator

low repetition laser

high repetition laser



repetitive

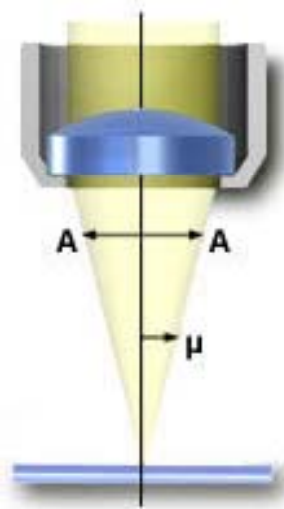
cumulative

fs-micromachining

high repetition rate micromachining

- material's change cause by accumulative effects
- spherical structuring region
- structured region exceeds the focal volume

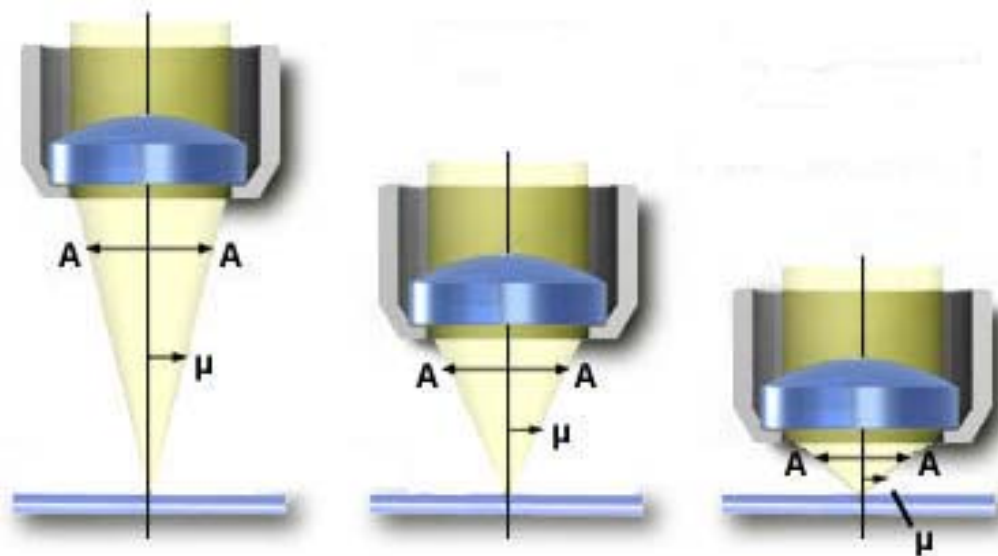
fs-micromachining: focusing



The angle μ is one-half the angular aperture A

$$NA = n \sin \mu$$

fs-micromachining: focusing



$NA = 0.12$

$\mu = 7^\circ$

$NA = 0.34$

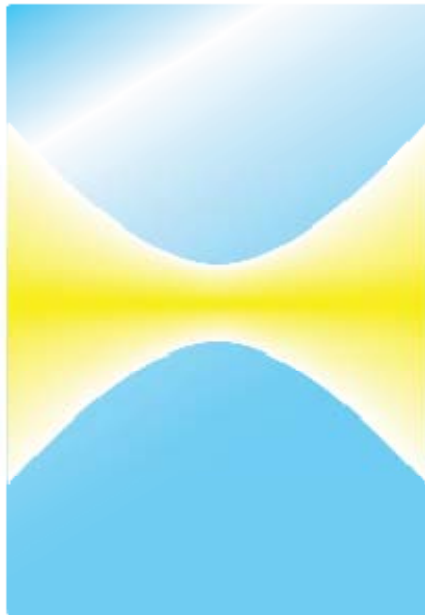
$\mu = 20^\circ$

$NA = 0.87$

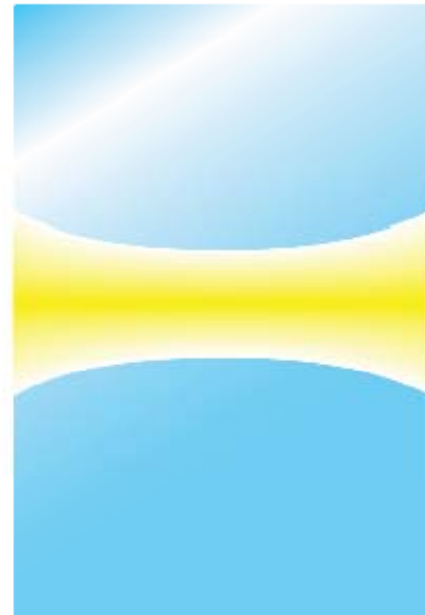
$\mu = 60^\circ$

what is the difference ?

high NA



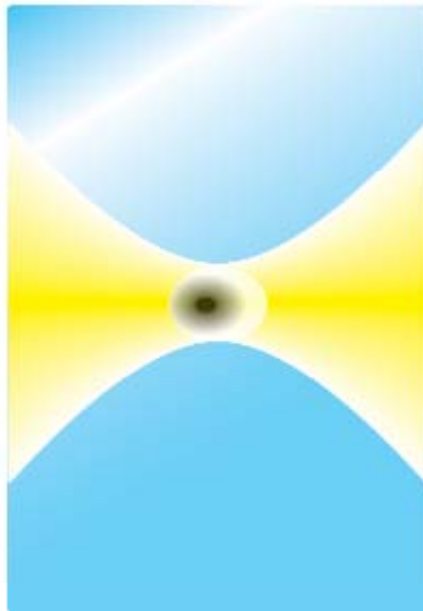
low NA



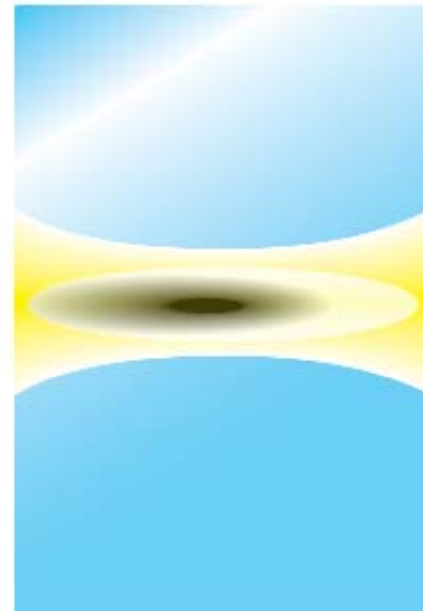
$$w_0 = \frac{\lambda}{\pi NA} \sqrt{1 - NA^2}$$

very different confocal length/interaction length

high NA



low NA



fs-micromachining

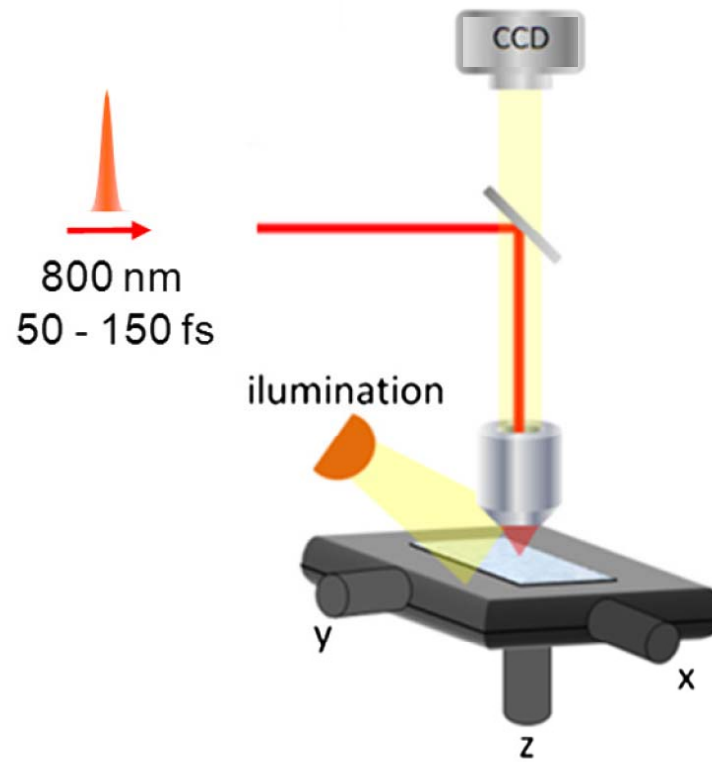
microfabrication can be controlled by

- objective NA
- number of pulses – scanning speed
- pulse energy

two main techniques

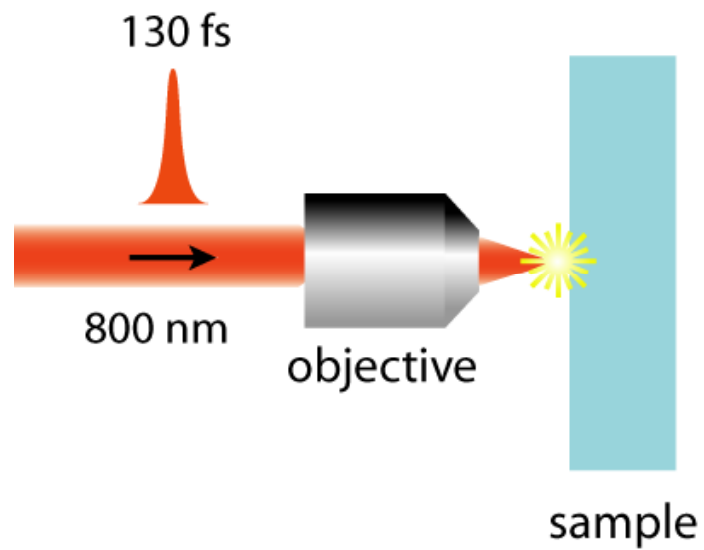
- fs-laser micromachining/microstructuring
- microfabrication via two-photon polymerization

fs-laser microstructuring experimental setup



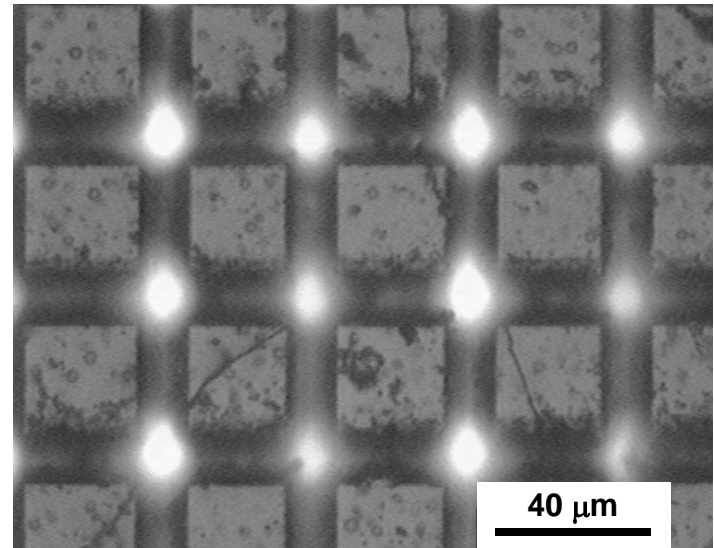
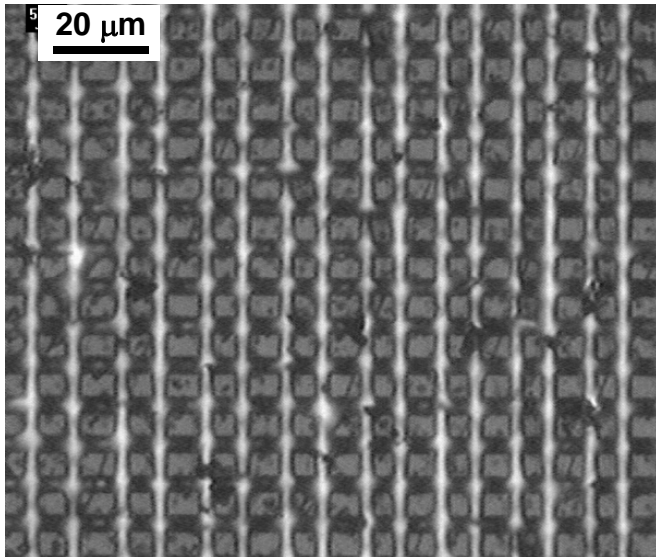
fs-laser micromachining

Surface

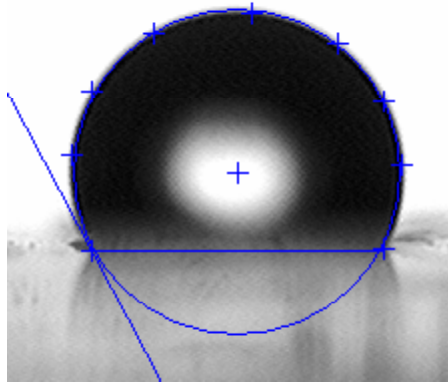


laser microfabrication: super hydrophobic surface

examples of fabricated surfaces

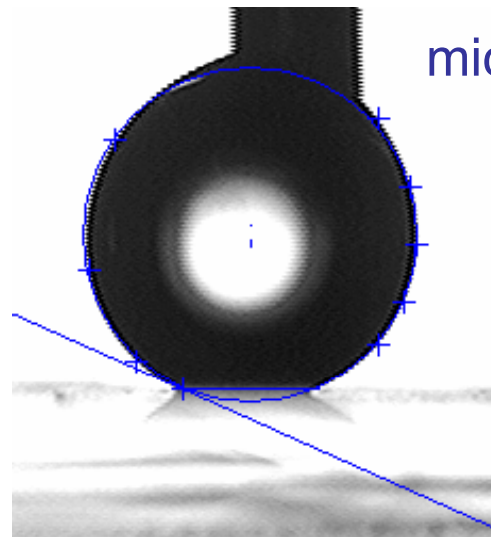


microstructuring polymer



flat surface

$$\theta = 118^\circ$$

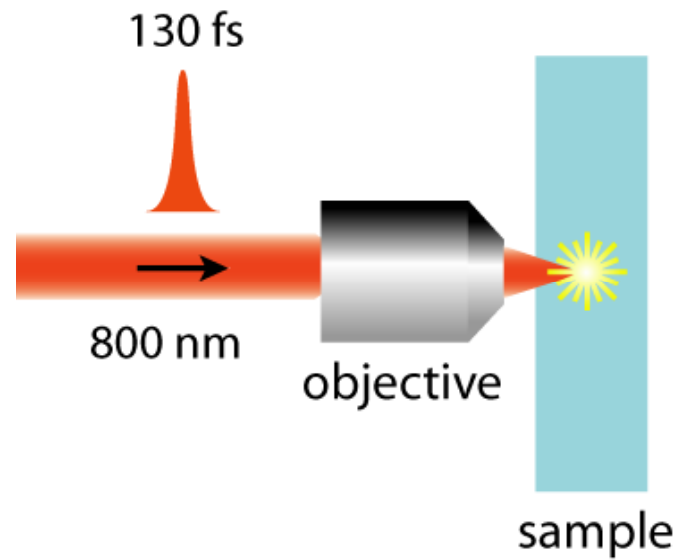


microstructured surface

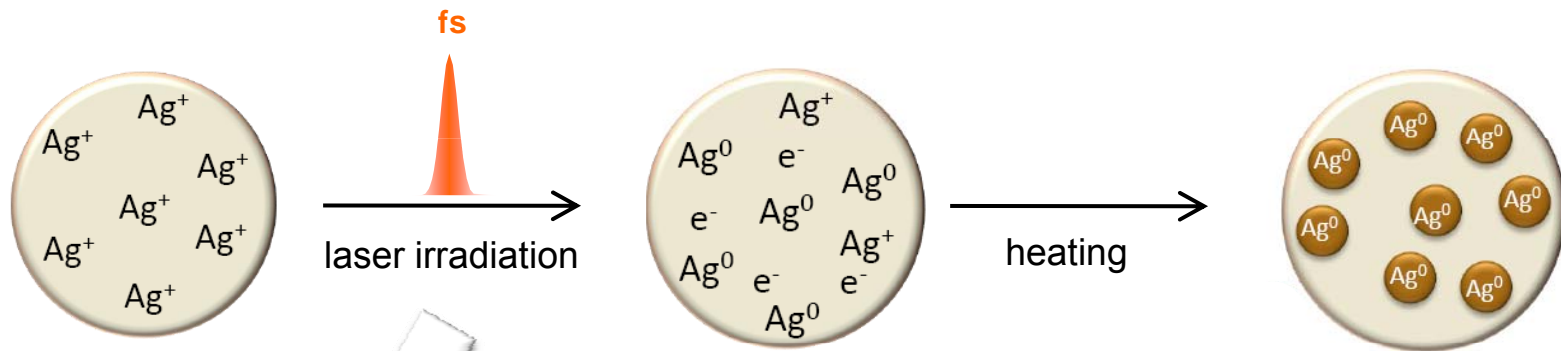
$$\theta = 160^\circ$$

fs-laser micromachining

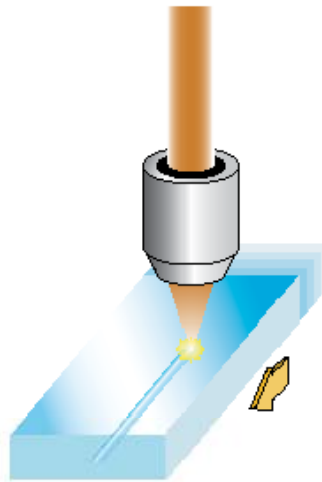
Volume



Generation of Ag nanoparticles

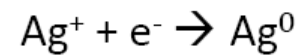


Ag nanoparticles are generated only in the irradiated area due to the **fs-laser induced photoreduction**



Free electron generation

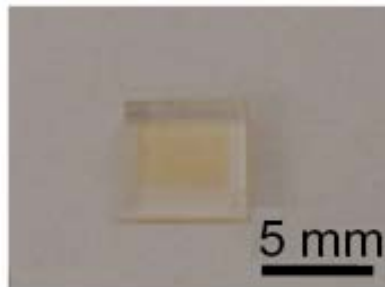
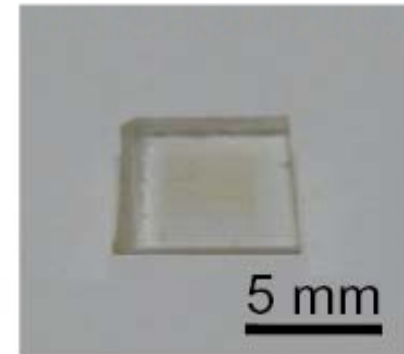
Photoreduction reaction



Generation of Ag nanoparticles

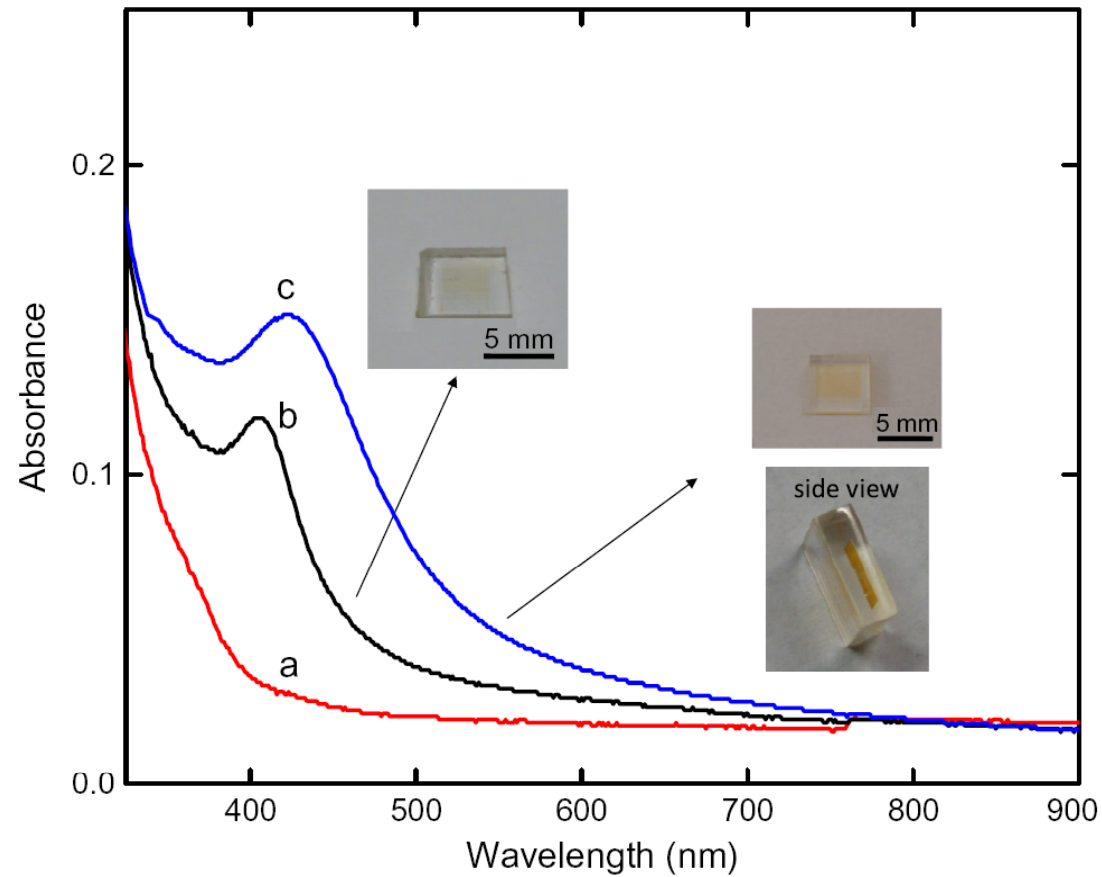
Silver doped barium borate glass (Ag:BBO)

Sample after irradiation with the amplified fs-laser (1 kHz) and subsequent thermal treatment at 400 C for 1 h



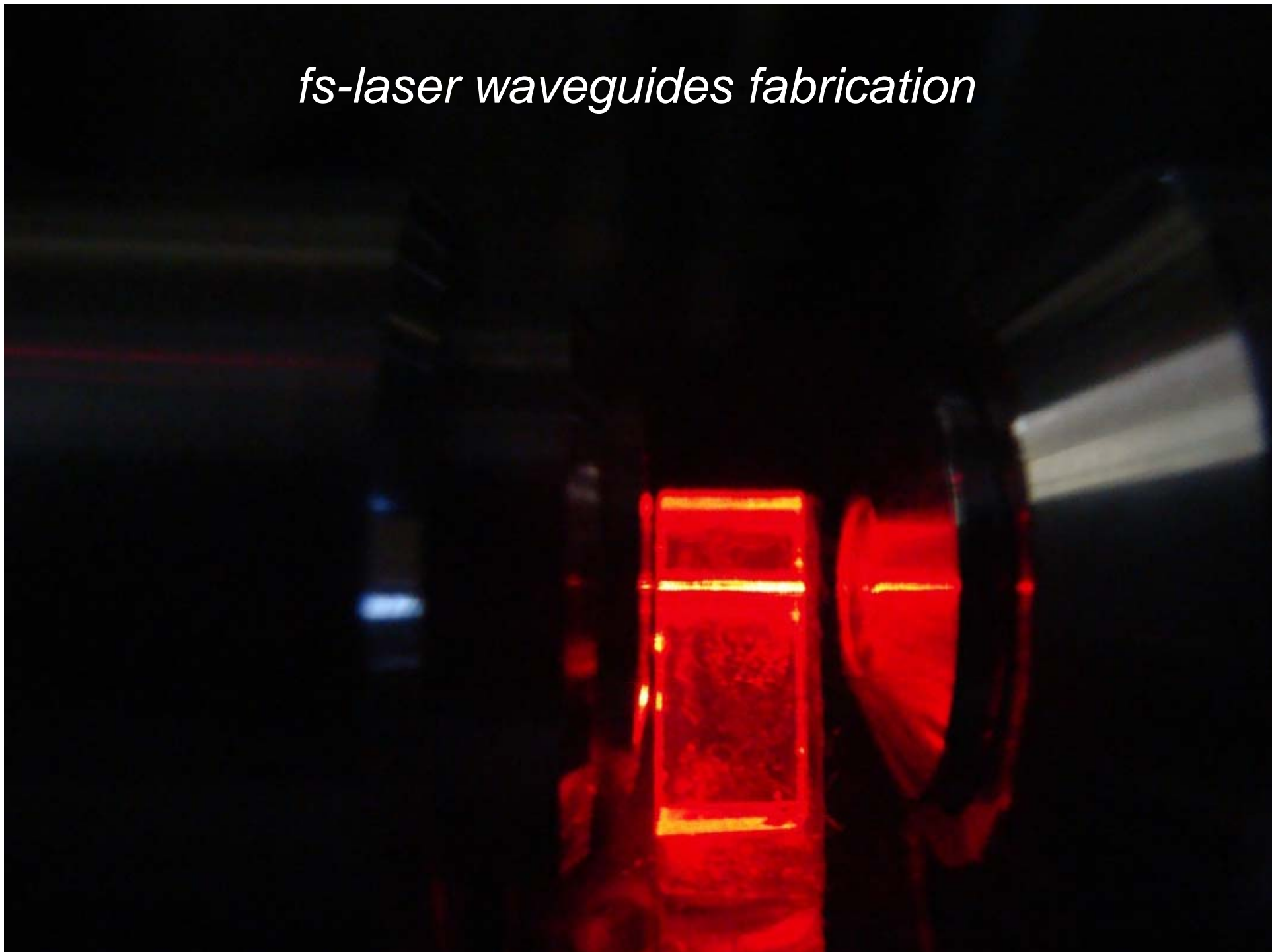
Sample after irradiation with the 5 MHz fs-laser

Generation of Ag nanoparticles



Absorption spectrum of the Ag:BBO sample as prepared (a), after irradiation with the 5 MHz fs-laser (b) and after irradiation with the amplified fs-laser (1 kHz) and subsequent thermal treatment.

fs-laser waveguides fabrication



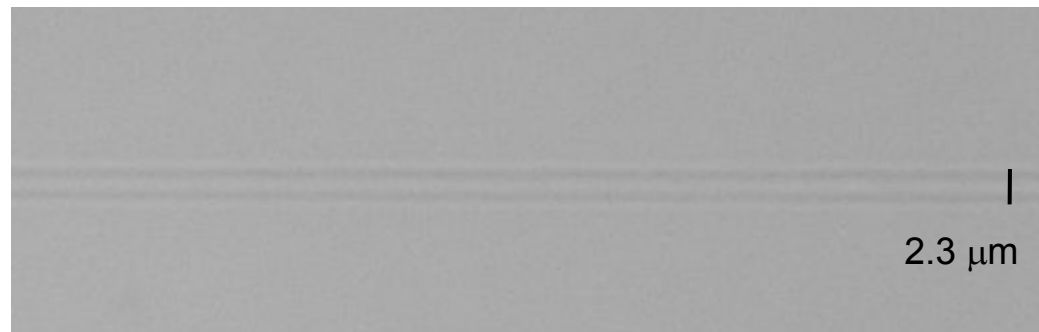
Waveguides fabrication

Sample:

Ag:P7W3

Tungsten lead pyrophosphate glass - $(70\text{Pb}_2\text{P}_2\text{O}_7-30\text{WO}_3):1\text{AgCl}$ (%mol)

Waveguides fabricated using the 5-MHz laser system (50 fs) with 37 nJ/pulse and $v = 10 \mu\text{m/s}$



Top view



Cross-section
view

Waveguides fabrication

Coupling light into the waveguides

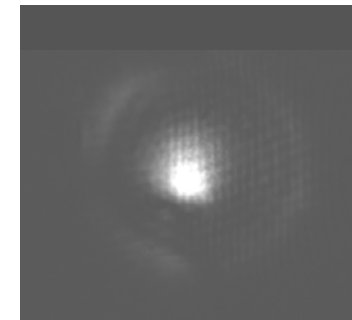
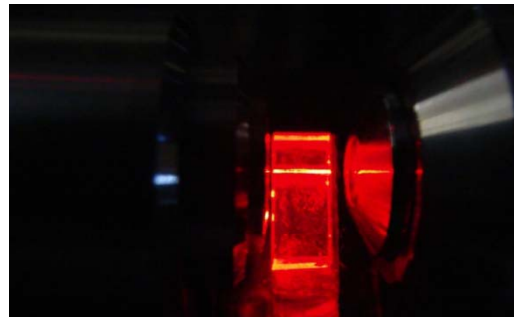
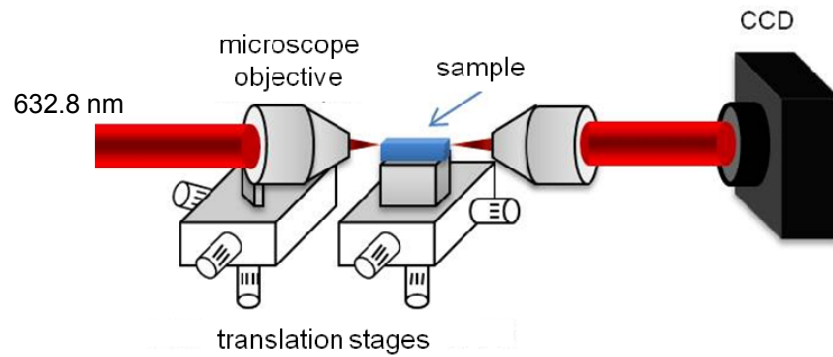


image of the waveguide output

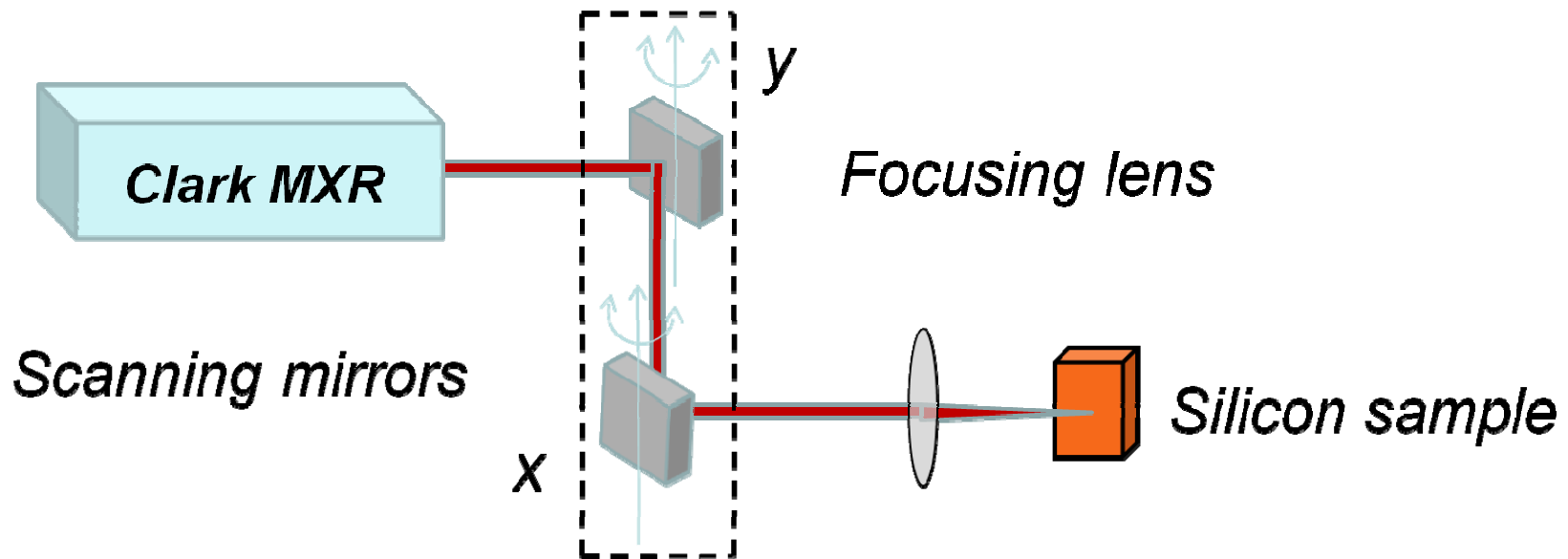
measured waveguide loss $L = 1.3 \text{ dB/mm}$



Structuring amorphous silicon films

structuring amorphous Si surface

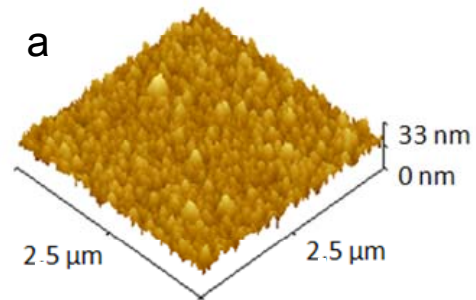
Experimental setup uses a pair of scanning mirrors



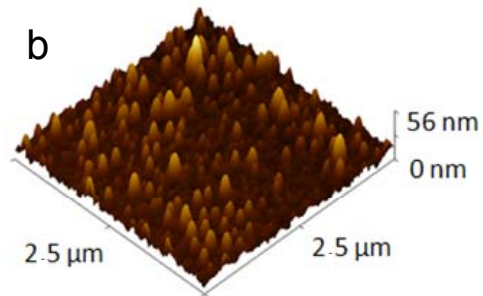
150 fs, 775 nm, 1 KHz, $v = 5$ mm/s, $f = 20$ cm

structuring amorphous Si surface

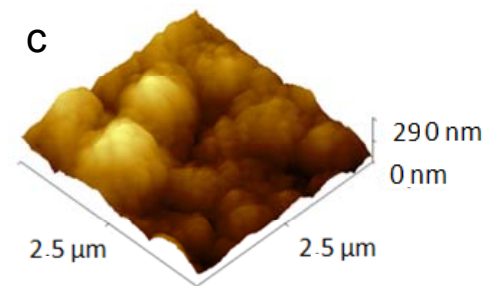
AFM micrographs of aSi microstructures at different laser intensities



before irradiation

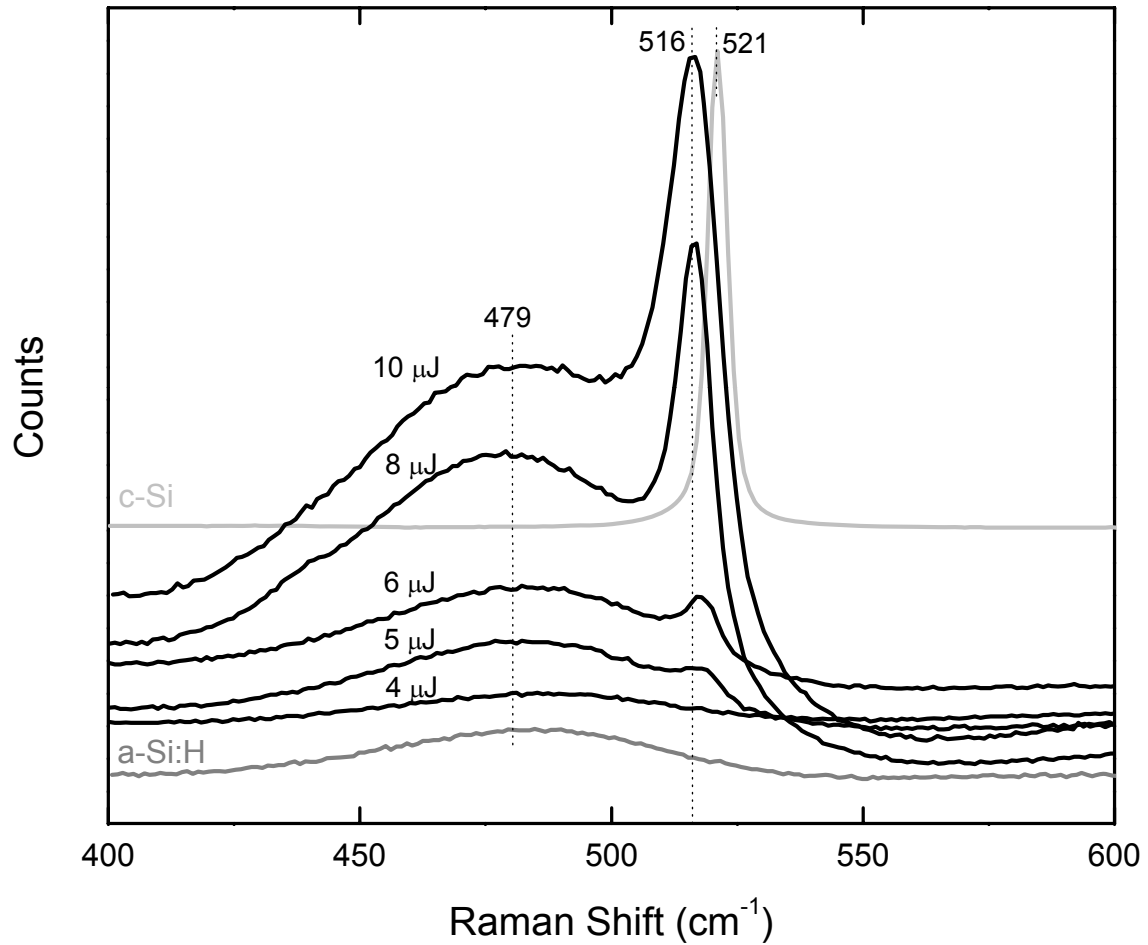


$E = 5 \mu\text{J/pulse}$



$E = 8 \mu\text{J/pulse}$

structuring amorphous Si surface



Micro-Raman analysis reveals the crystallization of the aSi upon fs-laser irradiation

structuring amorphous Si surface

The crystalline volume fraction

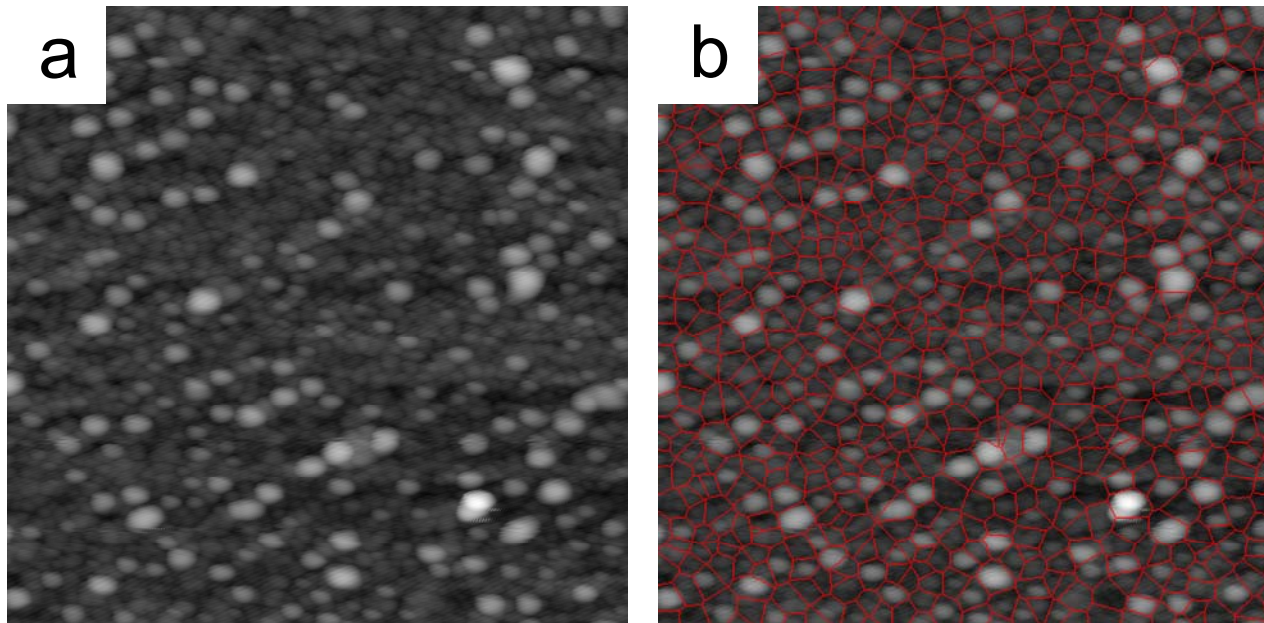
$$X_c = I_{cp} / (I_{cp} - \sigma I_{ap})$$

in which I_{cp} and I_{ap} are the intensities of the crystalline peak and at the center of the amorphous band

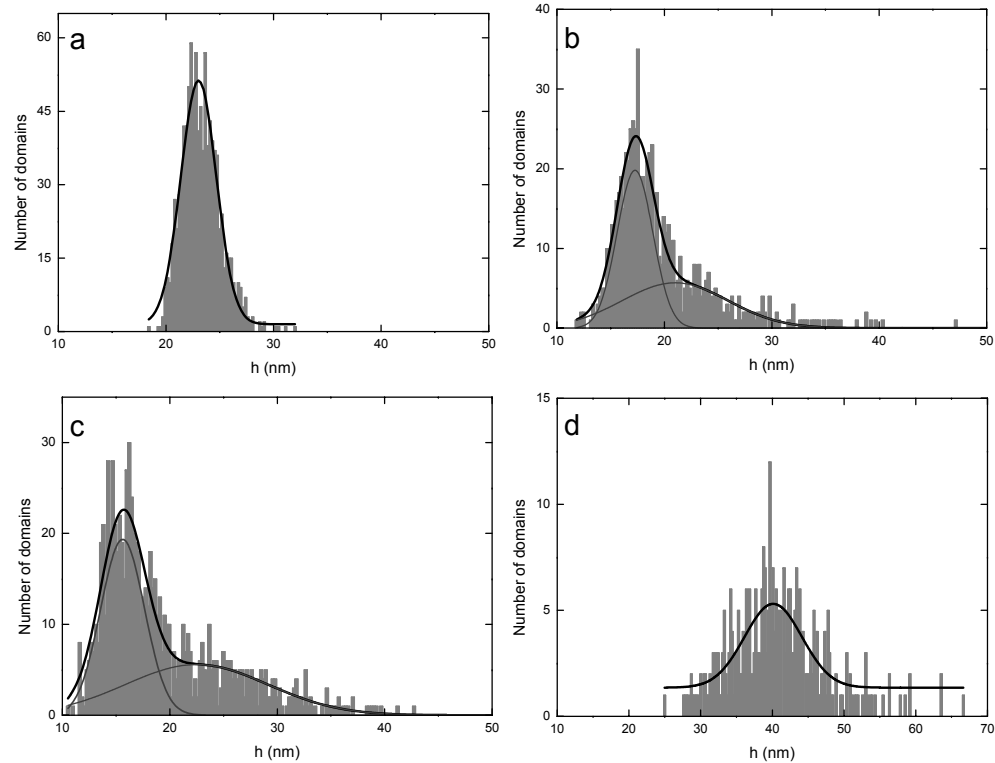
Pulse energy (μJ)	X_c (%)	Raman peak (cm^{-1})	Nanocrystal diameter (nm)
4	0	-	-
5	43	519	9
6	48	518	6
8	62	517	5
10	73	516	4

structuring amorphous Si surface

AFM image of a a-Si:H sample irradiated with 5 μJ (a) and its corresponding segmentation obtained using the Voroni's diagram method (b).



structuring amorphous Si surface



Height histograms of the domains on the sample surface

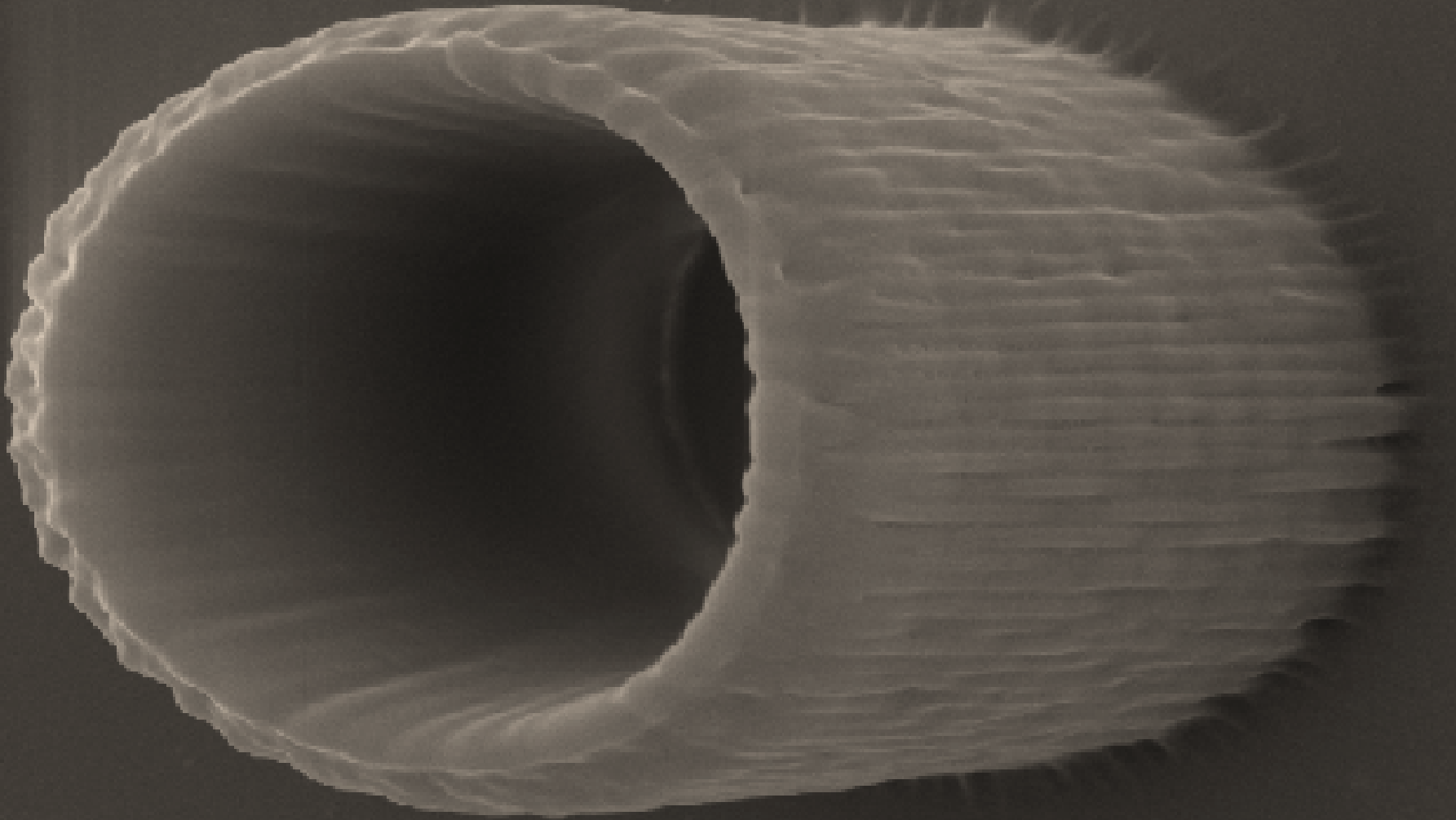
(a) before laser irradiation

(b) $E = 4 \mu\text{J}$

(c) $E = 5 \mu\text{J}$

(d) $E = 6 \mu\text{J}$

fs-laser microfabrication



fabrication of microstructure using fs-laser
and nonlinear optical processes

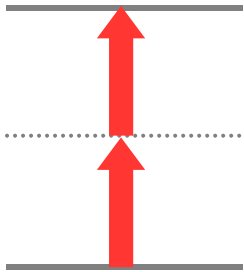
Two-photon polymerization



laser pulse

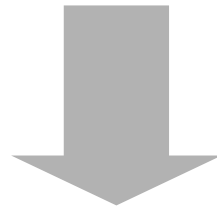
100 fs

Photoinitiator is excited by *two-photon absorption*

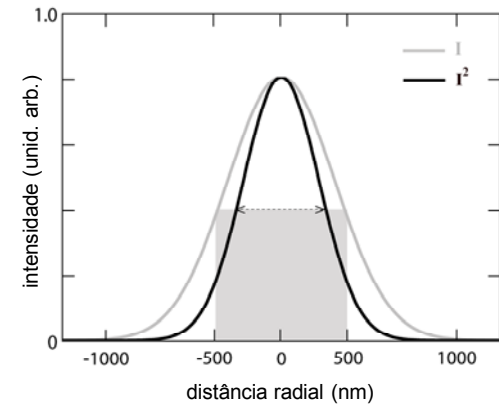


$$R \propto I^2$$

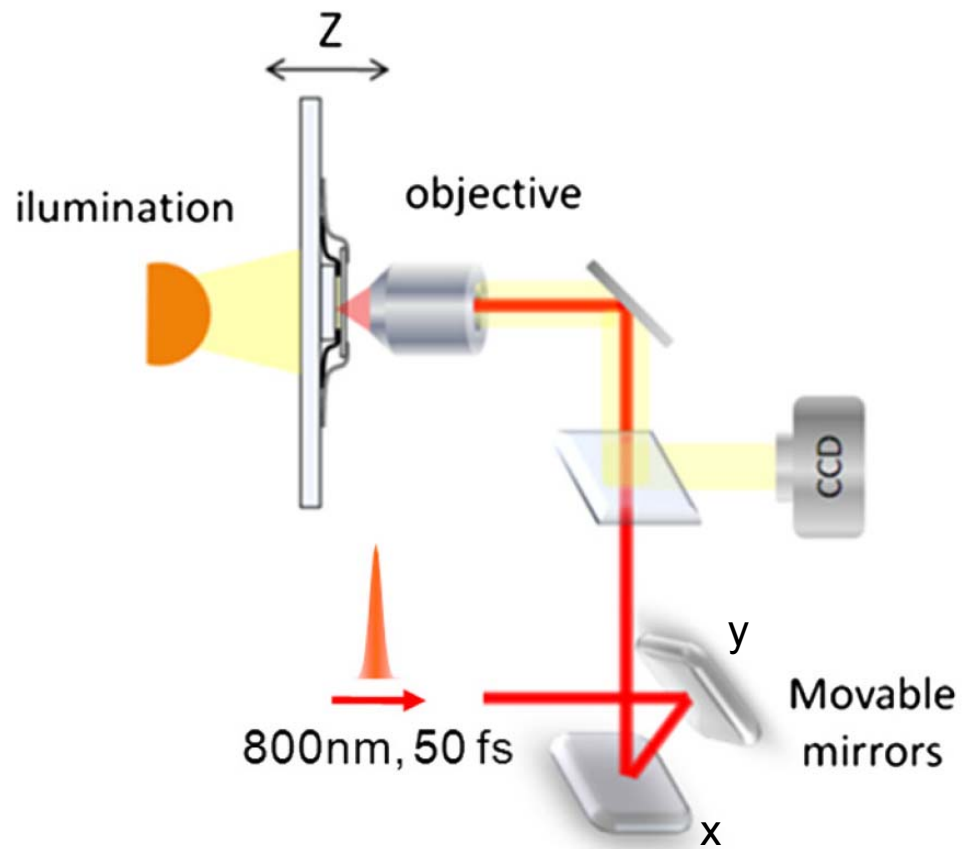
The polymerization is confined to the focal volume.



High spatial resolution



Two-photon polymerization setup



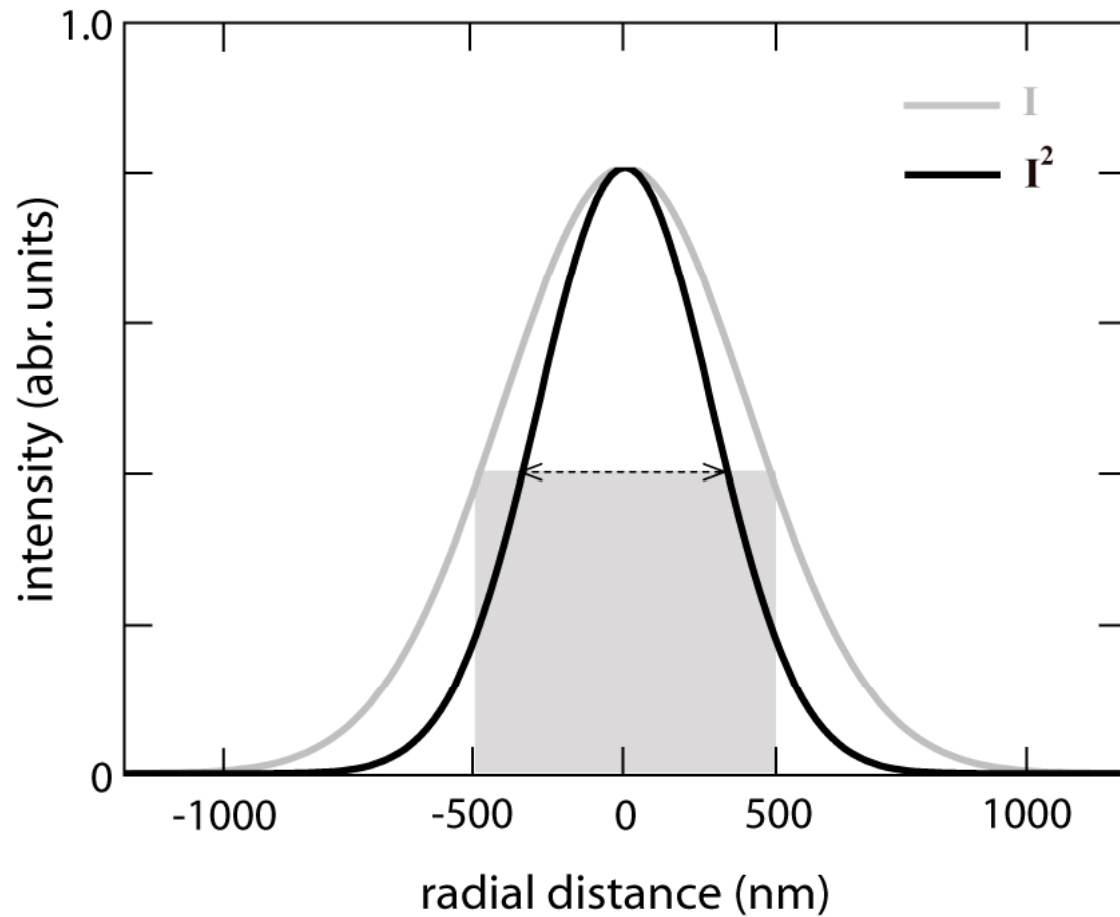
Ti:sapphire laser oscillator

- 50 fs
- 800 nm
- 80 MHz
- 20 mW

Objective

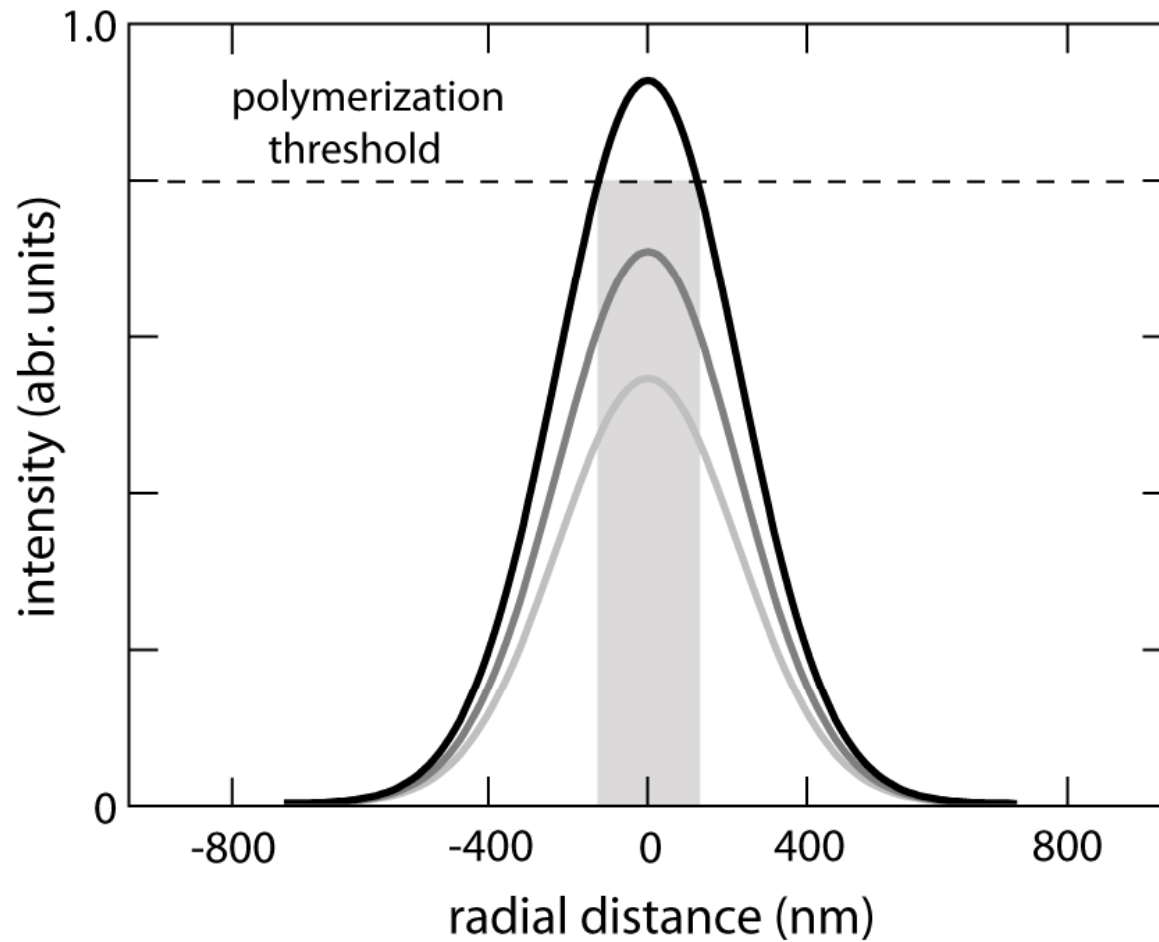
40 x
0.65 NA

Two-photon polymerization



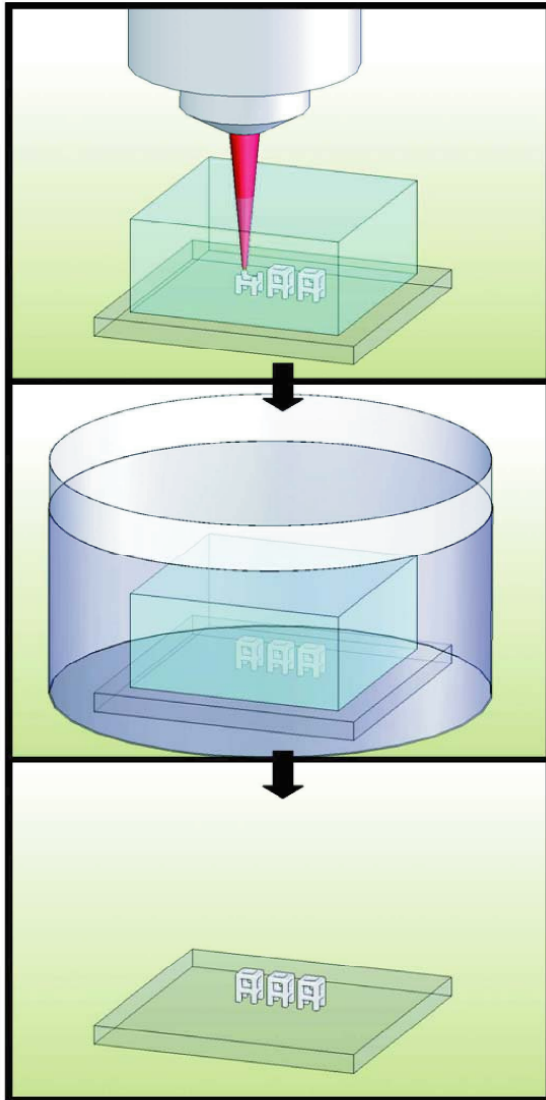
bellow the diffraction limit

Two-photon polymerization



even higher spatial resolution

Two-photon polymerization

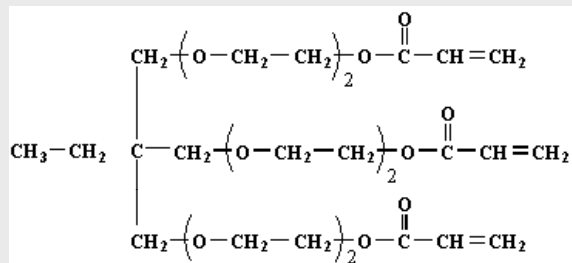


After the fabrication, the sample is immersed in ethanol to wash away any unsolidified resin and then dried

Resin preparation

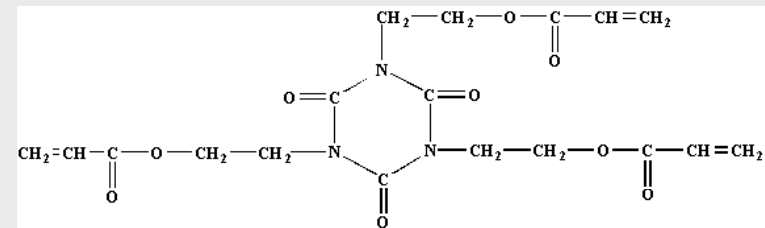
Monomers

Monomer A



reduces the shrinkage upon polymerization

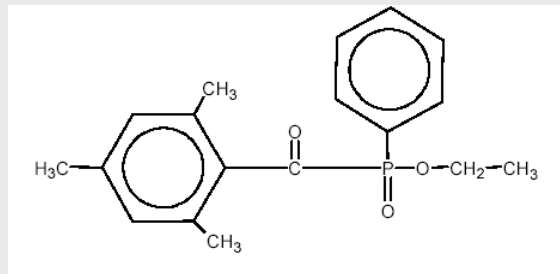
Monomer B



gives hardness to the polymeric structure

Photoinitiator

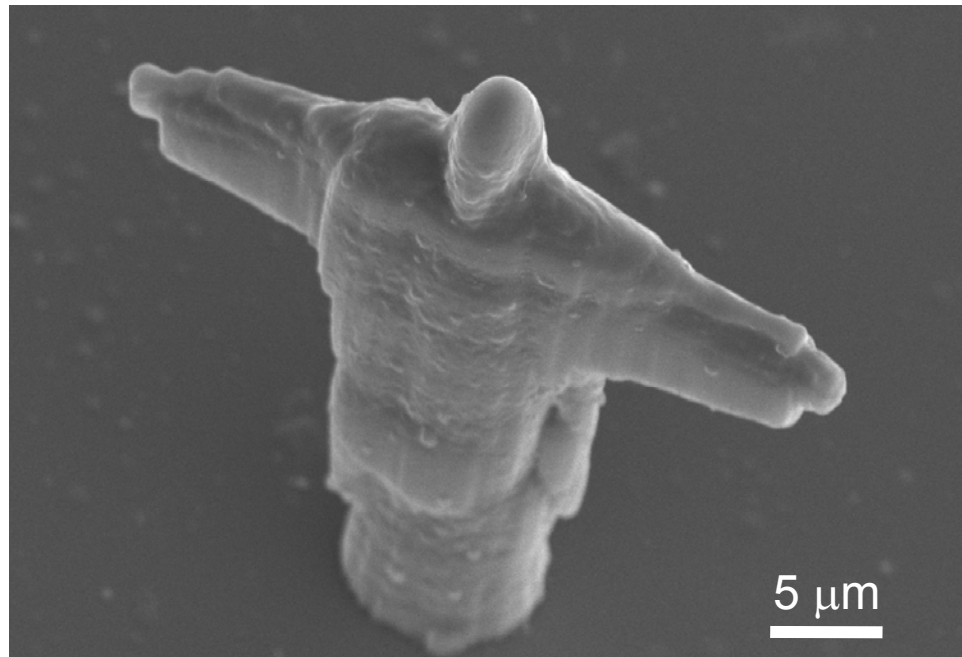
Lucirin TPO-L



Appl. Phys. A, 90, 633–636 (2008)

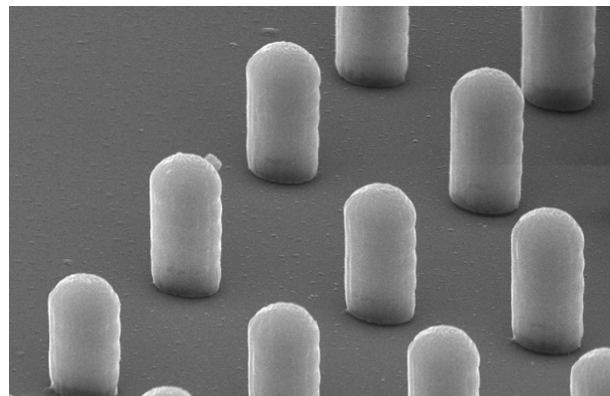
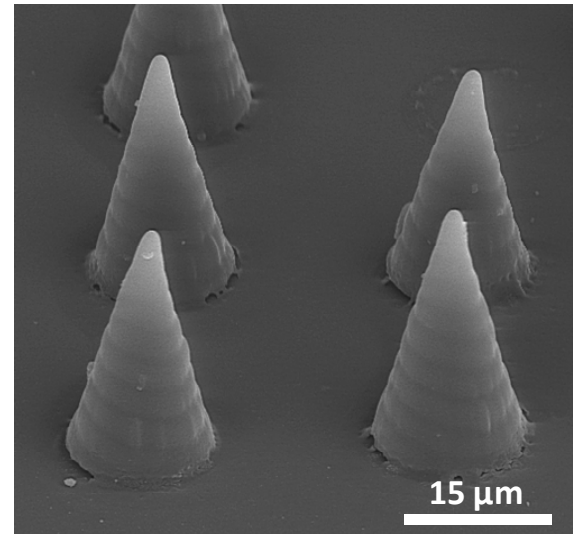
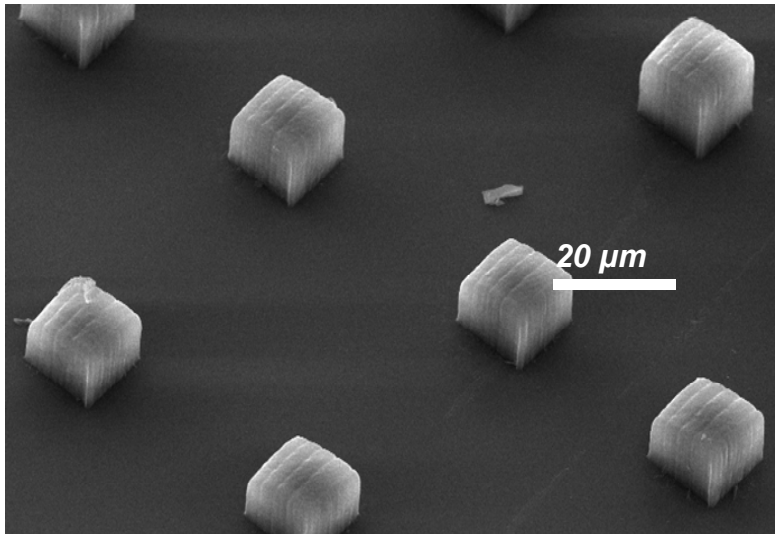
two-photon polymerization

Microstructure fabricated by two-photon polymerization



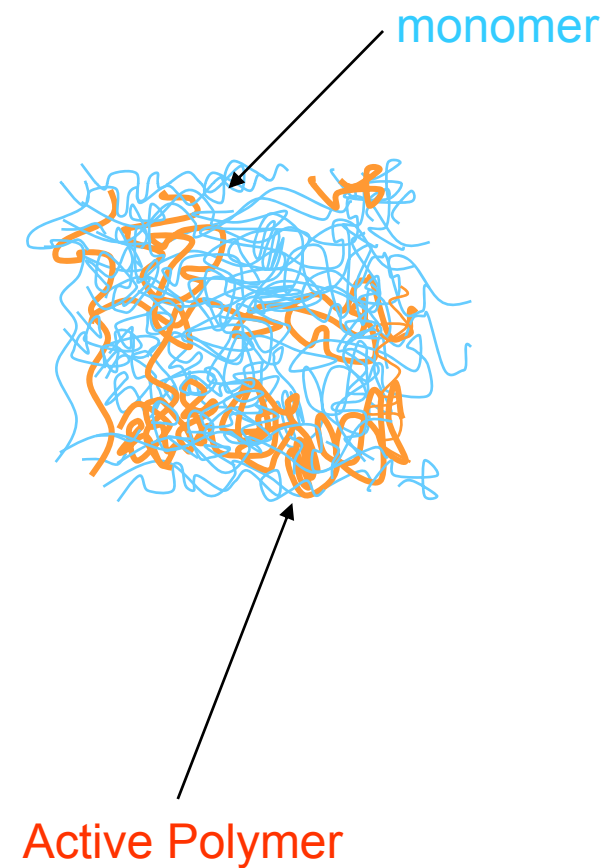
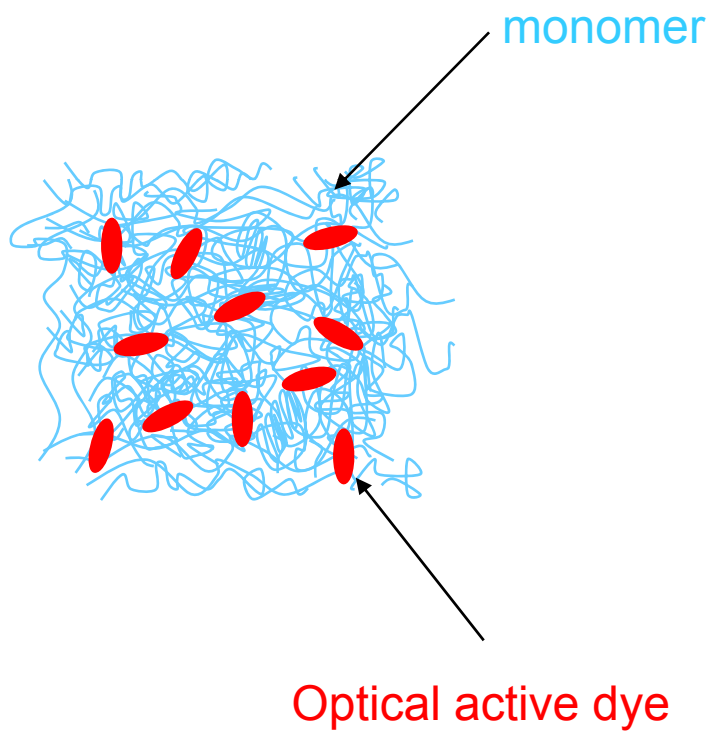
Two-photon polymerization

Microstructures fabricated by two-photon polymerization



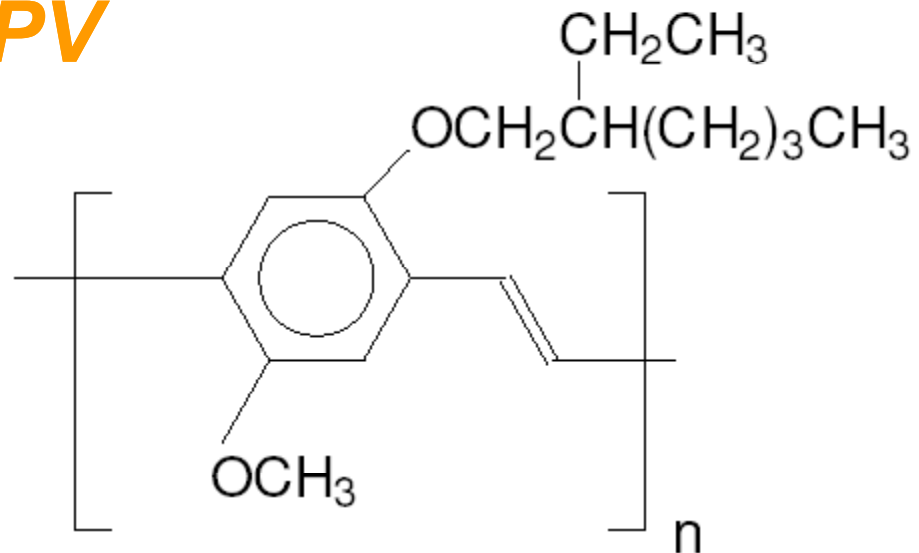
Doping microstructures

Microstructures containing active compounds



Microstructure containing MEH-PPV

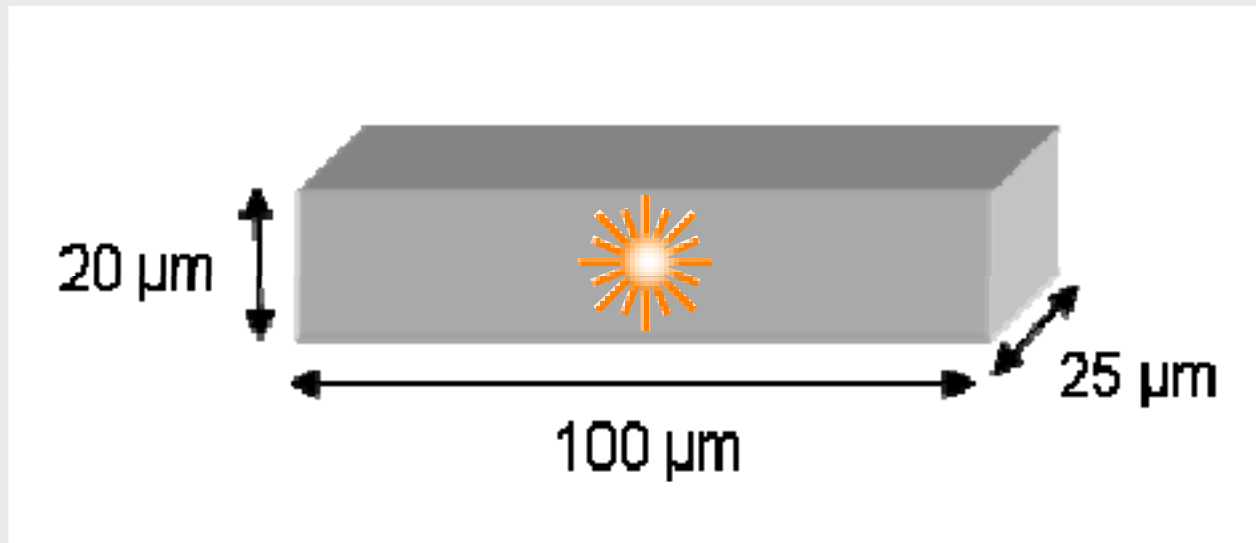
MEH-PPV



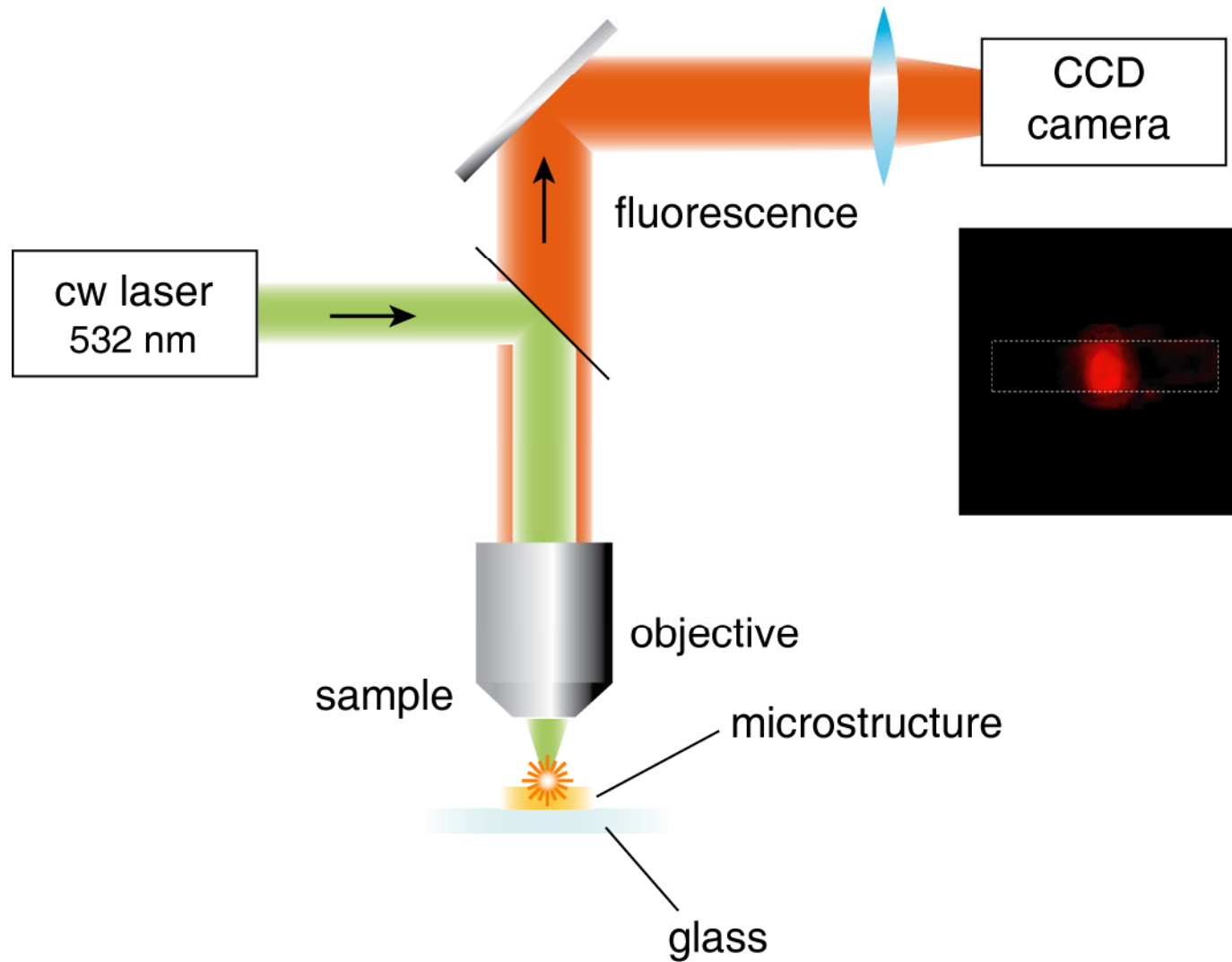
- Fluorescence
- Electro Luminescent
- Conductive

Microstructure containing MEH-PPV

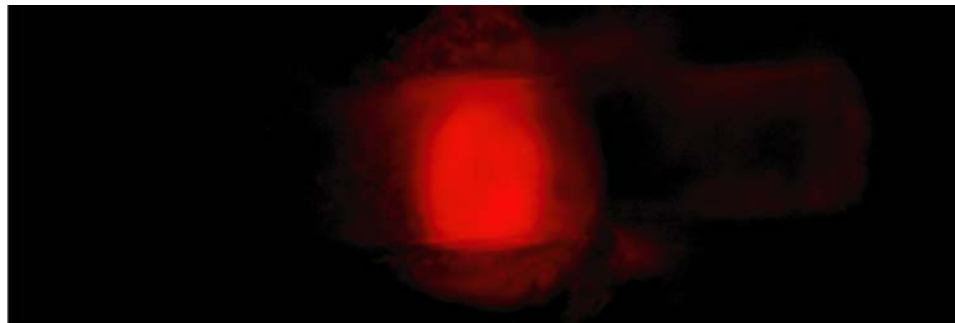
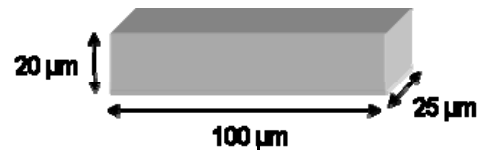
Do we have waveguiding in the microstructure ?



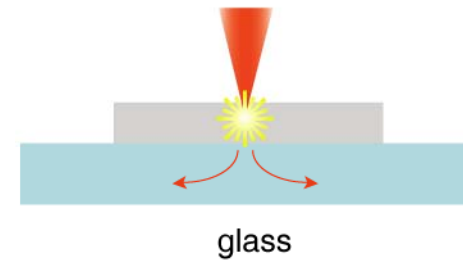
Microstructure containing MEH-PPV



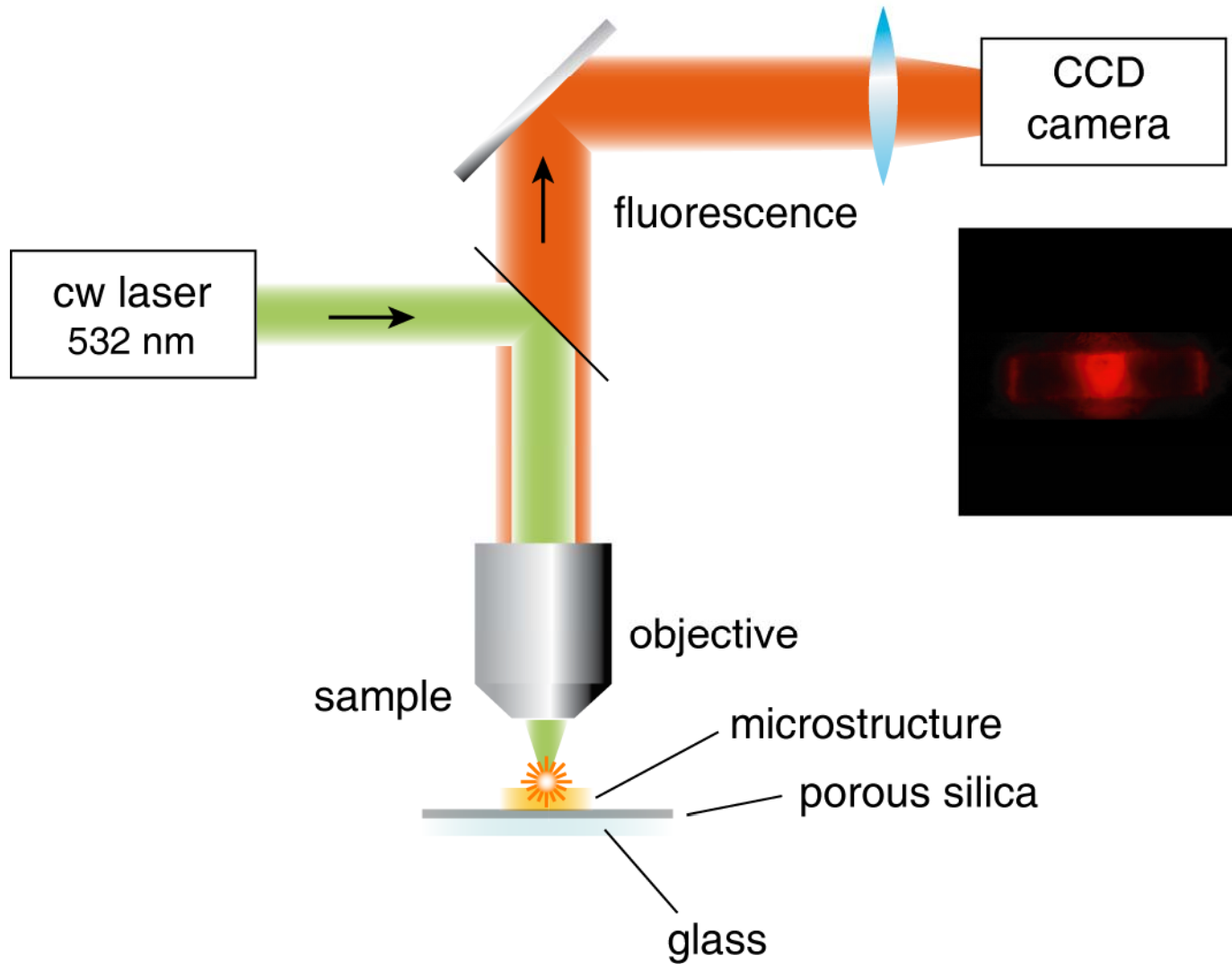
Microstructure containing MEH-PPV



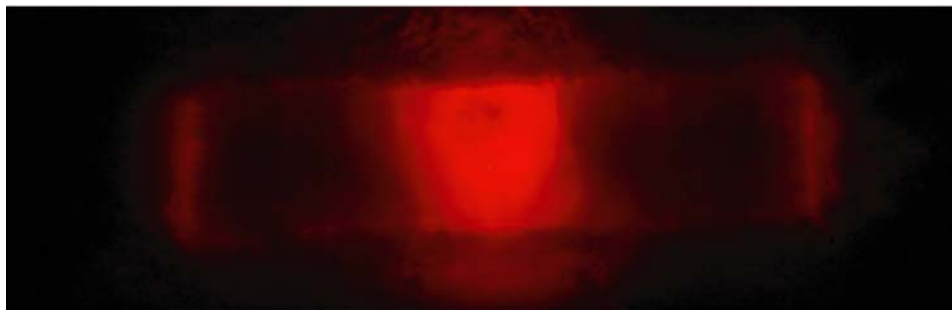
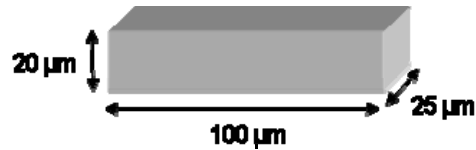
$20\ \mu\text{m}$ 



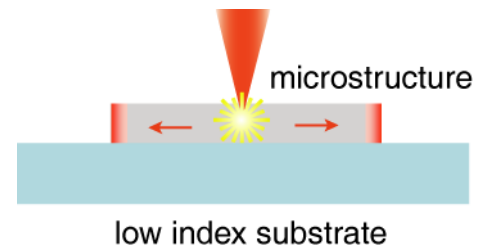
Microstructure containing MEH-PPV



Microstructure containing MEH-PPV



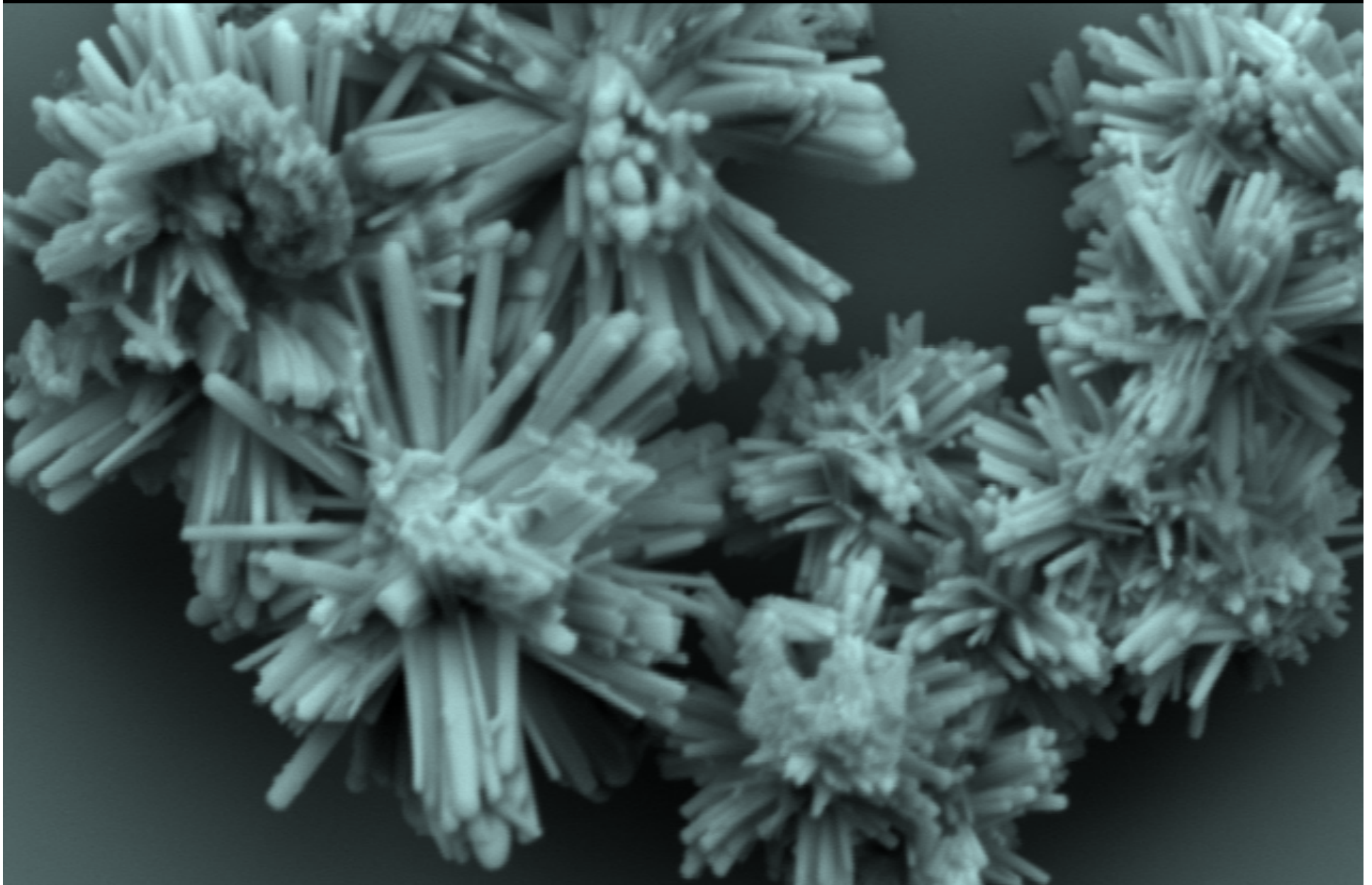
20 μm 



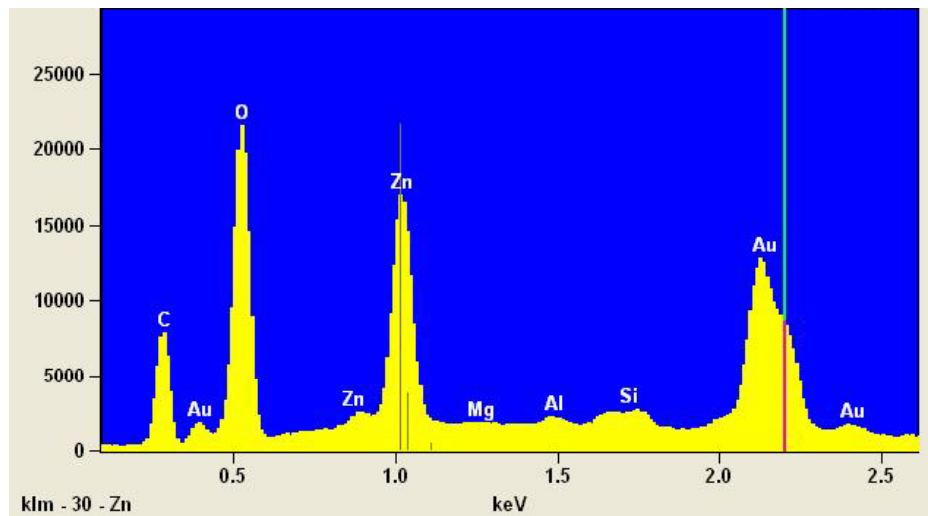
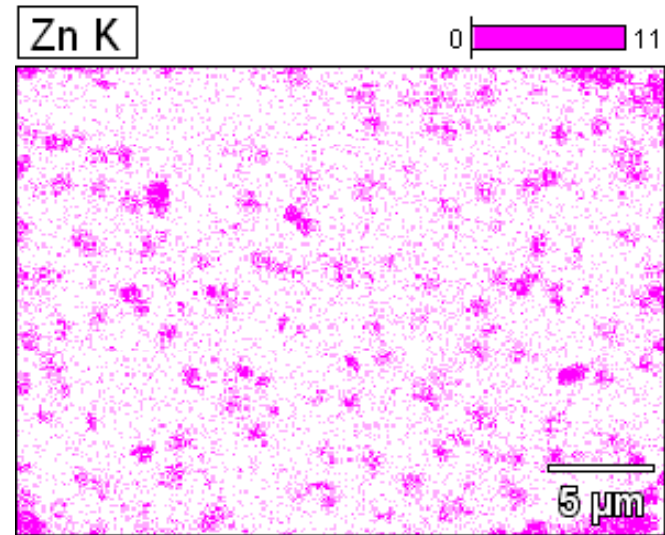
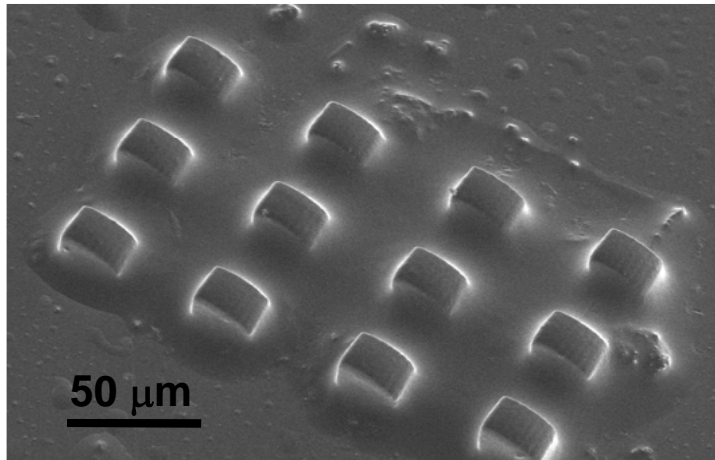
waveguiding of the microstructure fabricated on porous silica substrate ($n= 1.185$)

Applications: micro-laser; fluorescent microstructures; conductive microstructures

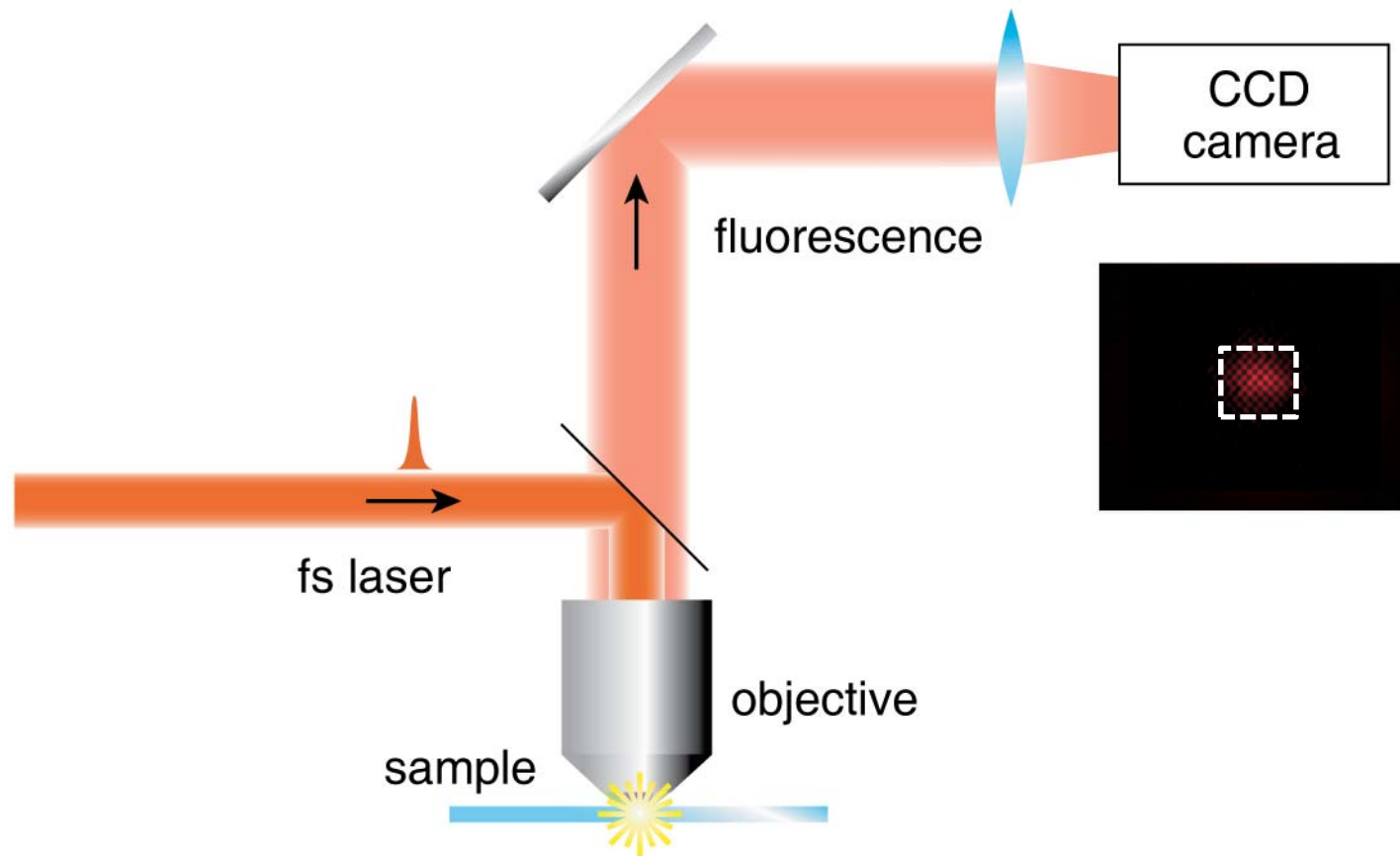
Microstructures with ZnO nanowires



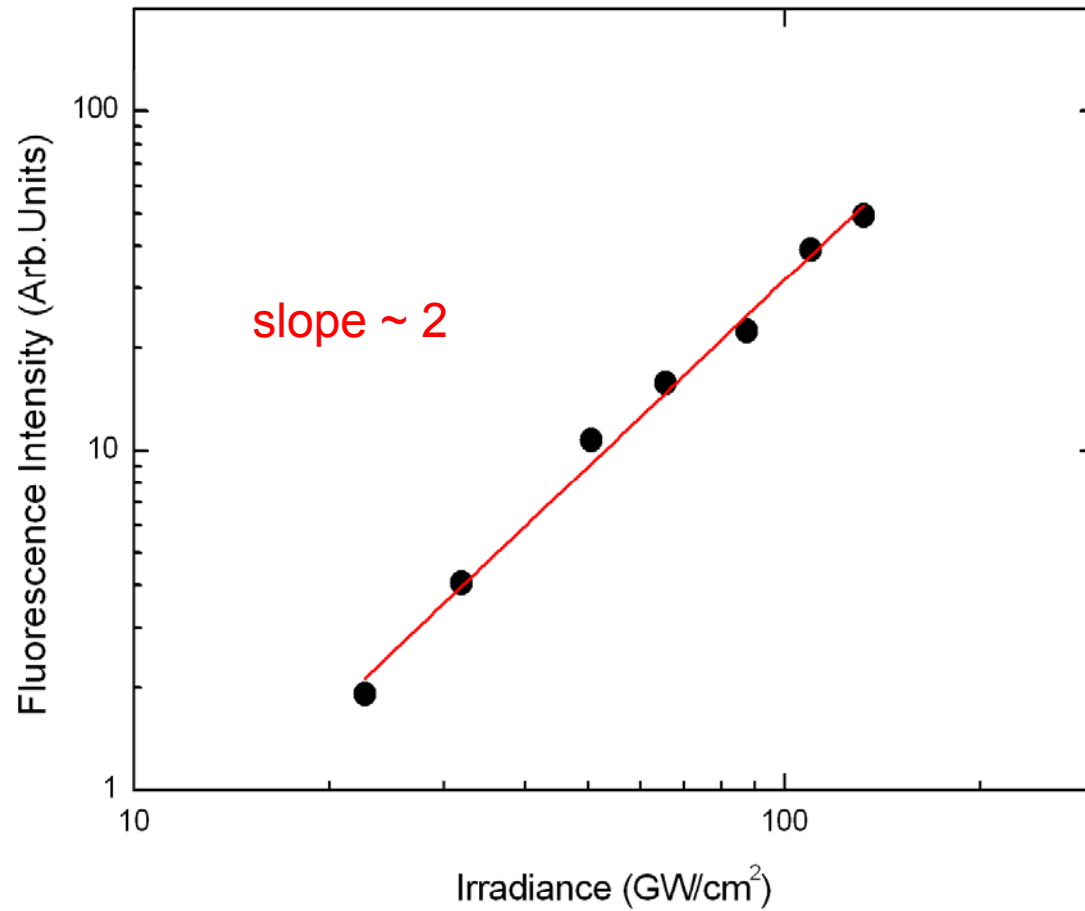
Microstructures with ZnO nanowires



Microstructures with ZnO nanowires

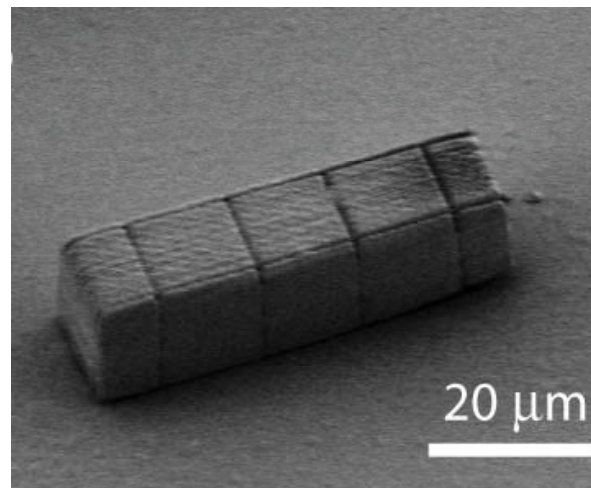
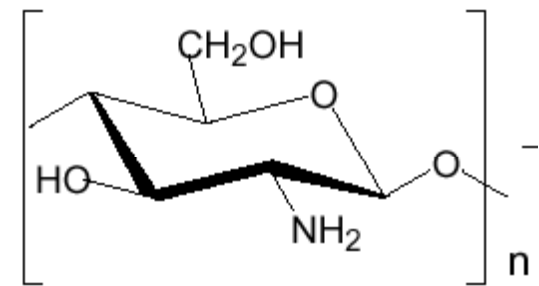
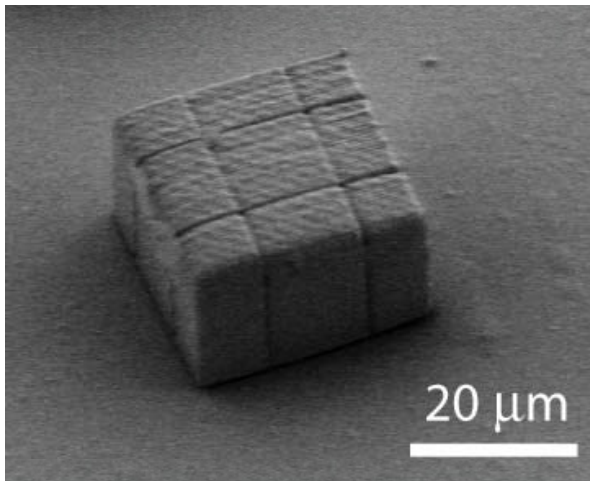


Microstructures with ZnO nanowires



Doping microstructures

- microstructures containing biopolymer - chitosan

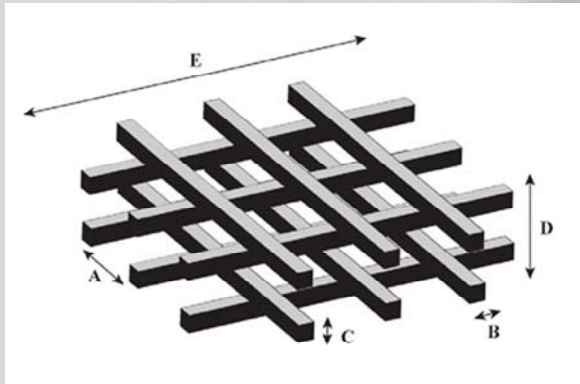


micro-environment to study cells and bacteria

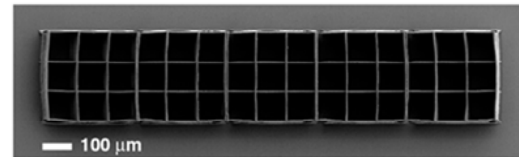
microfabrication of special
microstructures to biology

3D cell migration

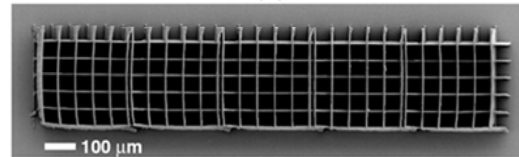
- 3D cell migration studies in micro-scaffolds



SEM of the scaffolds



110 μm pore size



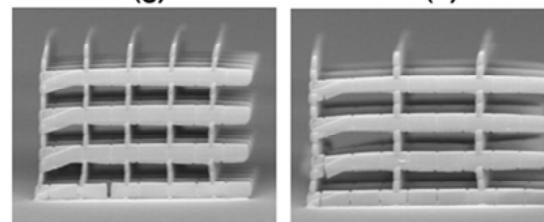
52 μm pore size



Top view



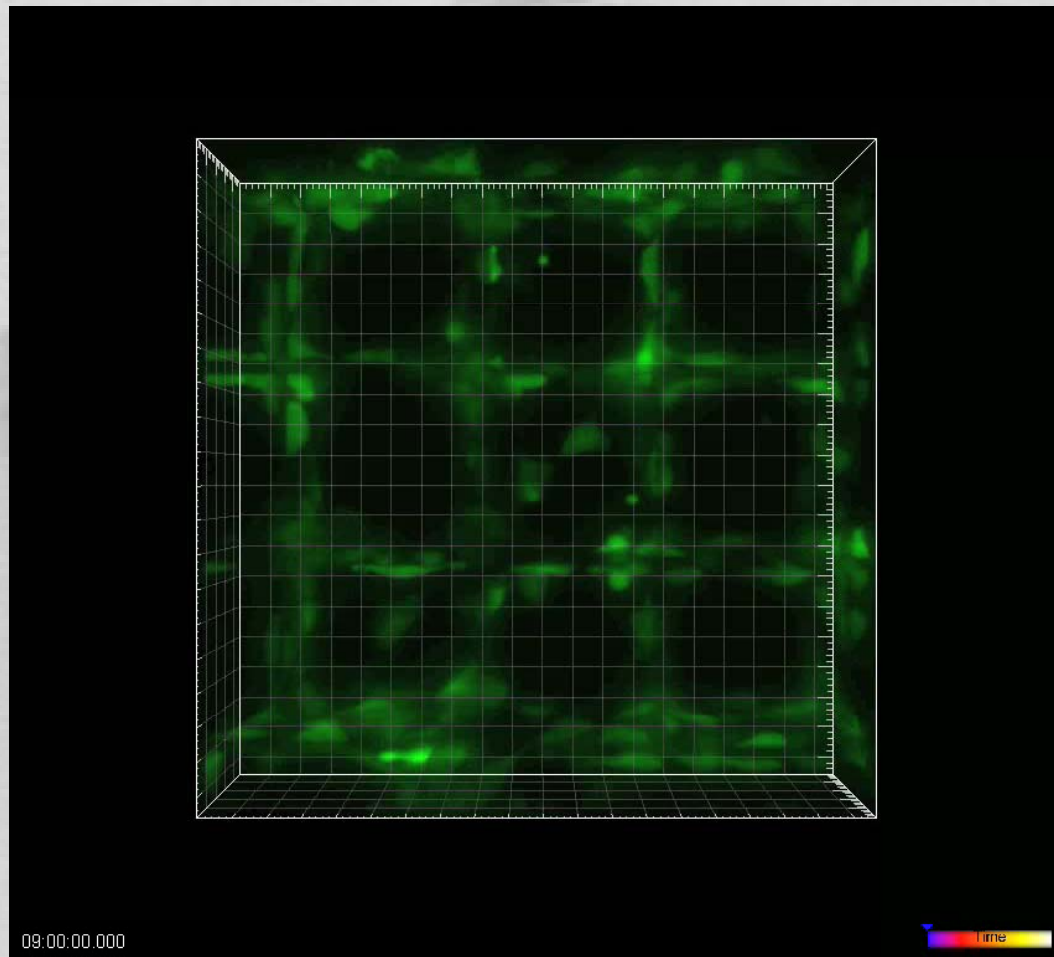
110, 52, 25, 12 μm pore size



Side view

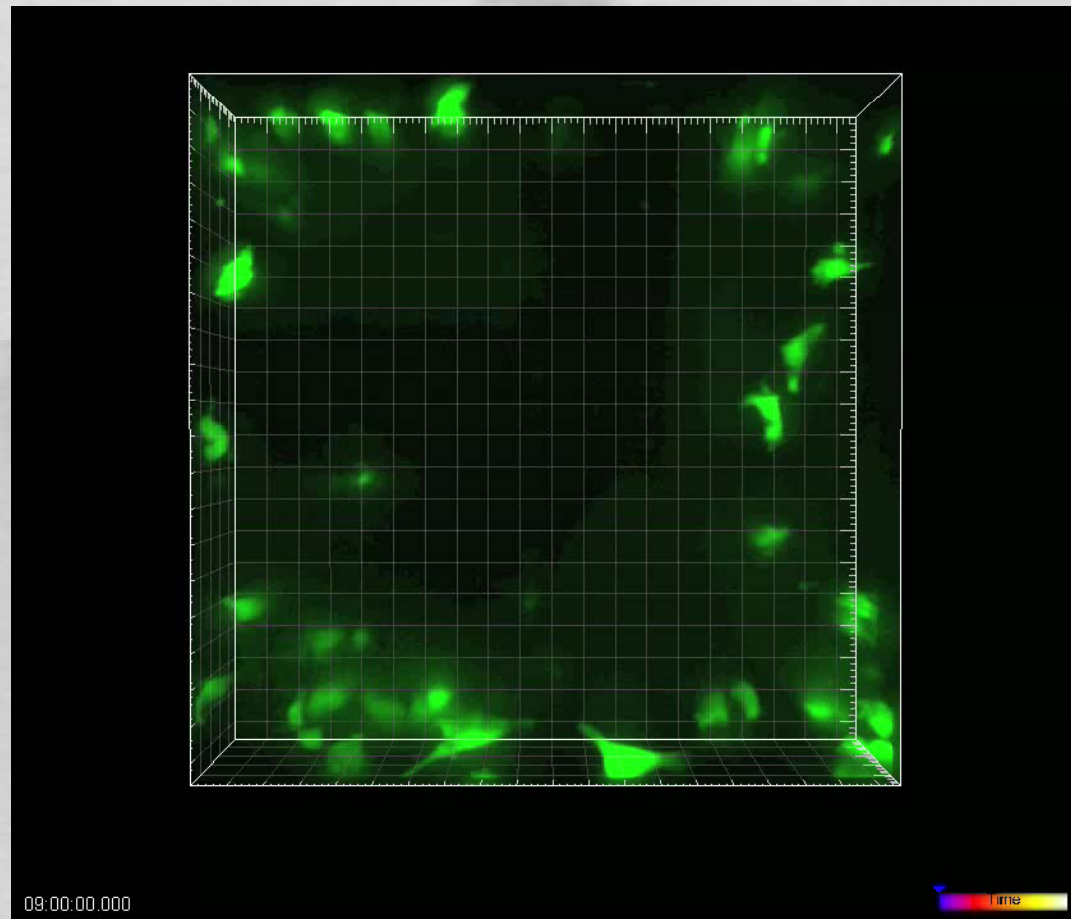
25, 52 μm pore size

3D cell migration



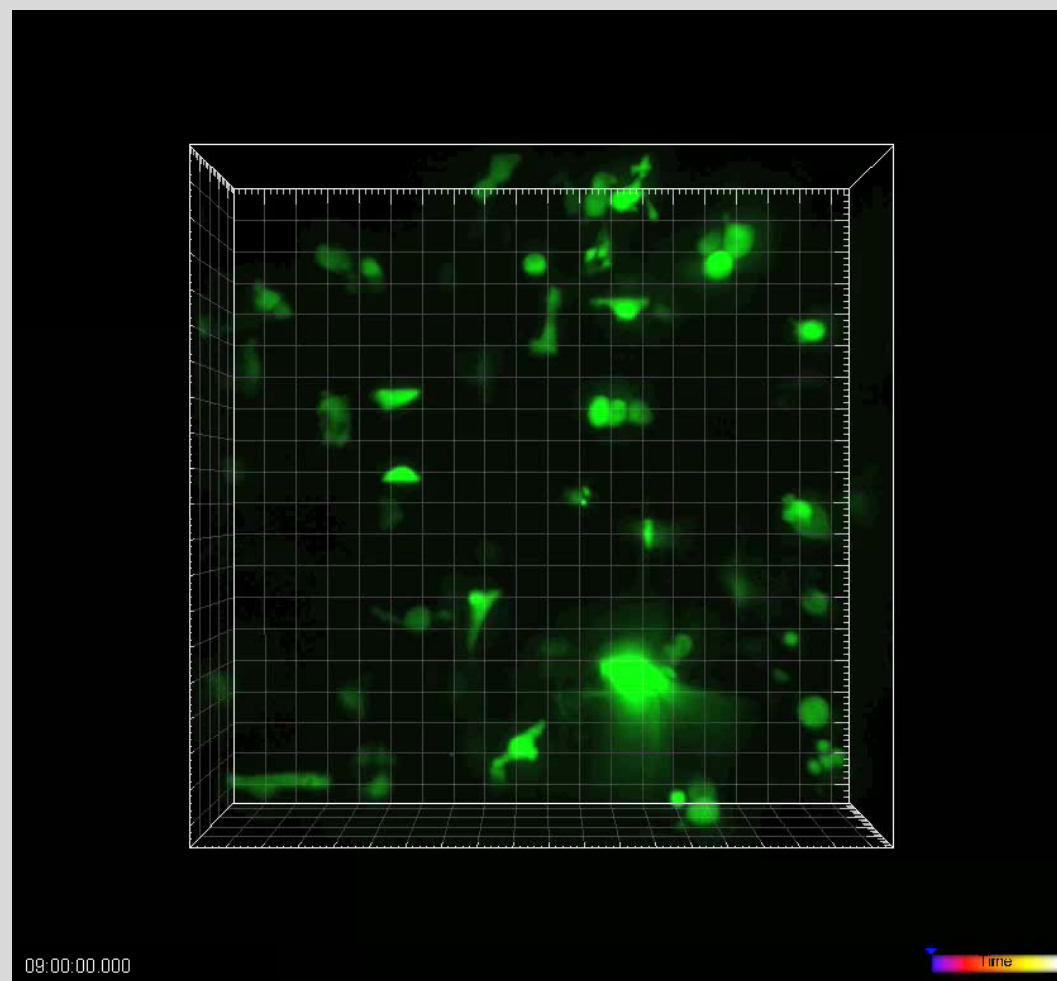
110 μm pore size

3D cell migration



12 μm pore size

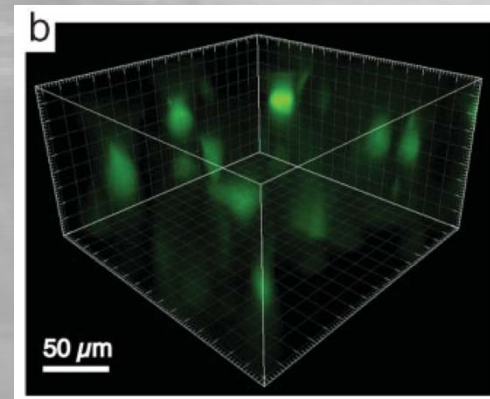
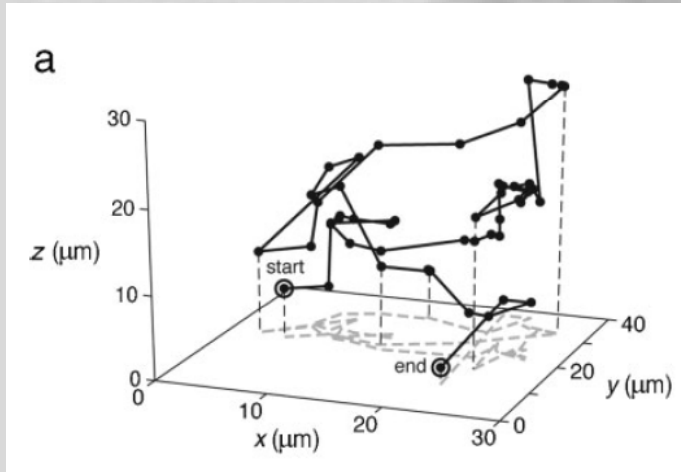
3D cell migration



52 μm pore size

3D cell migration

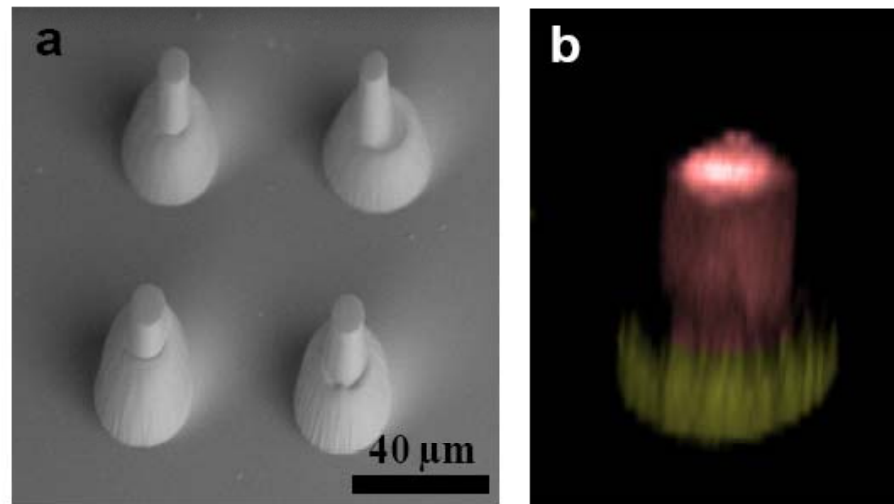
- 3D cell migration studies in micro-scaffolds



Guiding bacterial growth in a micro-environment

to study bacterial growth it was needed to develop **double doped microstructures**

microstructure containing Fluorescein and Rhodamine

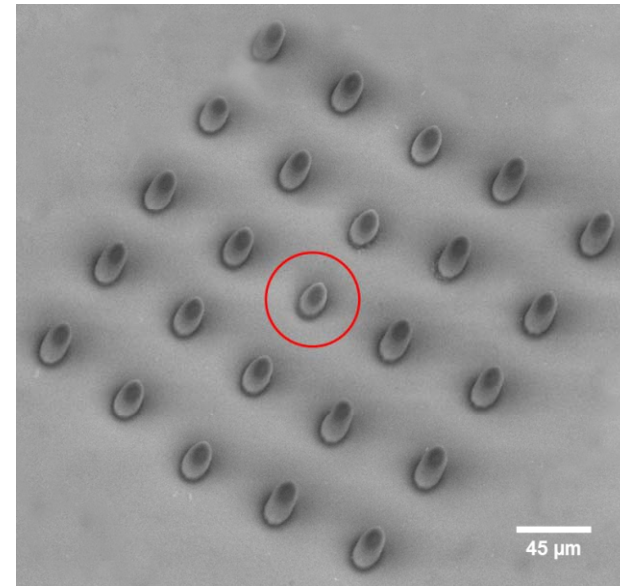
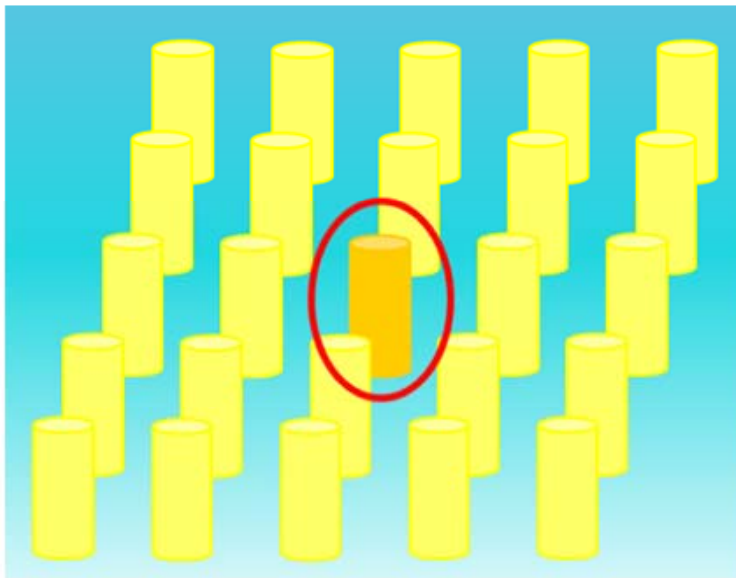


(a) SEM of a double-doped microstructure (top view).

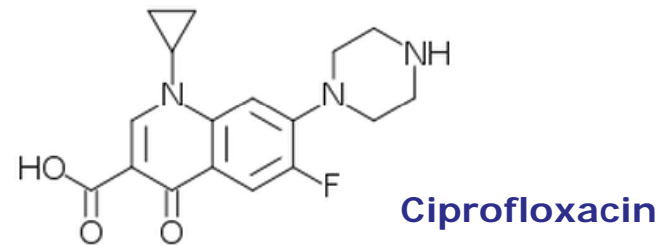
(b) Confocal fluorescent microscopy image of the same microstructure.

Guiding bacterial growth in a micro-environment

Study the development of *E. coli* in micro-environments:

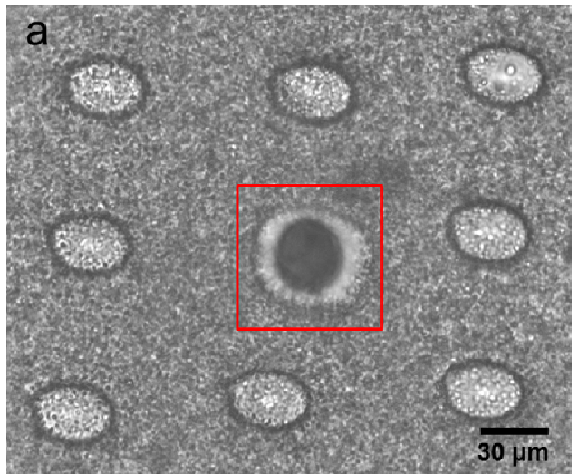


micro-environment in which the central structure contains antibiotic.

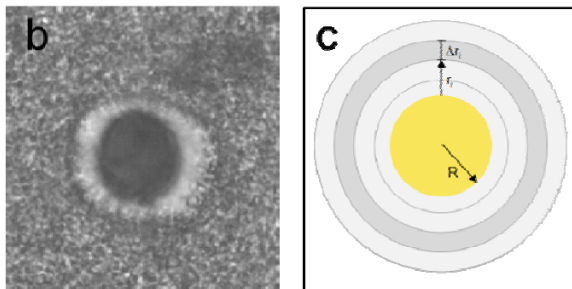


Guiding bacterial growth in a micro-environment

Study the development of *E. coli* in micro-environments:

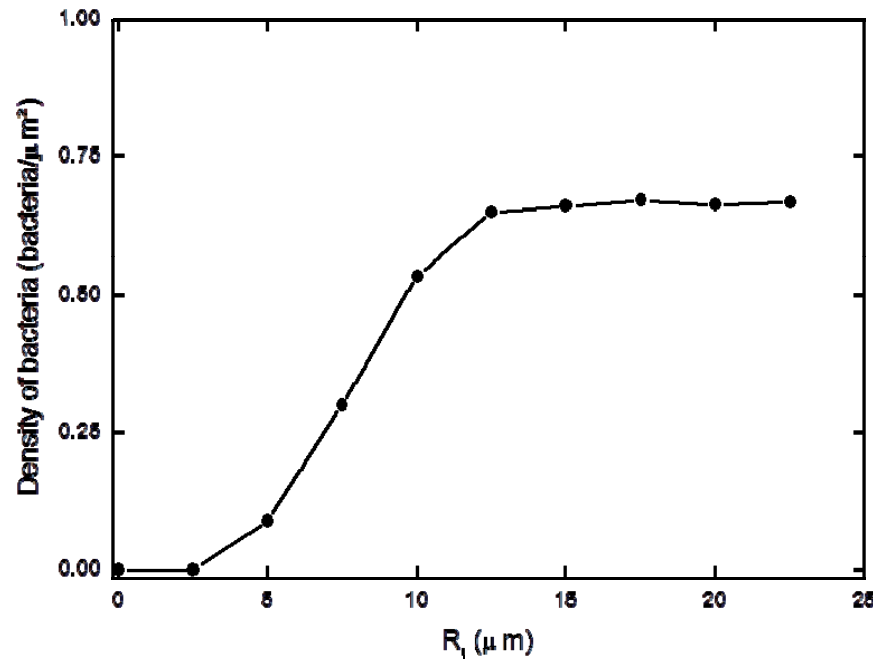


after 3 hours, we observed that a small region around the doped structure does not show bacterial growth.



such inhibition zone was analyzed by determining the bacterial density in concentric rings

Guiding bacterial growth in a micro-environment



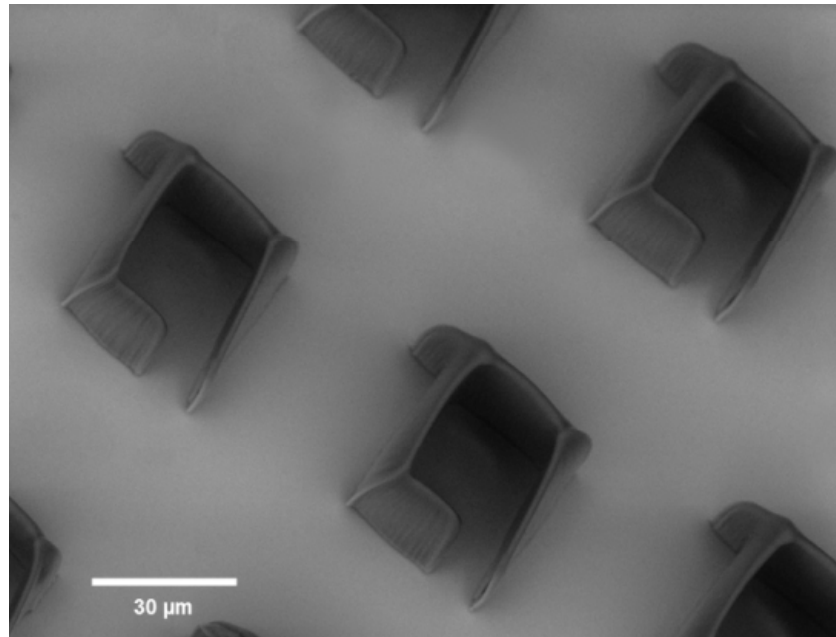
the density of bacteria grows monotonically with r_i

saturating when r_i reaches approximately 12 μm in about 0.7 bacteria/μm²

the inhibition zone has a maximum range of approximately 10 μm, being more effective as one gets closer to the microstructure impregnated with ciprofloxacin

Guiding bacterial growth in a micro-environment

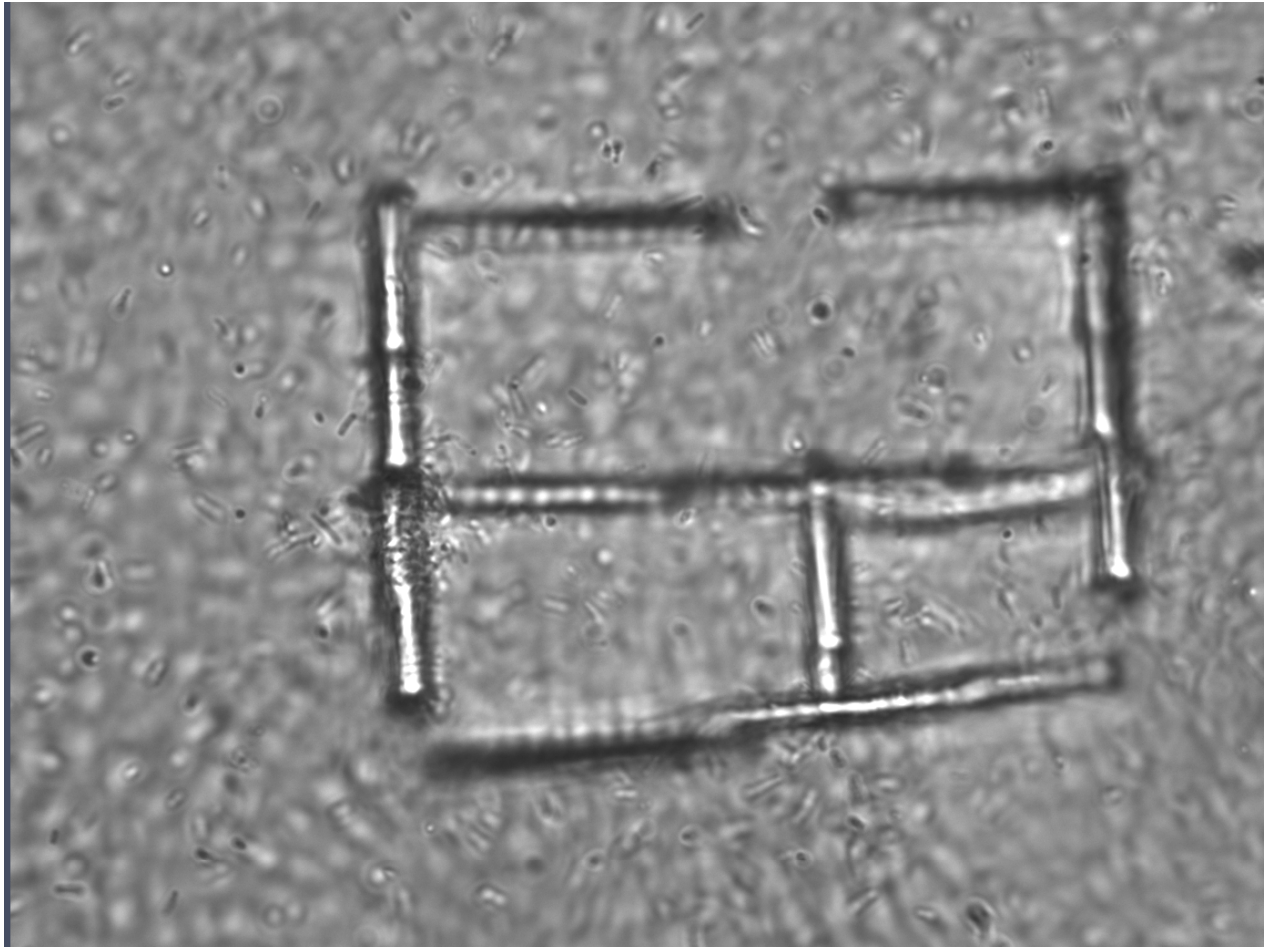
Bacteria microtraps



using micro-environments to study the dynamics of bacterial migration

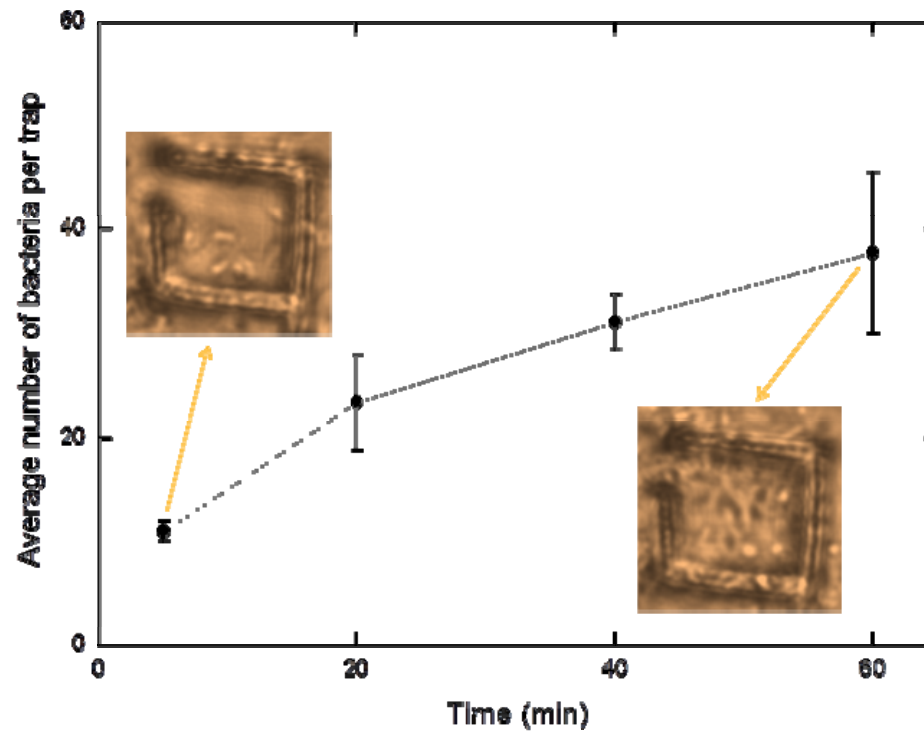
Guiding bacterial growth in a micro-environment

Bacteria microtraps



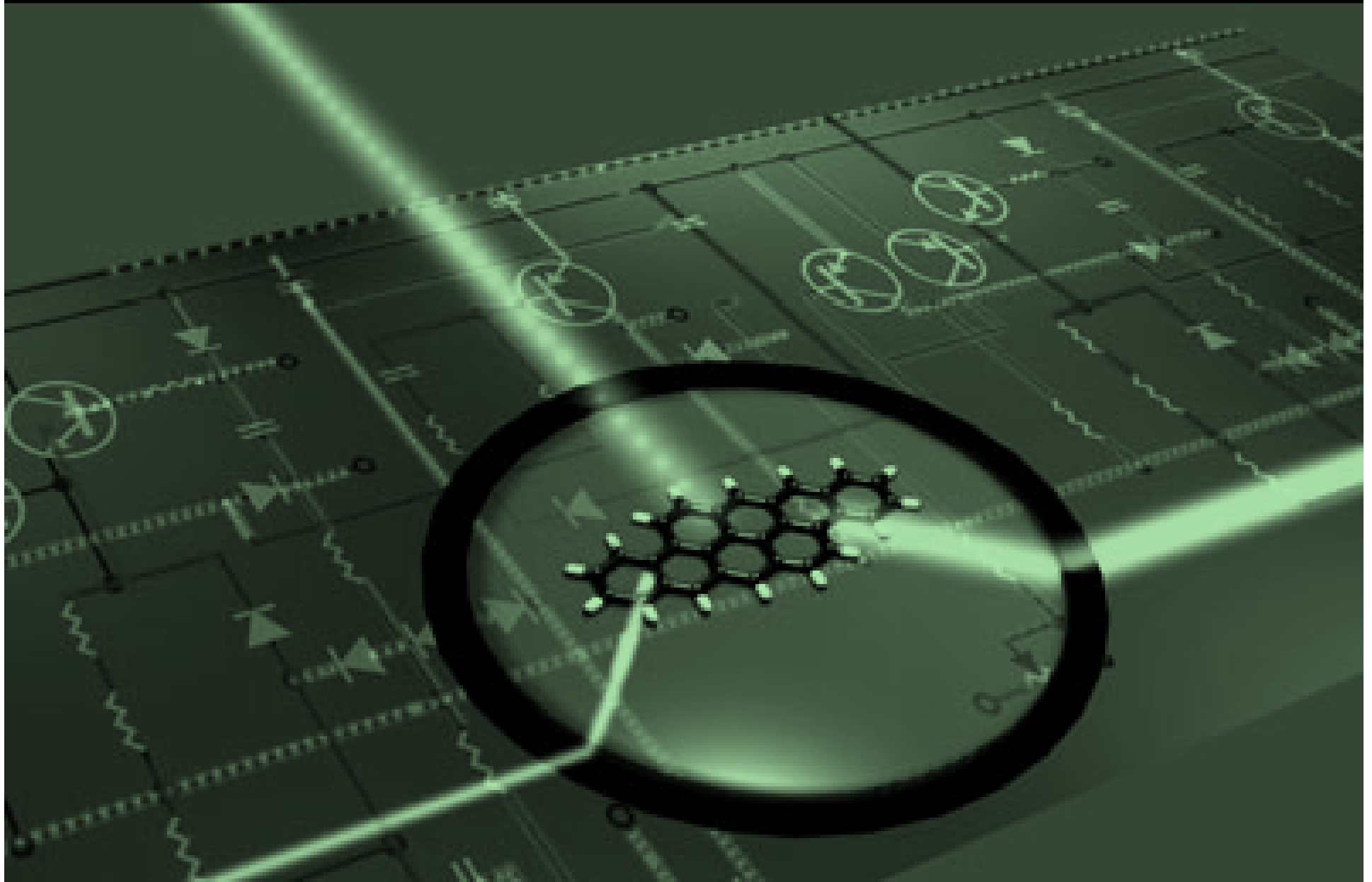
Guiding bacterial growth in a micro-environment

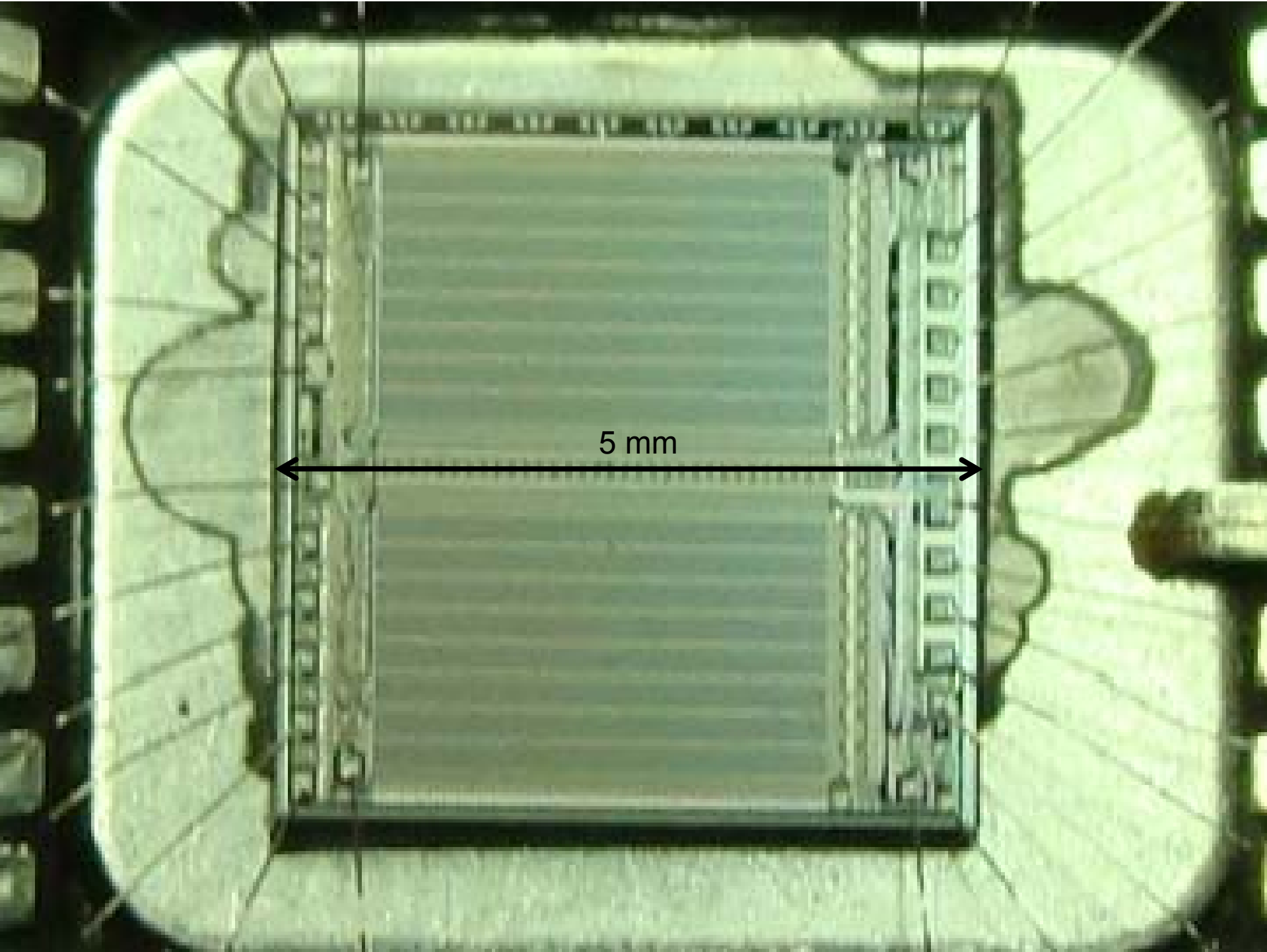
Bacteria microtraps

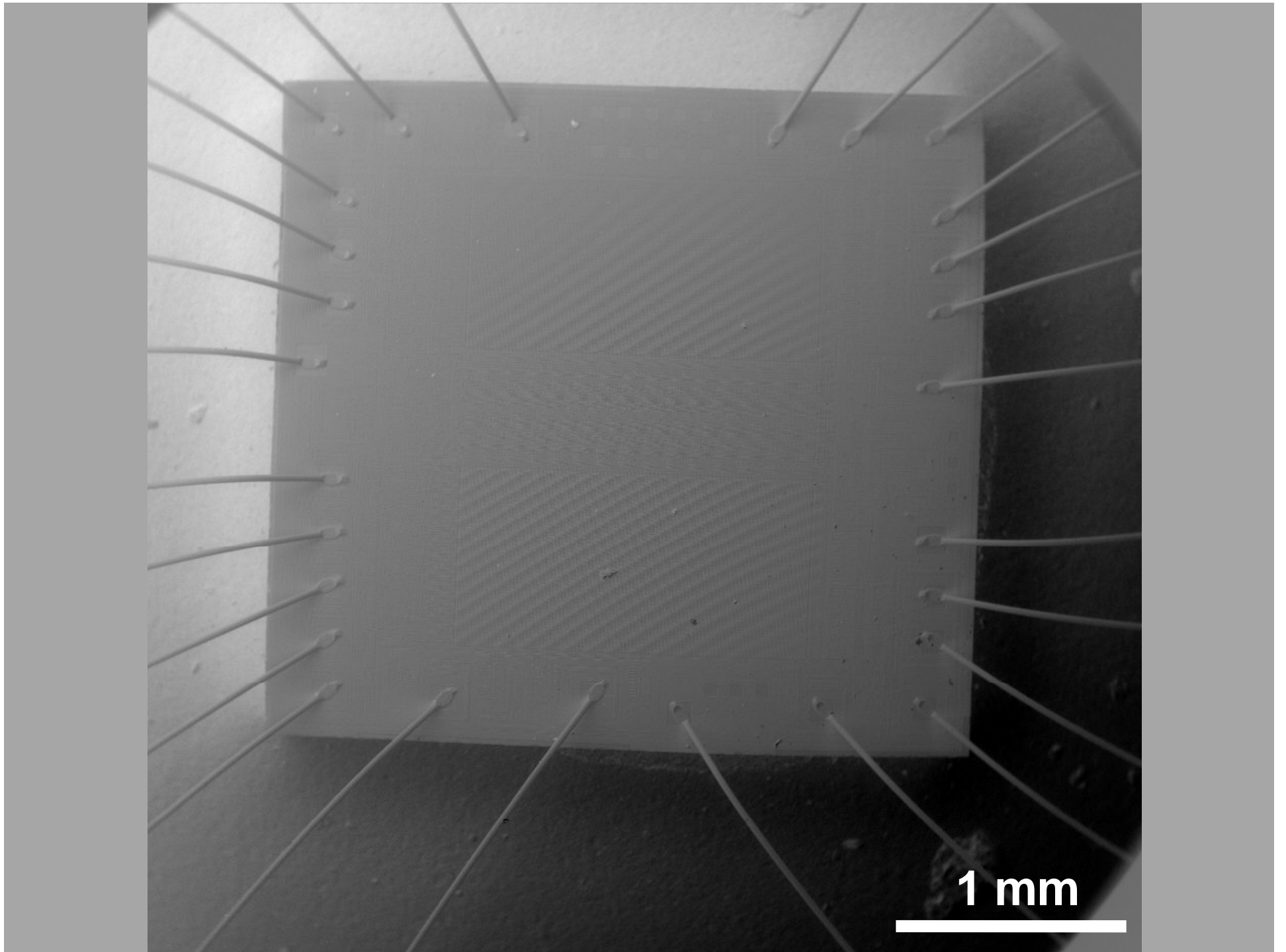


using micro-environments to study the dynamics of bacterial migration

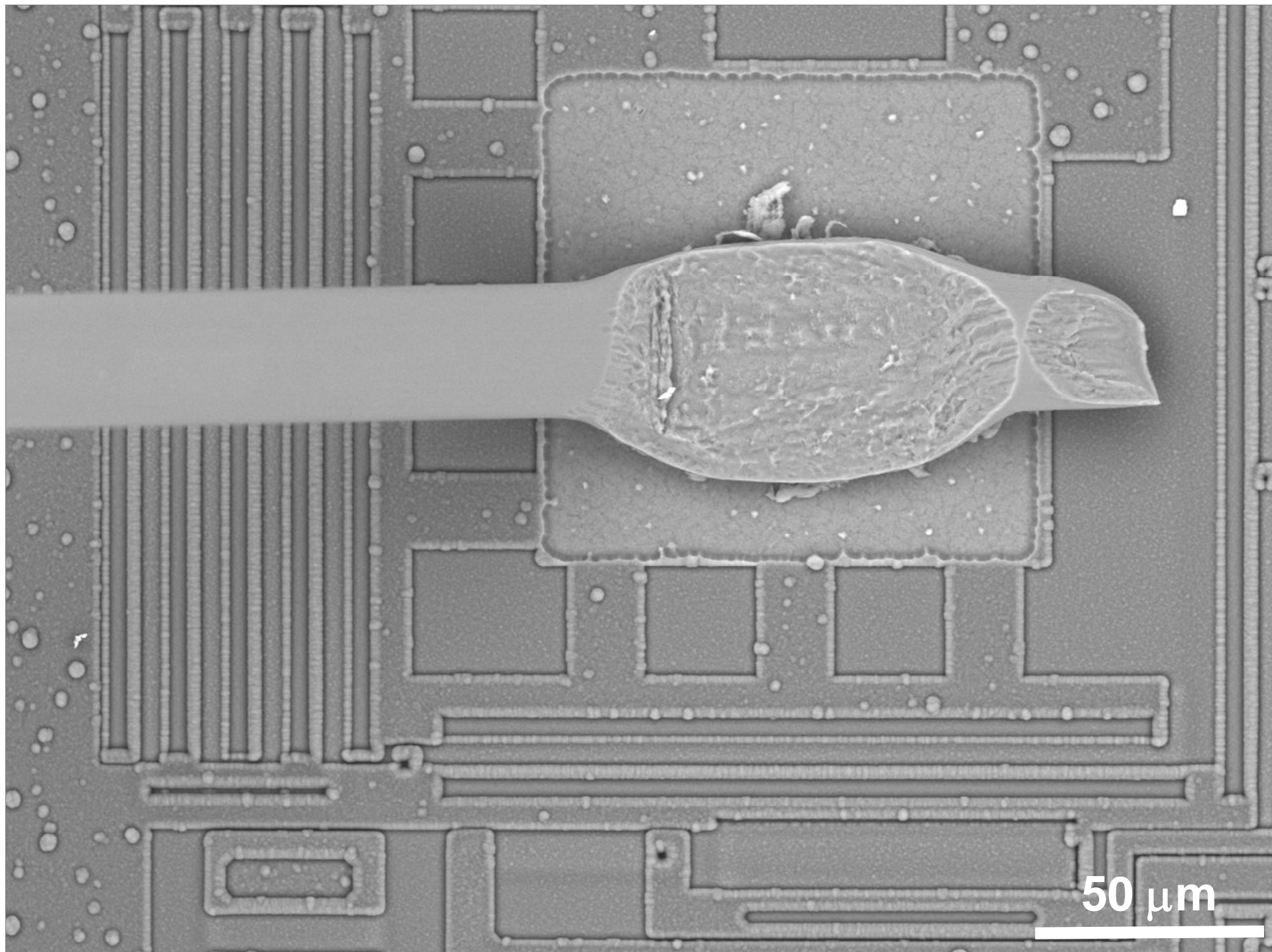
Optical circuit







1 mm



50 μm

Optical circuit

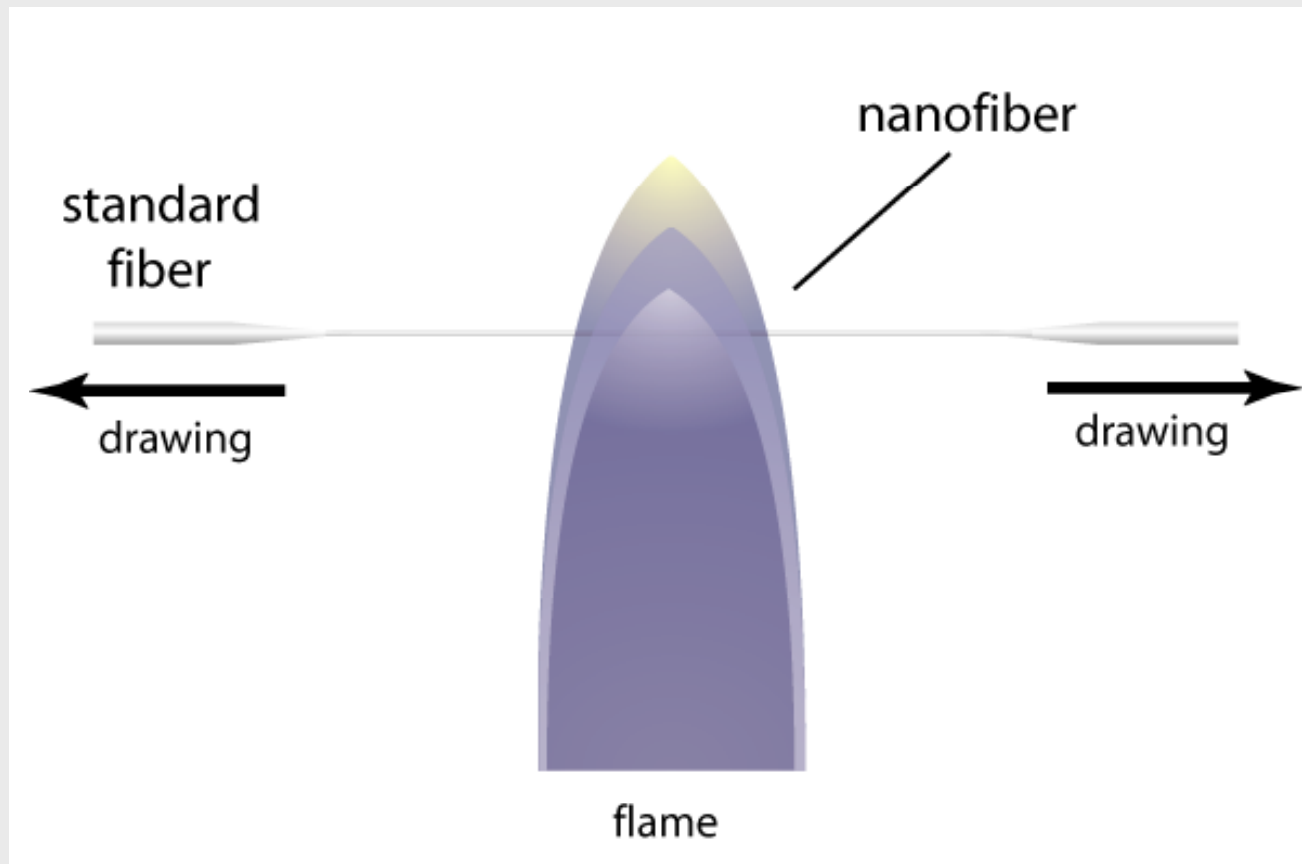
- microfabrication
- silica nanowires
- coupling microstructures

50 μm

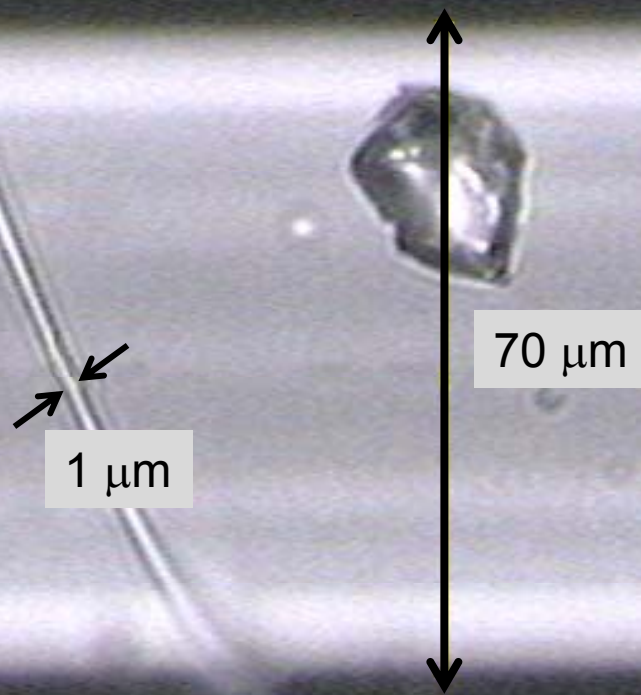


Silica nanowires

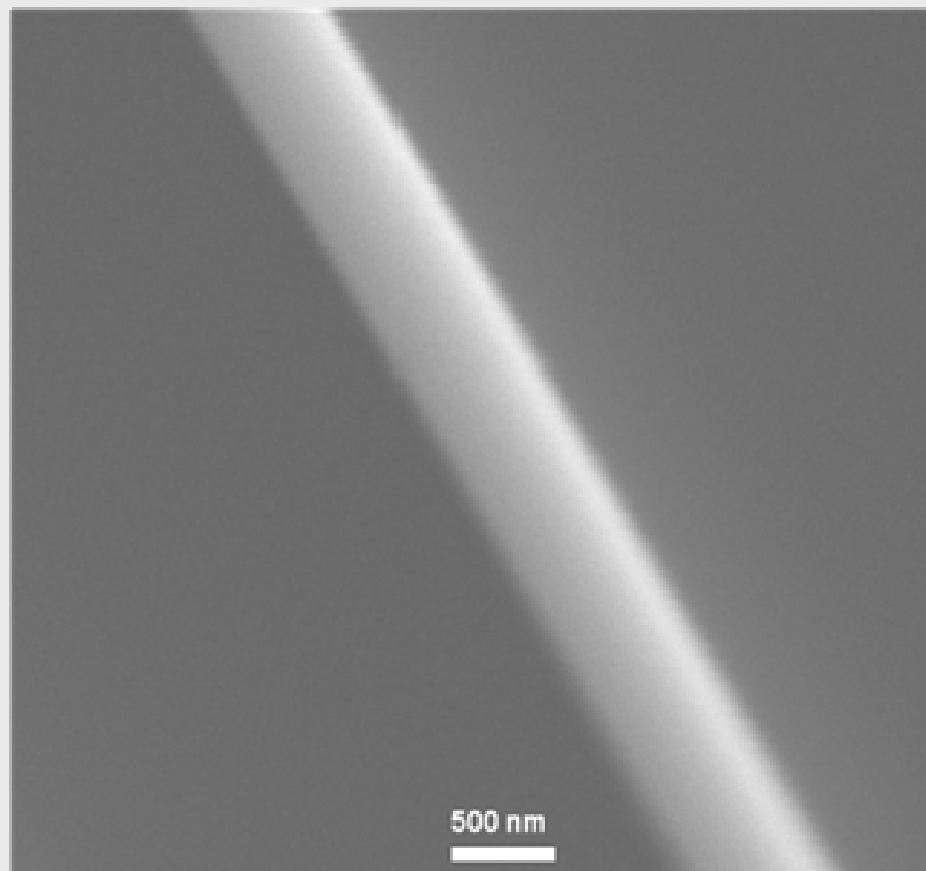
nanowires fabrication process



Silica nanowires

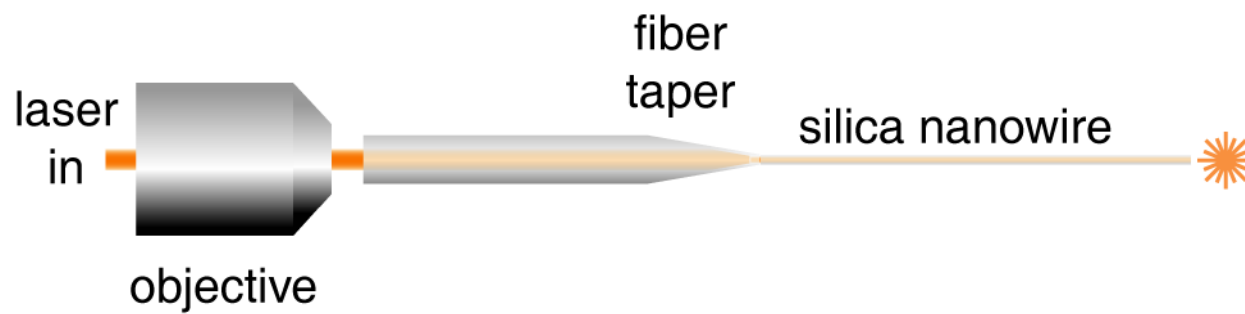


Silica nanowires



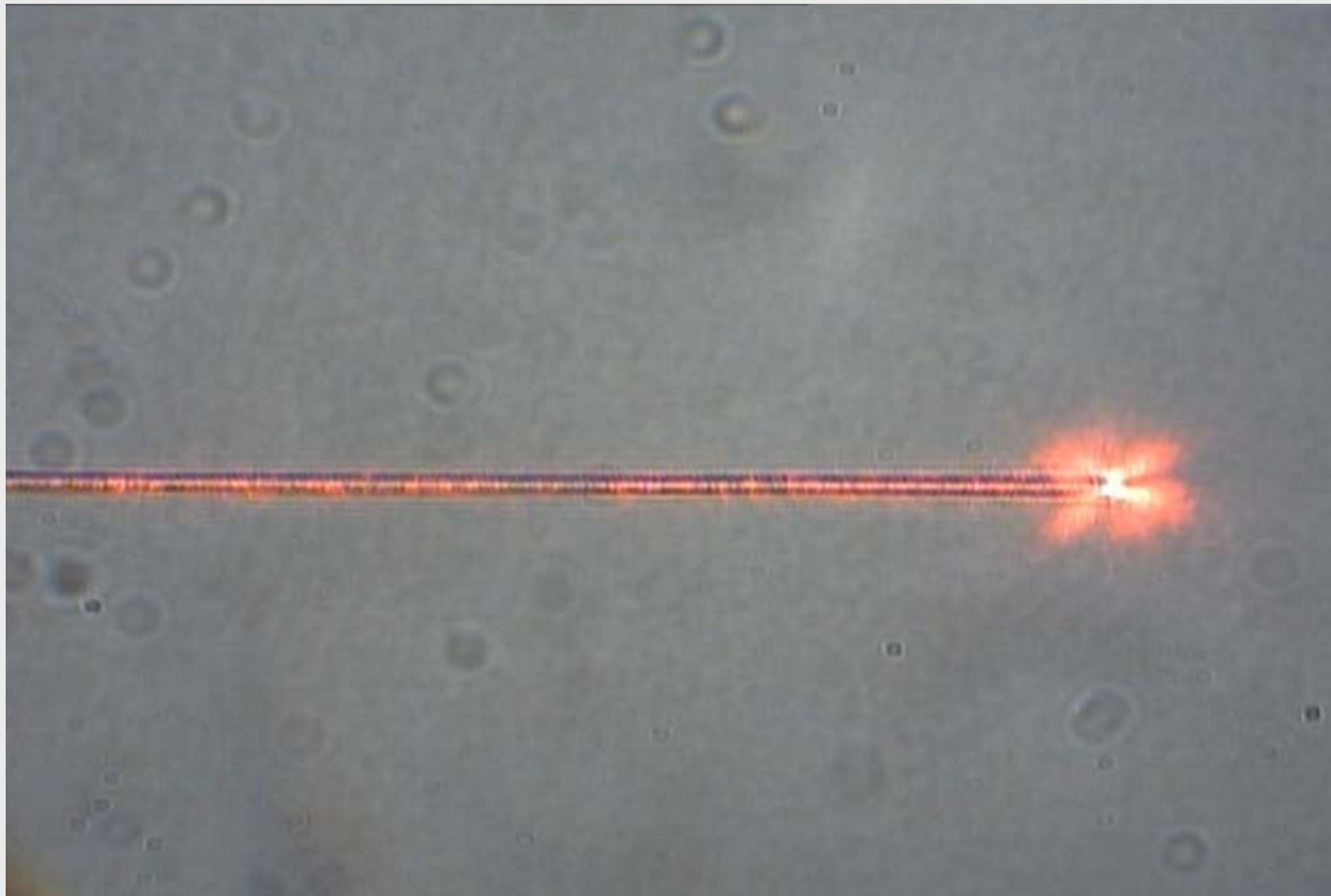
Silica nanowires

coupling light into nanowires



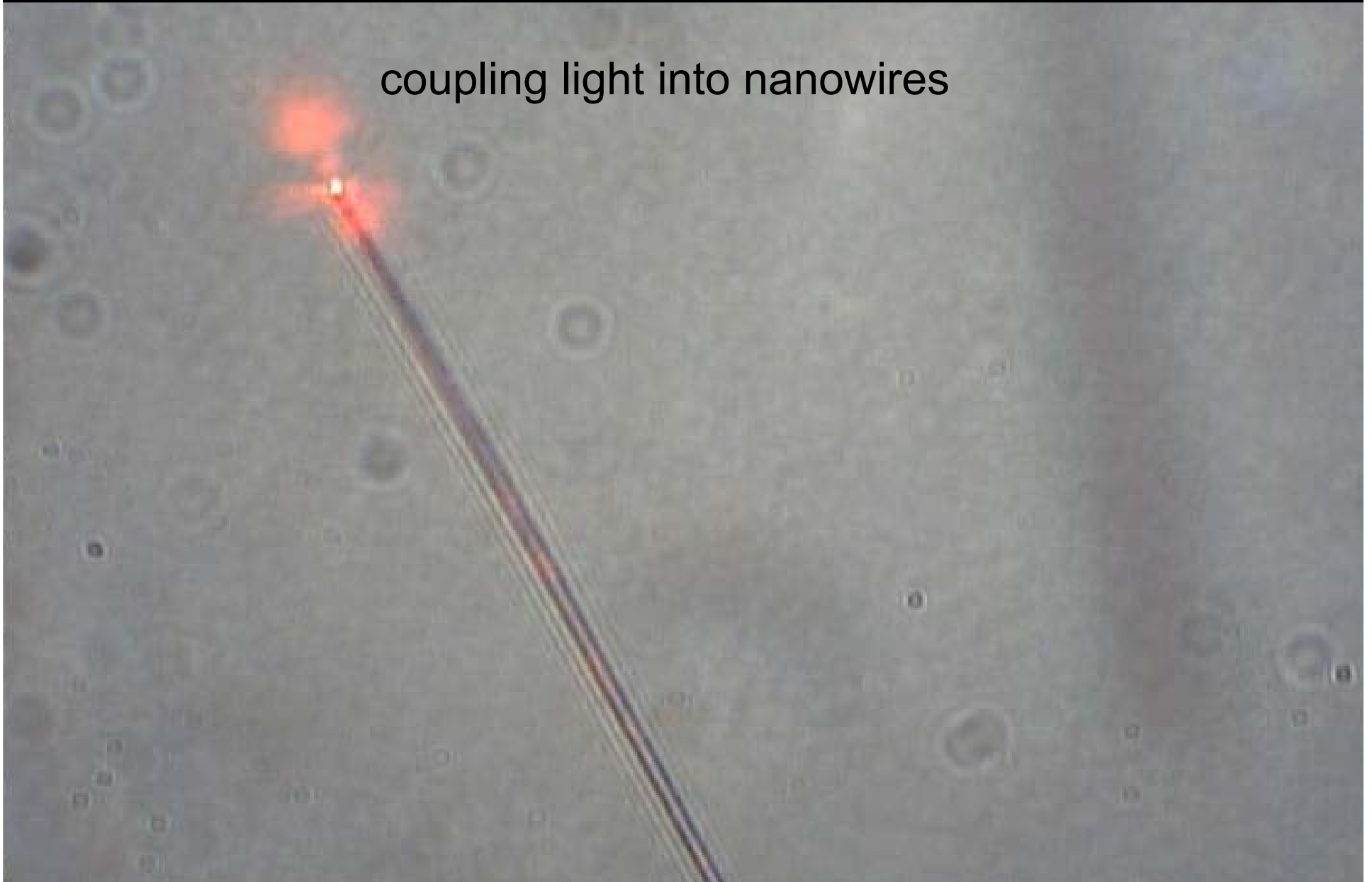
Silica nanowires

coupling light into nanowires



Silica nanowires

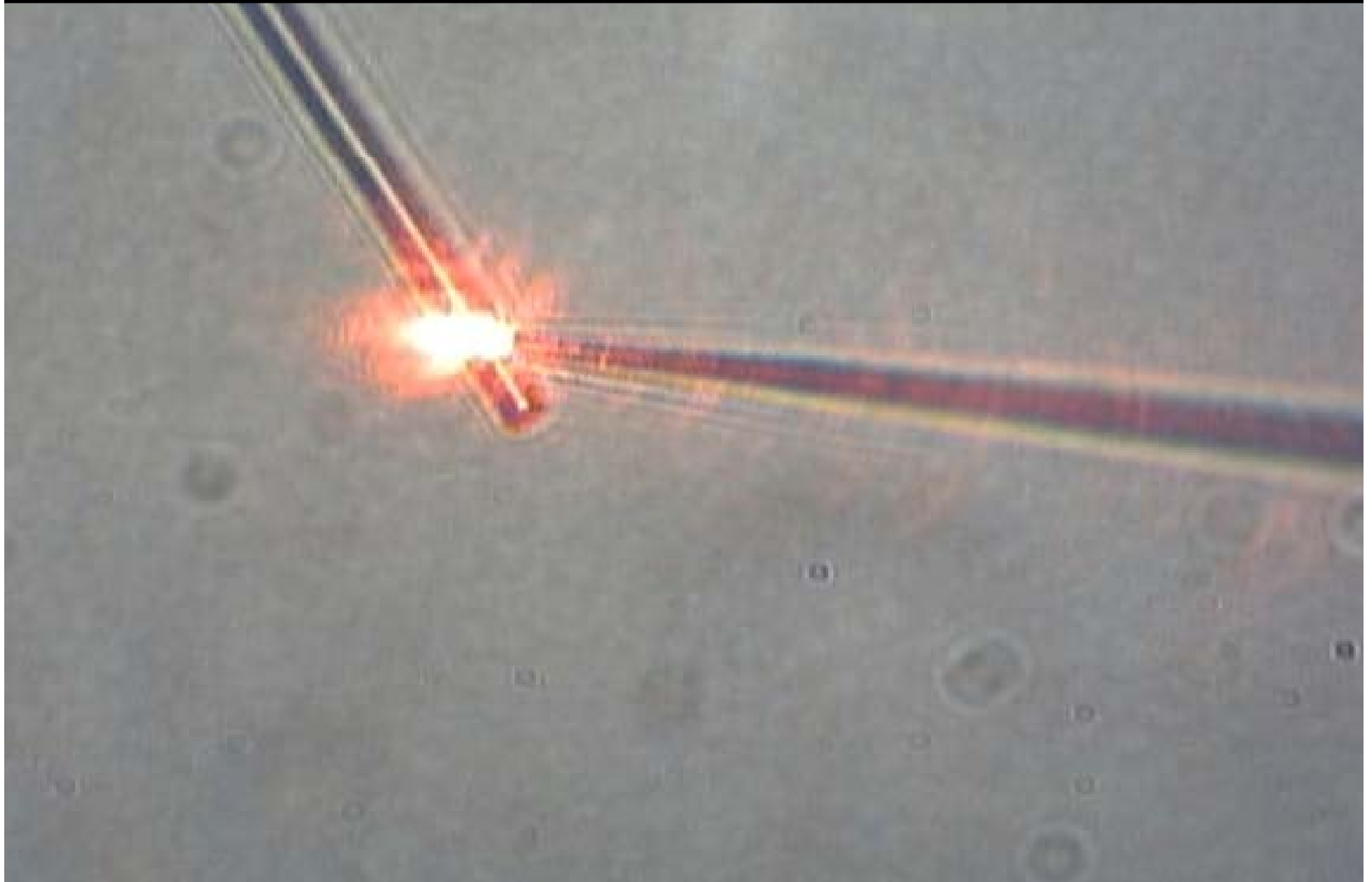
coupling light into nanowires



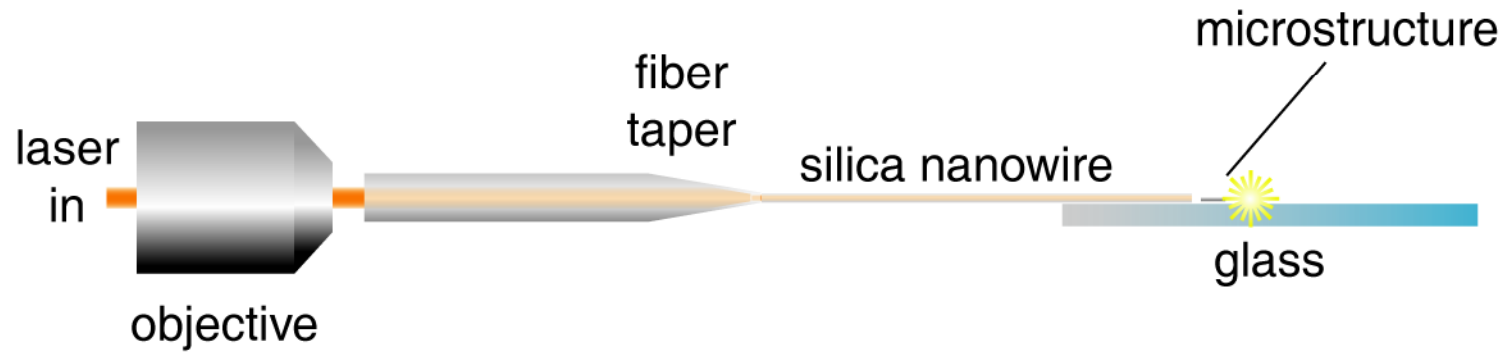
Silica nanowires

Manipulating the nanowires

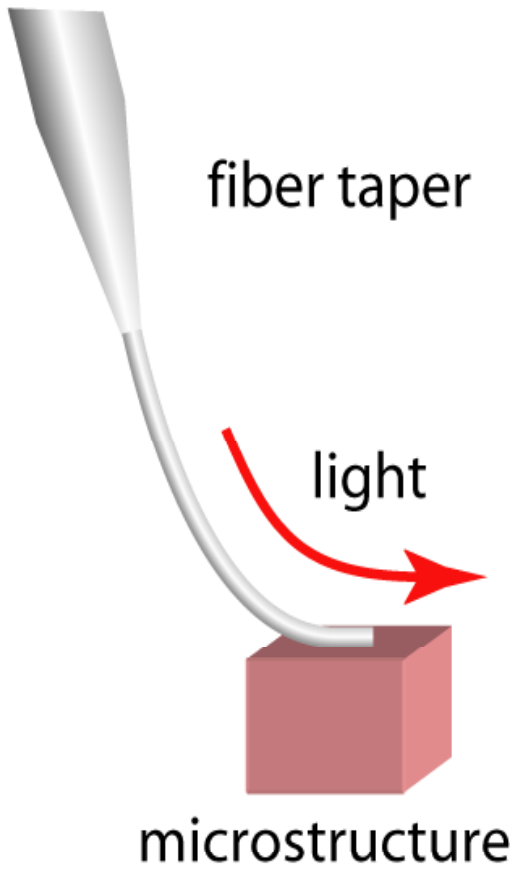
Silica nanowires



Coupling microstructures



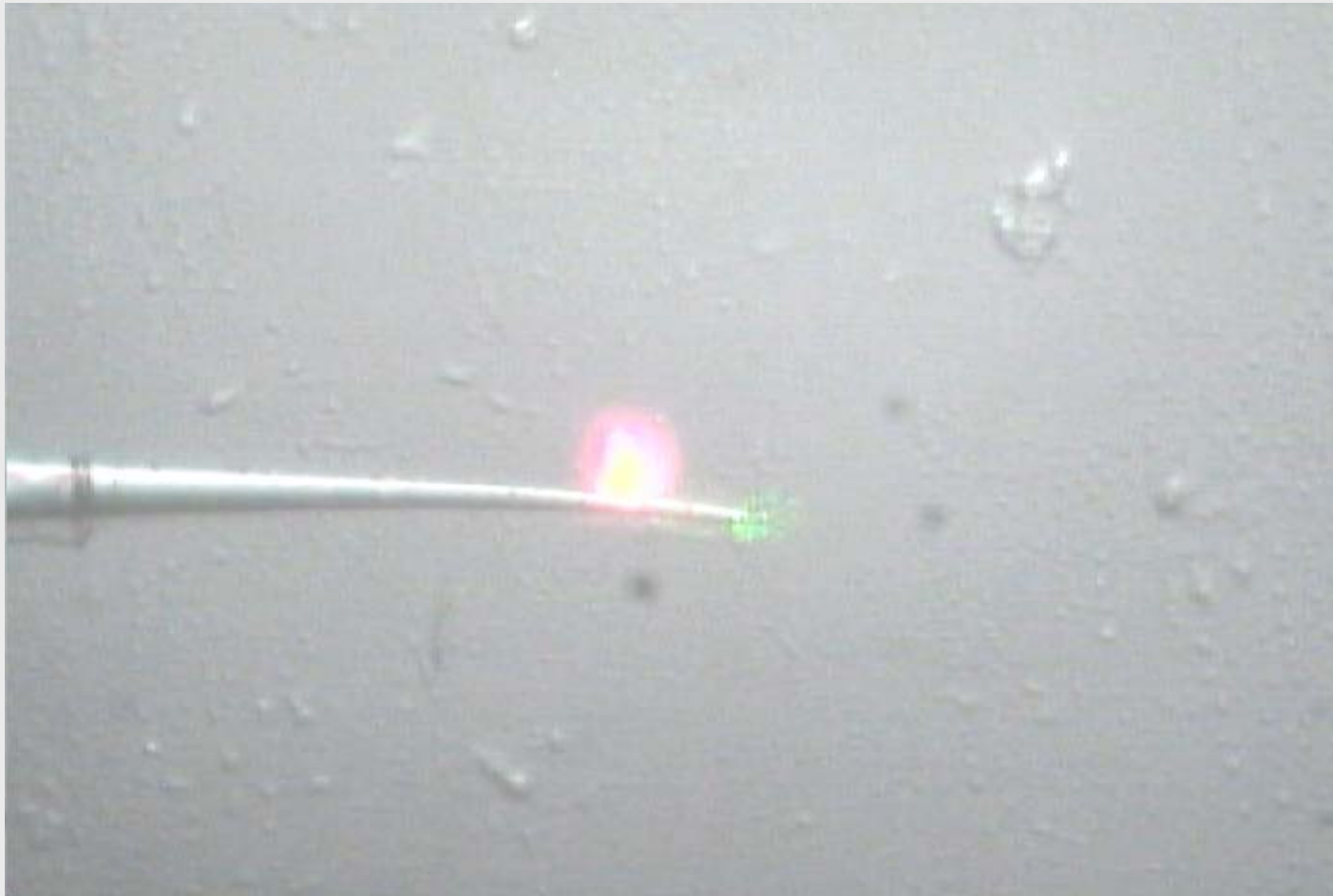
Coupling microstructures



Coupling microstructures



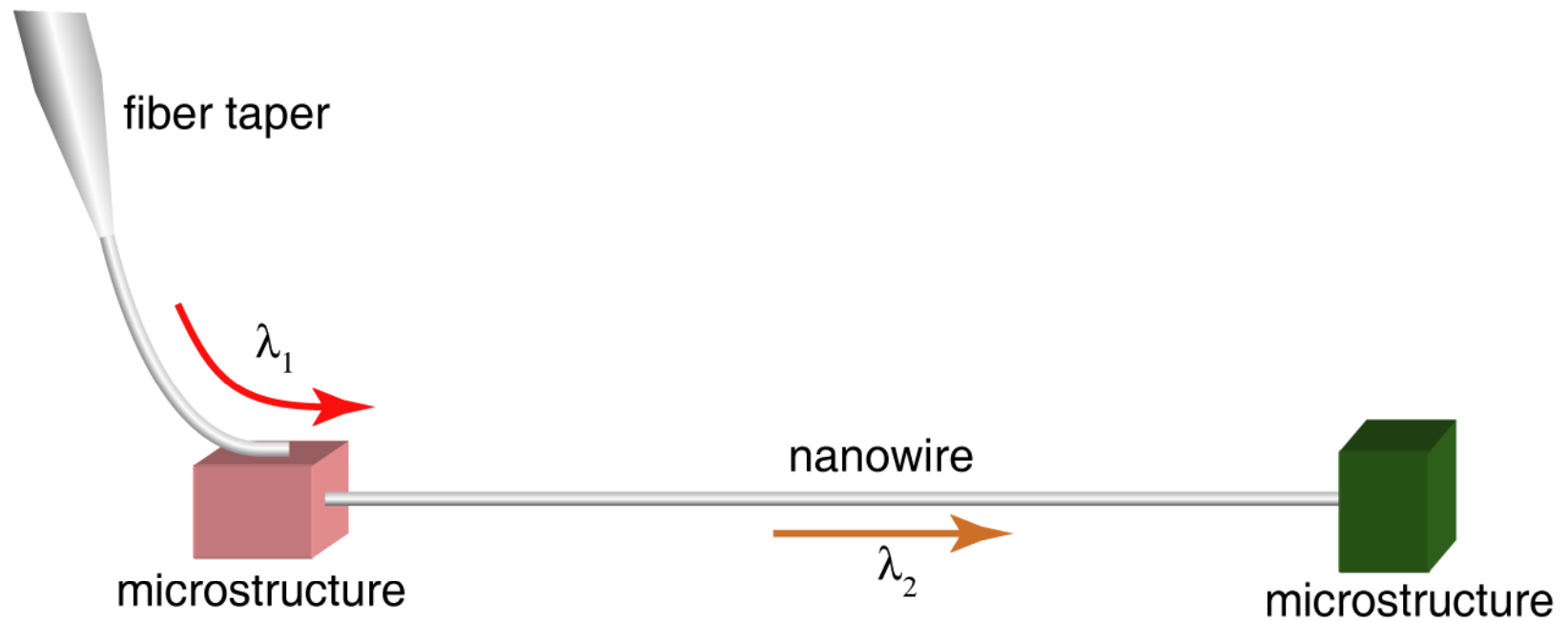
Coupling microstructures



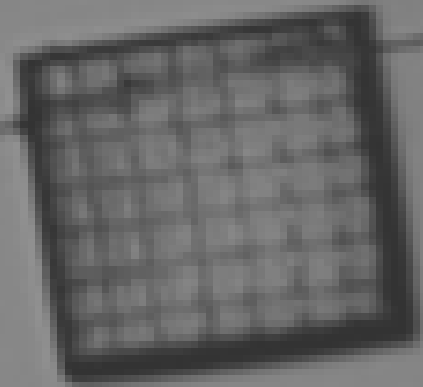
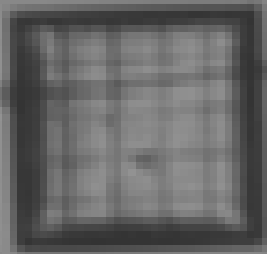
Coupling microstructures



Coupling microstructures



Coupling microstructures



Acknowledgments

Team

Daniel S Correa
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Gustavo Almeida
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Ruben Fonseca
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