

# Manufacturing Nano-optics

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[www.nanophotonics.ch](http://www.nanophotonics.ch)



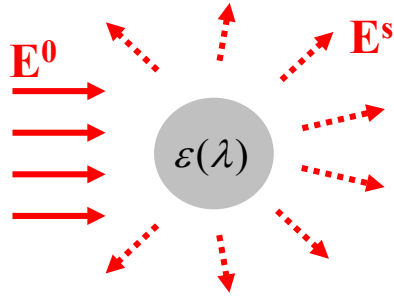
**EPFL**

# Outline

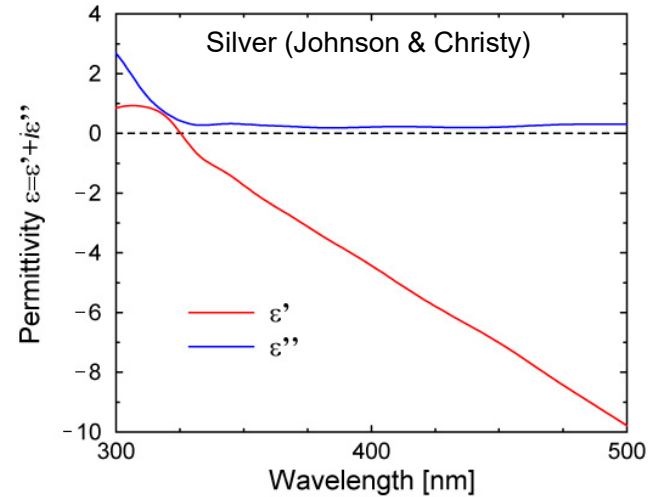
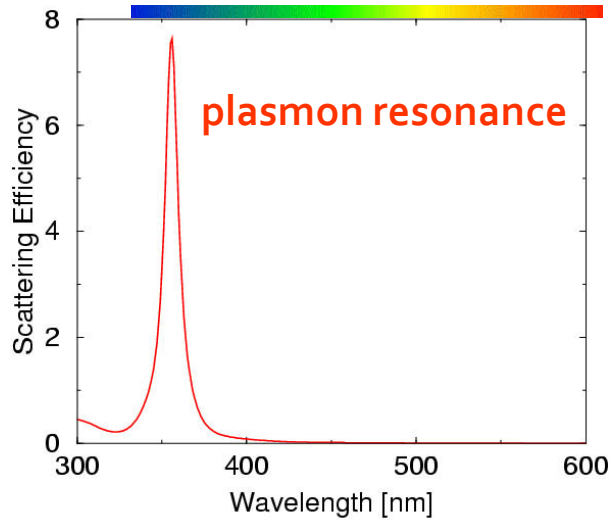
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- Plasmonics
- Basic fabrication principles with electron-beam lithography
- Adhesion issues
- Other metals than Au
- Au-Ag alloys
- Hybrid nanostructures
- Chemical synthesis
- Medieval stained-glass windows

# Mie scattering by a small sphere



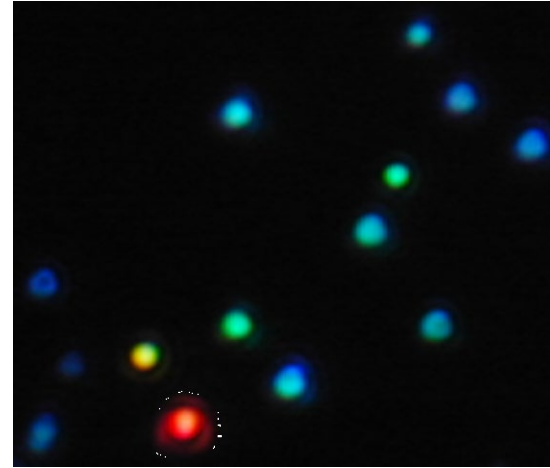
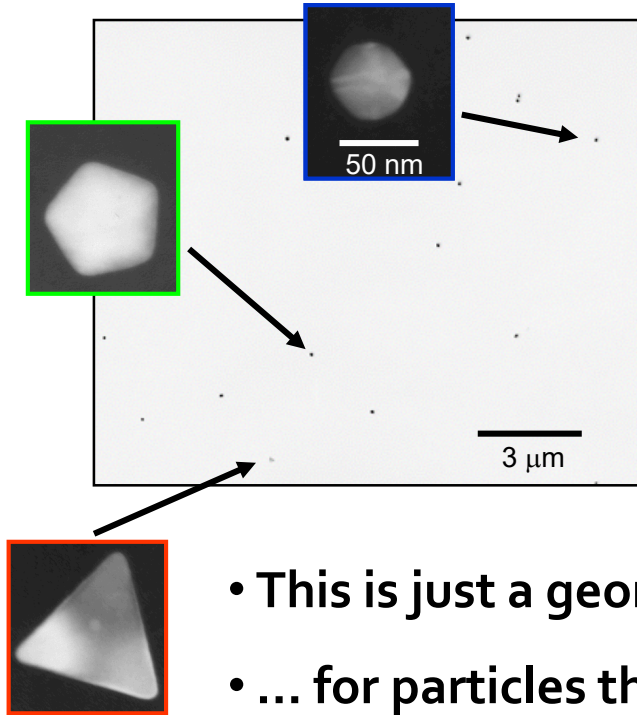
$$Q_{scat}(\lambda) = \frac{8}{3} \left( \frac{2\pi R}{\lambda} \right)^4 \left| \frac{\epsilon(\lambda) - 1}{\epsilon(\lambda) + 2} \right|^2$$



# Plasmon resonances

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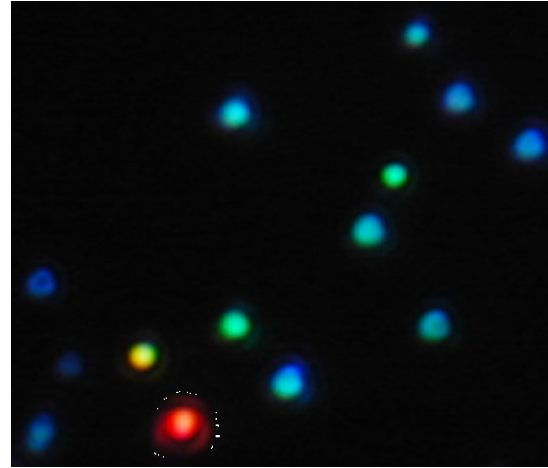
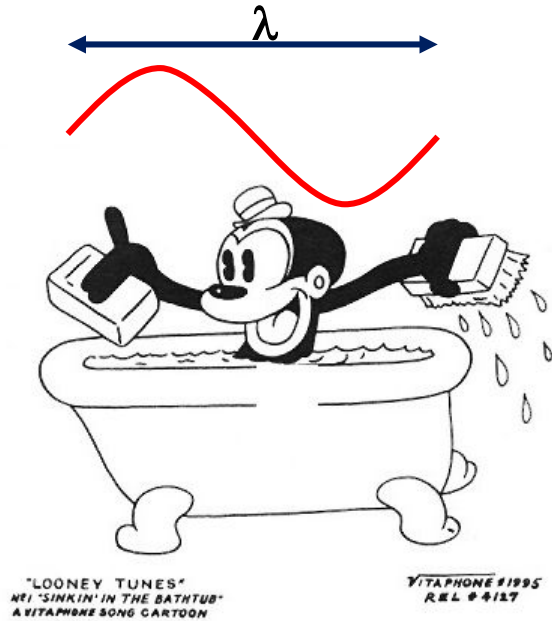
- Same material, similar sizes, but different colors ?



- This is just a geometrical optical resonance effect...
- ... for particles that are  $\ll \lambda$  !

# Plasmon resonances

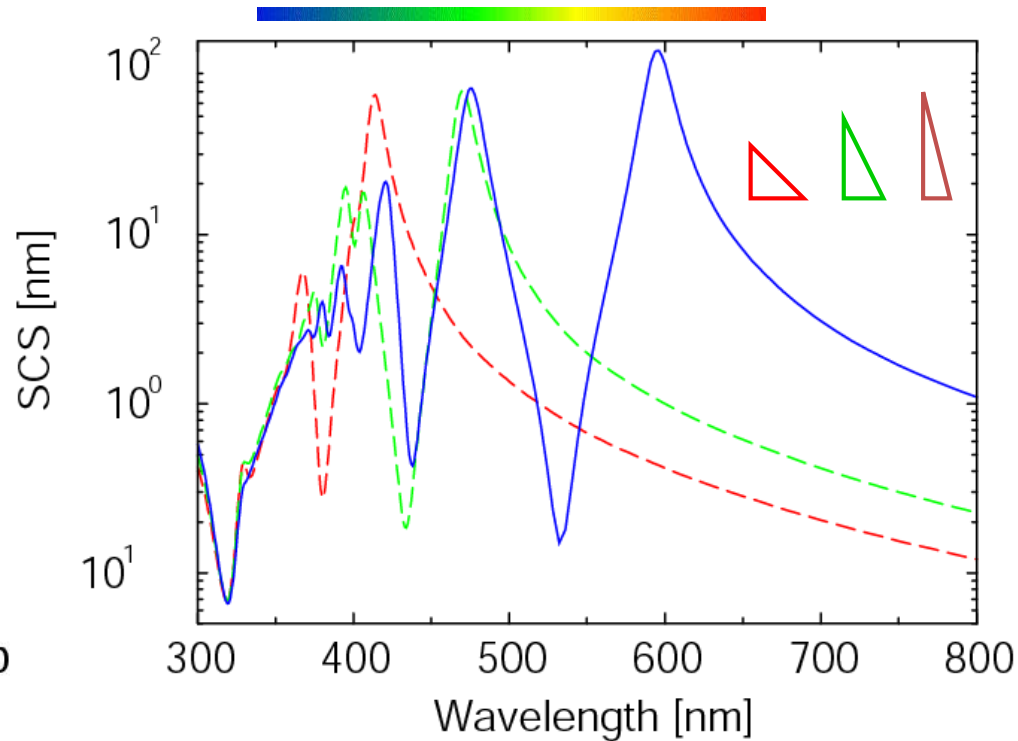
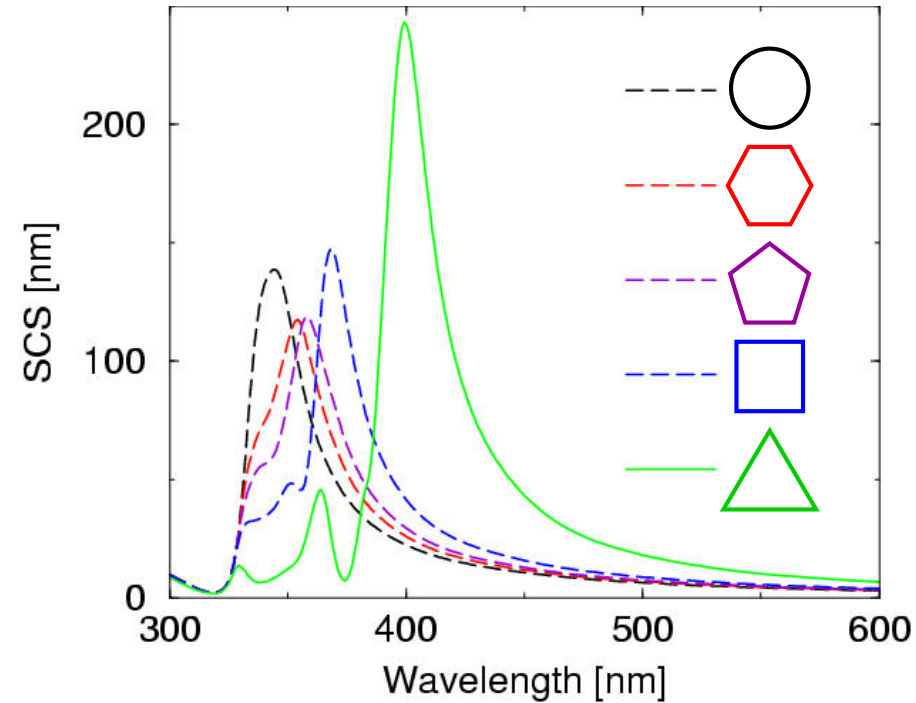
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- This is just a geometrical optical resonance effect...
- ... for particles that are  $\ll \lambda$  !

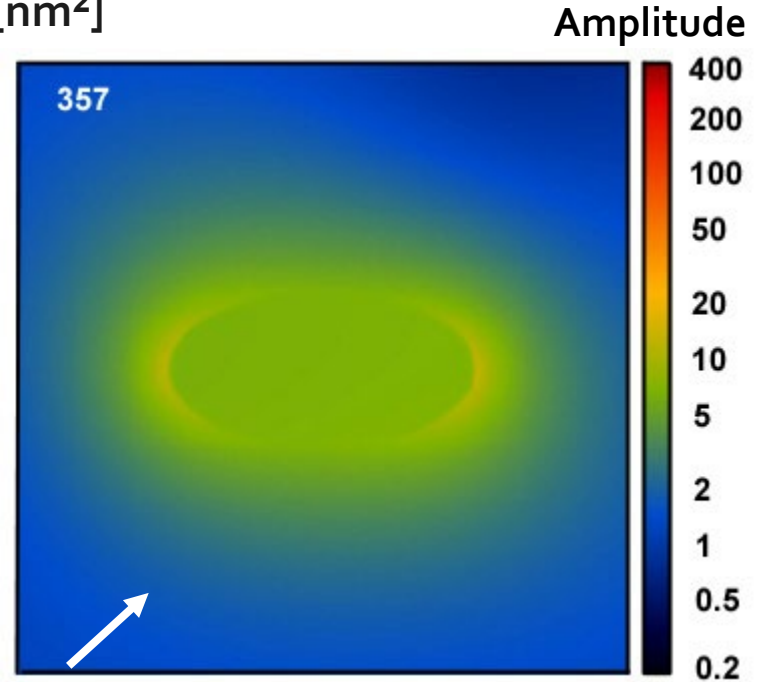
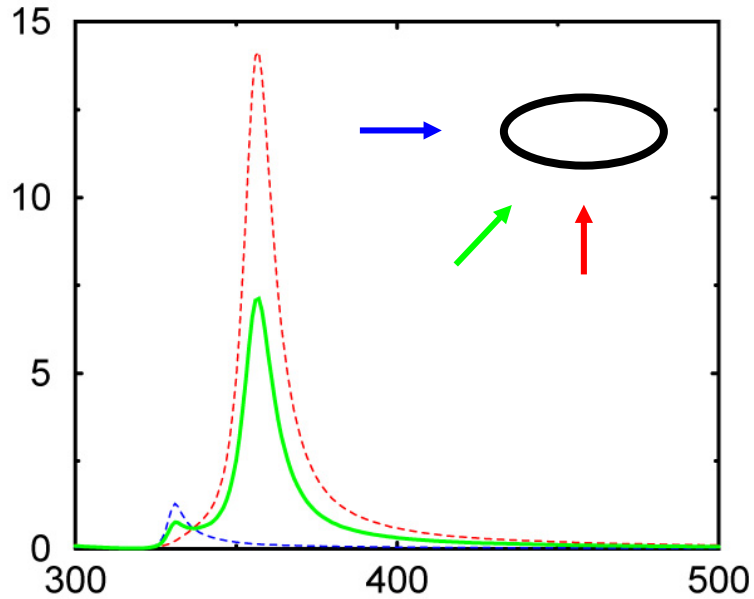
# Plasmonics simplexes

- More complex spectra as the particle symmetry decreases

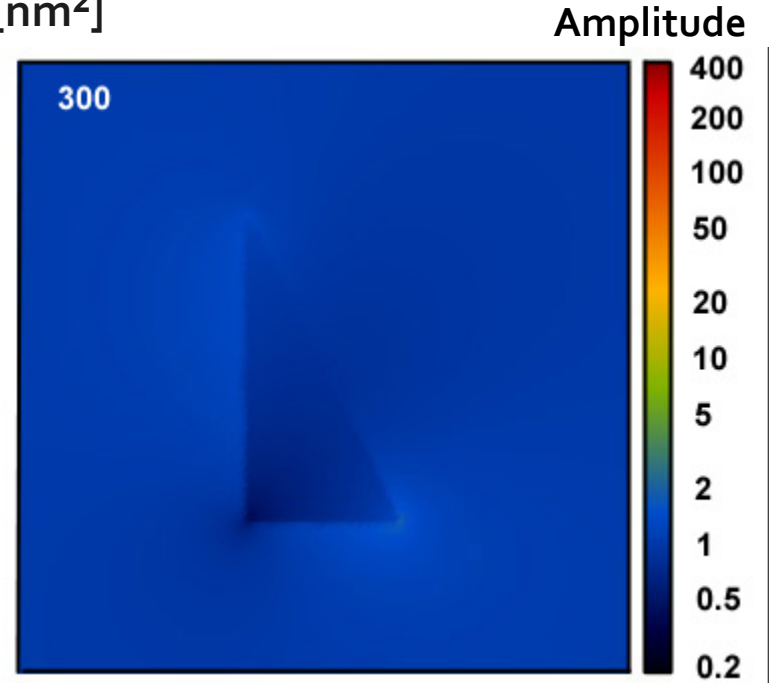
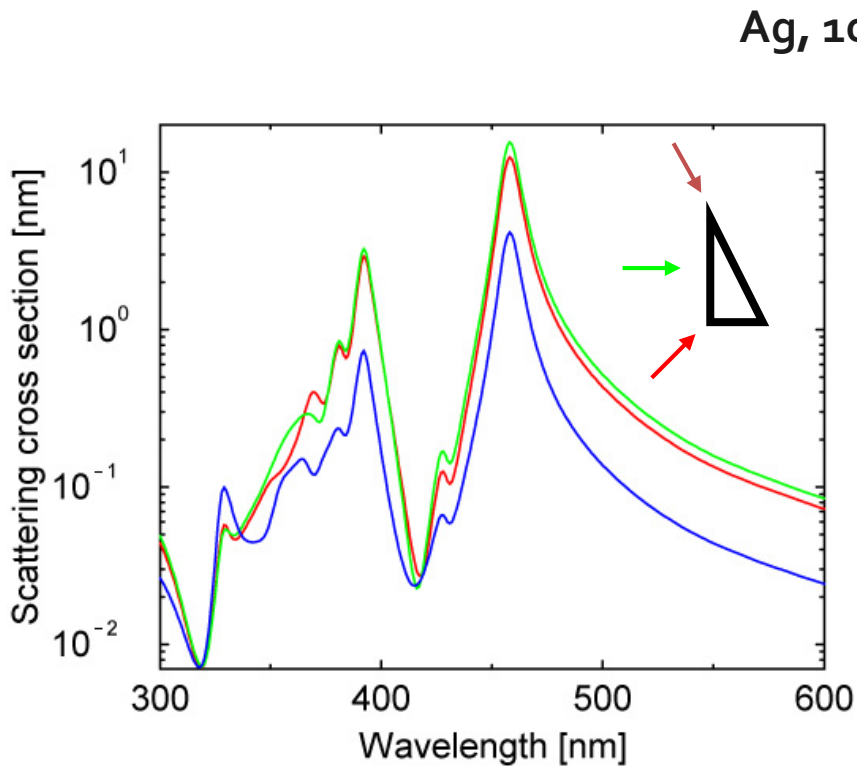


# Plasmon resonances – Elliptical particle

Ag, 20 x 10 [nm<sup>2</sup>]

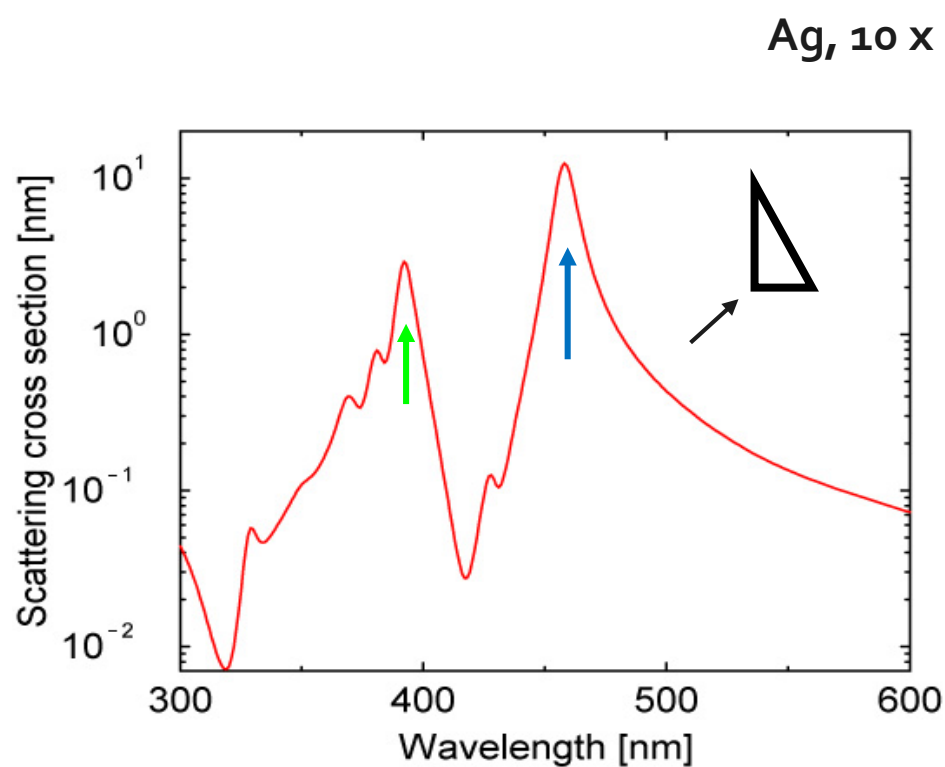


# Plasmon resonances – Triangular particle

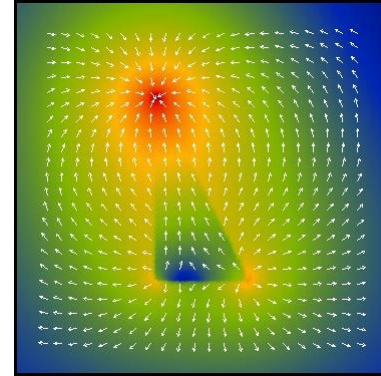


- Dramatic near-field enhancement

# Field distribution at different wavelengths



$\lambda = 458$  [nm]



Amplitude

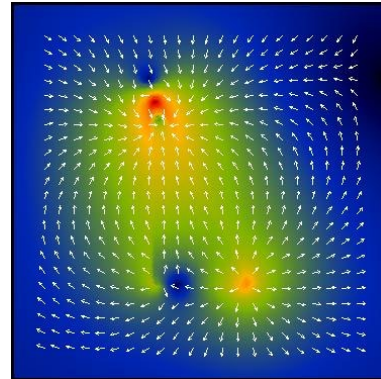
100

10

1

0.1

$\lambda = 392$  [nm]



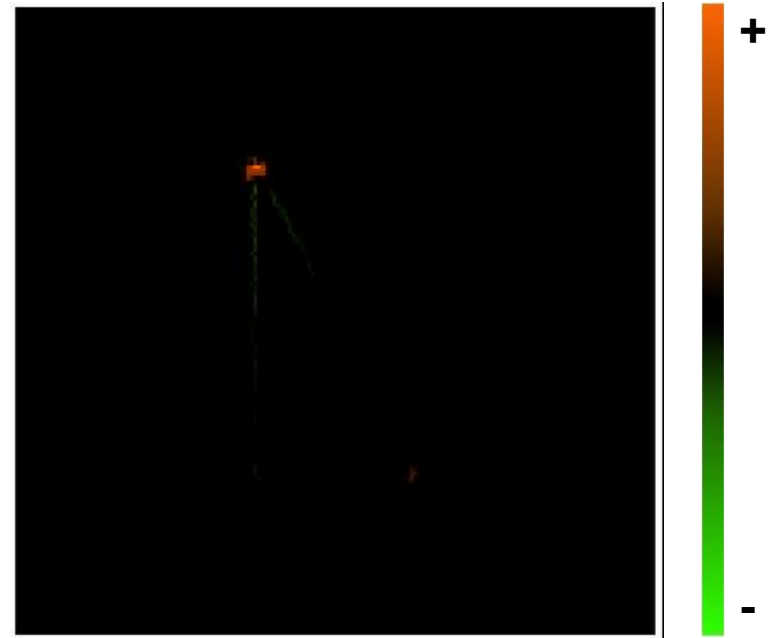
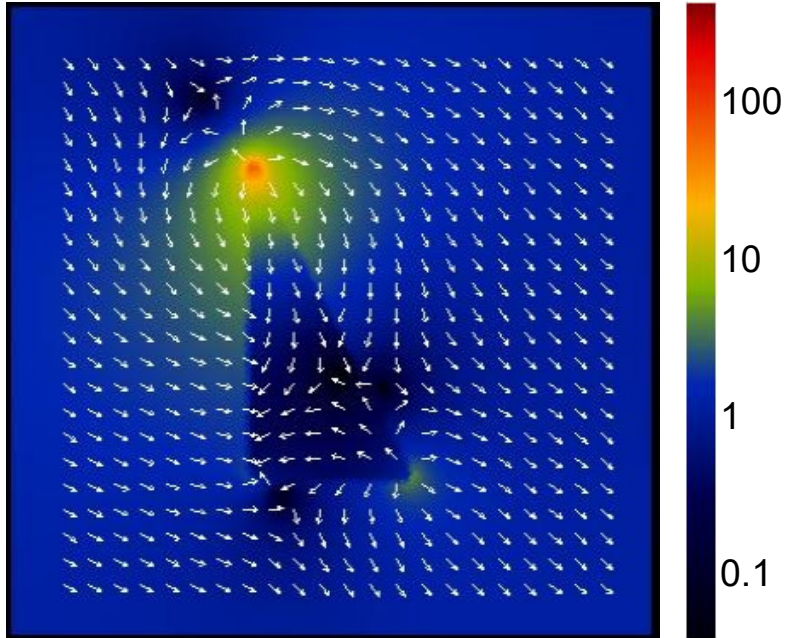
# Field distribution at different wavelengths

$\lambda = 458$  [nm]

Ag, 10 x 20 [nm<sup>2</sup>]

Amplitude

Polarization charge:  $\nabla \cdot \mathbf{E}$



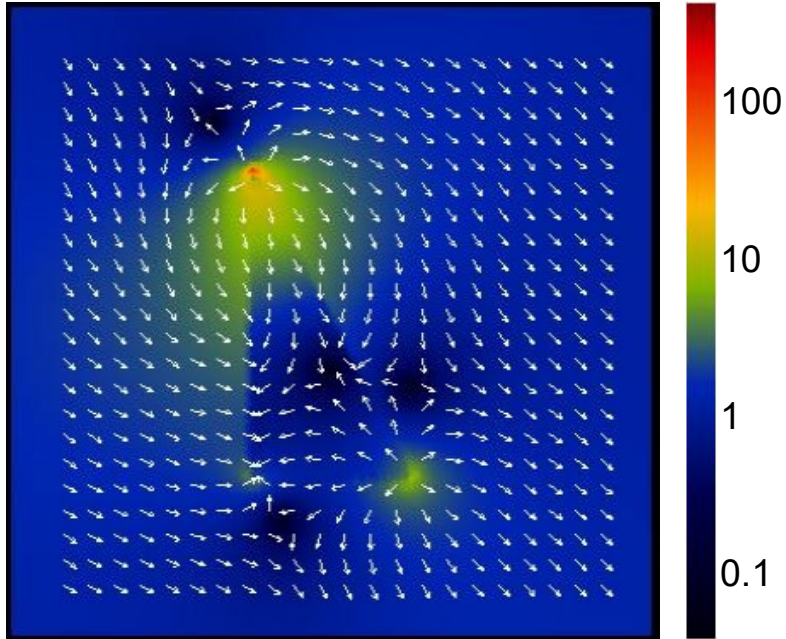
# Field distribution at different wavelengths

$\lambda = 392$  [nm]

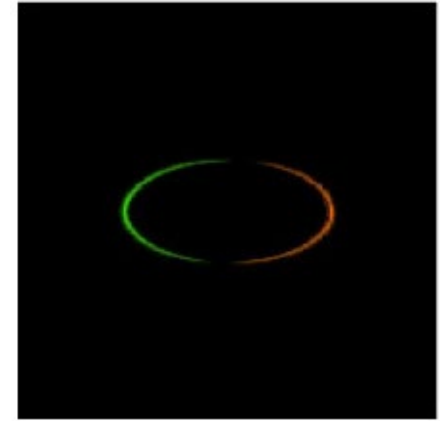
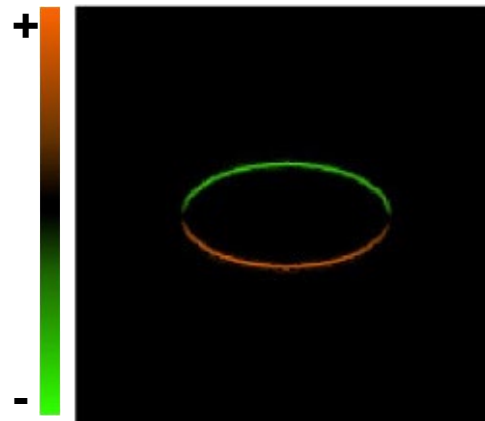
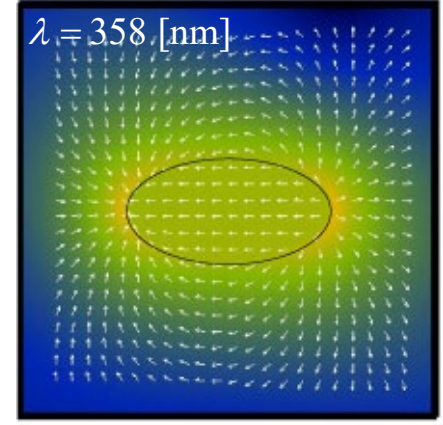
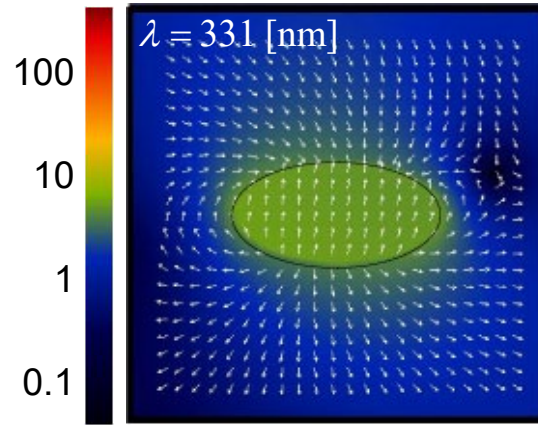
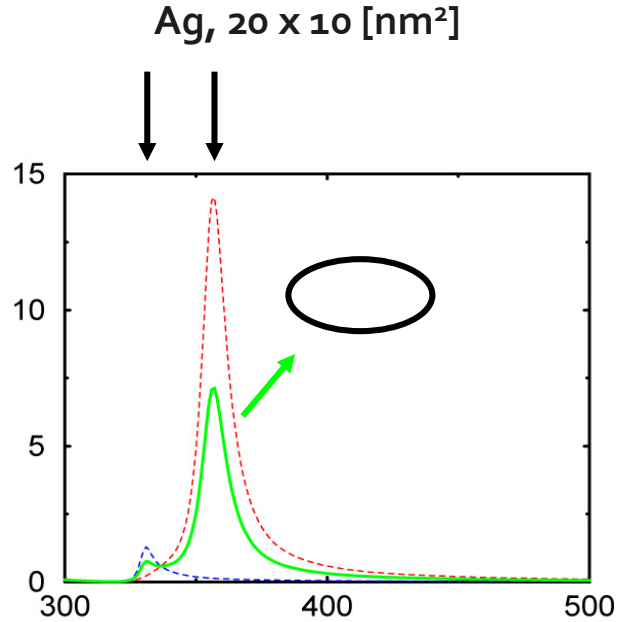
Ag, 10 x 20 [nm<sup>2</sup>]

Amplitude

Polarization charge:  $\nabla \cdot \mathbf{E}$

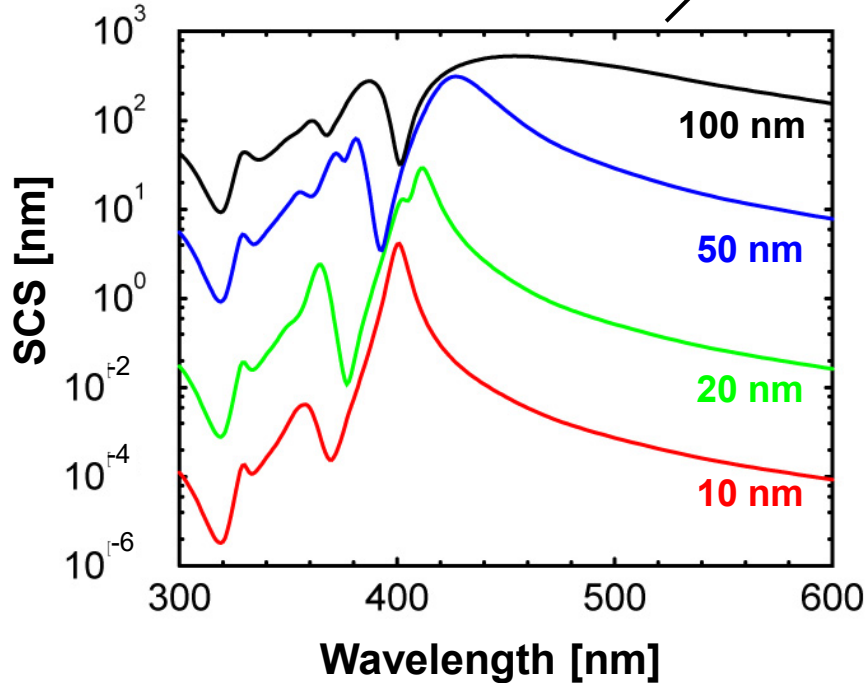
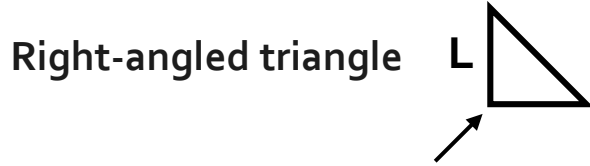


# Elliptical particle



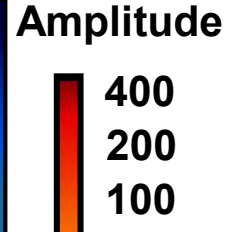
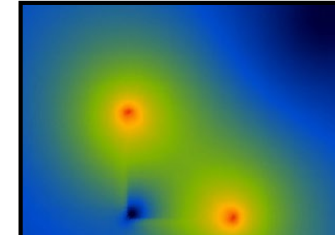
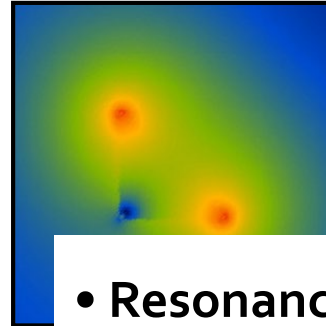
# Size effects

- The maximum enhancement decreases for particles  $> 50$  [nm]



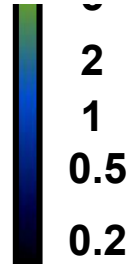
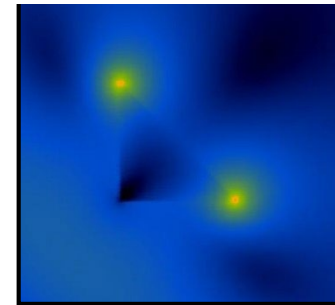
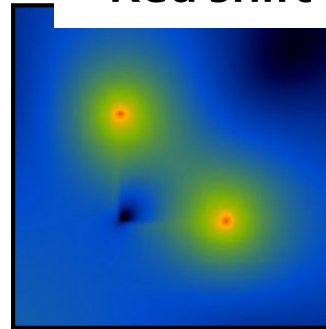
$L = 10$  nm

$L = 20$  nm



- Resonance broadening

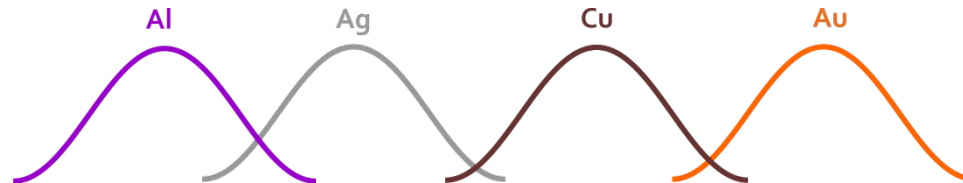
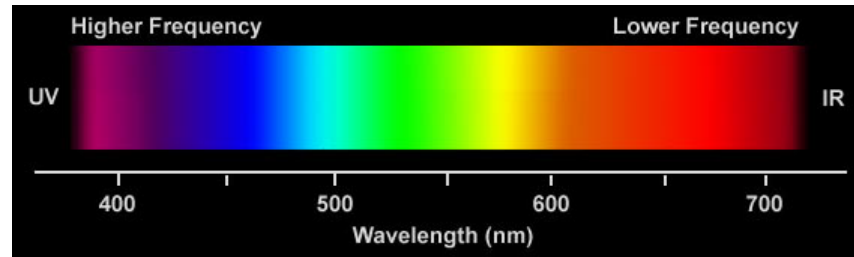
- Red shift



# Plasmonic metals

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- Only a few plasmonic metals are available
- Coinage metals: Au, Ag, Al, Cu, W
- Very high electron density ( $> 10^{22} / \text{cm}^3$ )
- Each metal works in a specific wavelength range determined by the real part of its permittivity

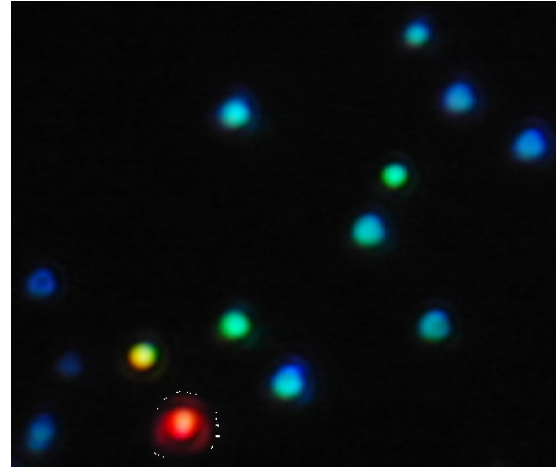


# Plasmon resonances

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- Nanostructures have a completely different optical response than bulk materials

- Silver bar:





## What have we learned so far...

- Plasmon resonances depend on the material and shape of the particle
- Each plasmon resonance is associated with a specific polarization charges distribution
- The field enhancement can be extremely significant
- The lower the symmetry of a particle, the more plasmon resonances and the strongest field enhancement
  
- Constrains for nanofabrication:
  - Sub-100 nm structures
  - Control dimensions down to 10 nm
  - Use appropriate materials



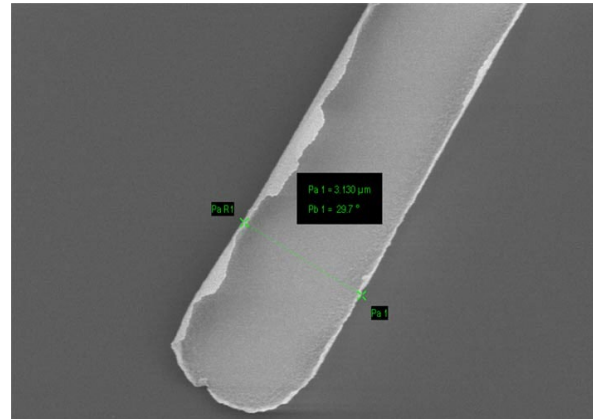
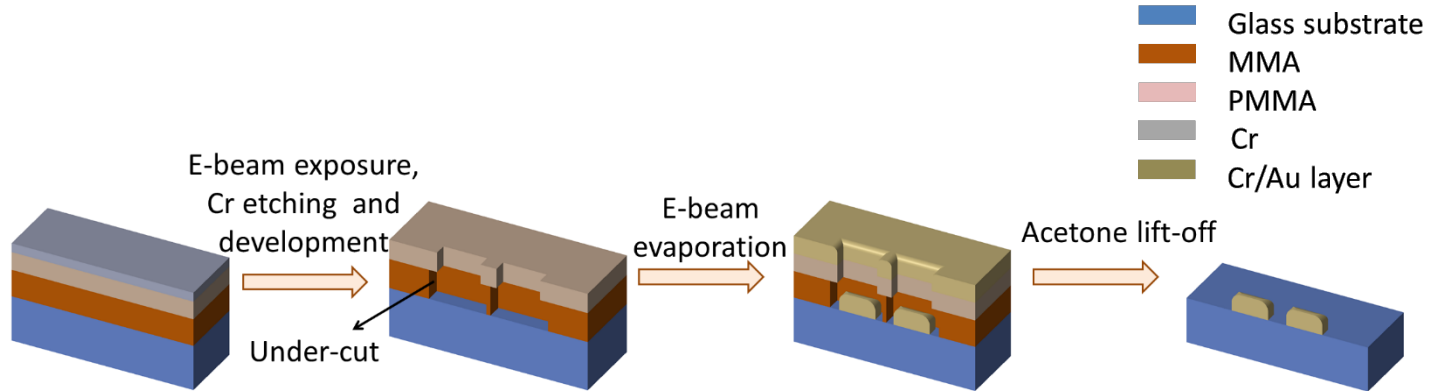
# Outline

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- Plasmonics
- Basic fabrication principles with electron-beam lithography
- Adhesion issues
- Other metals than Au
- Au-Ag alloys
- Hybrid nanostructures
- Chemical synthesis
- Medieval stained-glass windows

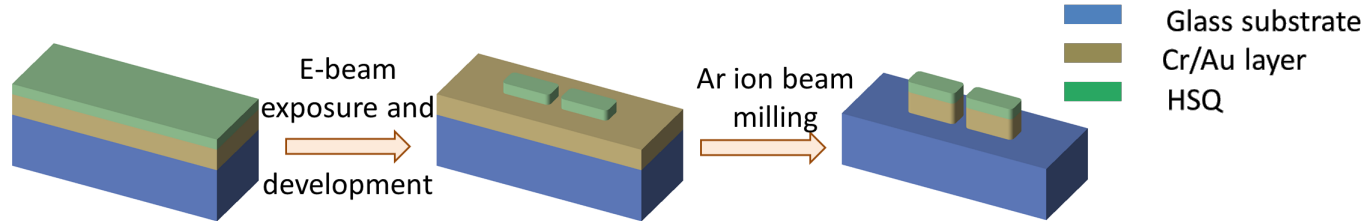
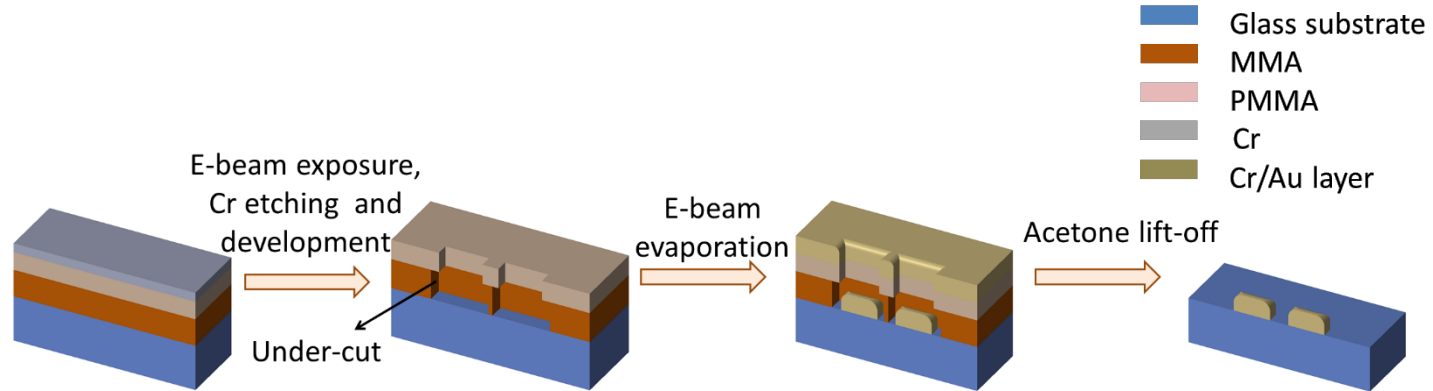
# Nanofabrication of plasmonic structures

- Lift-off (positive resist) vs. ion etching (negative resist)



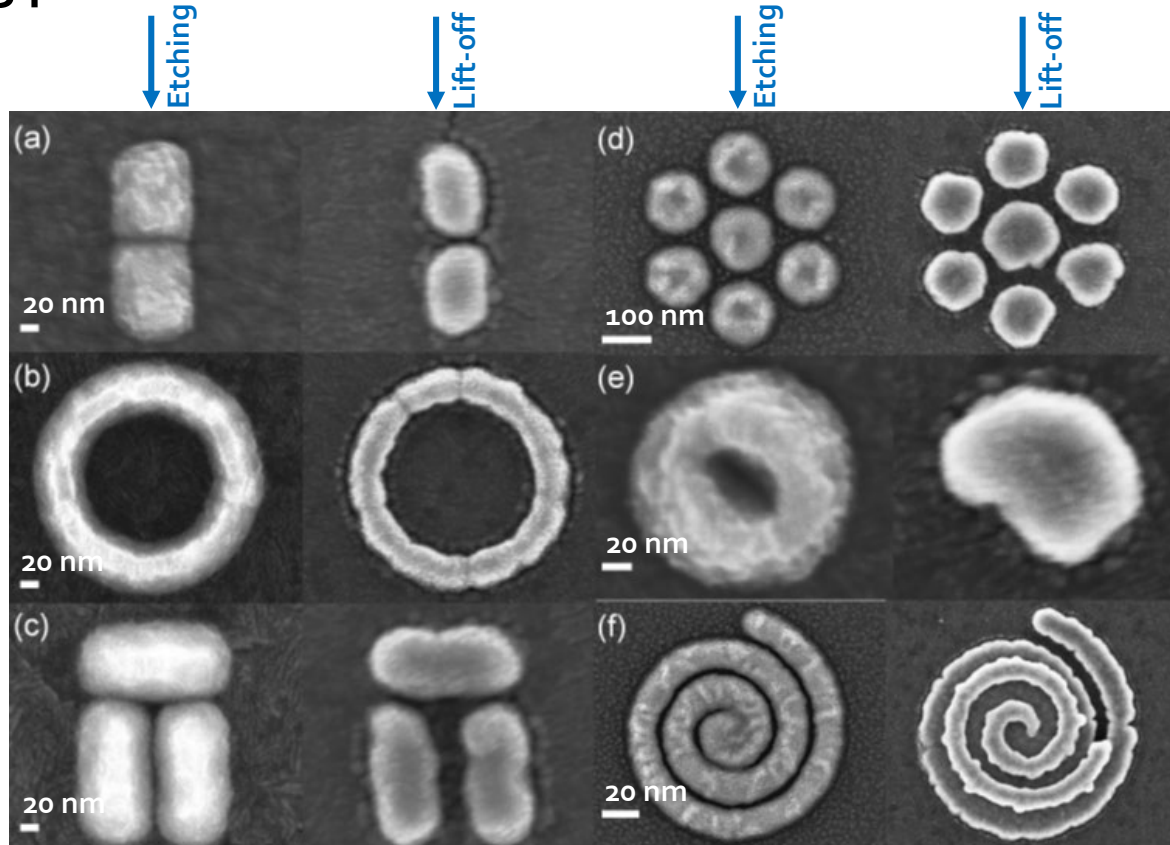
# Nanofabrication of plasmonic structures

- Lift-off (positive resist) vs. ion etching (negative resist)



# Lift-off vs. ion etching (gold nanostructures)

- Ion etching provides smaller features and better control

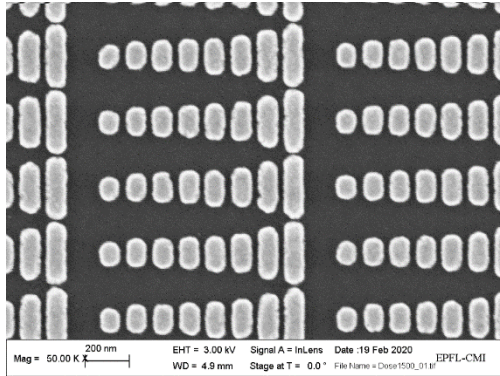


# Dose test

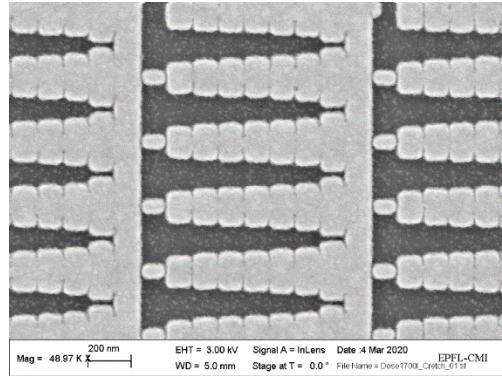
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- Both positive and negative resists require to perform a series of tests with different doses (for each sample geometry)

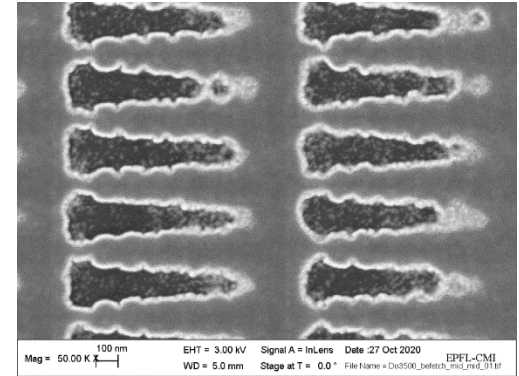
1'500  $\mu\text{C}/\text{cm}^2$



1'700  $\mu\text{C}/\text{cm}^2$



3'500  $\mu\text{C}/\text{cm}^2$



## What have we learned so far...

- Positive (lift-off) and negative (etching) electron beam resists
- Lift-off:
  - Double layer resist to facilitate lift-off
  - Metal diffusion within the mask creates "fuzzy" outlines
  - No residual resist (Oxygen plasma required, though)
- Etching:
  - Good outline definition
  - Very small features, including gaps
  - Residual resist on top of the structure (can be dealt with)
- Some tricky issues related to electron beam exposure (dose, proximity effects, stitching, fracturing,...)



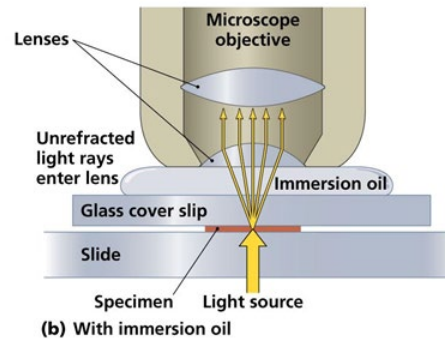
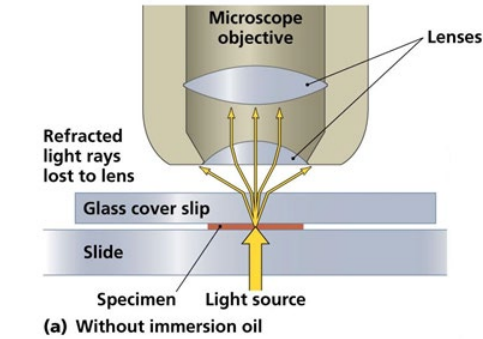
# Outline

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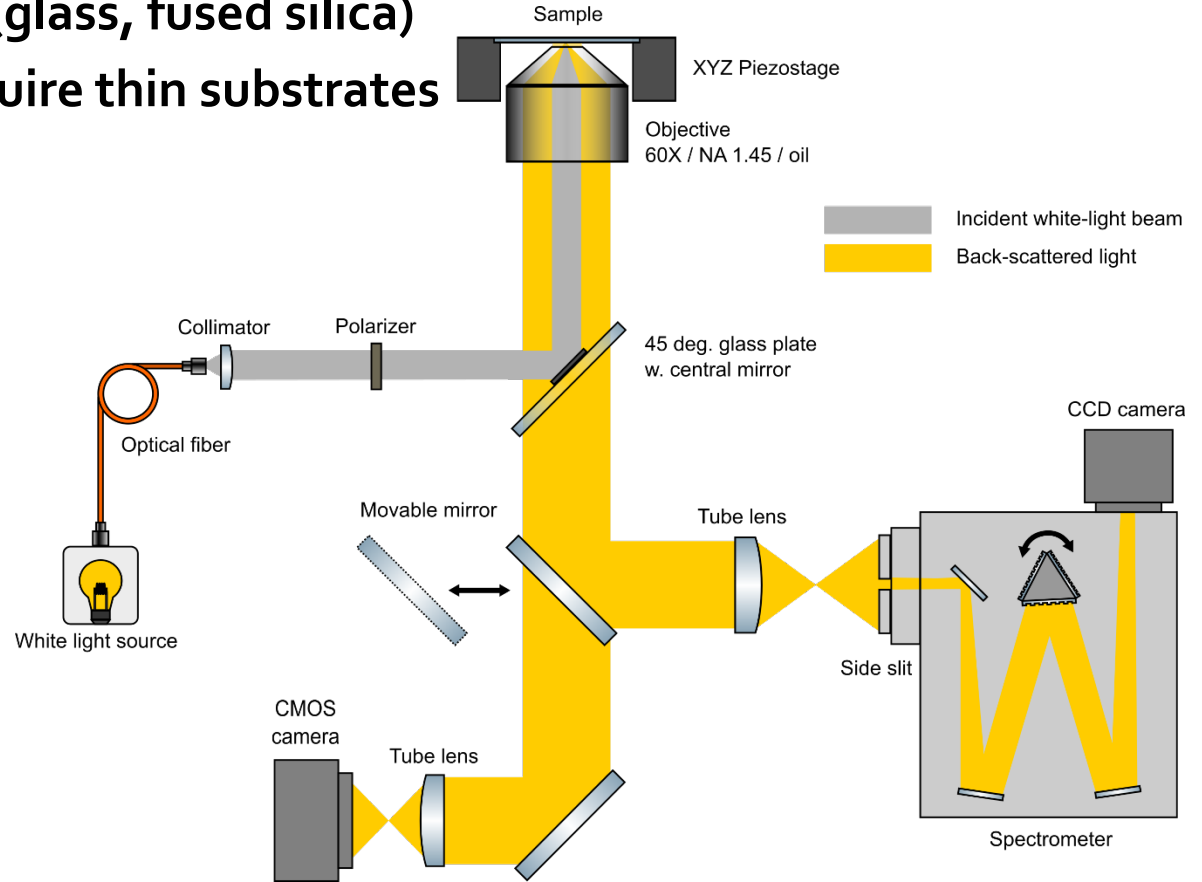
- Plasmonics
- Basic fabrication principles with electron-beam lithography
- **Adhesion issues**
- Other metals than Au
- Au-Ag alloys
- Hybrid nanostructures
- Chemical synthesis
- Medieval stained-glass windows

# Optical measurements

- Transparent substrate (glass, fused silica)
- High NA objectives require thin substrates

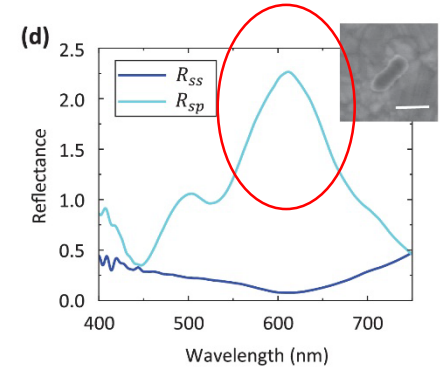
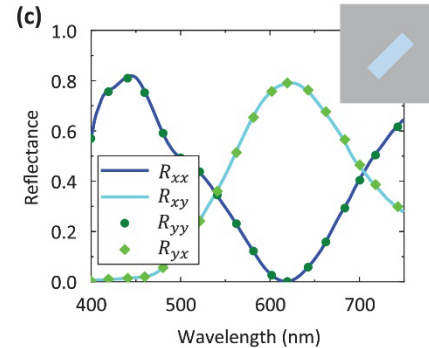
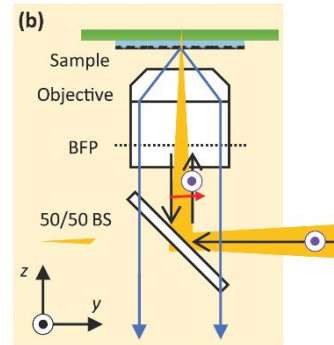
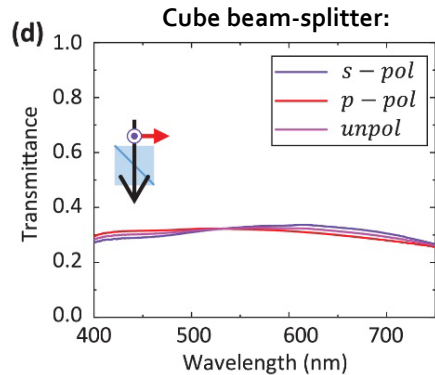
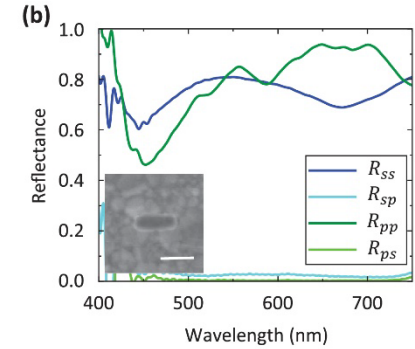
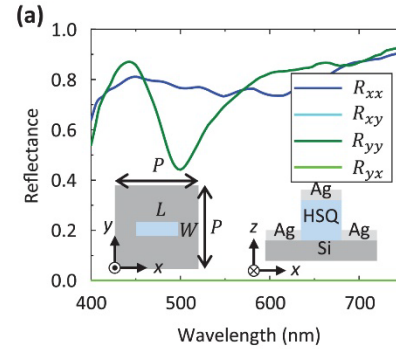
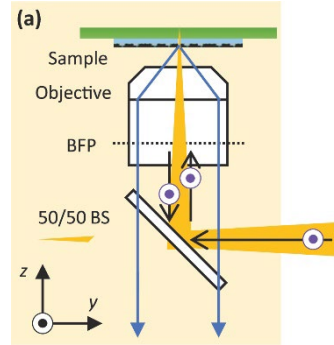
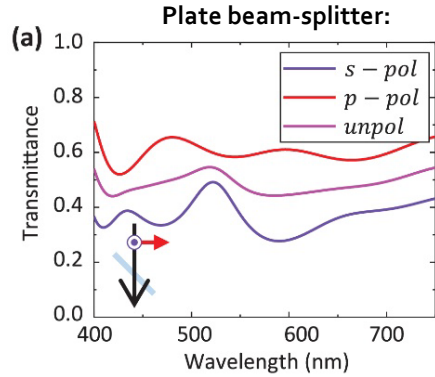


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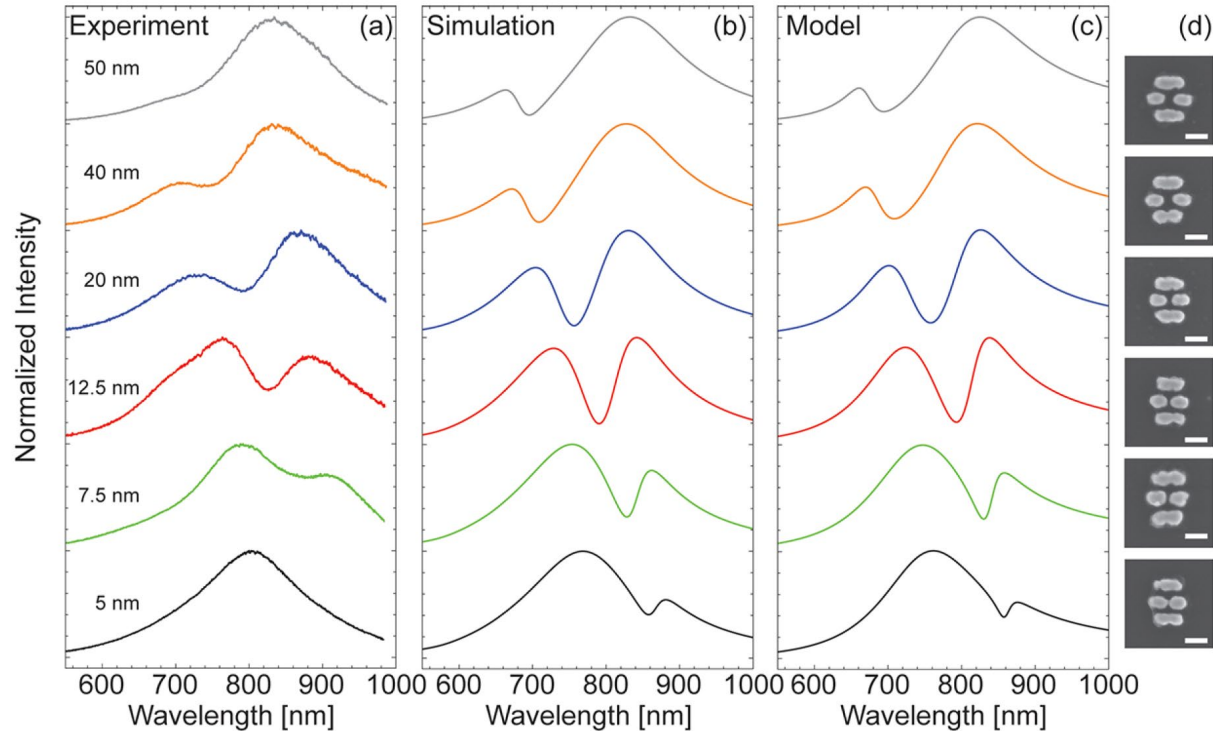
# Careful when measuring samples that alter the polarization

- Some components (e.g. beam-splitters) in a microscope do not transmit/reflect both polarizations in a similar way



# Optical measurements

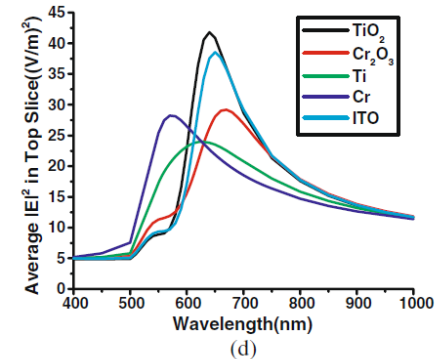
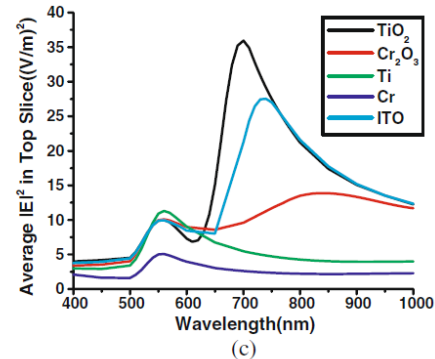
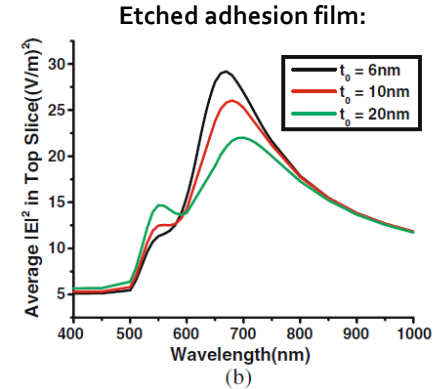
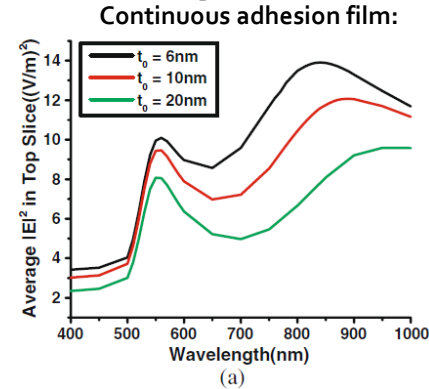
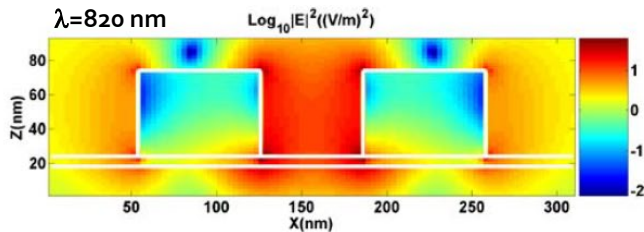
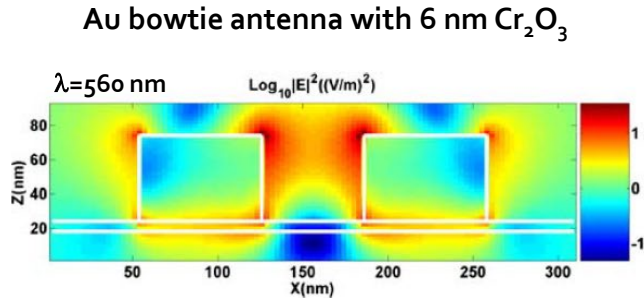
- Spectroscopy of individual nanostructures



- Gold nanostructures
- Central gap 5...50 nm
- Scalebar 100 nm

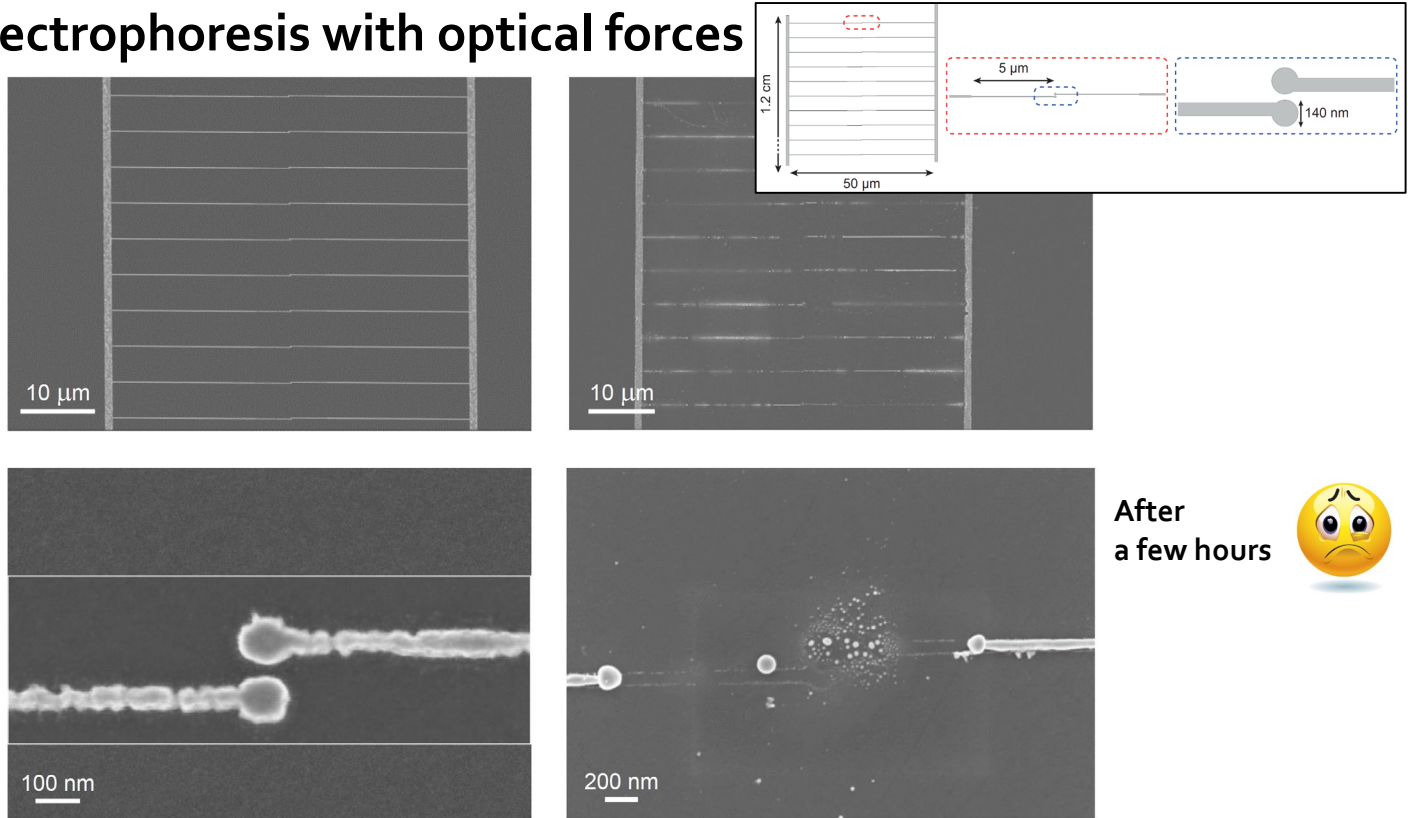
# Metal adhesion on SiO<sub>2</sub> is poor

- Usually, one resorts to an extremely thin inorganic adhesion layer: Cr, Ti
- Unfortunately, these layers can disturb the plasmon resonances



# Metal adhesion on SiO<sub>2</sub> is poor

- Fabrication with Cr adhesion of very high aspect ratio Au electrodes to combine dielectrophoresis with optical forces



# At the nanoscale, materials behave very differently

- When a gold particle shrinks, it becomes liquid!

## Size effect on the melting temperature of gold particles\*

Ph. Buffat and J-P. Borel

Laboratoire de Physique Expérimentale, Ecole Polytechnique Fédérale-Lausanne, Lausanne, Switzerland  
(Received 2 December 1975)

Recently, small particles have been shown to exhibit a melting temperature which depends on the particle size. The various possible experimental methods have been compared and measurements of the melting points of small gold particles have been made using a scanning electron-diffraction technique. This method was applied to particles having diameters down to 20 Å. Consideration of the size distribution over an entire sample makes it necessary to carry out a careful analysis of the experimental results in order to deduce the melting temperature of particles having a well-defined diameter. The experimental results are quantitatively in good agreement with two phenomenological models. The first model describes the equilibrium condition for a system formed by a solid particle, a liquid particle having the same mass, and their saturating vapor phase. The second model assumes the preexistence of a liquid layer surrounding the solid particle and describes the equilibrium of such a system in the presence of the vapor phase. In order to permit a better comparison between both models, a new expression for the thermodynamic equilibrium condition has been derived in the present work. In the case of the first model, the agreement was obtained using only the physical constants of massive gold. In applying the second model, however, one is compelled to assume the existence of a liquid layer having a thickness of about 6 Å.

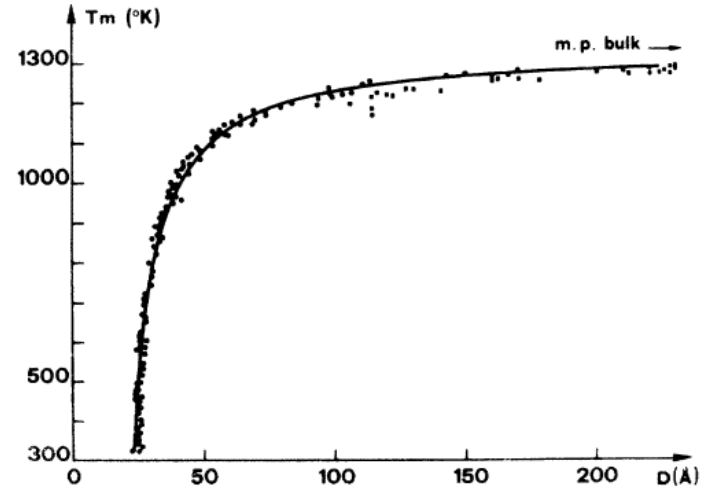
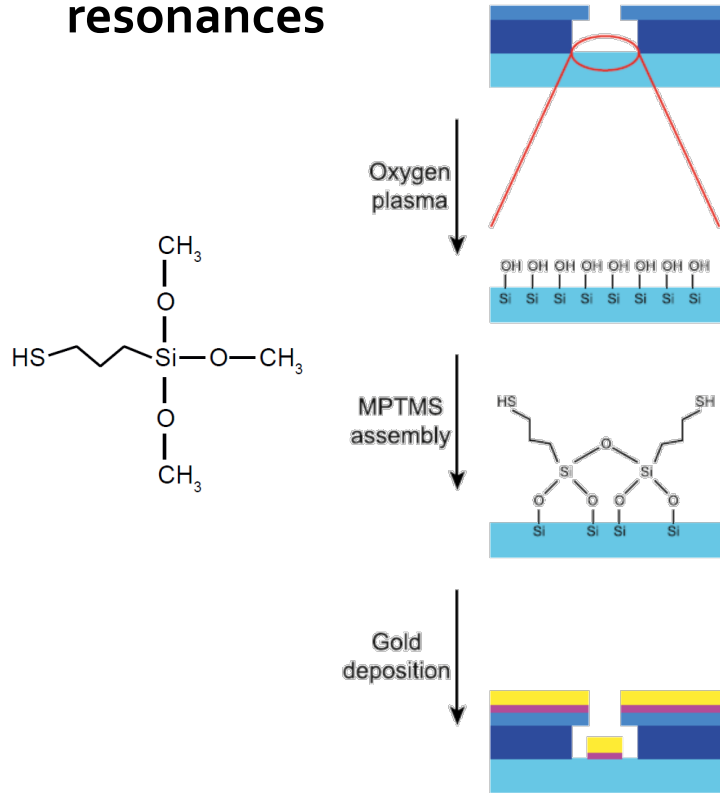


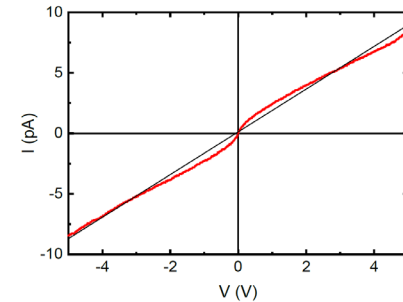
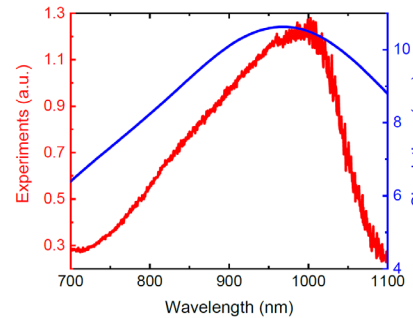
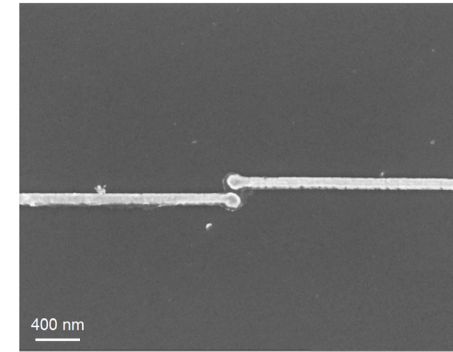
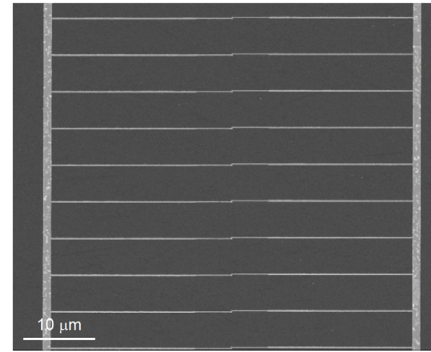
FIG. 6. Experimental and theoretical values of the melting-point temperature of gold particles, assuming validity of the first model in order to calculate the diffracted intensities and obtain the Debye-Waller factors: circles, present work; squares, Sambles (Ref. 28); the solid line results from a least-squares fit to Eq. (13) using all of the experimental points.

# Metal adhesion on SiO<sub>2</sub> is poor

- An organic adhesion layer can work better, without disturbing the optical resonances



Robust structures even after several weeks



# Cooking... disasters

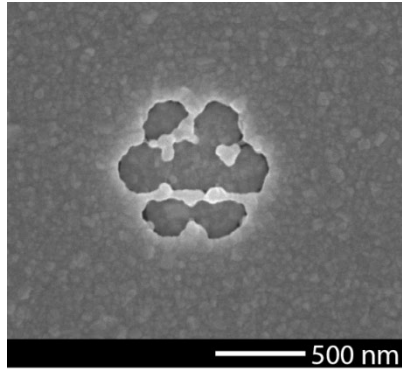
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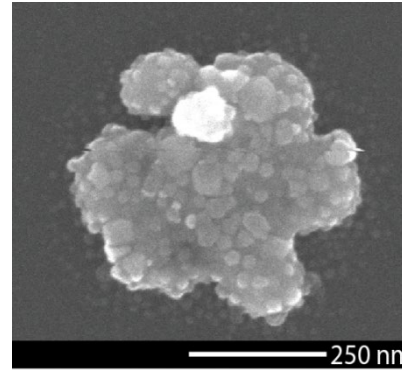
# Cooking... disasters with silver

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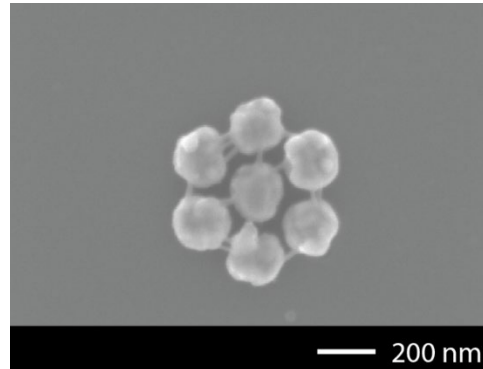
Poor lift-off



Large grains



Bridges between neighboring elements

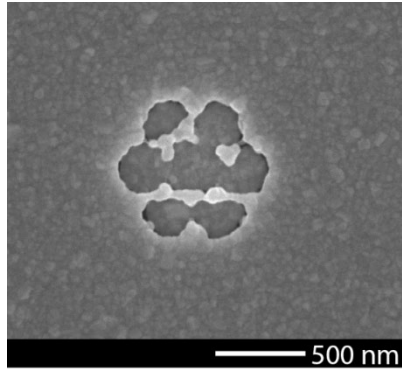


## Fabrication difficulties associated with silver

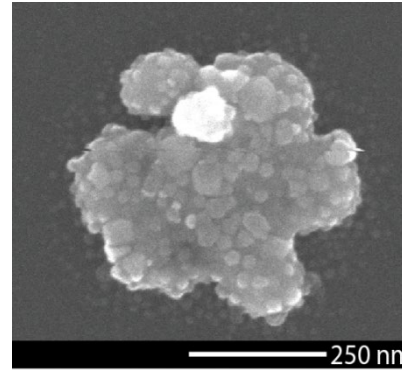
- Poor surface wetting of silver, leading to early nucleation and large grains
- Formation of silver oxide (reversible process at low O<sub>2</sub> pressure)
- Dissociation of Ag<sub>2</sub>O takes place to establish equilibrium
- Activated layer energetically favourable for incoming Ag atoms to produce metallic Ag
- A very thin Ag<sub>2</sub>O layer provides such an activated substrate

# Our first attempts at Ag nanostructures...

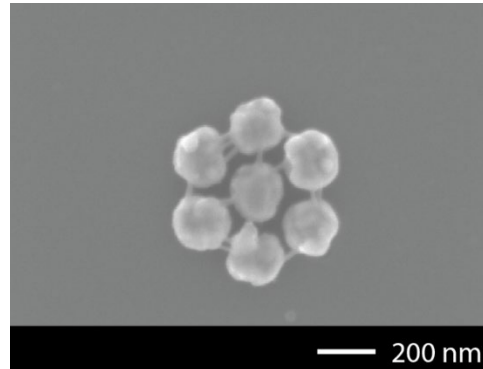
Poor lift-off



Large grains

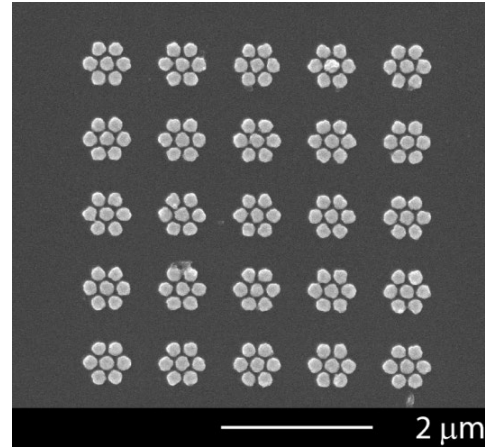
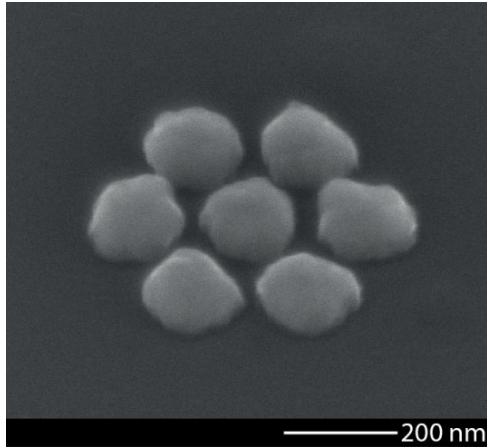


Bridges between neighboring elements



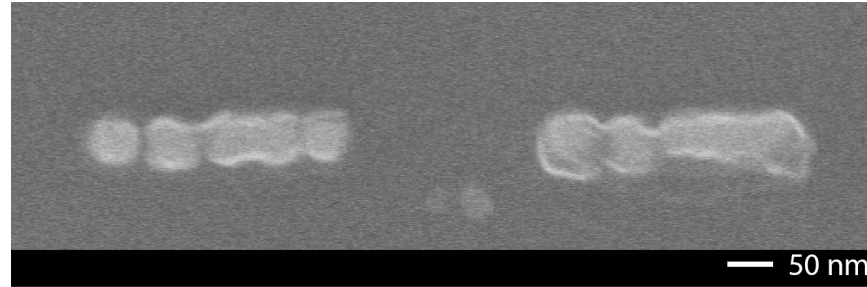
... and then we got better at it !

- Deposition of a very thin  $\text{Ag}_2\text{O}$  layer on the substrate enables the fabrication of Ag nanostructures with well-controlled dimensions, including for the gaps down to 10 nm

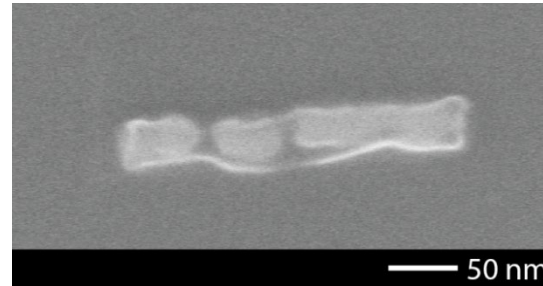


# Aluminum is not easy either...

Poor lift-off

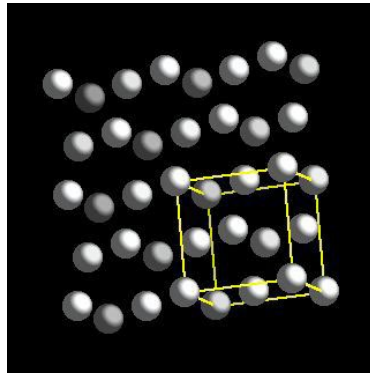


Large grains, bridges between neighboring elements

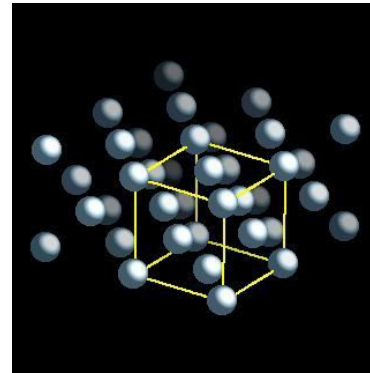


## Aluminum is not easy either...

- Poor surface wetting of aluminum → early nucleation and large grains
- Formation of aluminum oxide (non-reversible process)
- The same trick cannot be used with a thin  $\text{Al}_2\text{O}_3$  layer...
- However, Al and Ag have a very similar crystalline structure (cubic close-packed)
- We can use again silver oxide as activation layer



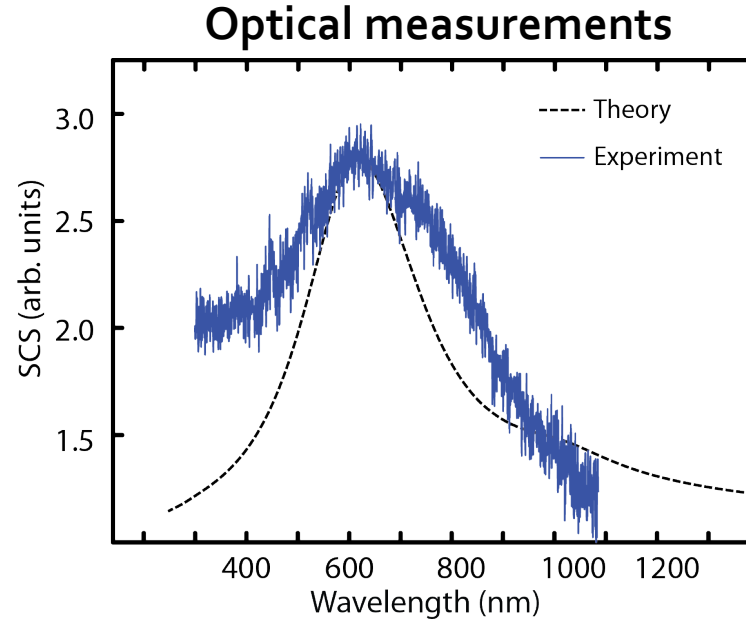
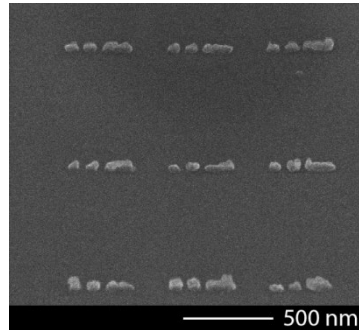
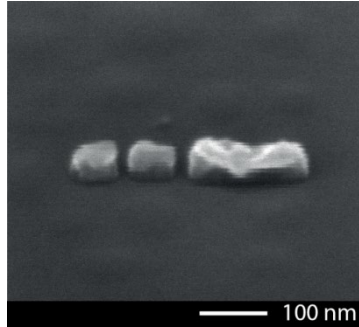
Silver 4.08 Å



Aluminum 4.04 Å

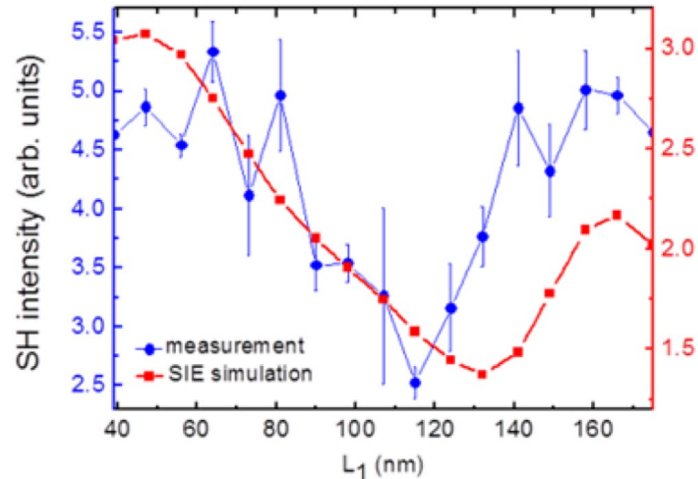
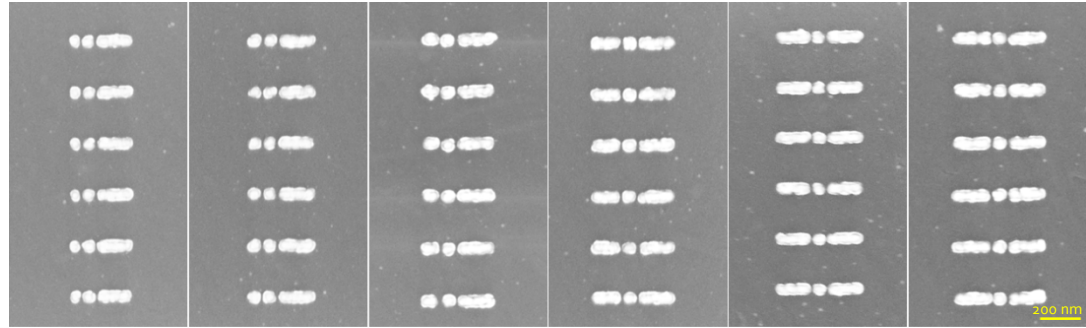
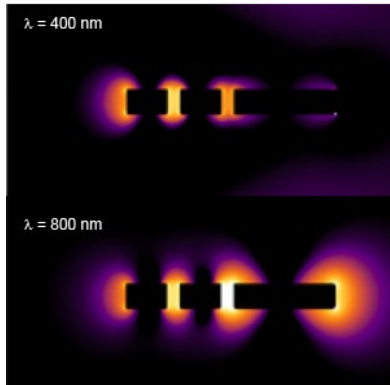
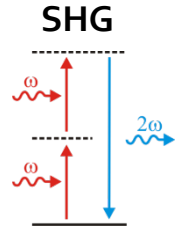
## Improved recipe for aluminum

- Fabrication of double resonant Al antennas, including small gaps and vertical sidewalls



# Double-resonant optical antenna in AI for SHG

- The second harmonic generation (SHG) strongly depends on the material and on the geometry

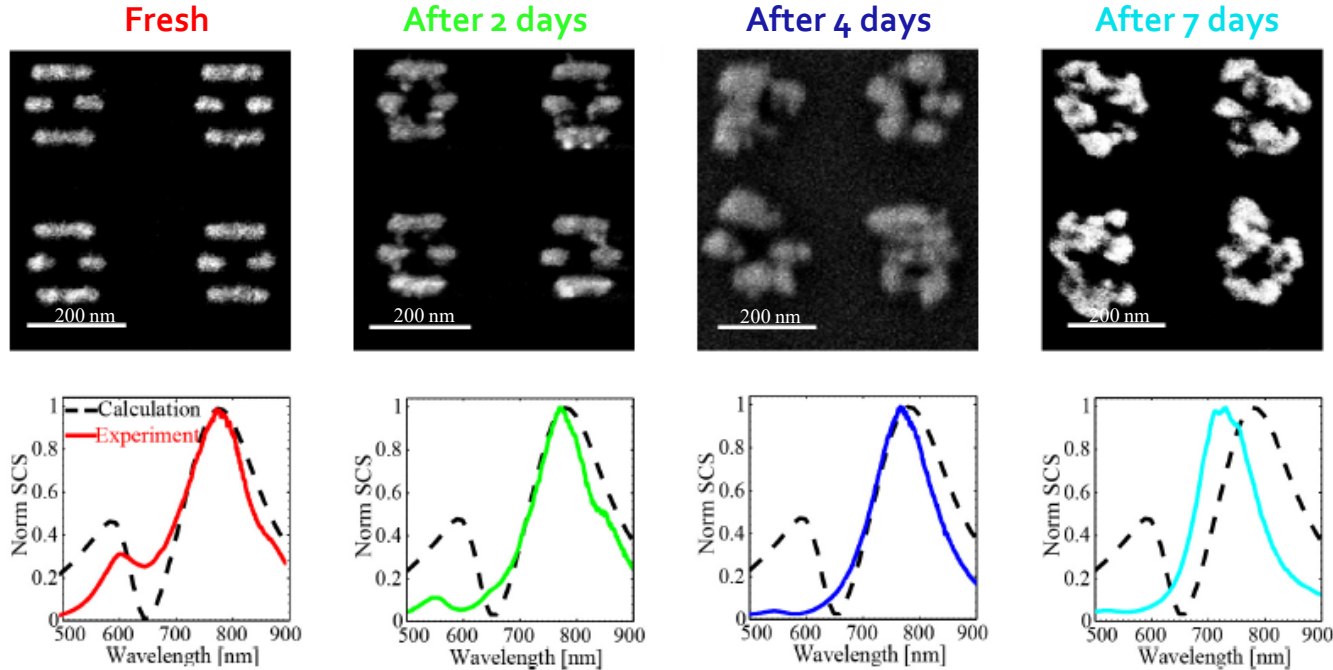


# Keeping your silver shiny!



# Rapid deterioration of Ag nanostructures

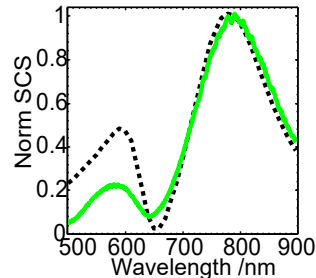
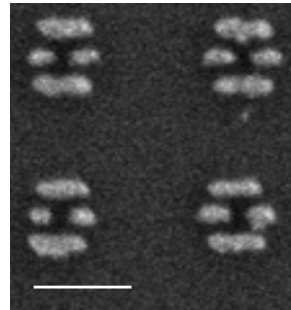
- After just a few days in ambient conditions, silver nanostructures are deteriorated with irreversible changes in their morphology and optical response



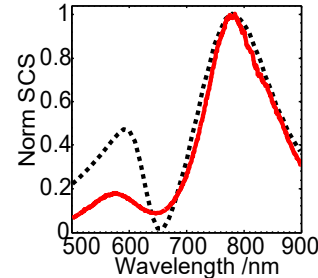
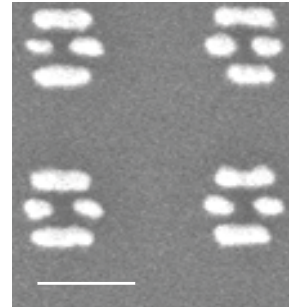
# Myth #1: Oxygen is responsible for the deterioration of Ag

...well, after two weeks exposure to dry oxygen, silver nanostructures do not exhibit any damage (similar to exposure to dry argon):

After two weeks in dry O<sub>2</sub>

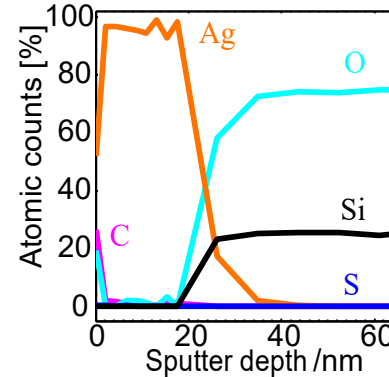
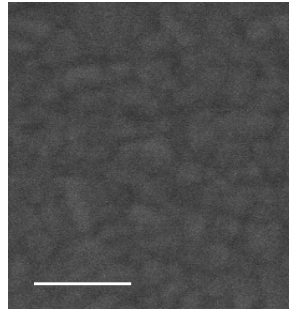


After two weeks in dry Ar



## Myth #2: Sulfidation is playing a key role for deterioration

...well, XPS analysis of a 40 nm thick silver film stored in ambient conditions does not reveal significant sulfur:



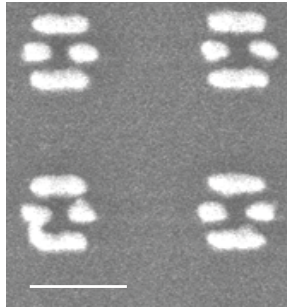
- **Even in rural areas, the total concentration of atmospheric sulfur compounds is as low as a few ppb**

*J.H. Seinfeld and S.N. Pandis, Atmospheric Chemistry and Physics from Air Pollution to Climate Change: Problem Solution Manual (Wiley, New York, 1989)*

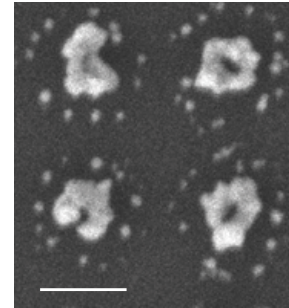
## ... so, what is responsible for Ag deterioration?

- Water !
- Silver nanostructures kept two weeks in 0 % relative humidity do not deteriorate, while those kept in 100 % relative humidity are destroyed:

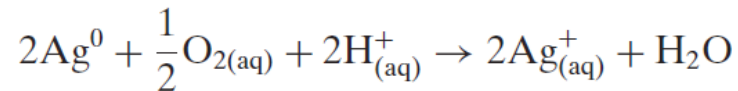
0 % Rel. humidity



100 % Rel. humidity

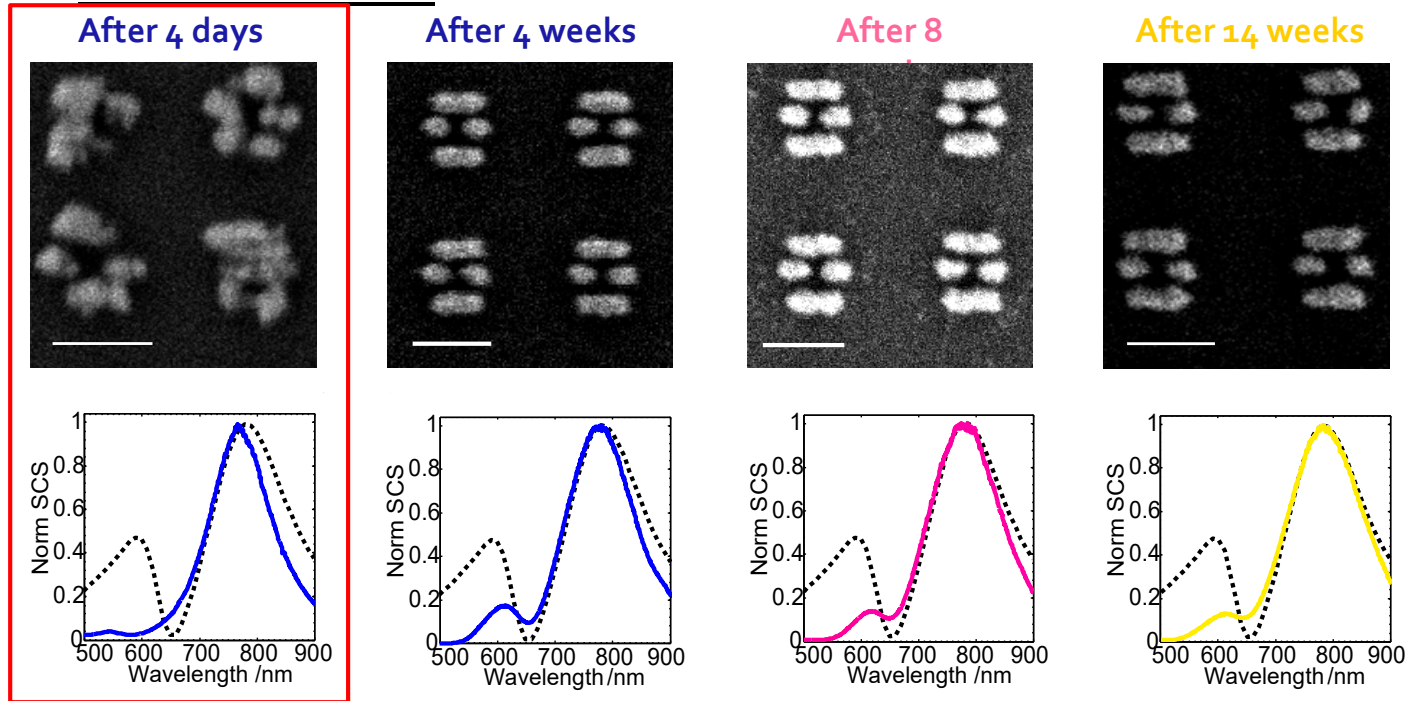


- Oxidation of silver by O<sub>2</sub> dissolved in water enables the migration of silver:



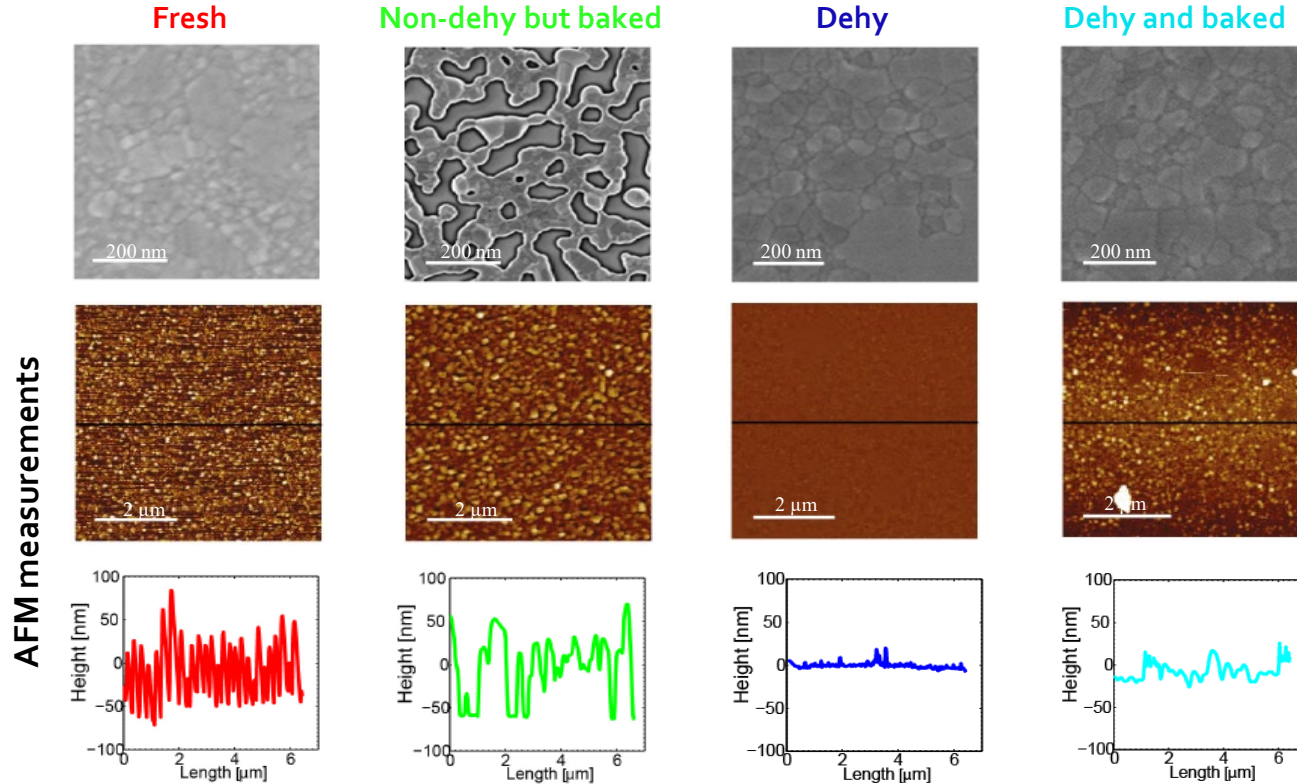
# Ag nanostructures can be stabilized with a dehydration process

- Fabricated nanostructures are transferred to a desiccator cabinet at 20 °C and purged continuously with a 5 nl/min Ar gas flow under a 1.013 Pa pressure for three weeks



# The dehydration process modifies the Ag surface

- Dramatic reduction of the surface roughness and thermally stable:



Baked 5 min @ 180°C

## What have we learned so far...

- The characterization techniques put constraints on the nanotechnology
  - Metal adhesion on glass is an issue
  - Look sideways: other fields can bring original solutions
- At the nanoscale, solids can behave very differently from what we are used to at our macroscopic scale
  - Don't be discouraged by disasters
  - Always question well-established Myths!
- It is possible to fabricate nice and robust nanostructures in Ag and Al
  - Read the "ancient" scientific literature



# Outline

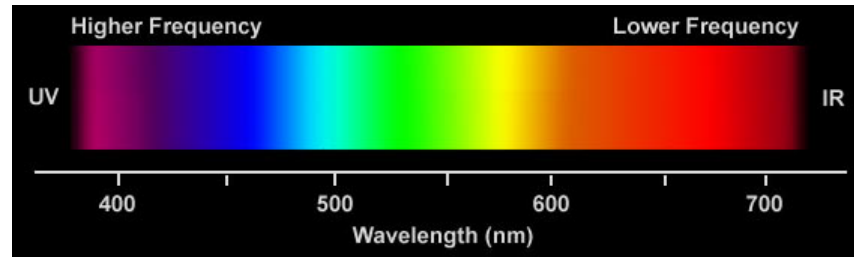
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- Plasmonics
- Basic fabrication principles with electron-beam lithography
- Adhesion issues
- Other metals than Au
- **Au-Ag alloys**
- Hybrid nanostructures
- Chemical synthesis
- Medieval stained-glass windows

# Plasmonic metals

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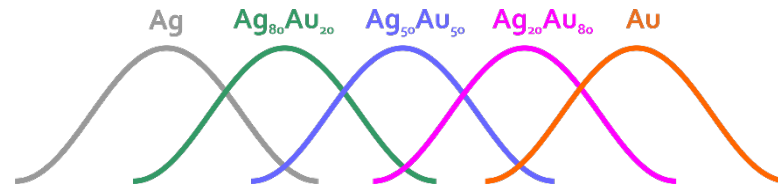
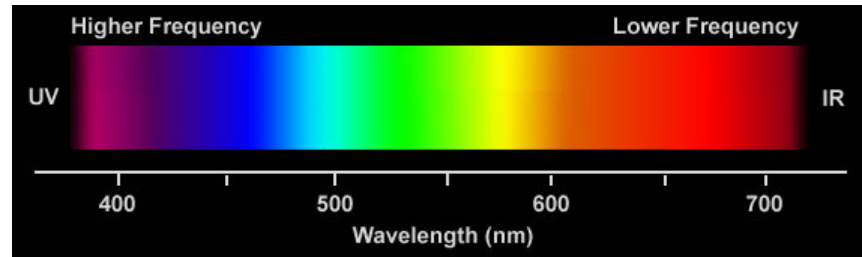
- Only a few plasmonic metals are available
- Coinage metals: Au, Ag, Al, Cu, W
- Very high electron density (  $> 10^{22} / \text{cm}^3$  )
- Each metal works in a specific wavelength range determined by the real part of its permittivity



# Plasmonic alloys

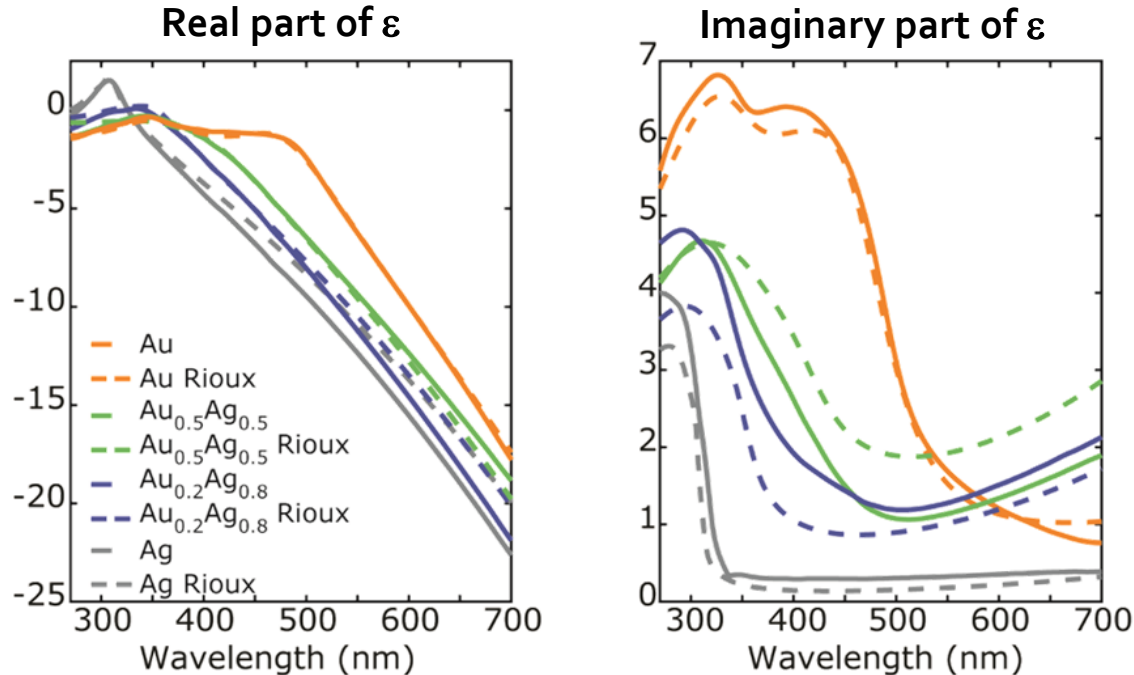
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- Can we extend the range of available plasmonic metals?



# Plasmonic alloys

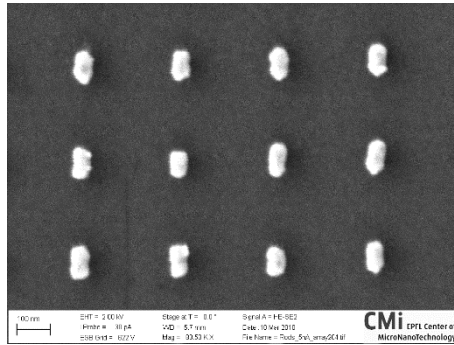
- Can we extend the range of available plasmonic metals?
- Alloys are certainly an interesting approach to engineer the dielectric function:



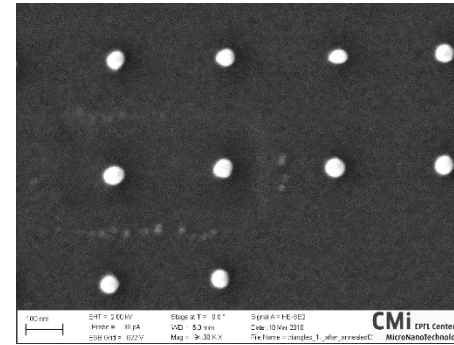
# Alloyed plasmonic nanostructures

- Normally,  $\text{Au}_x\text{Ag}_{1-x}$  alloying is performed at high temperature
- Can we maintain the shape of nanostructures during the alloying process...?

Before annealing



After annealing



- ... no! the shape is lost during the high temperature annealing

# Alloyed plasmonic nanostructures

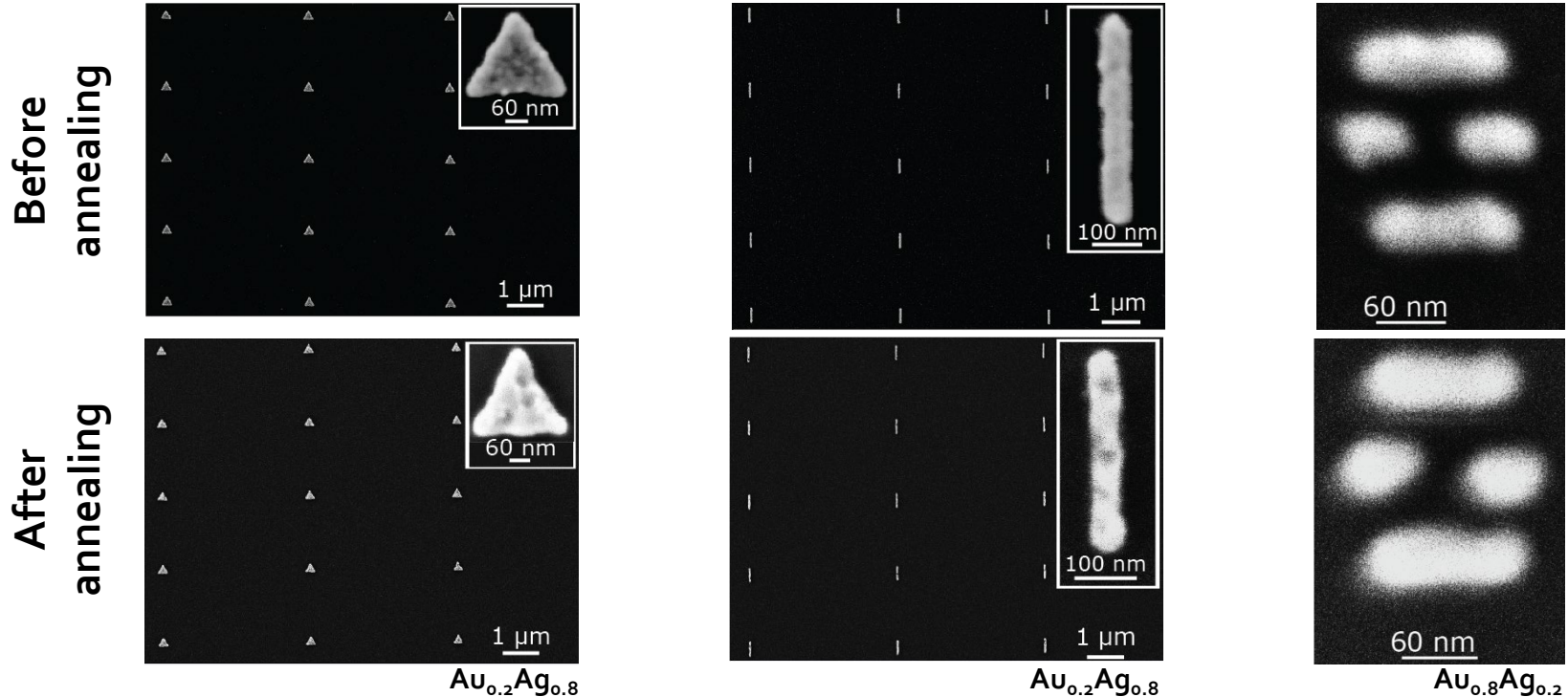
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- Challenge:
  - Find a low temperature alloying procedure...
  - ... that enables any alloy composition
- Solution:
  - Define the composition by the Au and Ag thicknesses
  - Alloy at 300°C for 8 hours and 450°C for 30 mins



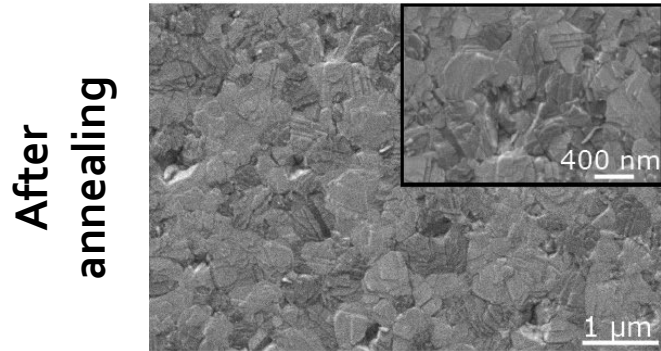
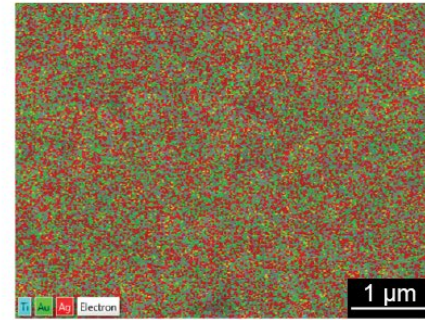
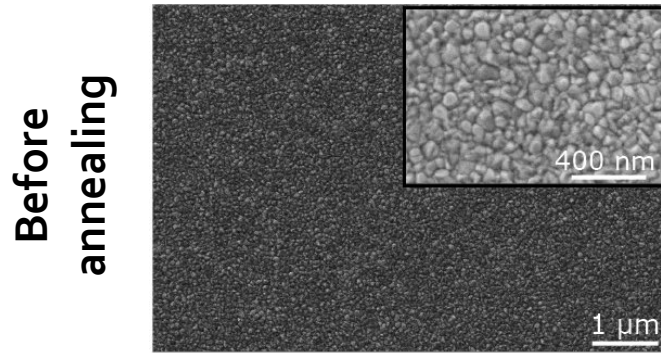
# Alloyed plasmonic nanostructures

- A low temperature annealing process can maintain the particle shape, whilst producing well alloyed materials

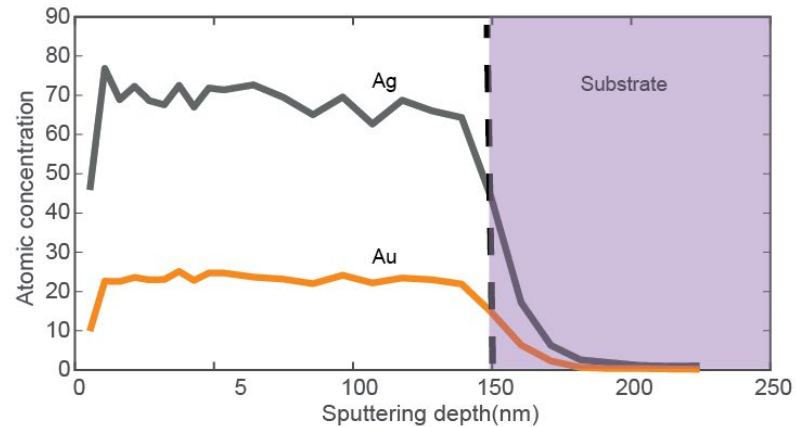


# Alloyed plasmonic nanostructures

- Well alloyed materials (EDX and XPS)

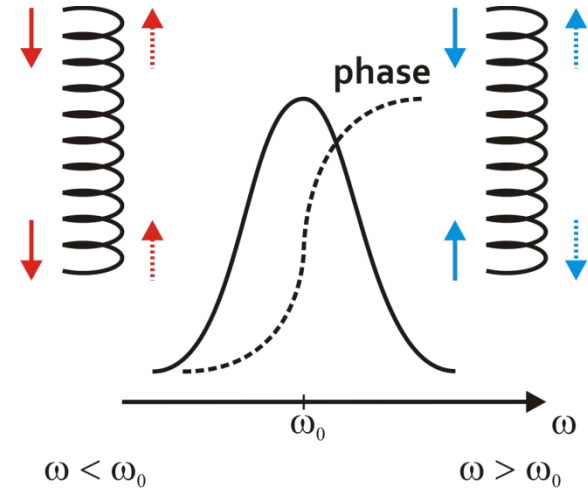
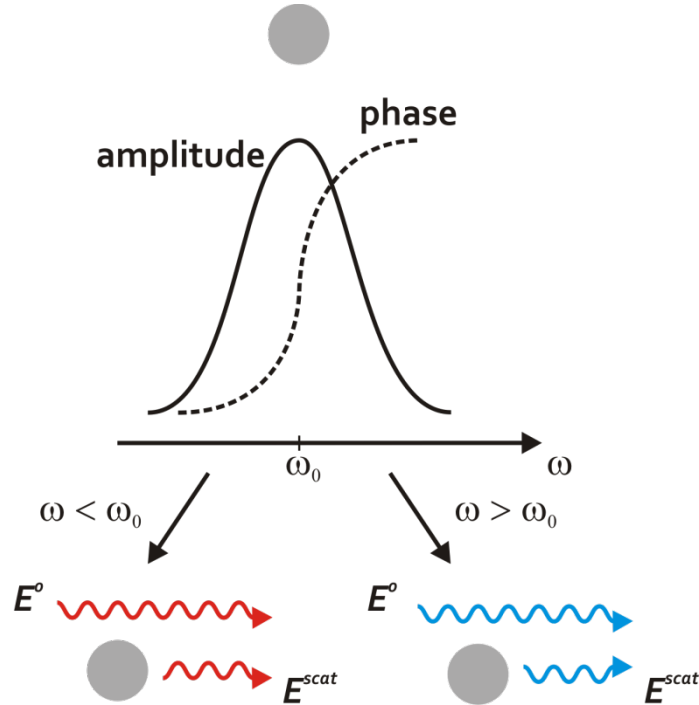


$\text{Au}_{0.2}\text{Ag}_{0.8}$



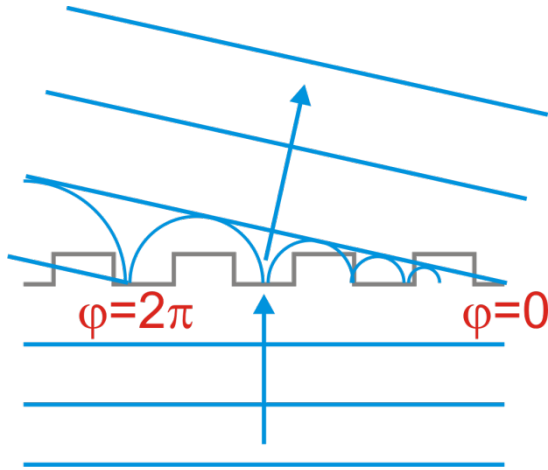
# Plasmonic metasurfaces

- Optical resonances can control the phase of the scattered light

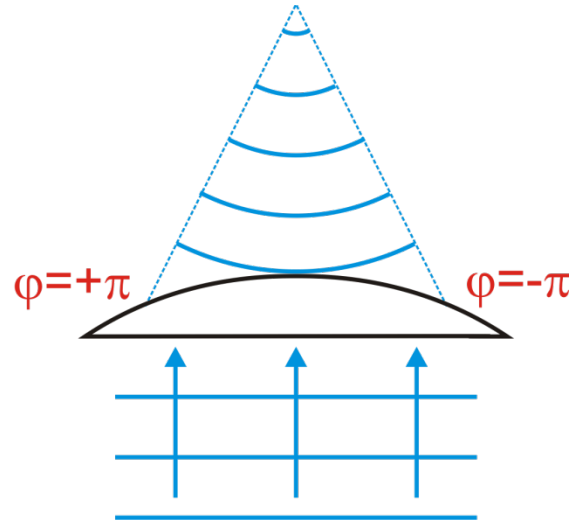


# All optical components manipulate the phase of light

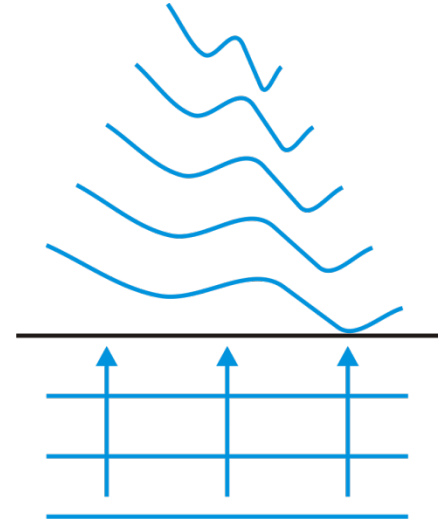
- Grating:



- Lens:



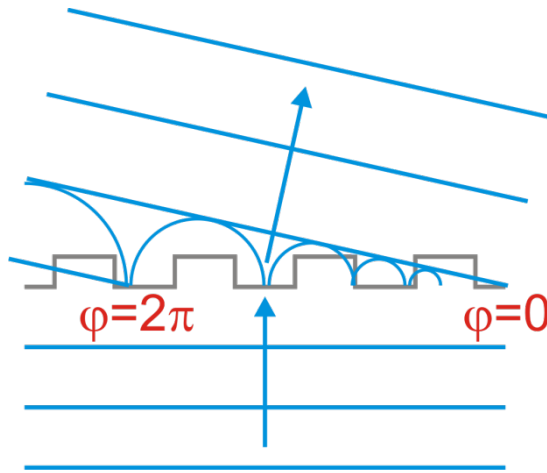
- Hologram:



# Plasmonic metasurfaces

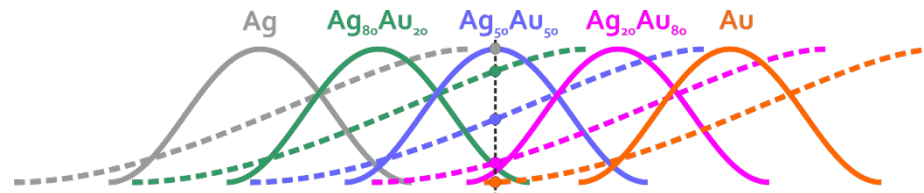
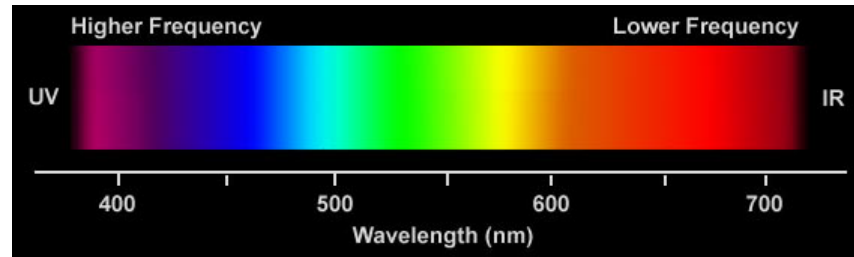
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- Can we mimic the response of an optical component using plasmonic nanostructures on a surface?
- Yes, if we use nanostructures that produce different phases! (different shapes, geometries, metals, or alloys)



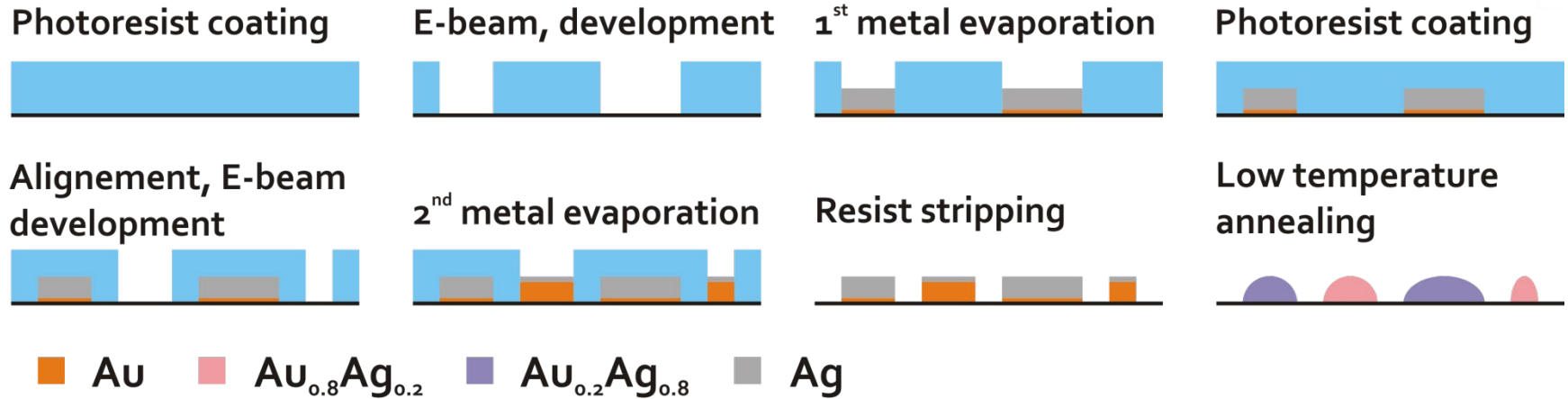
# Plasmonic metasurfaces

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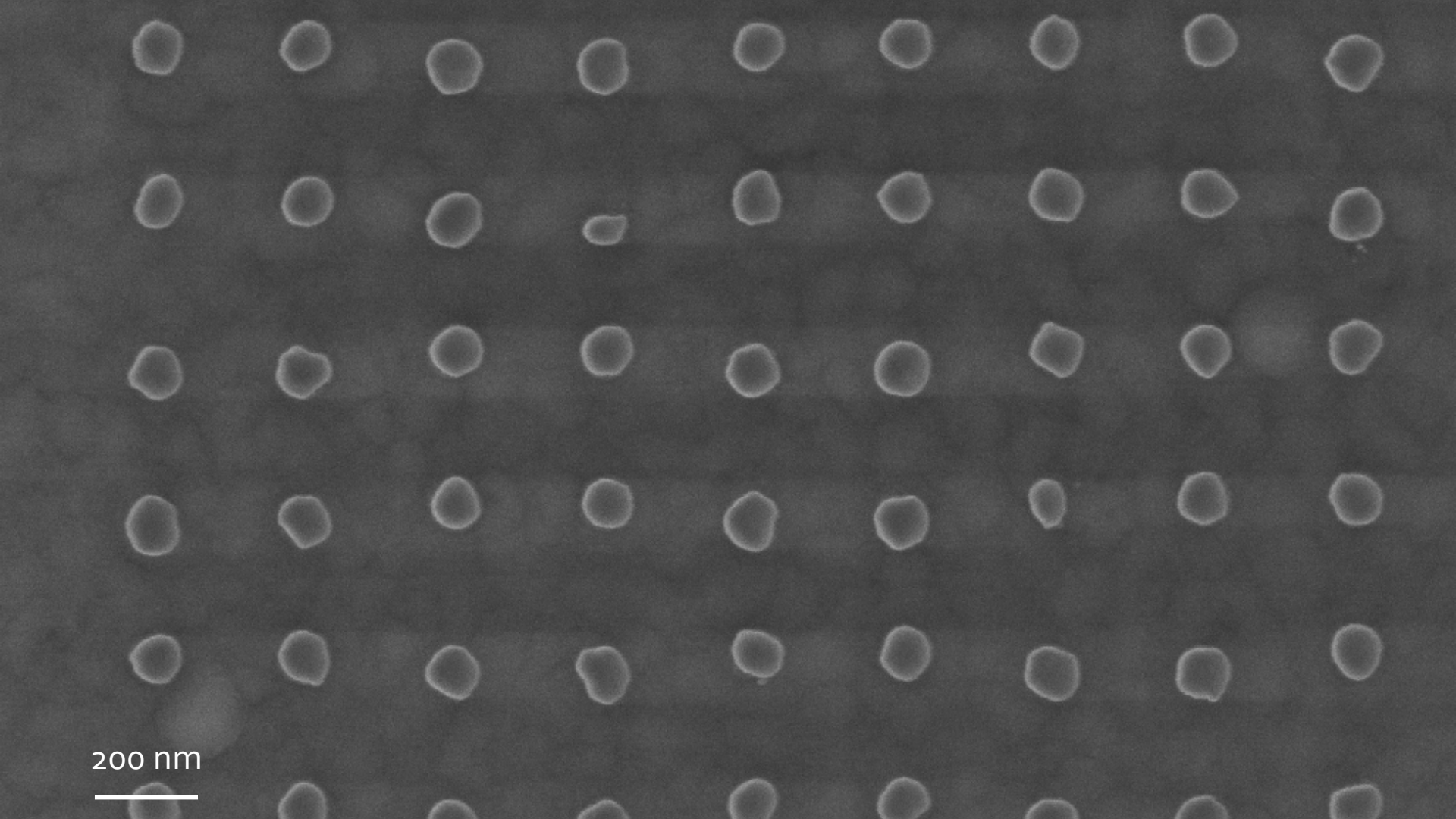
# Alloyed plasmonic metasurfaces

- Combinations of different alloy compositions on the same sample to produce more complex optical effects

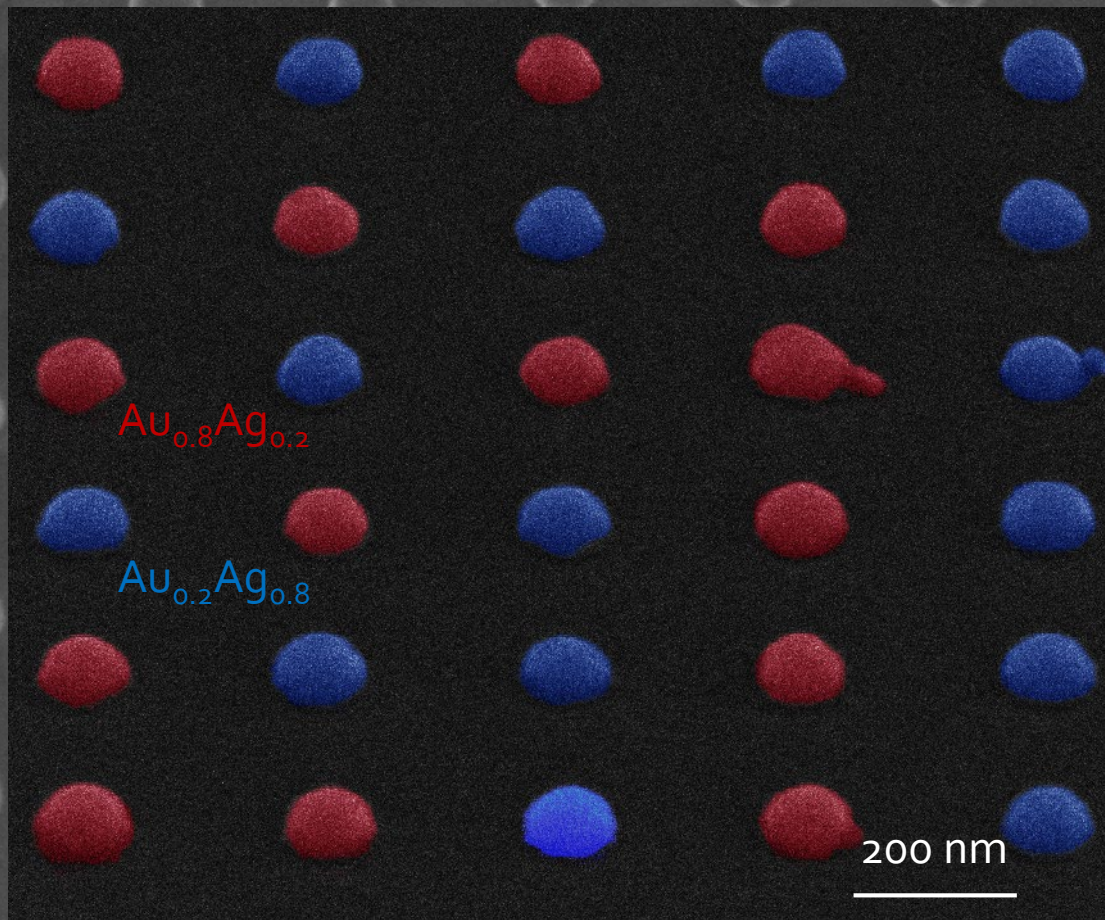


1  $\mu\text{m}$

A grayscale micrograph showing a highly ordered, periodic lattice structure. The lattice consists of small, dark, circular or square-like units arranged in a regular grid pattern. The spacing between these units is uniform across the field of view. In the bottom-left corner, there is a white horizontal scale bar. Above the scale bar, the text "1 μm" is printed in white, indicating the length of the scale bar.



200 nm



# Complex metasurfaces

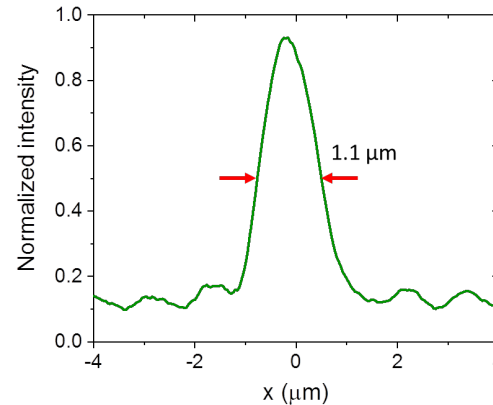
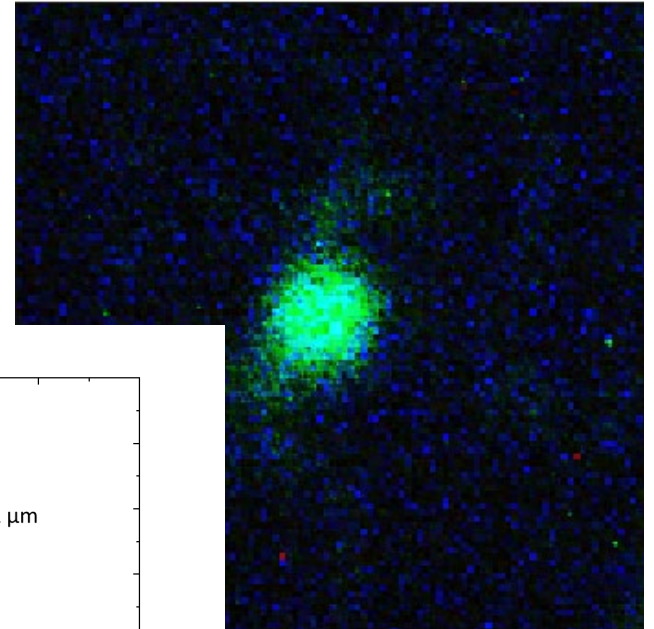
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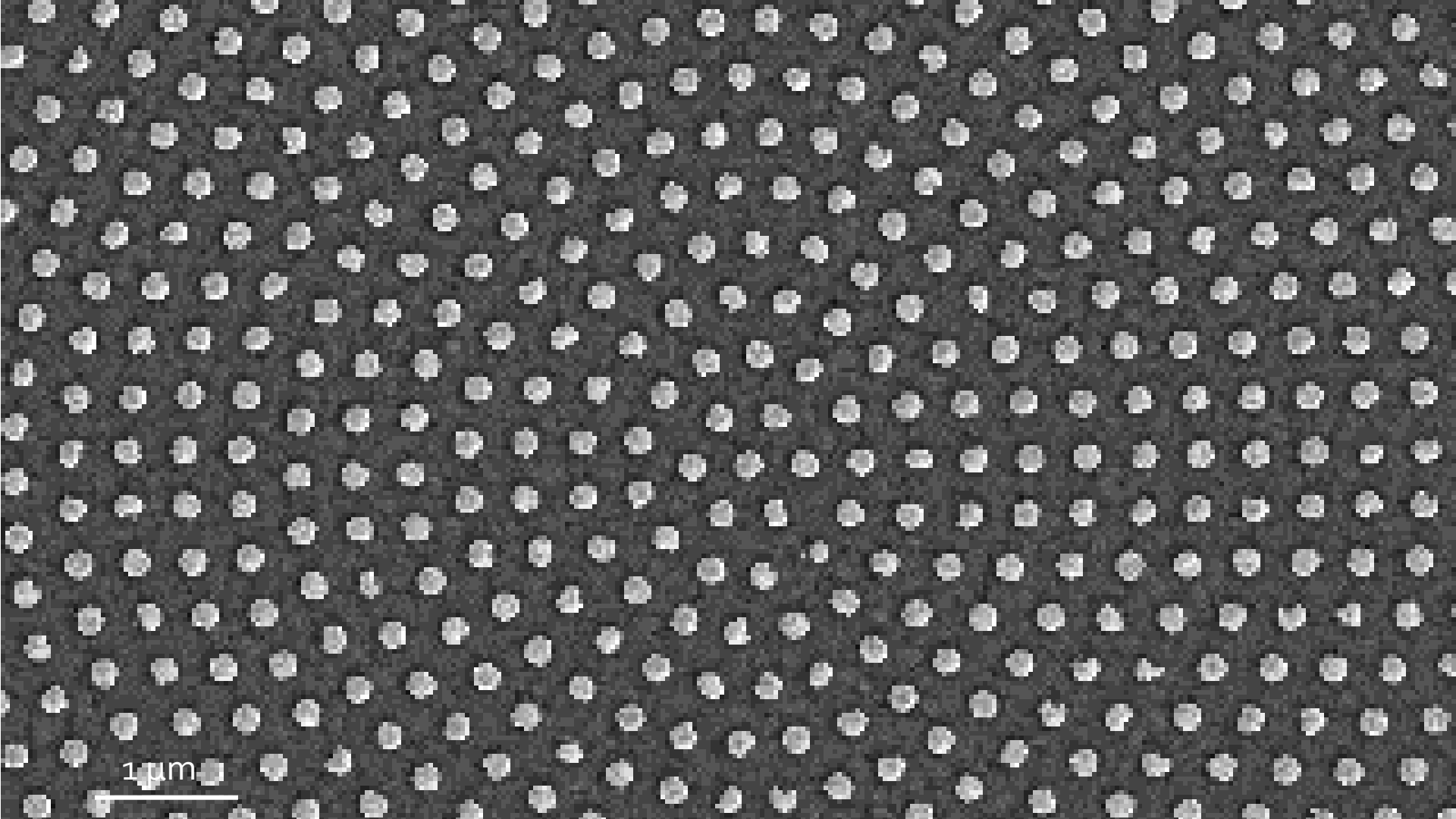
- Hologram:



$\lambda=532$  nm

- Lens:





## What have we learned so far...

- If you don't get the property you wish from pure materials, why not mix several together?
- You don't need to follow well-established recipes, you can try completely novel approaches (if you have time and money...)
  - Alloying does not necessarily require high temperature
  - Scattering from a nanostructure also manipulates the phase of light
    - This can be used to produce all sorts of flat optical elements (metasurfaces)
    - Multiple Ebeam lithography opens new possibilities



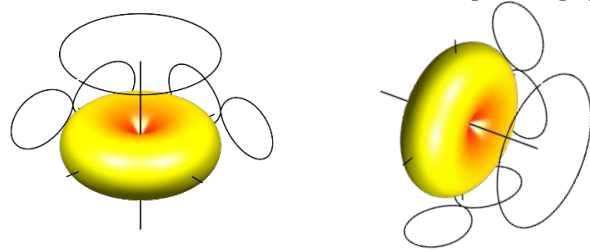
# Outline

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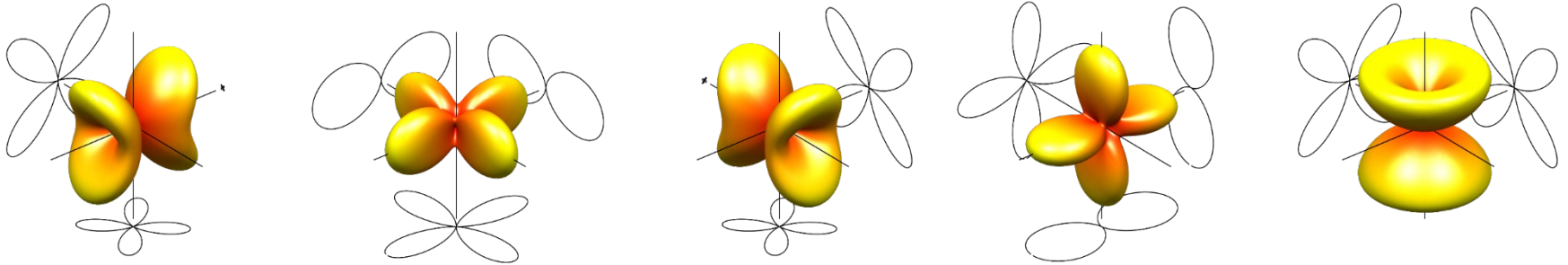
- Plasmonics
- Basic fabrication principles with electron-beam lithography
- Adhesion issues
- Other metals than Au
- Au-Ag alloys
- **Hybrid nanostructures**
- Chemical synthesis
- Medieval stained-glass windows

## Optical modes determine the response of the system

- Simple plasmonic nanostructures essentially support electric dipolar modes



- Dielectric resonators can support magnetic and higher order modes

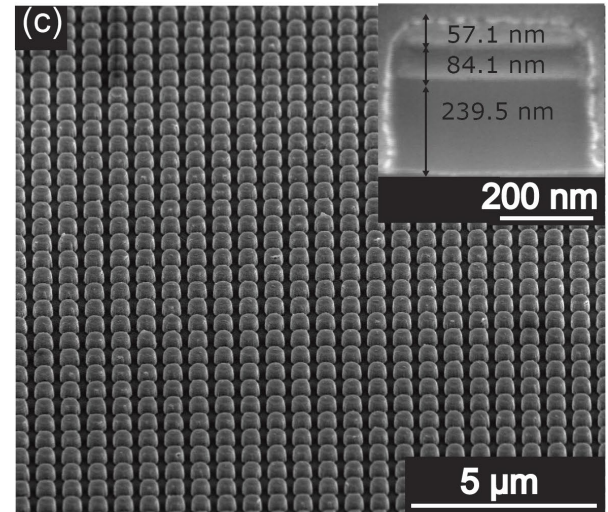
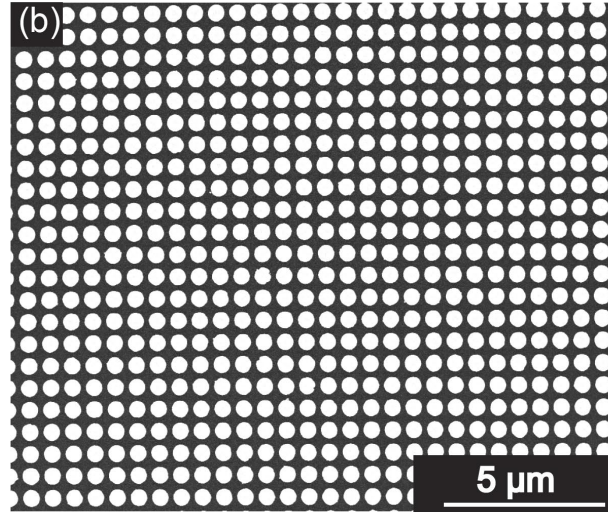
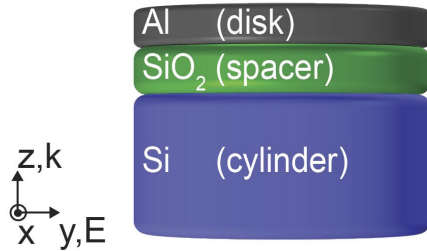


- Can we combine both to access interesting effects?

# Hybrid metasurface for sensing

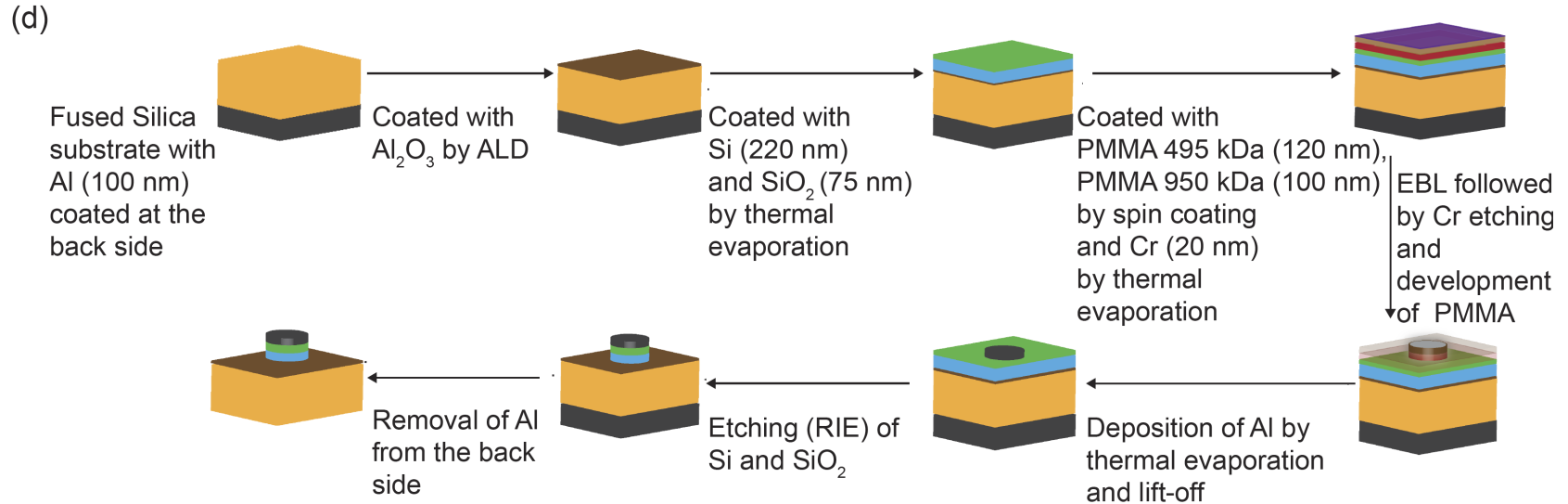
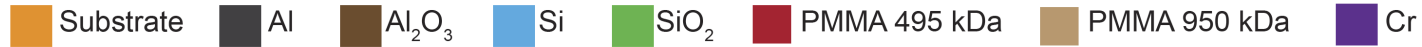
- Combining a dielectric resonator (Si) with a plasmonic disk (Al)

(a)



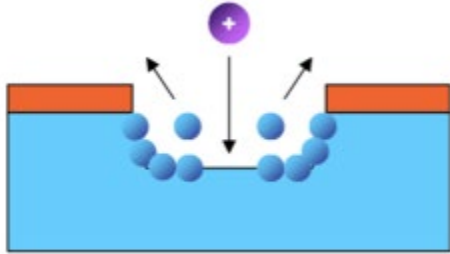
# Hybrid metasurface for sensing

- The Al disk serves both as plasmonic structure and mask for etching



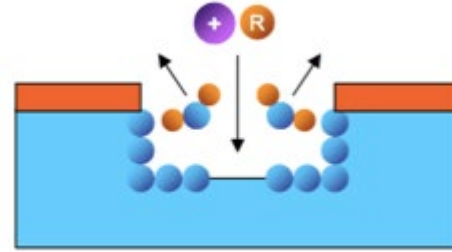
## Ion etching vs. Reactive ion etching (RIE)

- Ion etching, physical sputtering



- High anisotropy
- Poor selectivity

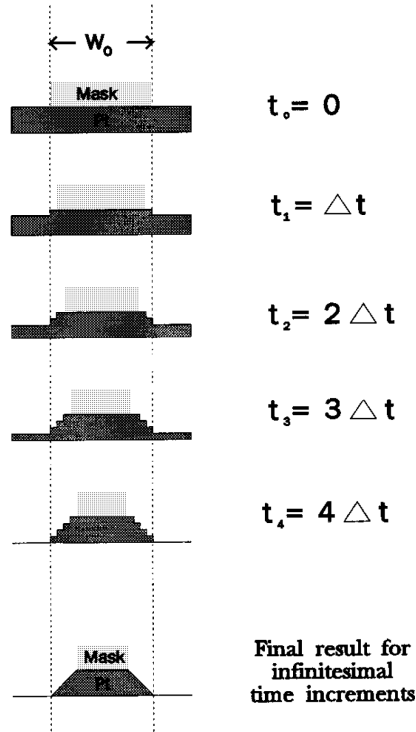
- Reactive ion etching



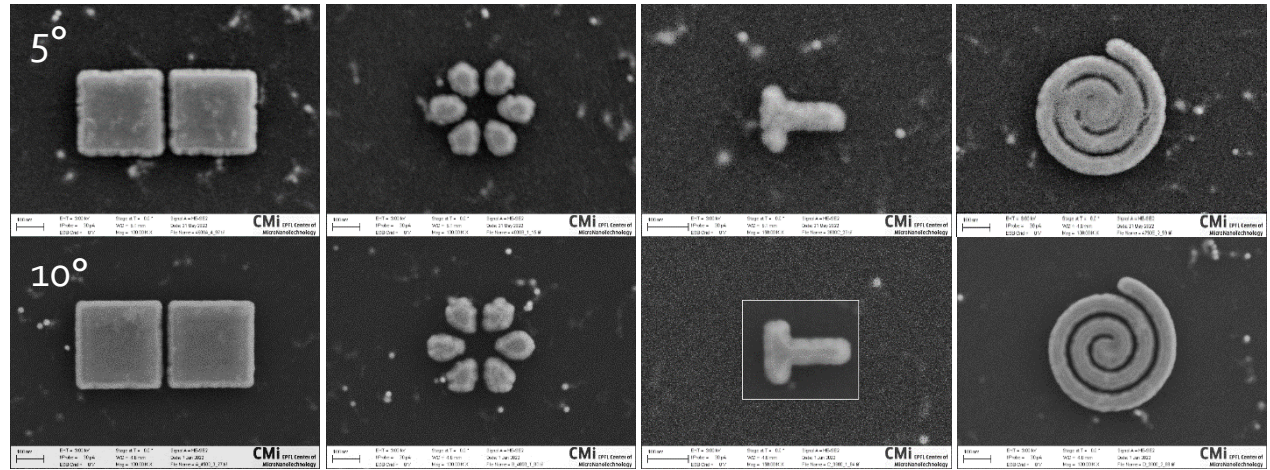
- Medium anisotropy (can be controlled)
- Good selectivity
- Cl or F chemistry
- Not well-suited for Au (but possible)

# Fencing and redeposition

- During dry etching, some of the etched material can redeposit
- The mask can also be etched, producing slanted sidewalls



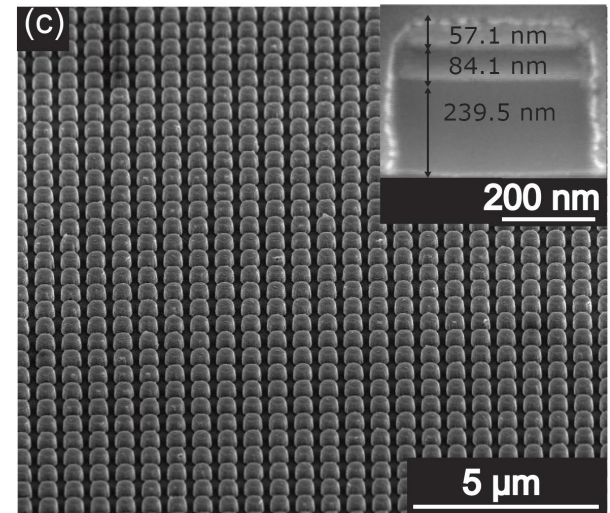
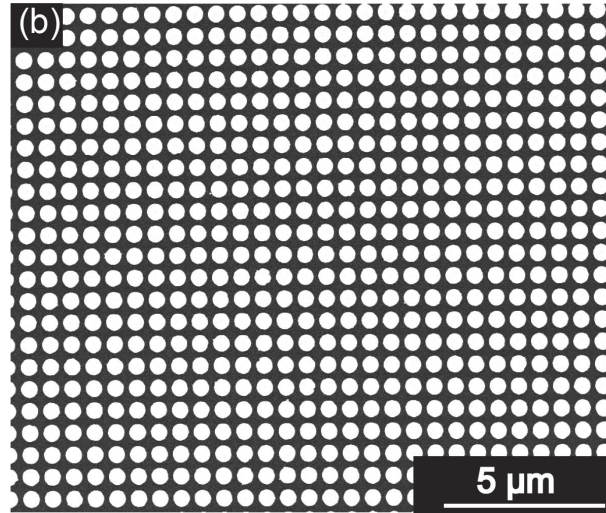
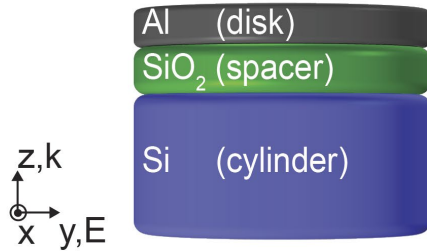
Tilting and rotating the substrate during etching helps avoid redeposition



# Hybrid metasurface for sensing

- Combining a dielectric resonator (Si) with a plasmonic disk (Al)

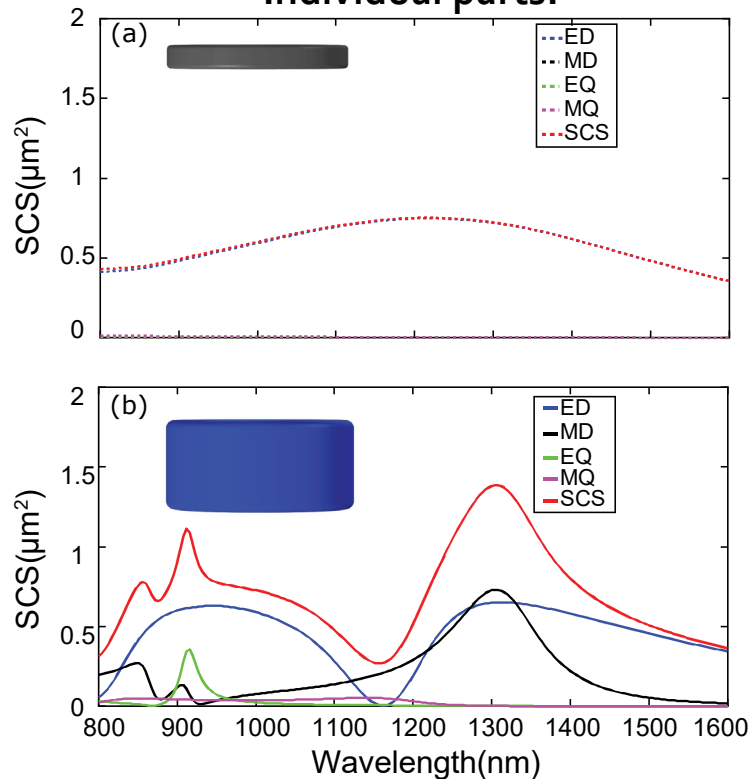
(a)



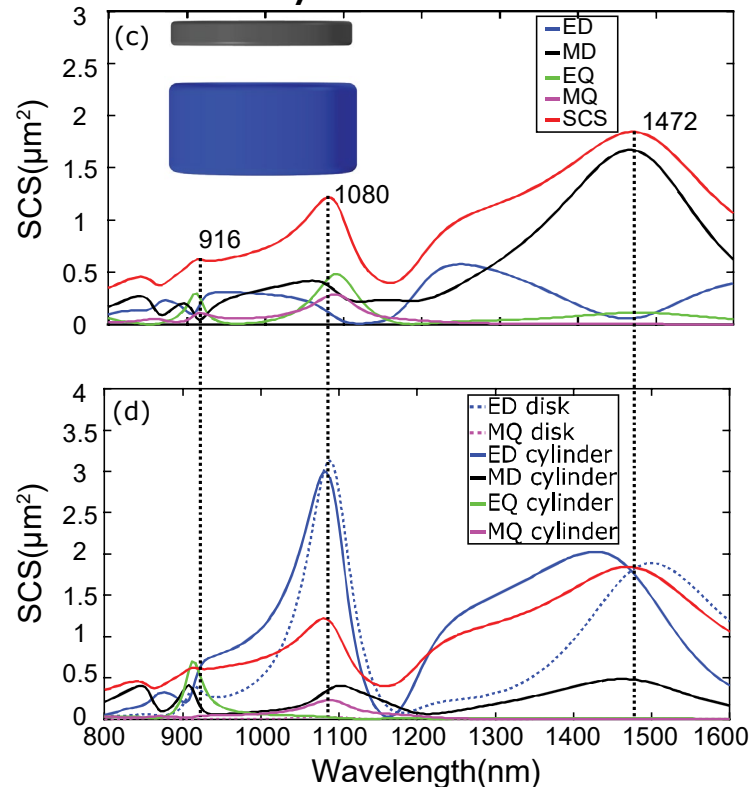
# Hybrid metasurface for sensing

- Interplay between the modes supported by the different parts

Individual parts:

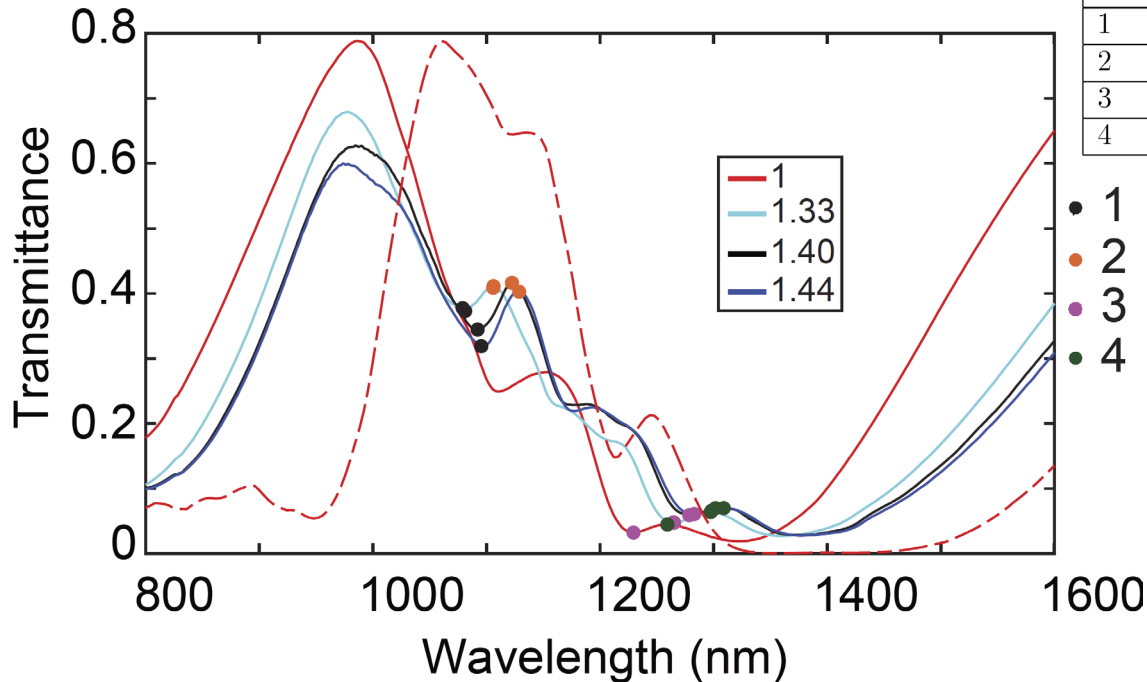


Hybrid structure:



# Hybrid metasurface for sensing

- Multiple features can be used for bulk refractive index sensing (experimental data)



Sensitivity:

Feature no.	$S = \Delta\lambda / \Delta n$ (nm/RIU)	Range (nm)
1	144	1079–1095
2	208	1106–1129
3	120	1229–1283
4	111	1259–1309

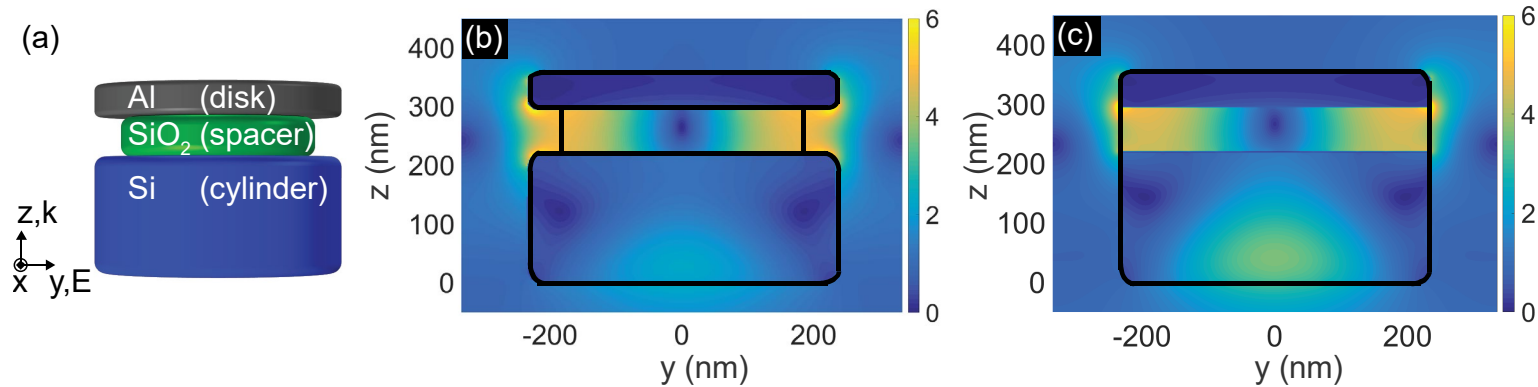
- 1
- 2
- 3
- 4

Reference sensitivities:

- Dielectric: 300 nm/RIU
- Plasmonic: 600 nm/RIU

# Hybrid metasurface for sensing

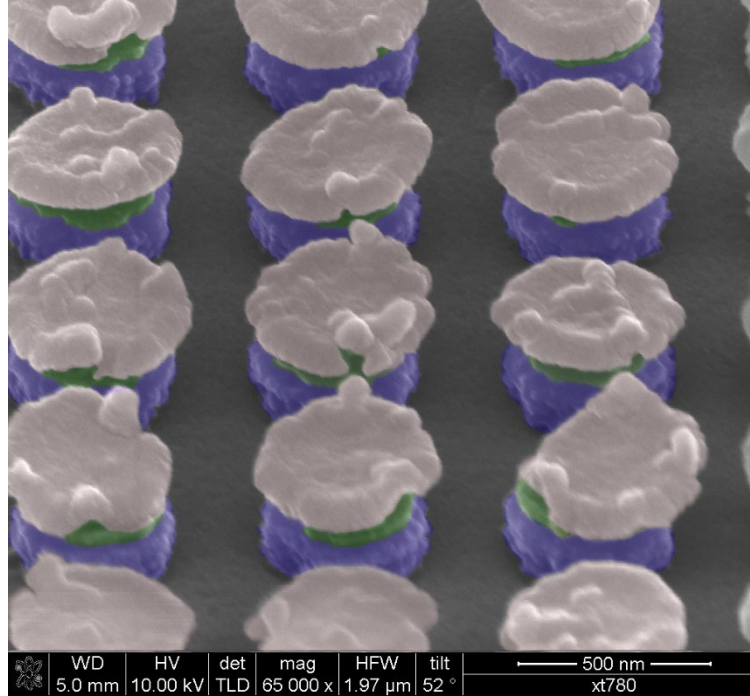
- Can we improve the overlap between near-field and analyte?



- Additional wet etch step to produce an undercut

## Hybrid metasurface for sensing

- Can we improve the overlap between near-field and analyte?



Sensitivity improvement  
of 40 nm/RIU  
( $S=245$  nm/RIU)

## What have we learned so far...

- Ion etching (reactive or not) is an interesting, but subtle, approach
  - A variety of materials can be processed this way
- Do not hesitate to combine several processes to produce your samples
- Close the loop: numerical simulations → fabrication → experiments → numerical simulations → fabrication again → ...
- Don't be disappointed if at the end all your efforts provide only limited improvements



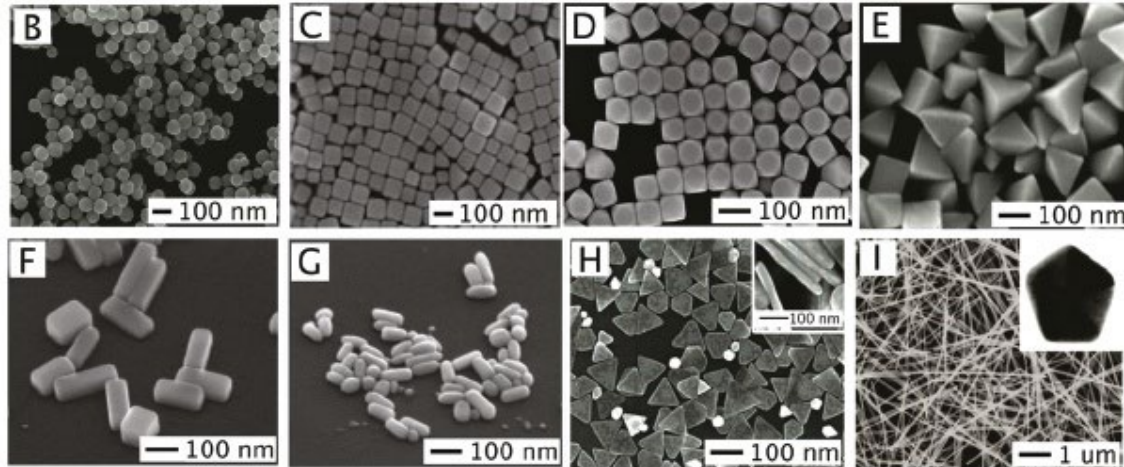
# Outline

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- Plasmonics
- Basic fabrication principles with electron-beam lithography
- Adhesion issues
- Other metals than Au
- Au-Ag alloys
- Hybrid nanostructures
- **Chemical synthesis**
- Medieval stained-glass windows

## Different types of plasmonic nanostructures

- Two approaches exist for fabricating plasmonic nanostructures:
  - Top-down nanofabrication (conventional clean-room fabrication)
  - Bottom-up (chemical, self-assembled fabrication):

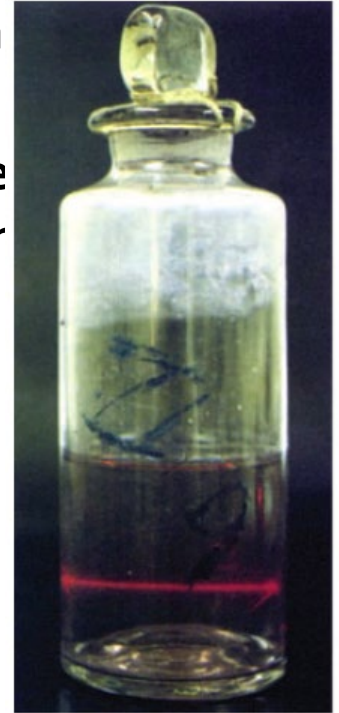
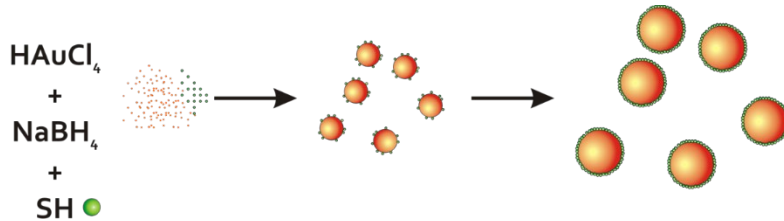


# Conventional gold nanoparticle synthesis

- Reduction of  $\text{Au}^{\text{III}}$  compounds to form metallic Au (e.g. with borohydride)
- Ostwald ripening process: over time small particles dissolve and redeposit onto larger particles, which are more favorable than one single large particle:



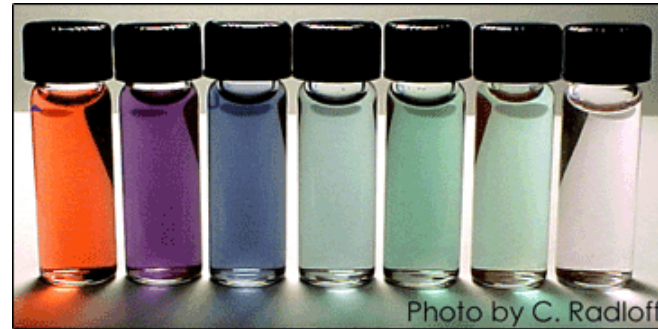
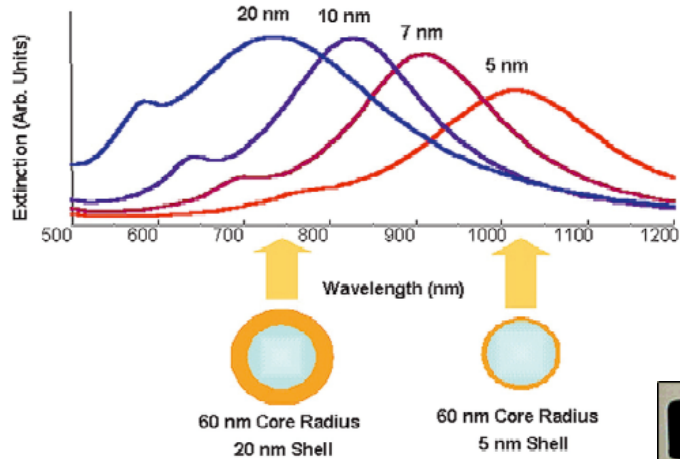
- A capping agent (thiol-based for Au) stabilizes the process, monodispersed nanoparticles:



Faraday's colloidal suspension of gold or « gold fluid »

# Different types of plasmonic nanostructures

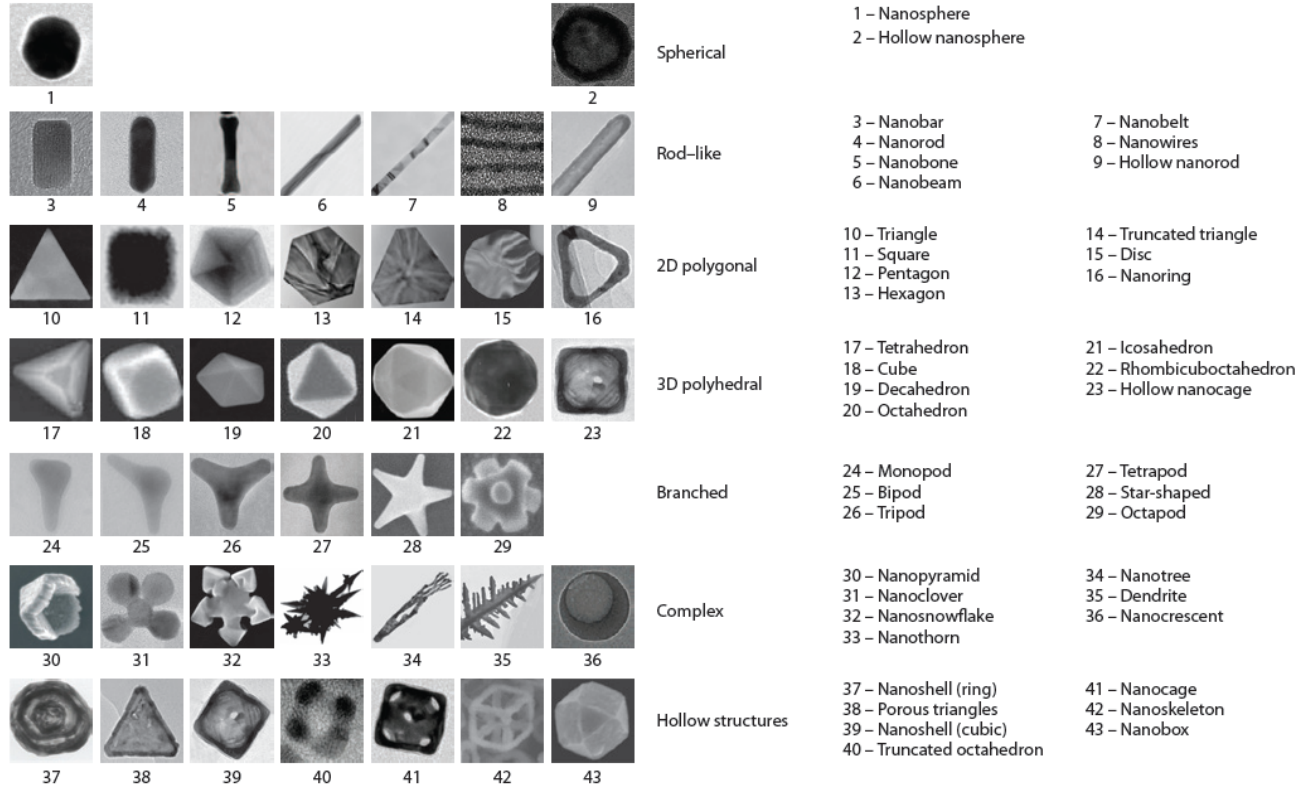
- Core-shell nanoparticles are highly tunable:



# A broad variety of plasmonic "atoms" can be fabricated

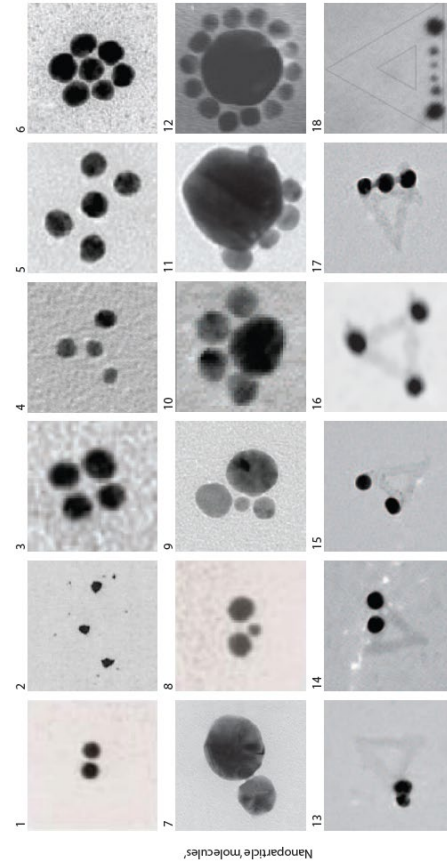
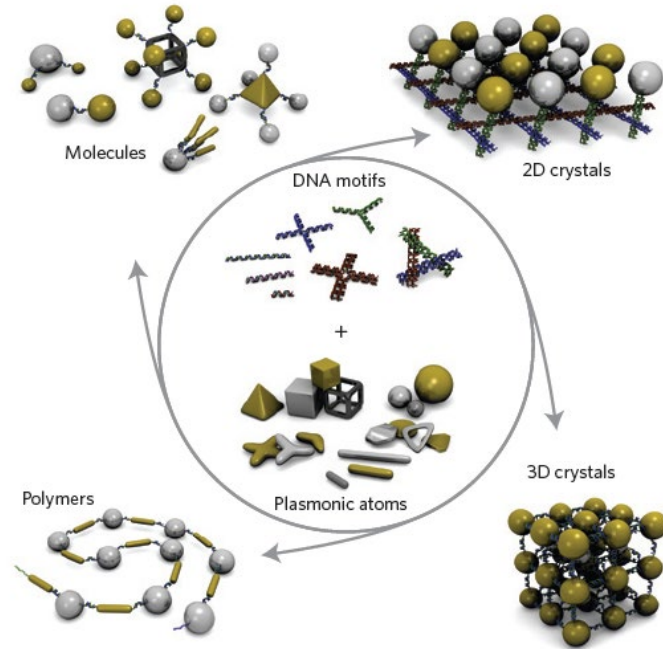
REVIEW ARTICLE

NATURE NANOTECHNOLOGY DOI: 10.1038/NNANO.2011.49



# Larger structures can be realized from these "atoms"

- DNA origami



Plasmonic colors in medieval stained-glass windows:  
Myth or reality?



# Medieval stained-glass windows

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# Medieval stained-glass windows

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Stained-glass window  
ca 1480

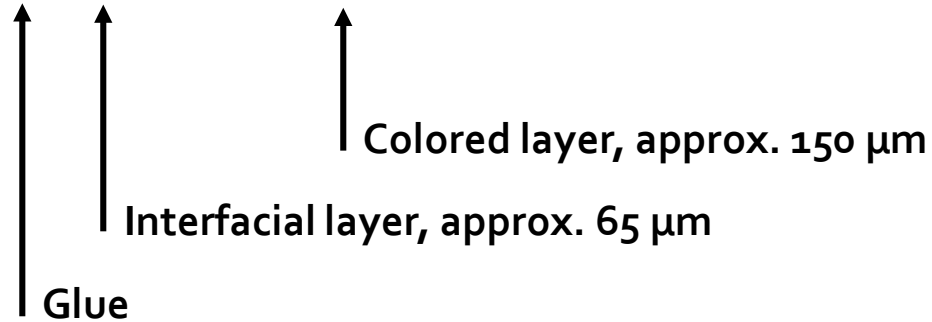
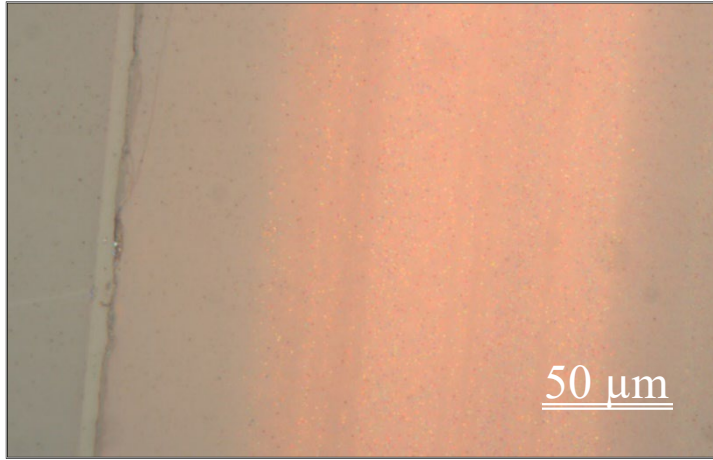


Ulm minster  
built from 1377  
*wikipedia*

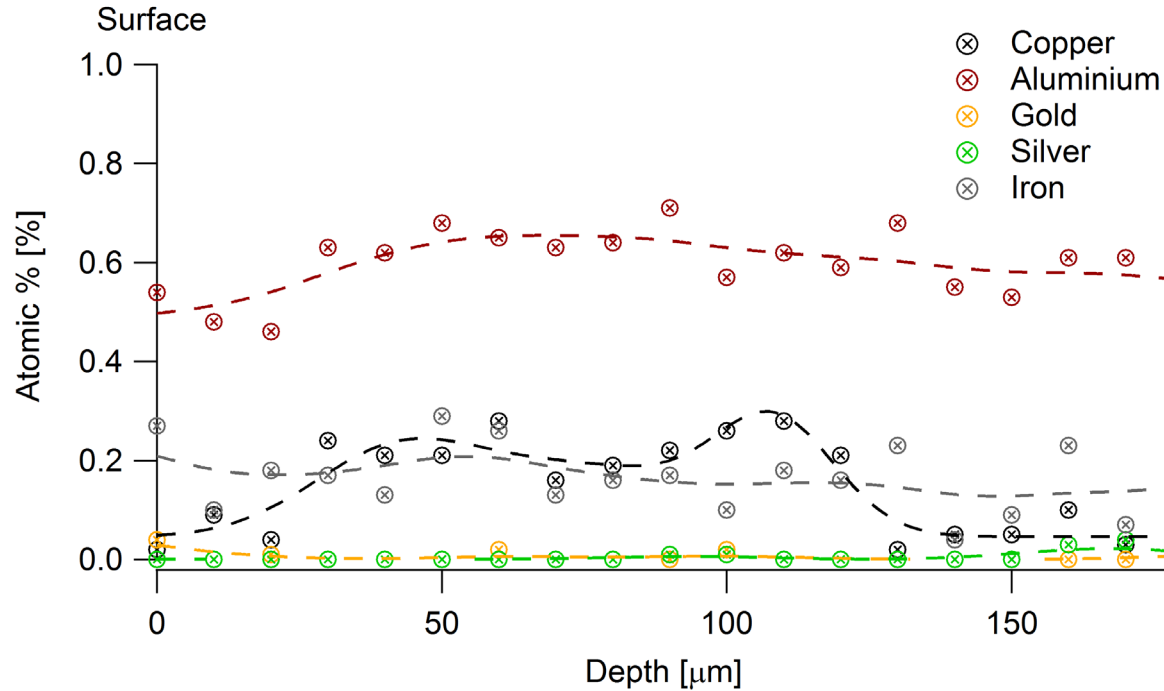


# Medieval stained-glass windows

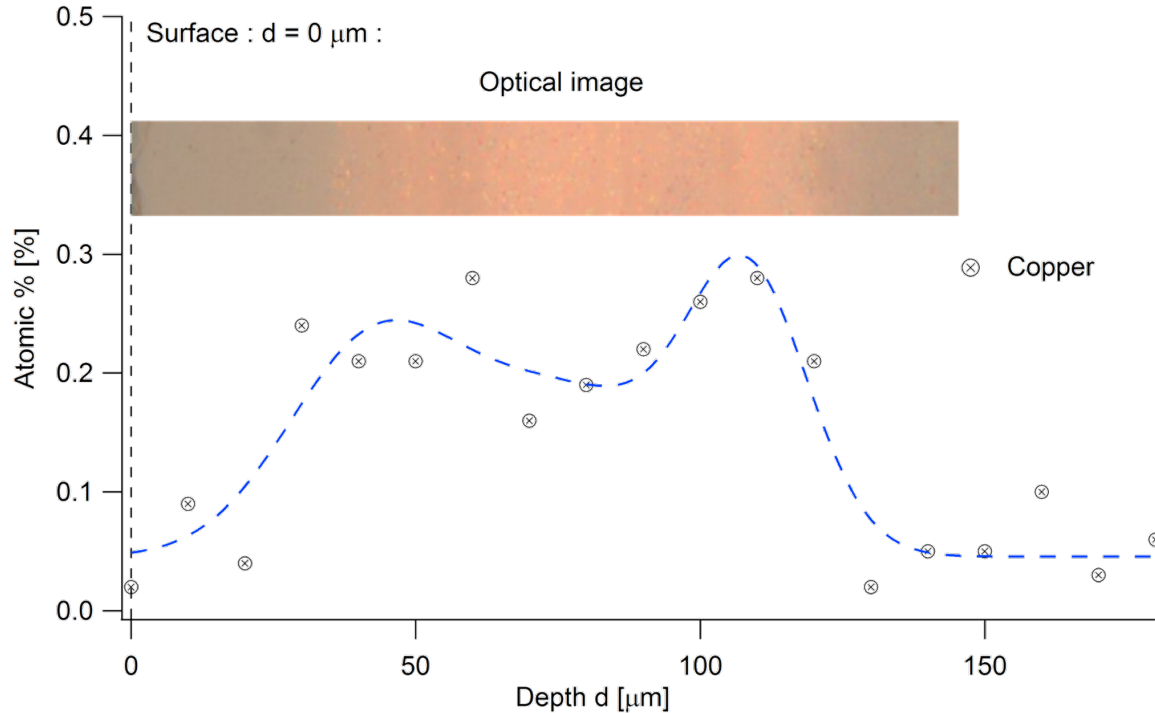
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# Energy dispersive X-ray spectroscopy (EDX)



# Correlation between color and density of Cu particles



**Cu particles:**

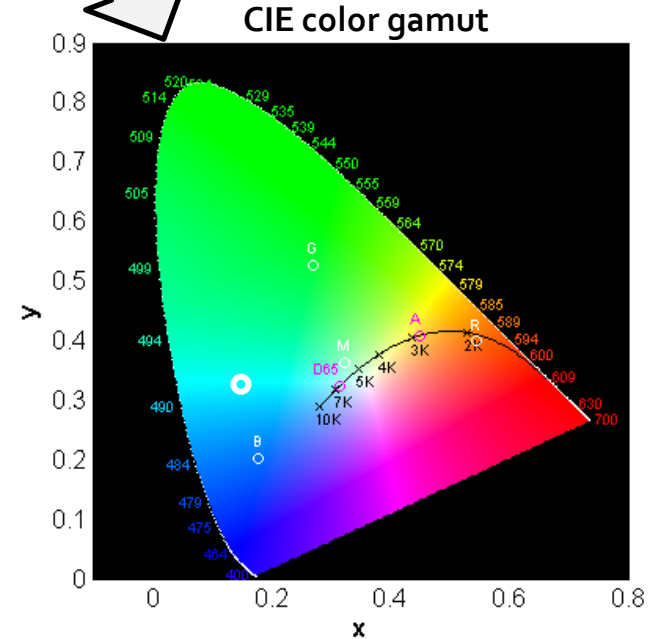
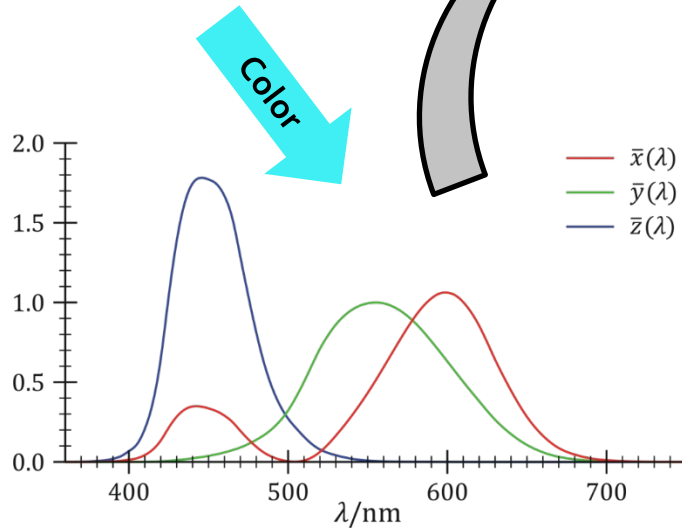
– size:  $63 \text{ nm} \pm 15 \text{ nm}$

– density:  $0.38 \mu\text{m}^{-3}$

– average distance between particles  $3.6 \mu\text{m}$

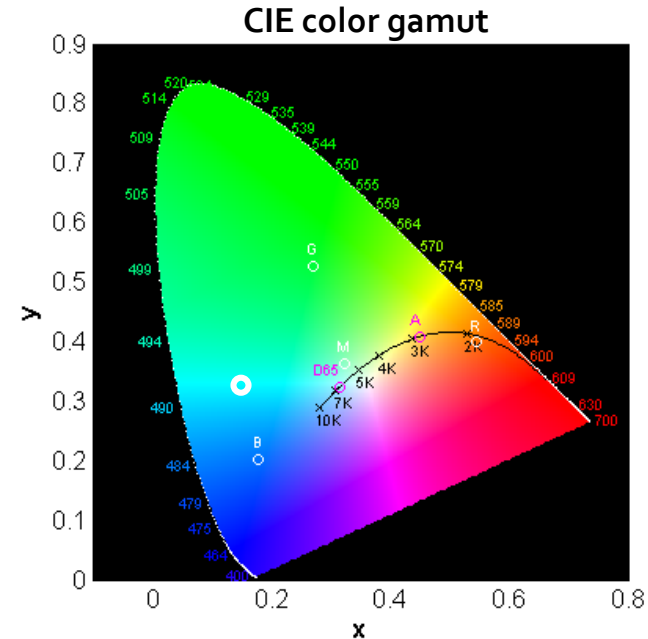
# Color perception

- Two families of receptors enable human vision:
  - Rods (high sensitivity, black & white)
  - Cones (3 types with max. sensitivity @  $\lambda=420$ ,  $530$  or  $560$  nm)



# Color perception

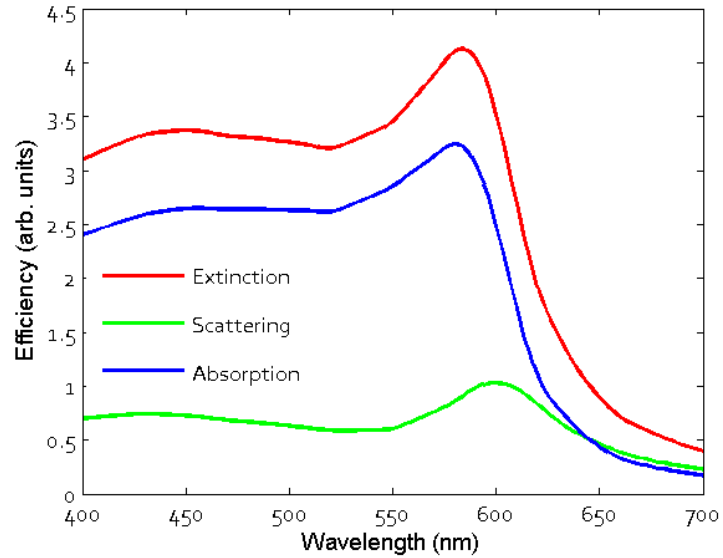
- Two families of receptors enable human vision:
  - Rods (high sensitivity, black & white)
  - Cones (3 types with max. sensitivity @  $\lambda=420, 530$  or  $560$  nm)
- Any color can be represented as a point (x,y) in the color gamut



# Comparison with Mie calculations

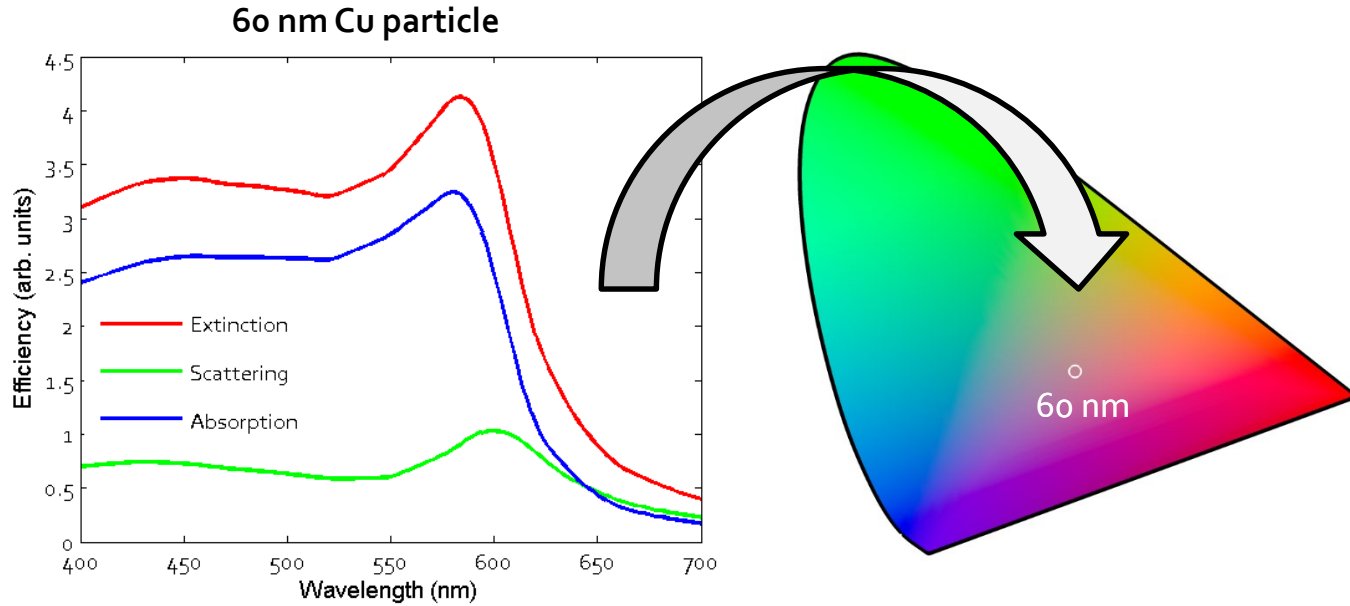
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60 nm Cu particle



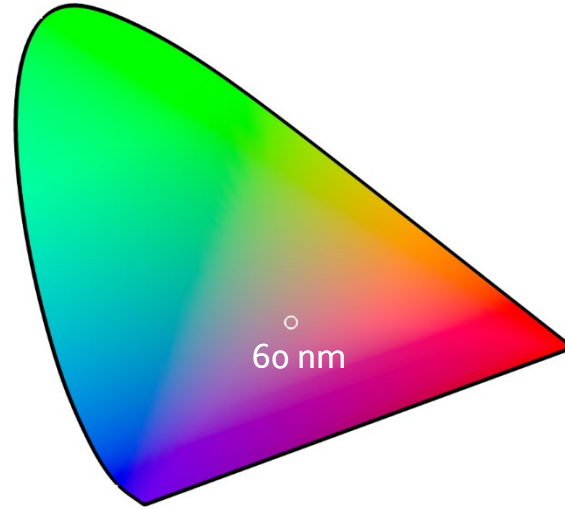
# Comparison with Mie calculations

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# Comparison with Mie calculations

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- Good agreement with the observed color

## What have we learned so far...

- Chemical synthesis provides an alternative route to cleanroom nanotechnology
- This approach can be used independently or together with top-down approaches
  - Quite deterministic nanostructures can be produced this way
    - Interesting LEGO approach to build complex structures
    - Remain fascinated by Nature and the world around us !





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[www.nanophotonics.ch](http://www.nanophotonics.ch)