Synthesis of substrates for SERS sensors with DE-LIL (Double Exposition Laser Interference Lithography) technique

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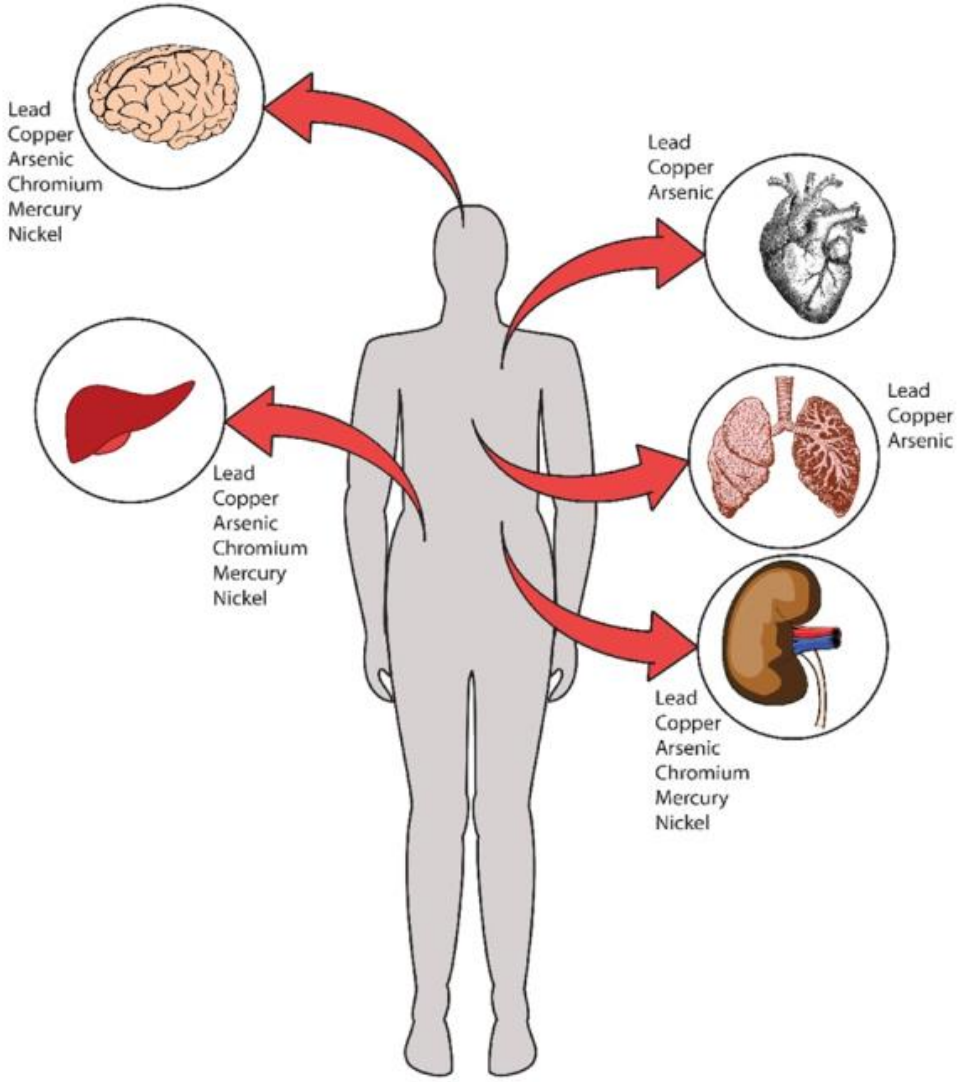
Heavy metals contamination

- Industrial waste
- Contaminations of water and soil
- Propagations/Accumulation in the food chain
- Health damage (degenerative diseases, tumors, breathing problems …)



Mahakalkar, M. A., Gupta, M. R., & Nandeshwar, M. S. (2013). Bioaccumulation of Heavy metal toxicity in the vegetables of Mahalgaon, Nagpur, Maharashtra (India). *Current World Environment*, *8*(3), 463-468.

https://www.epa.gov/ground-water-and-drinking-water/table-regulated-drinking-water-contaminants

Heavy metals contamination

Heavy metals	EU mg/l
Magnesium (Mg)	50
Sodium (Na)	150
Potassium (K)	12
Aluminum (Al)	0.2
Nitrate (NO3)	50
Nitrite (NO2)	0.1
Nitrogen (N)	1
Iron (Fe)	0.2
Manganese (Mn)	0.05
Copper (Cu)	1
Zinc (Zn)	0.1
Fluorine (F)	25-30°C 0.7
Silver (Ag)	0.01
Arsenic (As)	0.05
Cadmium (Cd)	0.005
Chromium (Cr)	0.05
Mercury (Hg)	0.001
Nickel (Ni)	0.05
Lead (Pb)	0.05
Antimony (Sb)	0.01
Selenium (Se)	0.01

Maximum permitted concentrations for heavy metals in natural waters according to the EU (Europea)

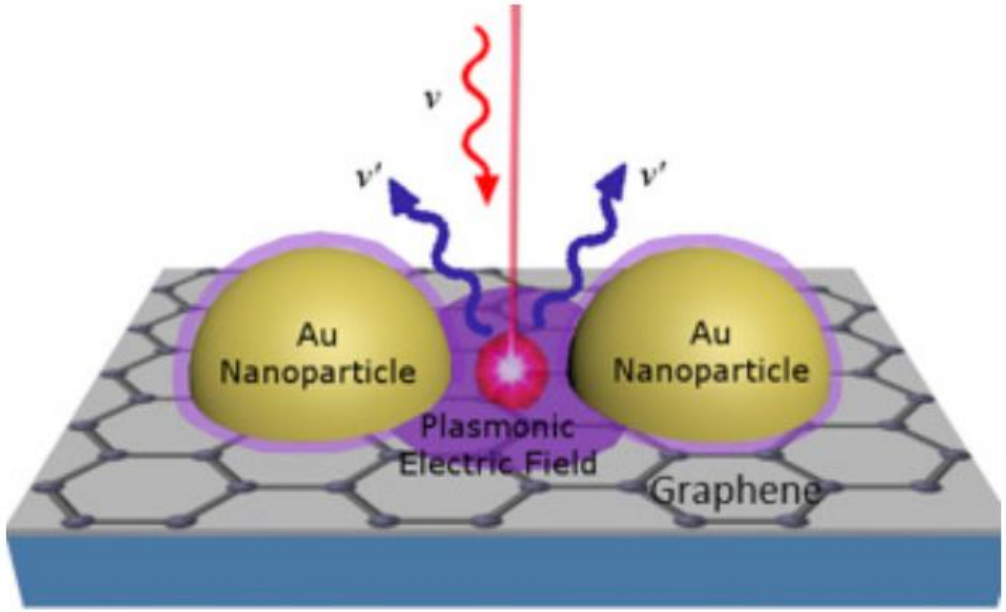
COUNCIL DIRECTIVE of 15 July 1980 relating to the quality of water intended for human consumption (80/778/EEC)

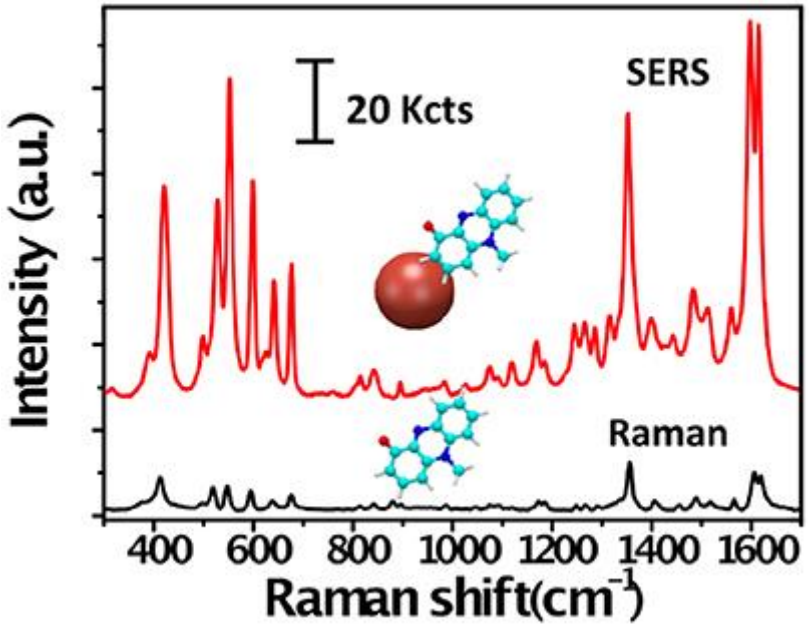
Heavy metals detection

- Fluorescent sensors
- FET sensors
- SERS sensors
- Plasmonic sensors
- Electrochemical sensors

SERS (surface enhanced raman spectroscopy)

Thanks to Raman diffusion amplification of molecules adsorbed on metal nanostructures with particular optical properties (localized surface plasmonic resonances), SERS technique allows to reveal vibrational spectra of the species in contact with the nanoparticles with high sensitivity, also the determination of few or single molecules



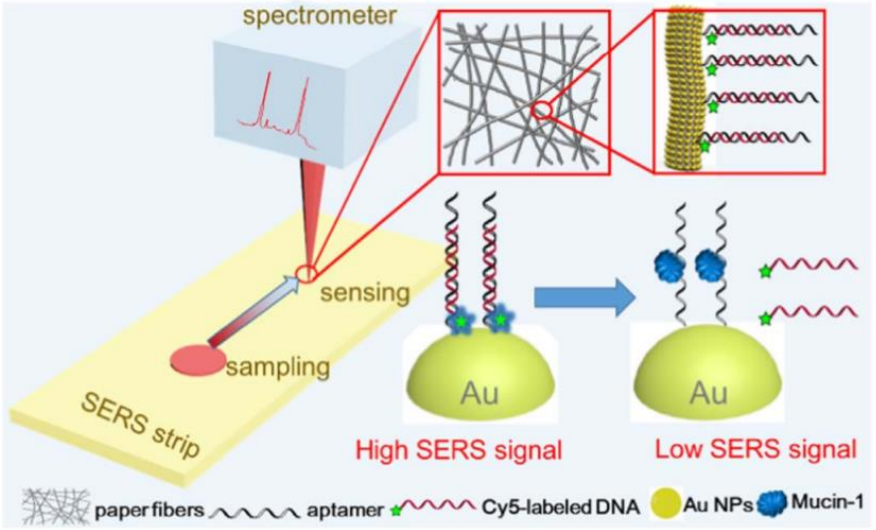


Raman (black) and SERS (red) spectra of pyocyanin bound to a plasmonic gold nanoparticle (red sphere)

Surface-Enhanced Raman Scattering Spectroscopy for Label-Free Analysis of *P. aeruginosa* Quorum Sensing

SERS detection of arsenic in water: A review

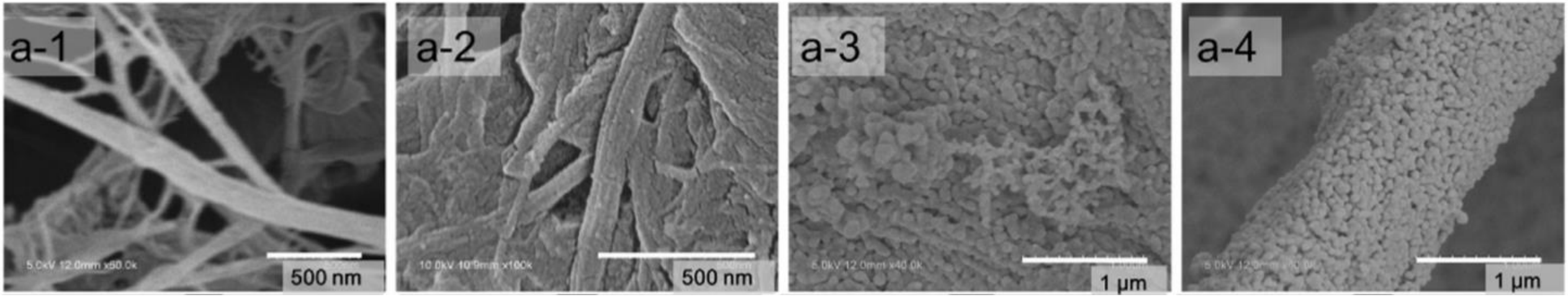
State of art



Scheme 1. Concept and design of the paper-based SERS test strip.

Detection	Detection
Mucin-1	50 ng/mL to 50 µg/mL

Carbon dots synthesized on paper by hydro thermal reaction play an essential role during the following Au NPs deposition



1. The morphology and hydrophilicity control of paper-based SERS strip. (A) SEM images of (a1) bare paper, (a2) carbon dots modified paper, (a3) Au NPs deposited on bare paper, (a4) Au NPs deposited on carbonized paper

A paper-based SERS test strip for quantitative detection of Mucin-1 in whole blood

State of art

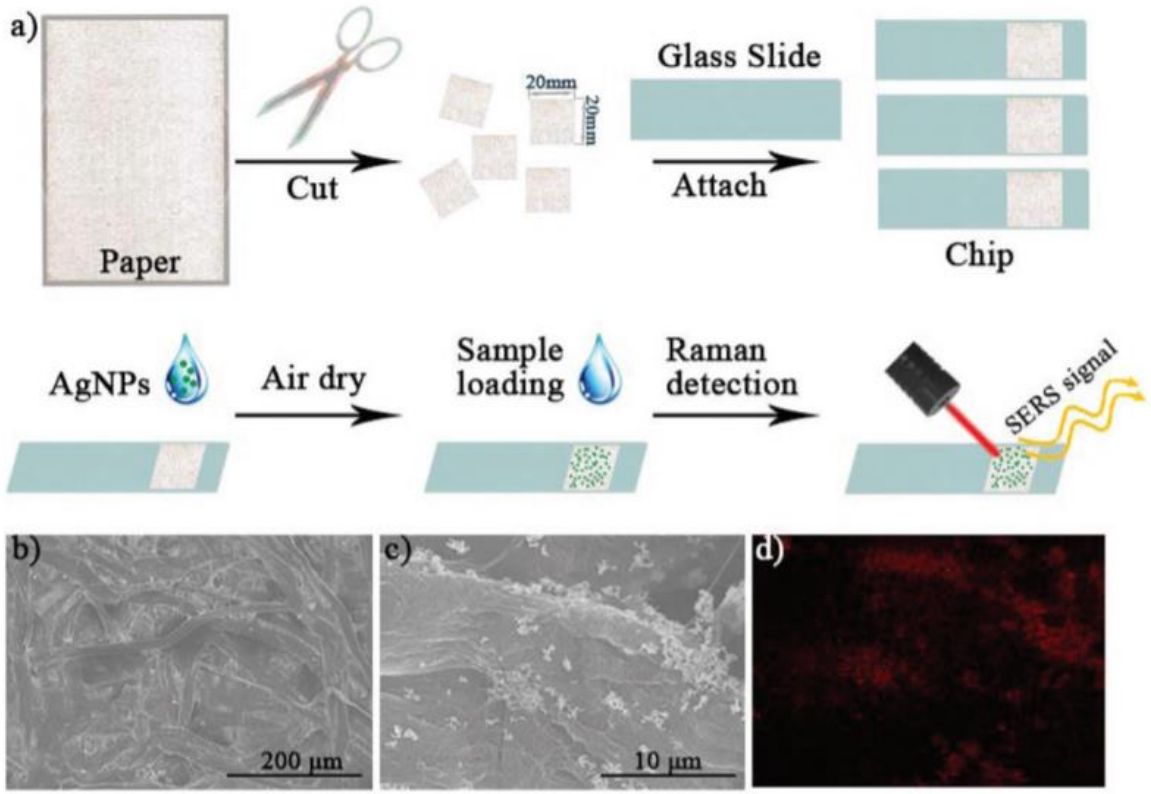


Fig. 3 (a) Schematic illustration of the preparation of the paper based SERS chips and Raman measurement; (b) SEM image of the surface morphology of the filter paper (b) before and (c) after addition of AgNPs; (d) EDX mapping of the Ag element distributed on the paper chip shown in (c).

Detection	Detection
Crystal violet	10^-1 M – 10^-2 M
RDM 6G	10^-5 M

State of art

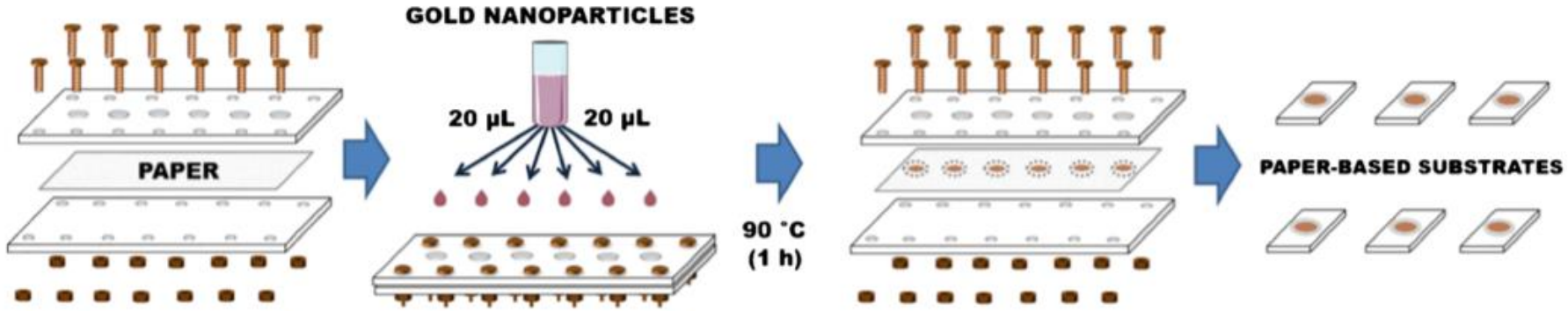


Fig. 1 Procedure for rapid preparation of paper-based SERS substrates

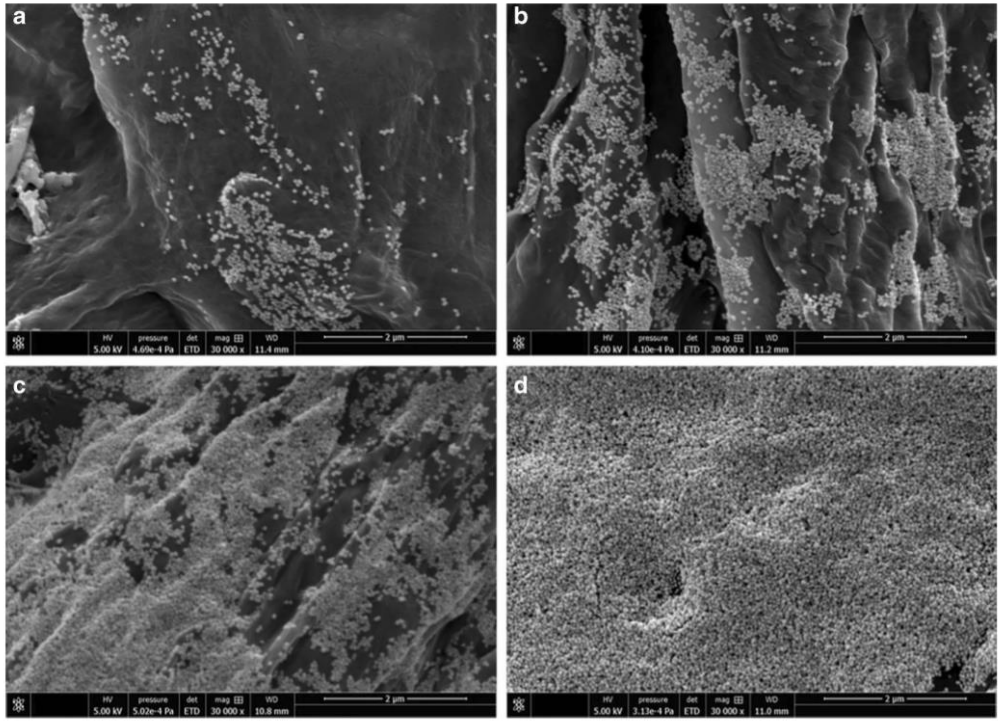
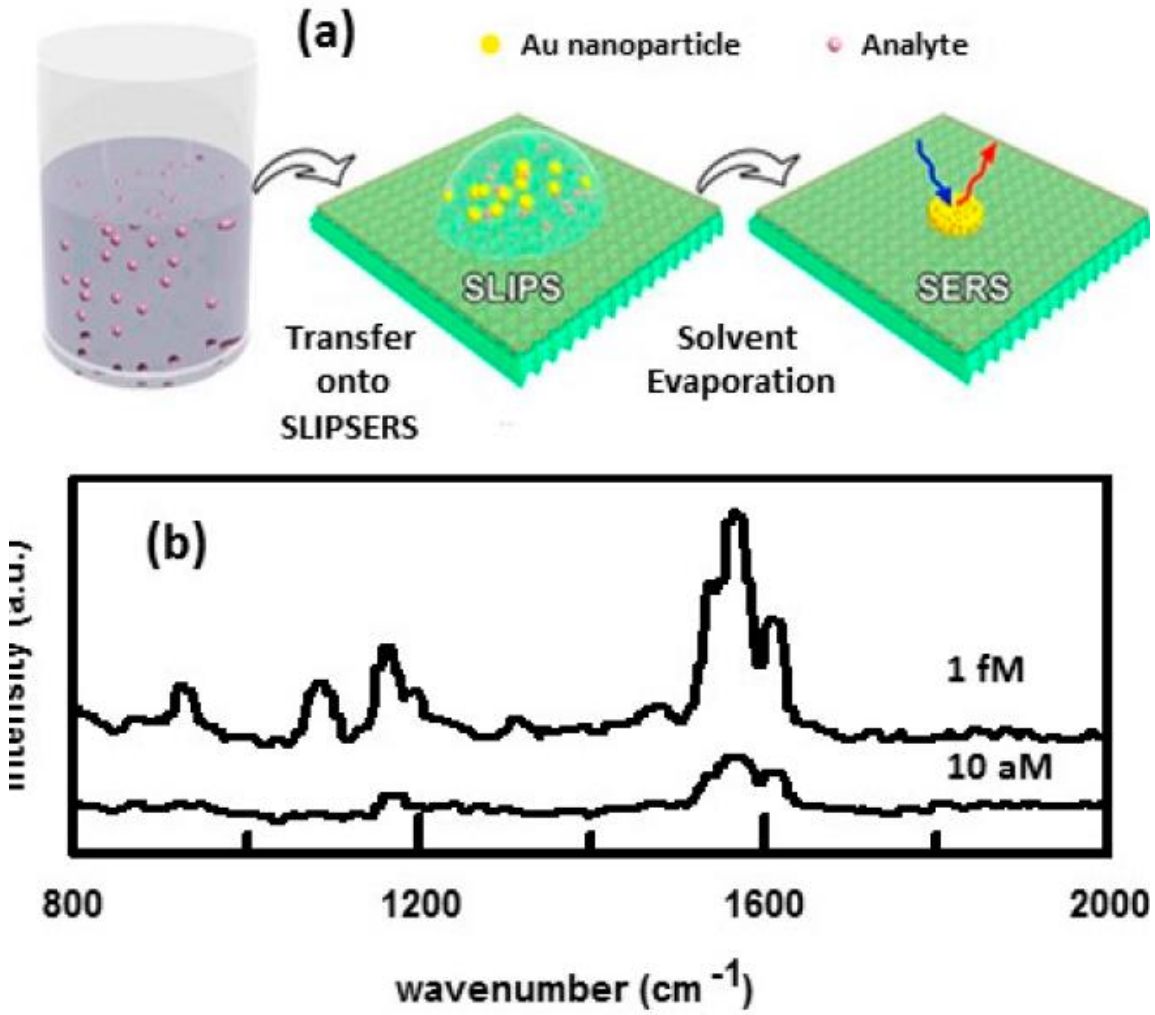


Fig. 3 FE-SEM studies for the paper-based substrates prepared with colloidal GNPs after **a** 3-fold dilution, **b** no preconcentration, **c** 10-fold preconcentration and **d** 50-fold preconcentration

Detection	Detection
nicotine	0,02 mg/L
Uric acid	0,03 mg/L

Fabrication of gold nanoparticle-coated paper and its use as a sensitive substrate for quantitative SERS analysis

State of art

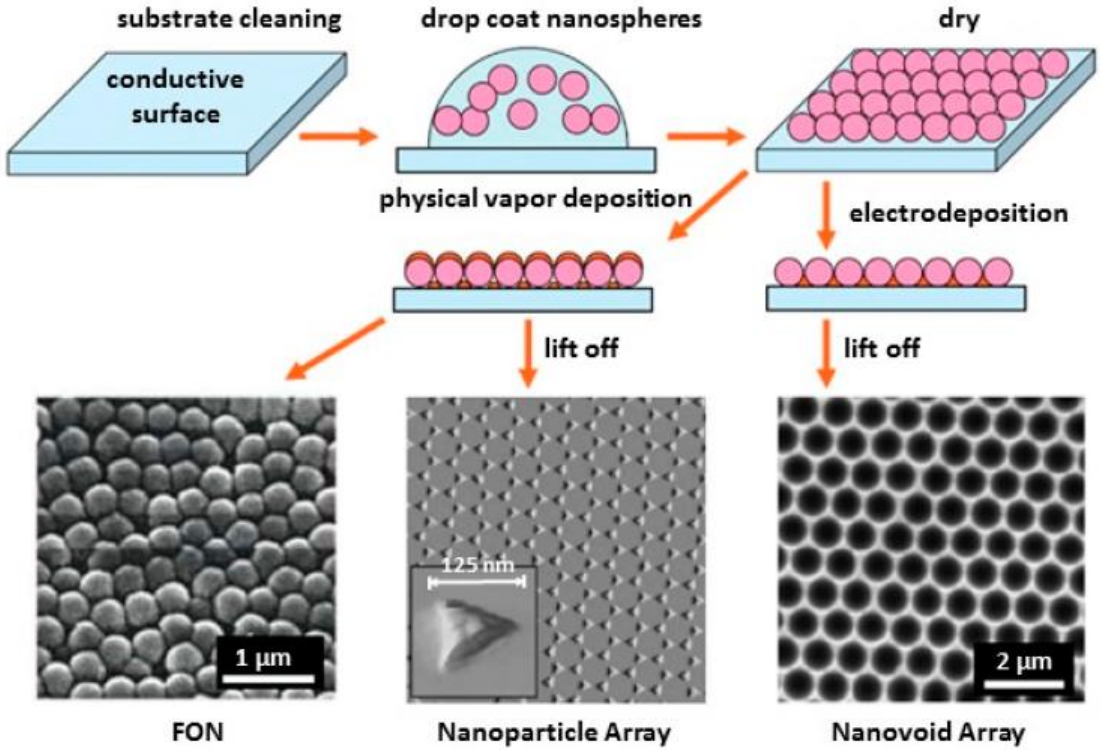


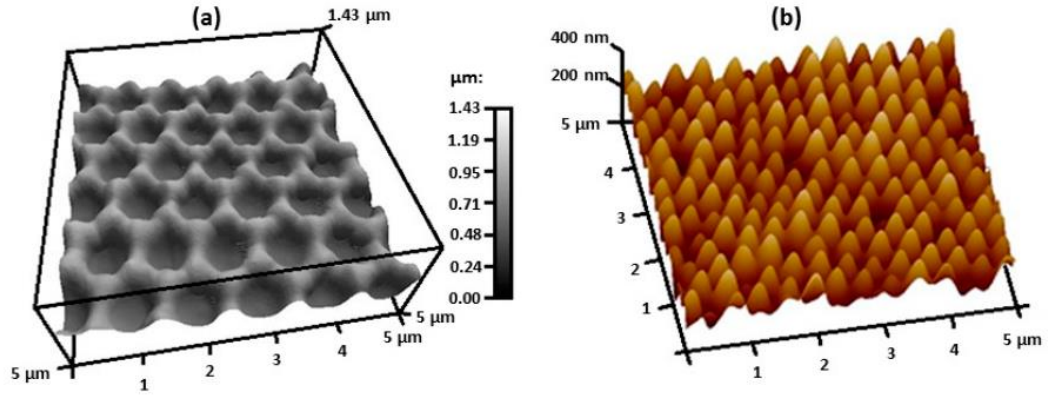
A slippery liquid-infused porous surface (SLIPS) consists of a film of lubricating fluid locked in place by a micro/nanoporous substrate that creates a smooth and stable interface that nearly eliminates pinning of the liquid contact line.

Slippery liquid-infused porous surface-enhanced Raman scattering (SLIPSERS)

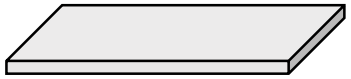
State of art

Nanosphere lithography process

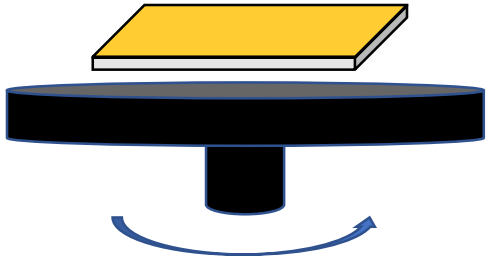




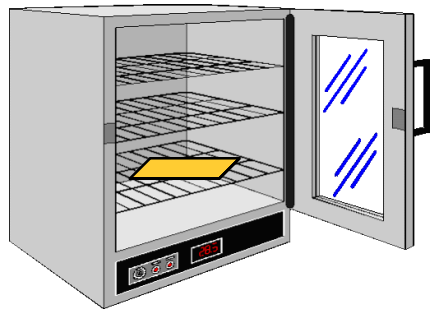
Lithography



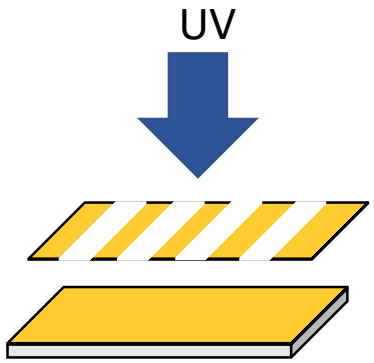
Cleaning



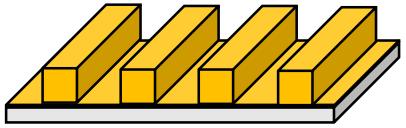
Resist deposition
and spin-coating



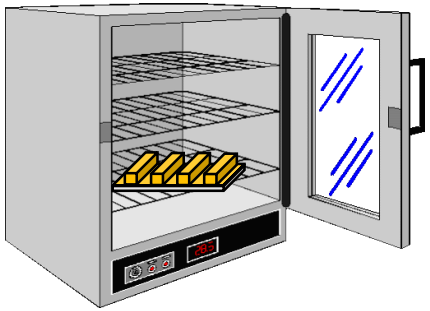
Pre Baking



UV exposition

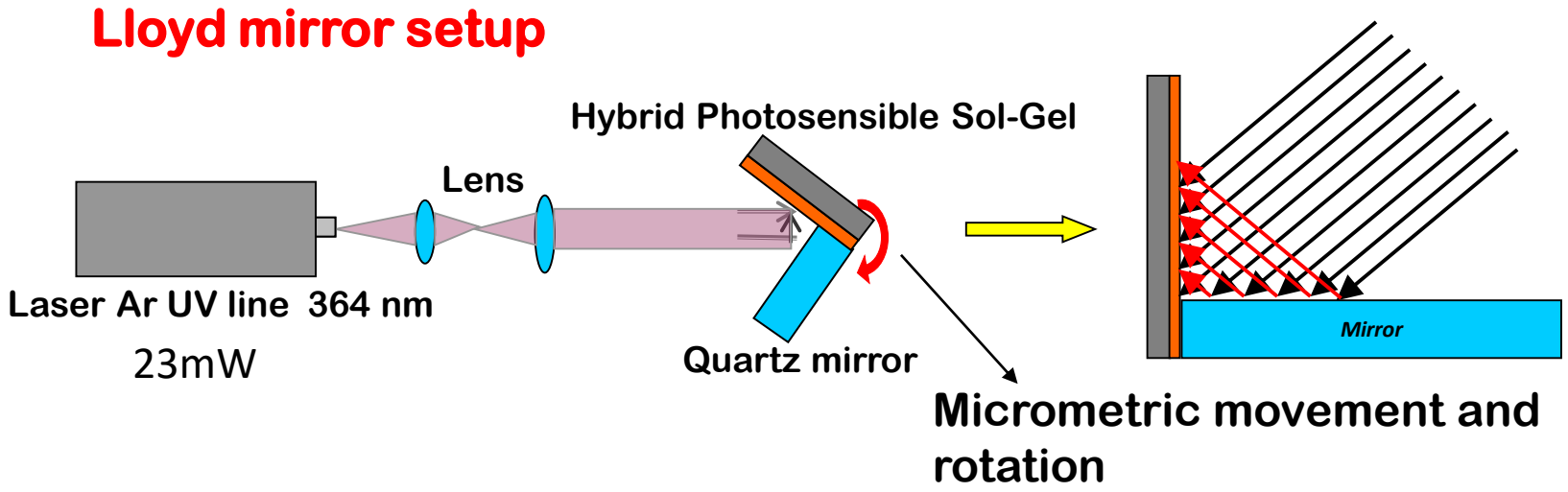


Developement



Post Baking

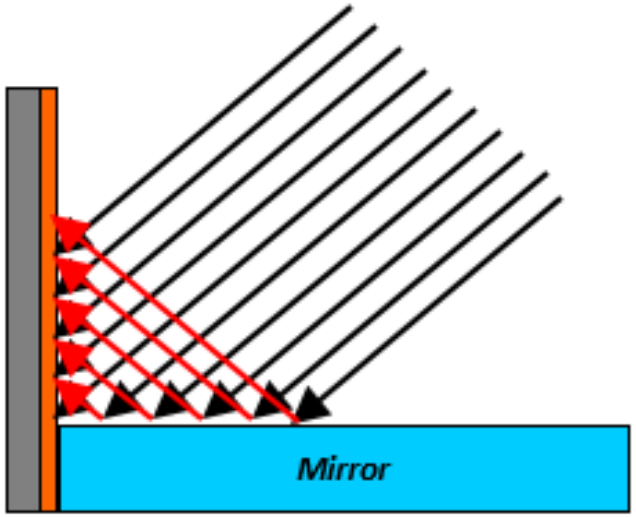
Double exposition laser interference lithography (DE-LIL)

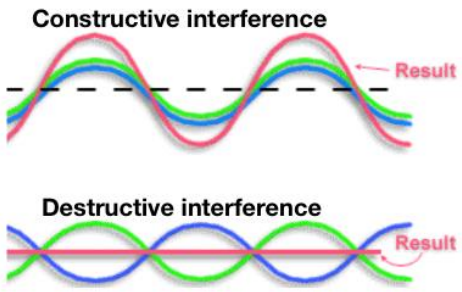


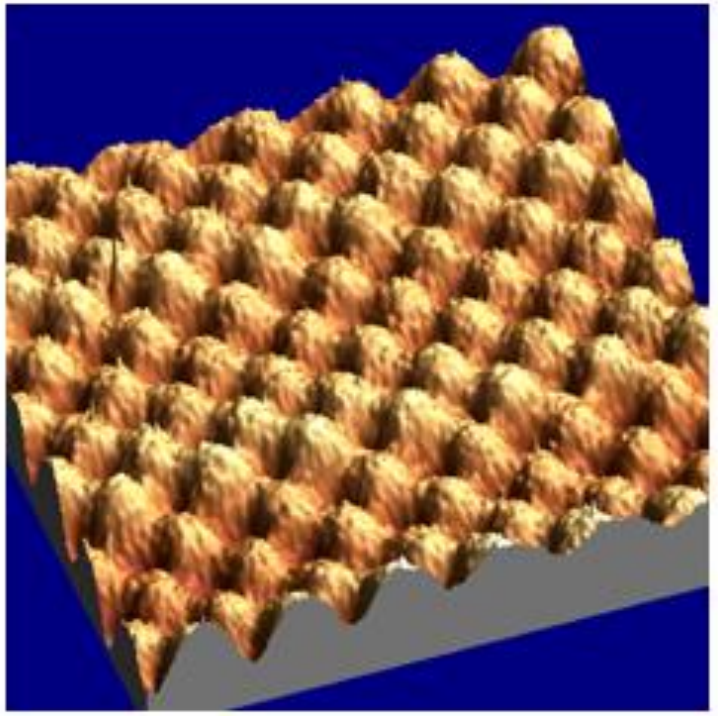
photosensitive Sol-gel (Ti / TMSPM) illuminated with Lloyd's mirror in two phases. First exposure at 0 ° angle and subsequent exposure at 90 ° with respect to the first.

TMSPM(3-(Trimethoxysilyl)propyl methacrylate

Double exposition laser interference lithography (DE-LIL)

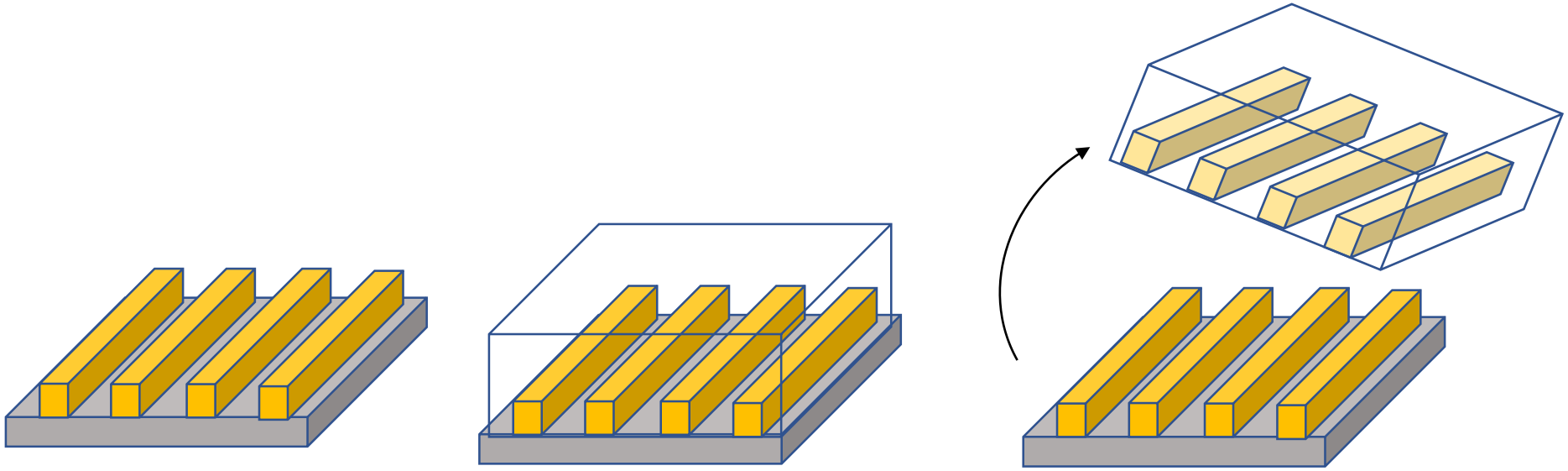






photosensitive Sol-gel (Ti / TMSPM (3-(Trimethoxysilyl)propyl methacrylate)

Soft lithography – master replication



MASTER FROM LIL

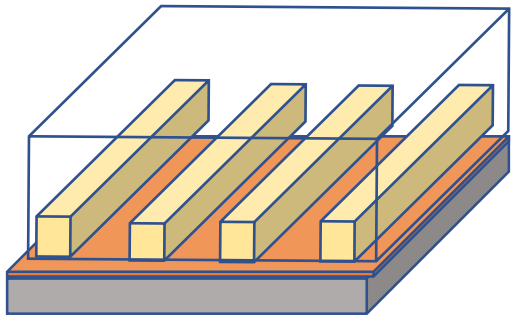
PDMS pouring

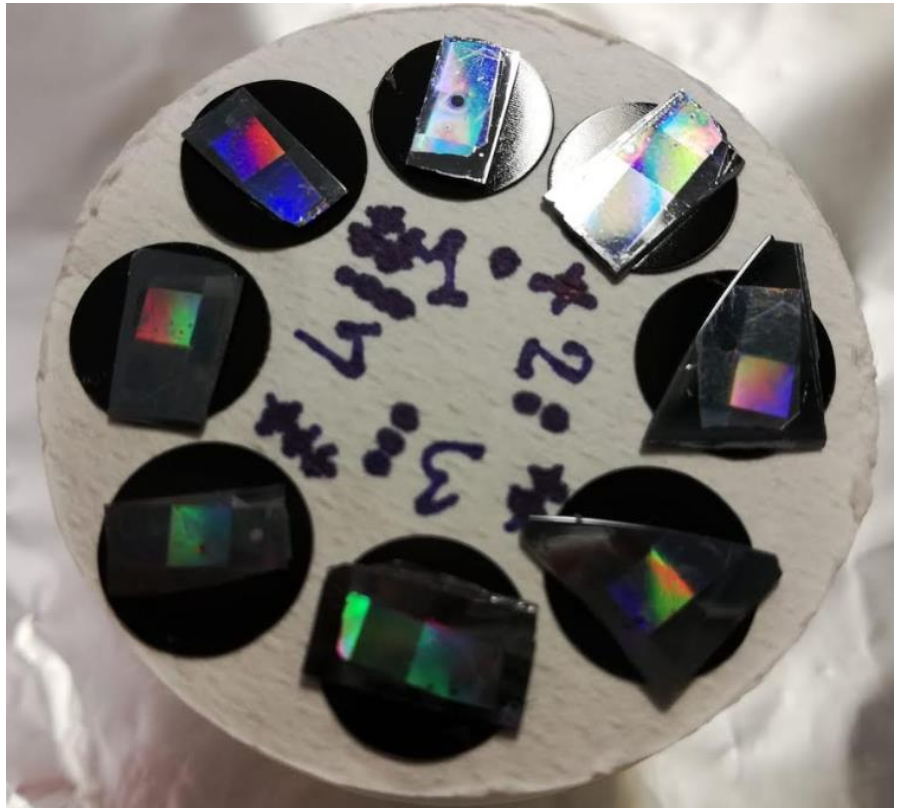
peeling

PDMS(Polydimethylsiloxane) mold can replicate nanostructures

3D - grating

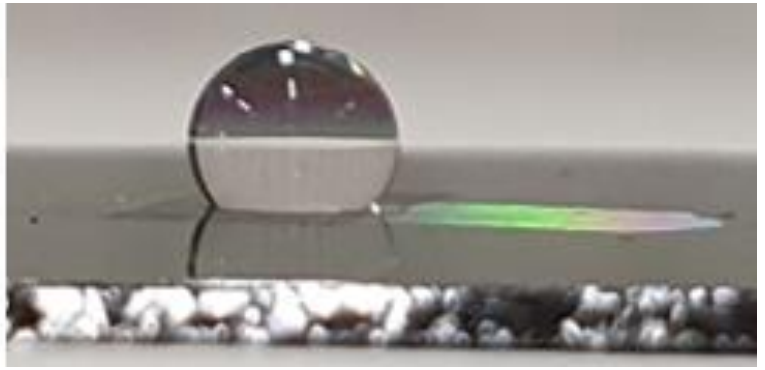
UV





Bagnability

No 3D grating



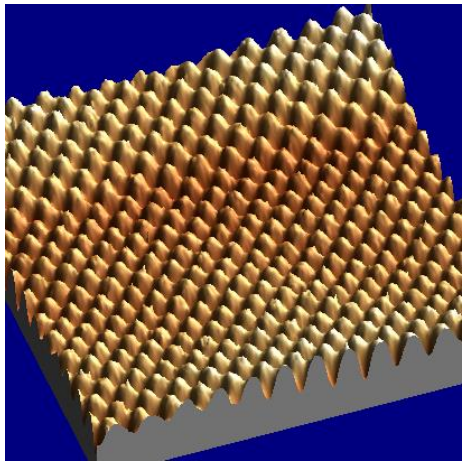
3D grating

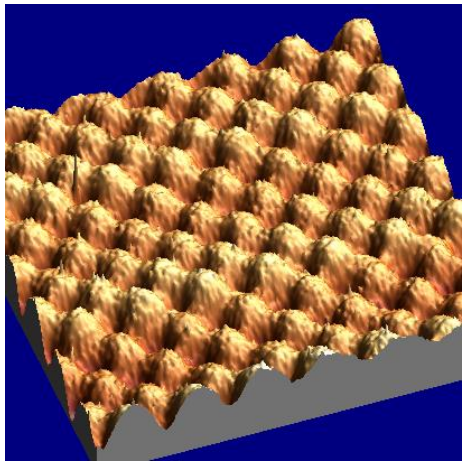
Morphological characterization

AFM

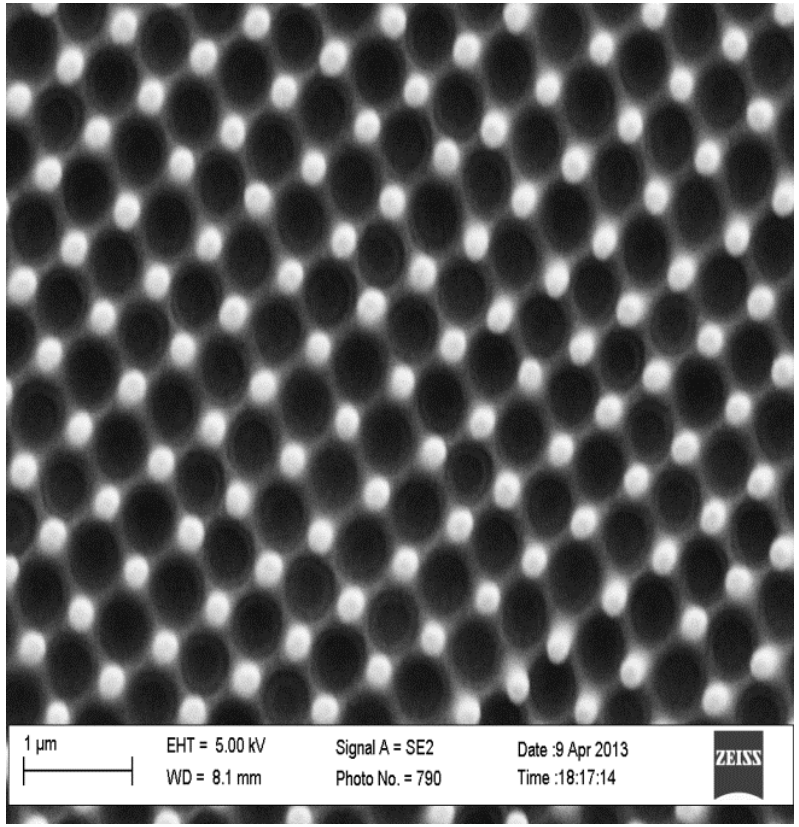
SEM

2.0µm

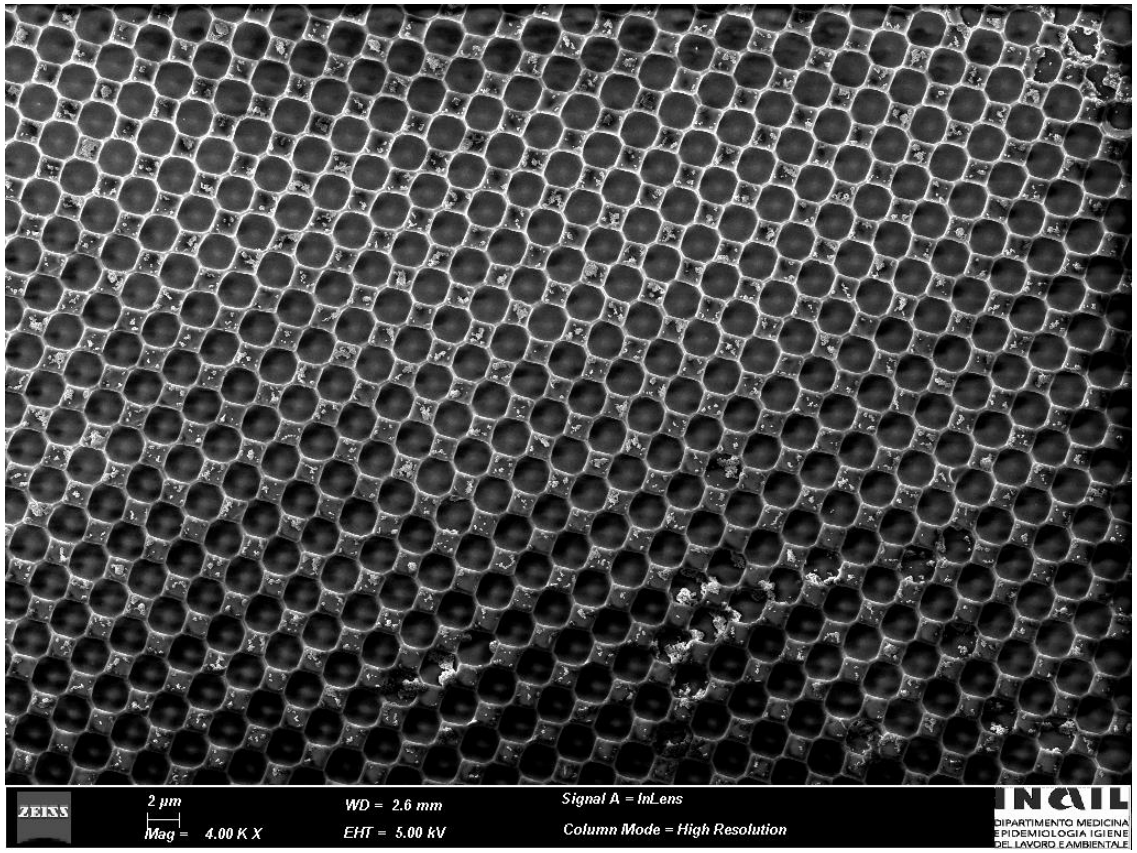


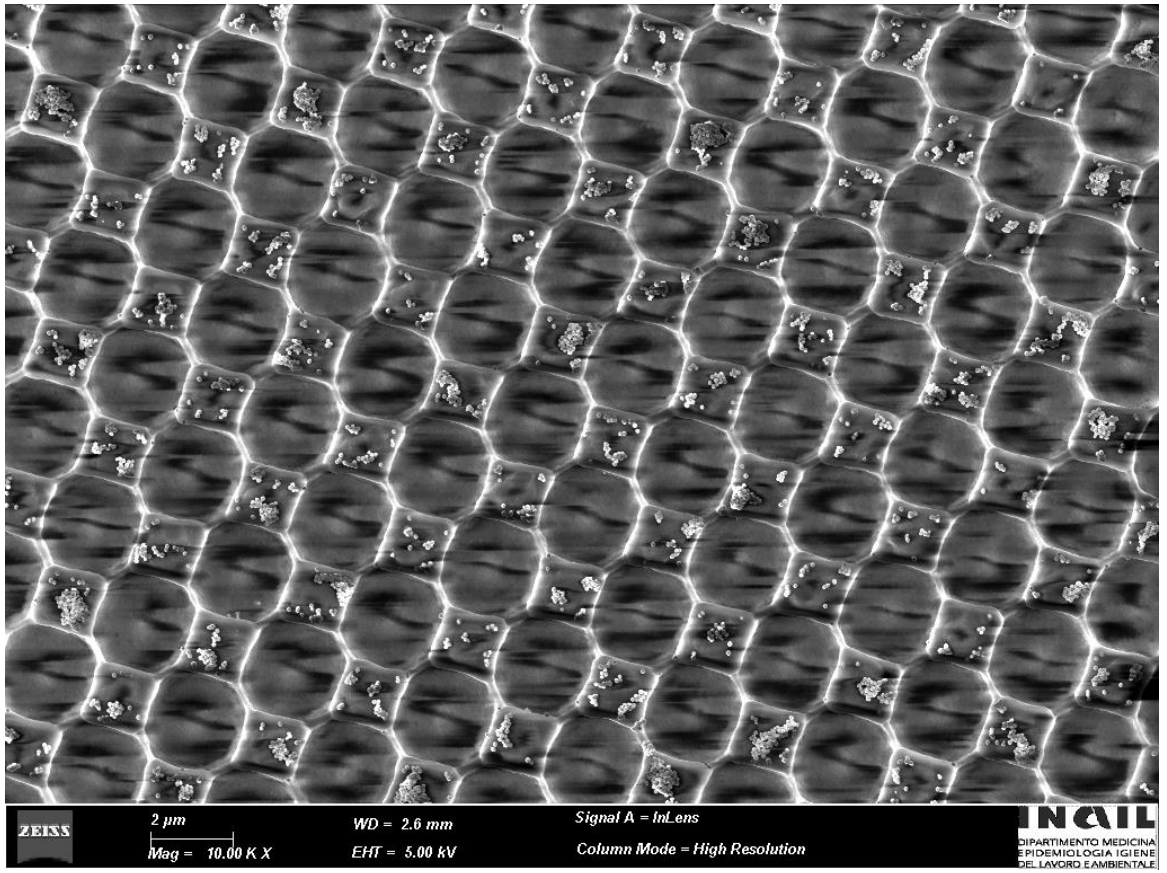


h = 100-200 nm



SEM images

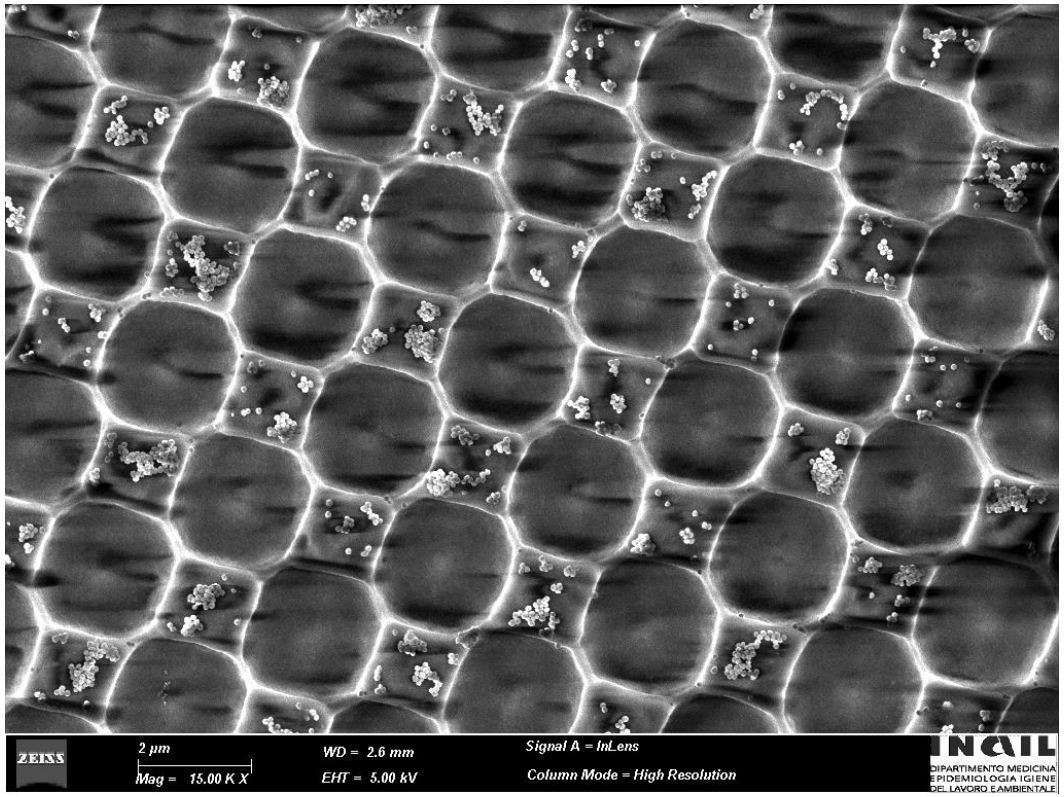


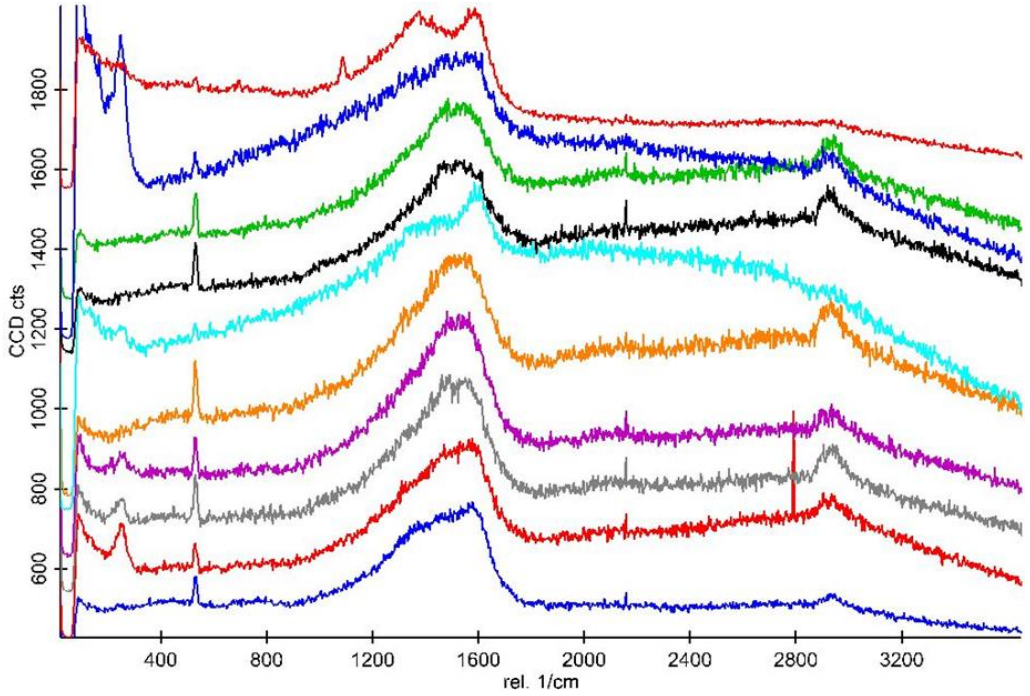


- Ti/TMSPM grating
- Ag NPs 100 nm

- Ag NPs concentrate0,06M
- Slow evaporation of water

SEM images


ZEISS
2 µm
Mag = 15.00 K X
WD = 2.6 mm
EHT = 5.00 kV
Signal A = InLens
Column Mode = High Resolution
INAIL
DIPARTIMENTO MEDICINA EPIDEMIOLOGIA IGIENE DEL LAVORO E AMBIENTALE


CCD cts
600
800
1000
1200
1400
1600
1800
400
800
1200
1600
2000
2400
2800
3200
rel. 1/cm

Conclusions and future prospects

Realization of a substrate for AgNPs deposition

Increase AgNPs concentration

As detection test

Thanks to:
NeMO laboratories of Tor Vergata and NARAs project

Teams: Erica Ciotta, Thu Ha Dao, Fabio De Matteis, Roberto Francini, Paolo Prosposito, Mauro Casalboni
Thanks to Dr. Stefano Casciardi

http://webnemo.uniroma2.it/Nemo/

NEW MATERIALS FOR OPTOELECTRONICS

LAZIO INNOVA

NARAs

NARAs - Nanotecnologie **A**pplicate alla **R**ilevazione di **As** nelle acque

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