instituto imdea nanociencia

EXCELENCIA SEVERO OCHOA

Nanoimprint lithography and hybrid processes

to produce complex nanostructures

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EUROPEAN UNION
STRUCTURAL FUNDS

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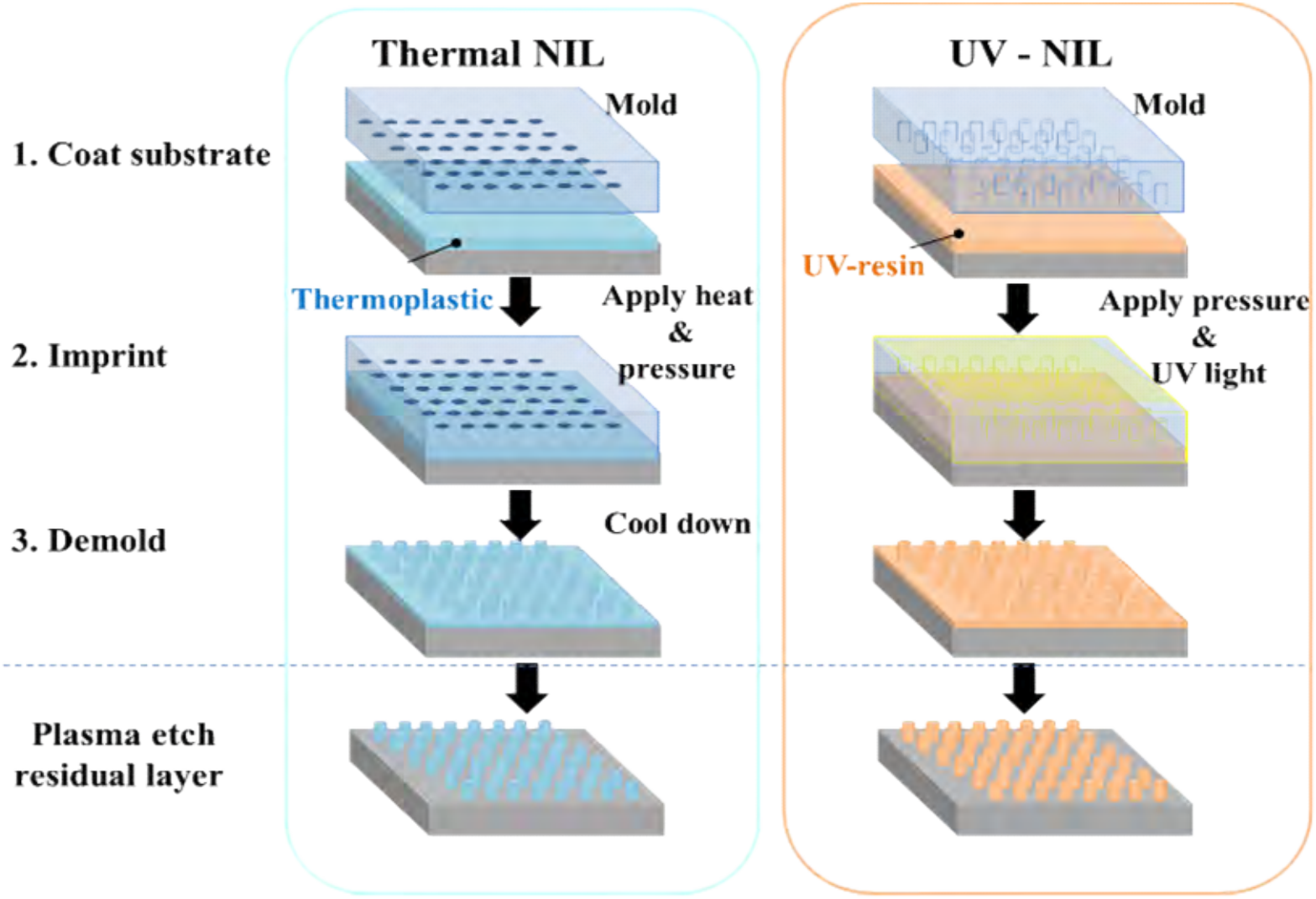
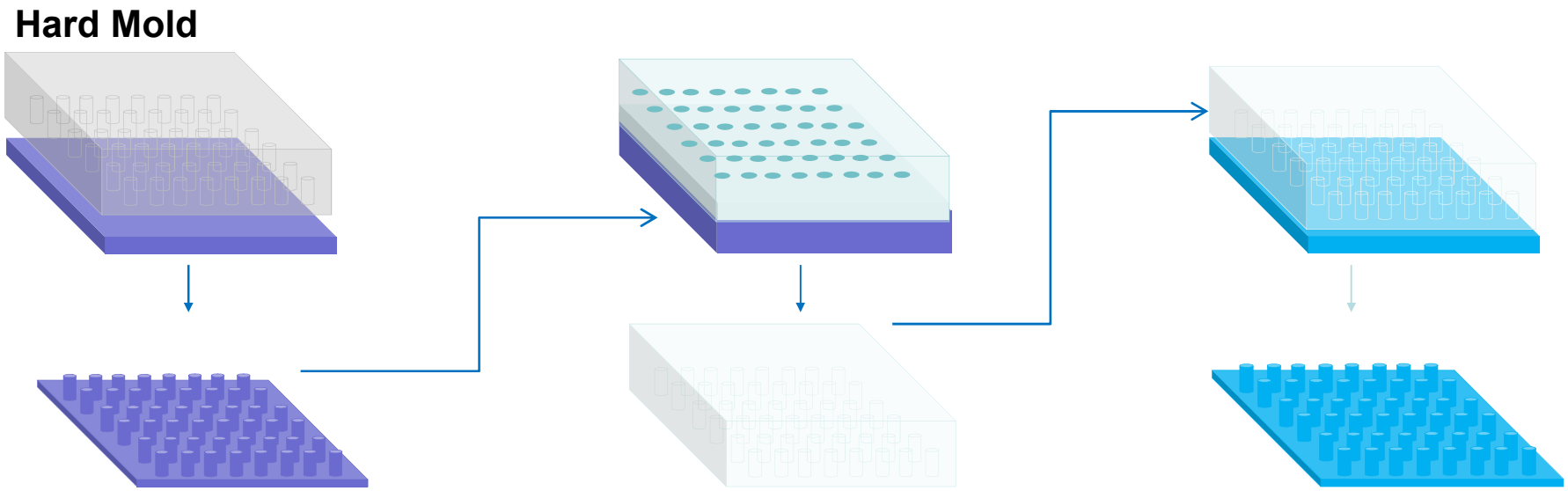
Nano Imprint Lithography - NIL

A replication process : Pattern transfer by mechanical deformation of a flowing resist material



Soft –NIL

Thermal-NIL | **Soft lithography** | **Soft NIL**



IPS
Intermediate polymer stamp

Soft Mold
PDMS
PFPE

Patterned polymer

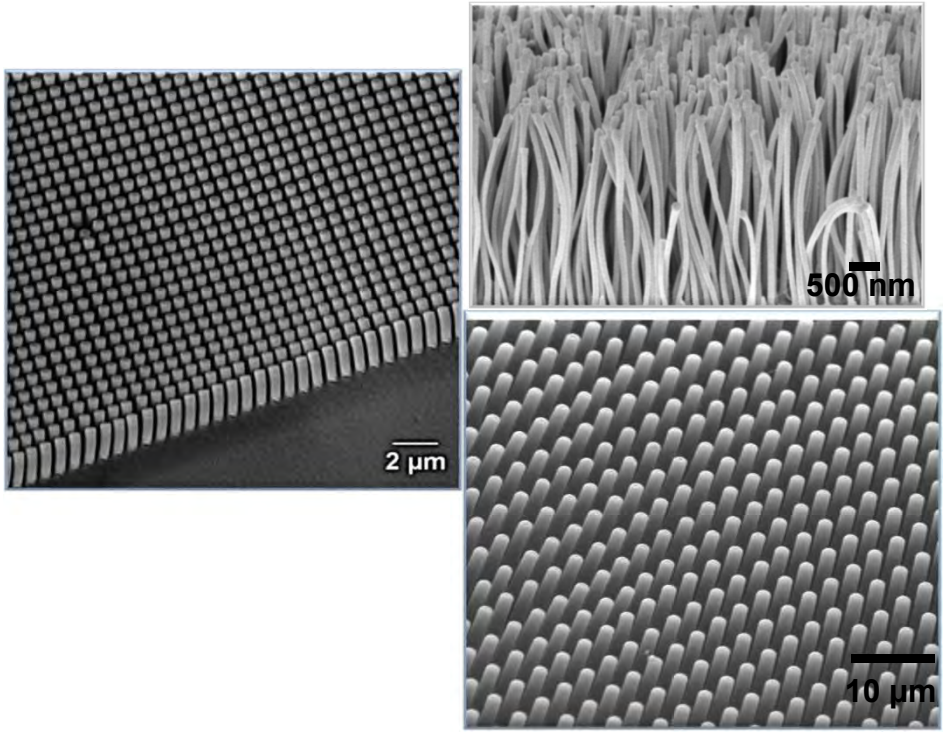
Soft –NIL:

- Sequential action of peeling the mold from substrate
- High gas permeability
- Conformal contact to substrate
- Low surface energy

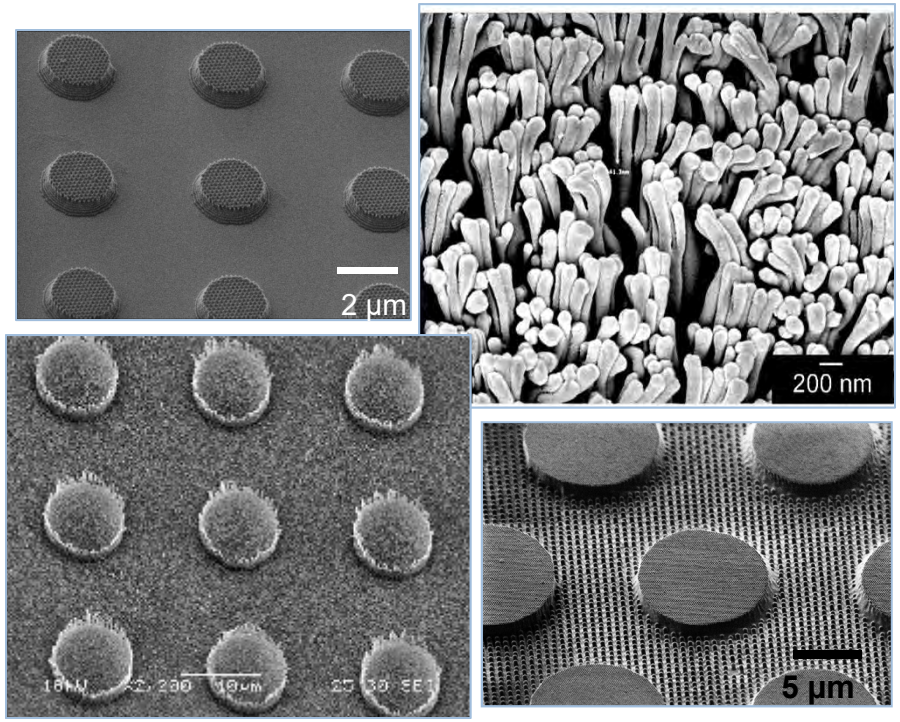
PDMS: Polydimetilsiloxane
PFPE: Polyurethane based on perfluoropolyether

Key Technological Expertise

High Aspect Ratio Topographies



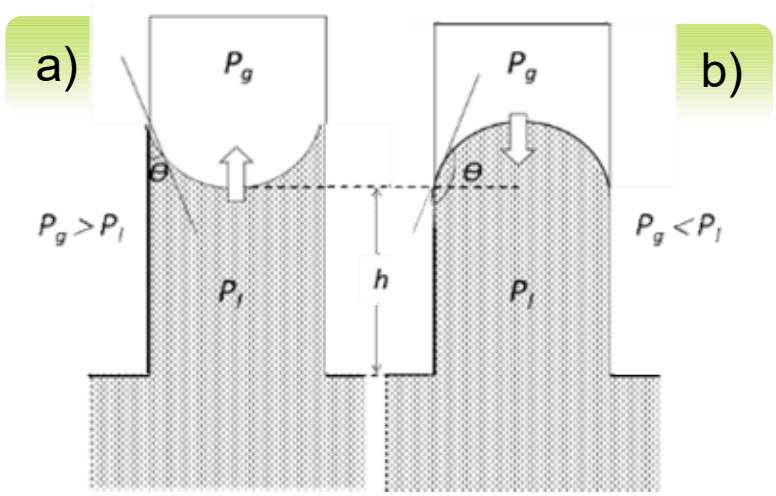
Complex Hierarchical Topographies

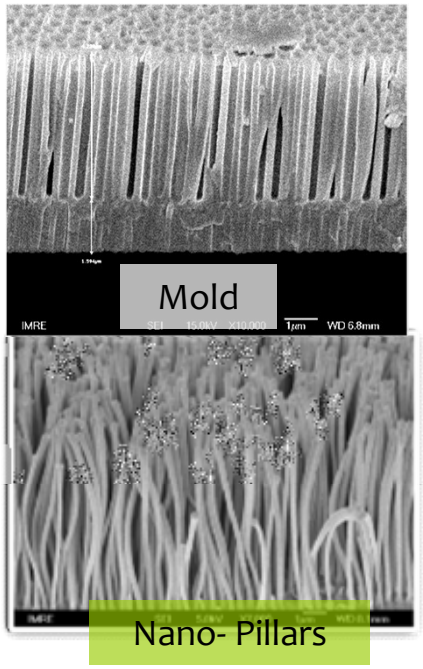


High Aspect Ratio NIL

Laplace pressure - Capillary forces

$$\Delta P_c = P_g - P_l = \frac{2\,\gamma cos\theta}{r}$$





a) polymer wets the capillary (θ < 90°)

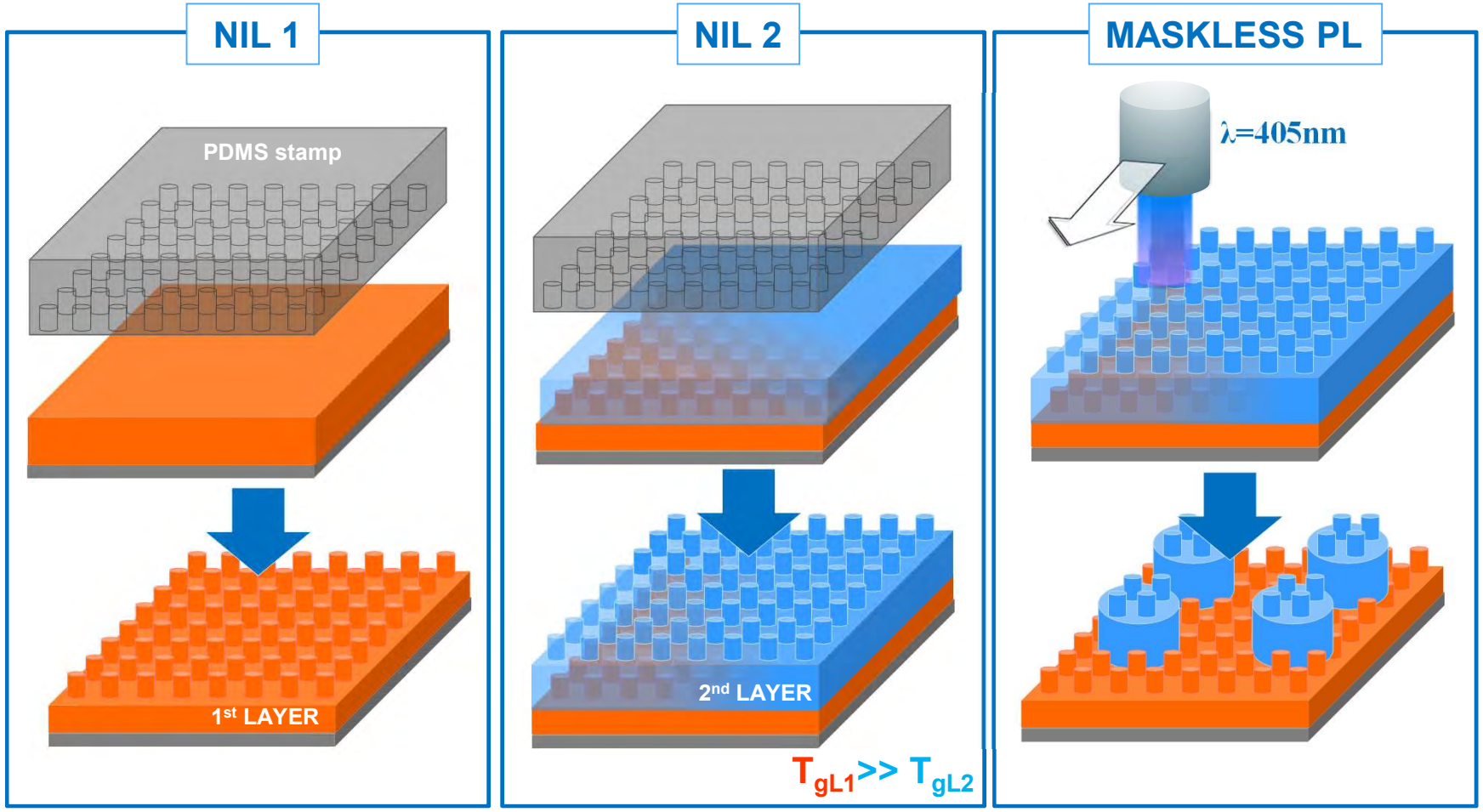
b) polymer does not wet the capillary (θ > 90°)

Lucas-Washburn equation:

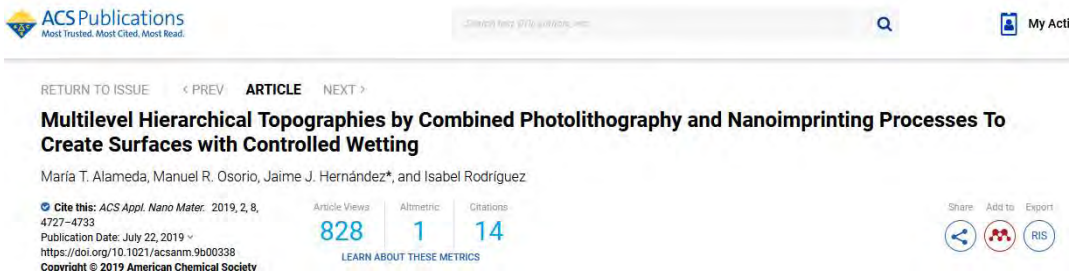
$$\frac{dh}{dt} = \frac{r\,\gamma cos\theta}{4\eta h} \qquad t = \frac{2\,\eta\,h^2}{r\,\gamma\,cos\theta}$$

γ_p - surface tension of the viscous polymer
η - the viscosity
θ - the solid-liquid contact angle
r - the capillary radius.

Hybrid fabrication proccess: NIL + PL



NIL: Nanoimprint Lithography ; PL: Photolithography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Multilevel Hierarchical Topographies by Combined Photolithography and Nanoimprinting Processes To Create Surfaces with Controlled Wetting

María T. Alameda, Manuel R. Osorio, Jaime J. Hernández*, and Isabel Rodríguez

Cite this: *ACS Appl. Nano Mater.* 2019, 2, 8, 4727−4733
Publication Date: July 22, 2019
https://doi.org/10.1021/acsanm.9b00338
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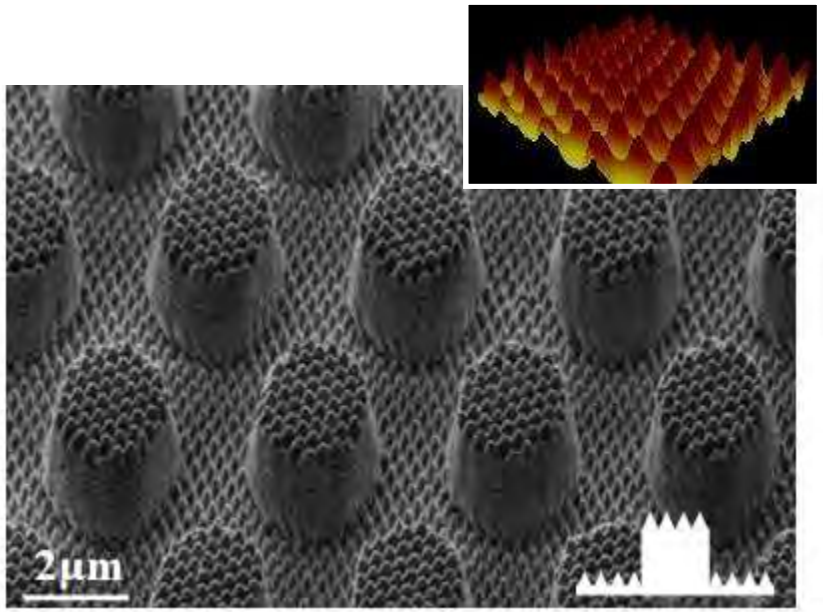
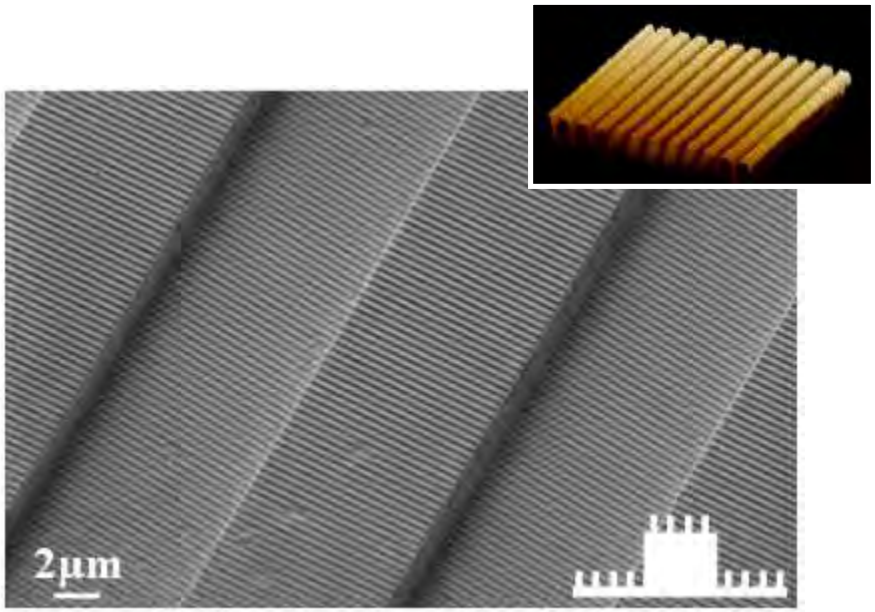
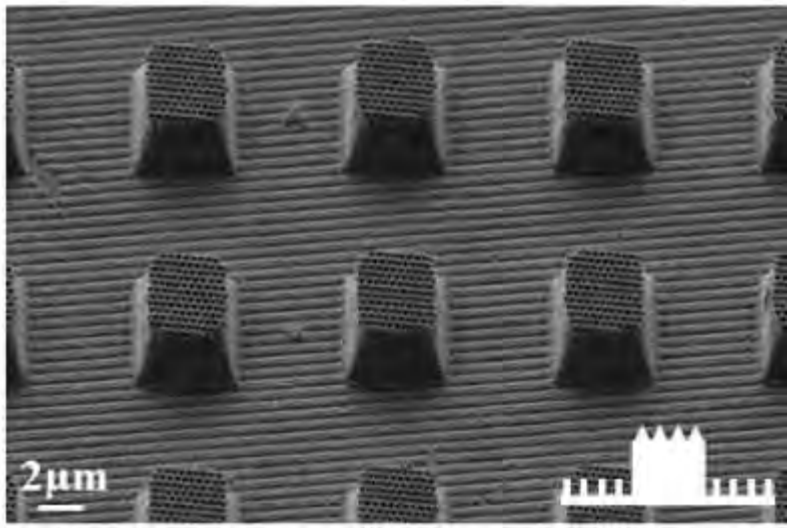
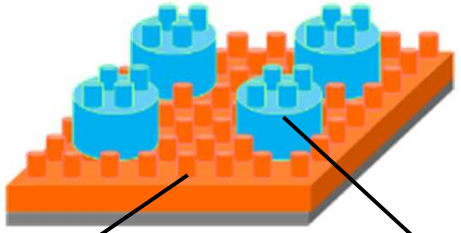
Micro & Nano Hierarchical Structures

1ST LAYER: THERMOPLASTIC







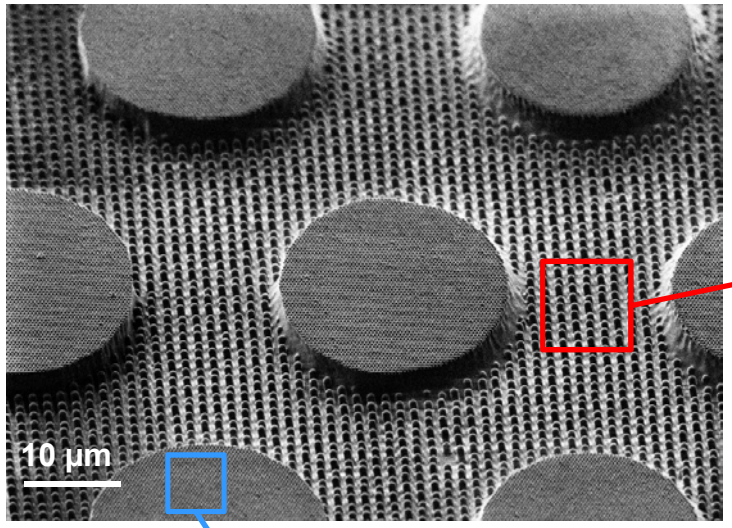


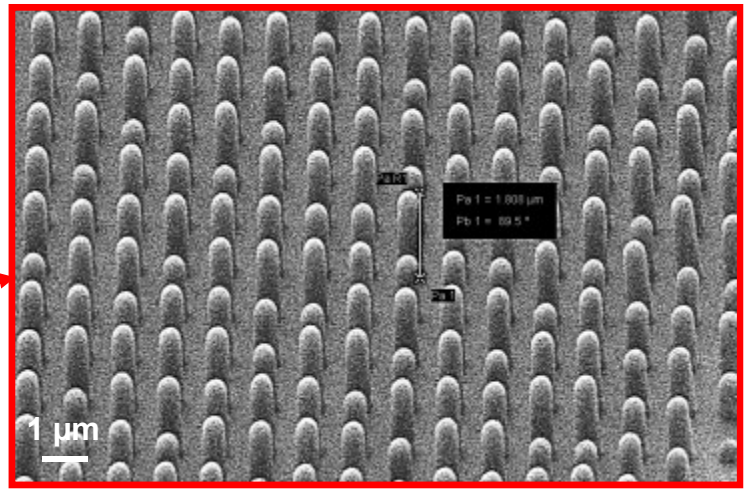
PMMA-A4
T_g=98-106ºC
T_{NIL}=170ºC
P_{NIL}=30bar
t=5min

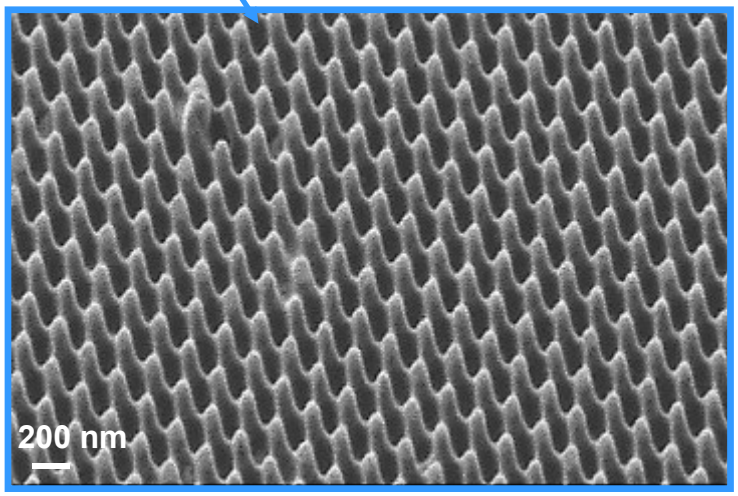
AZ1512
S.C: 2000rpm, 1min
T_{NIL}=100ºC
P_{NIL}=30bar
t=5min

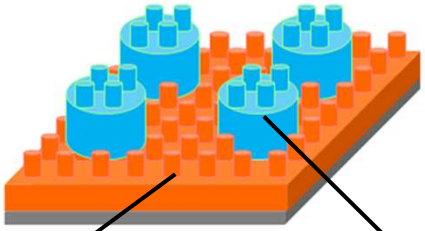
Micro & Nano Hierarchical Structures

1ST LAYER: THERMOSET





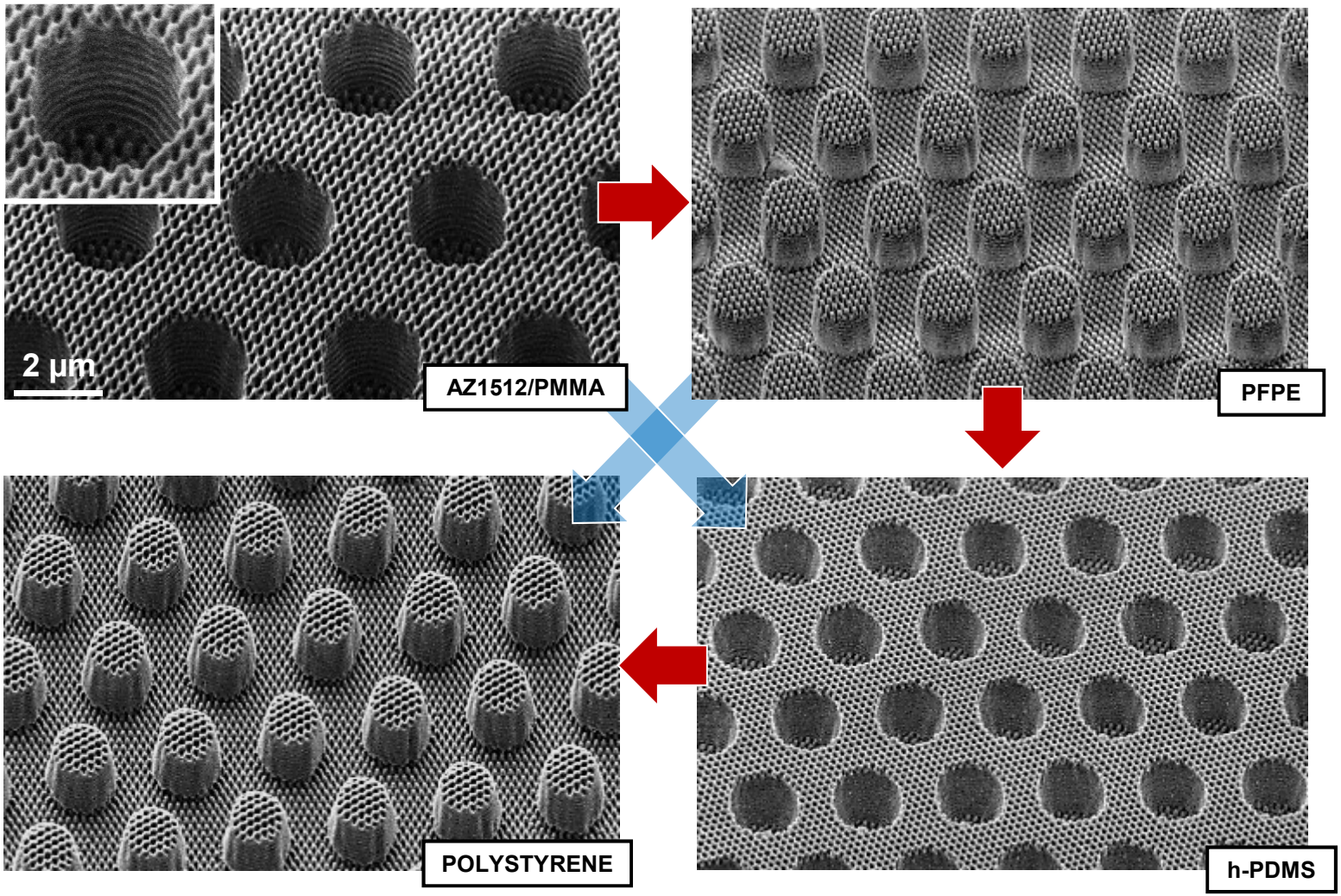




EpoTek
$T_{UV\text{-}NIL}$=60ºC
P_{NIL}=30bar
t=5min

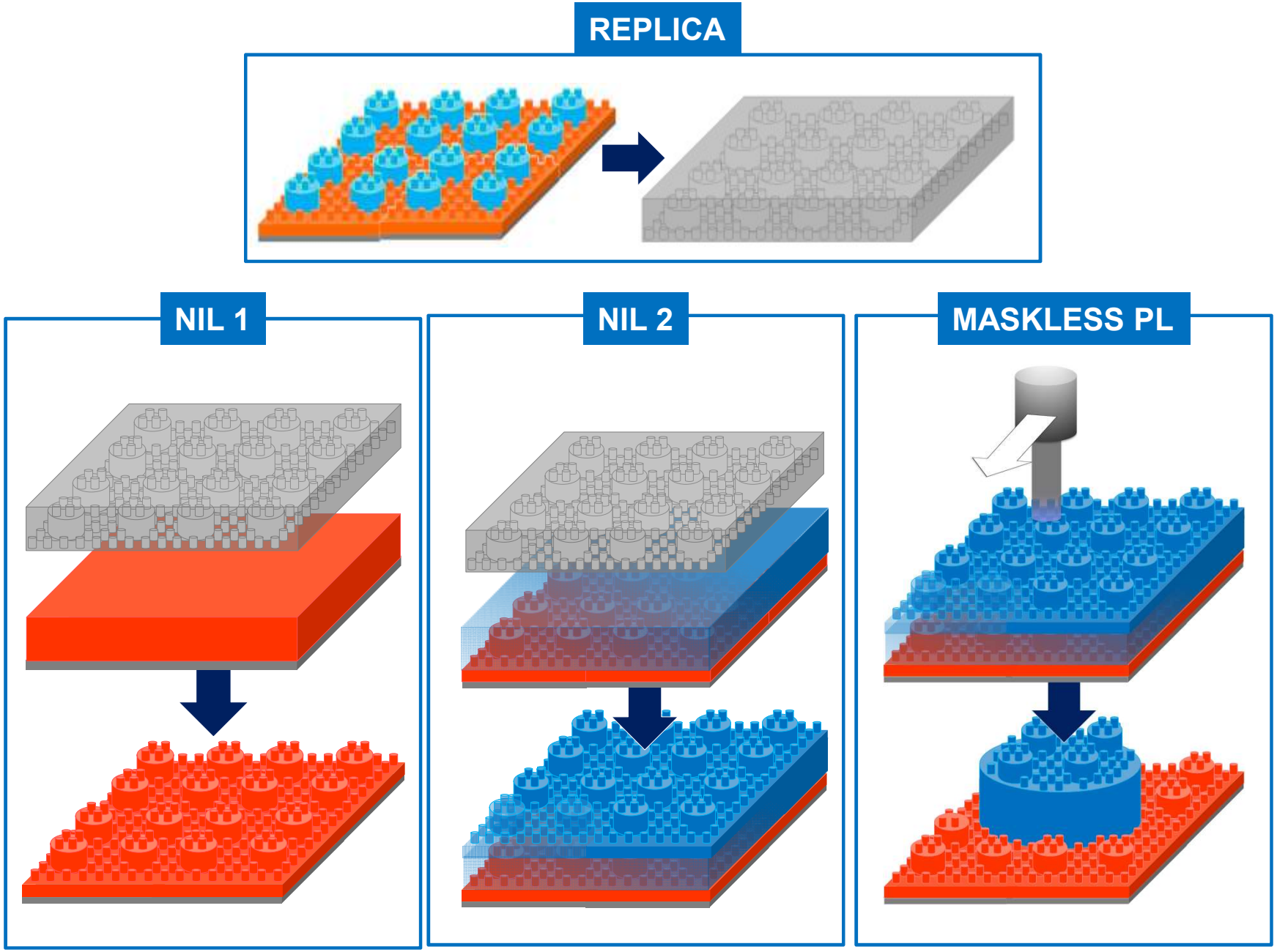
AZ1512
S.C: 2000rpm, 1min
T_{NIL}=100ºC
P_{NIL}=30bar
t=5min

Soft-NIL - Polymer replication

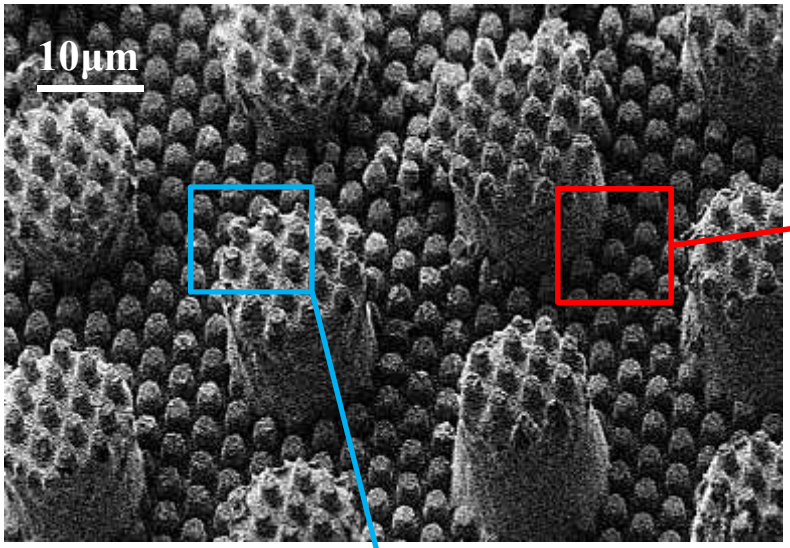


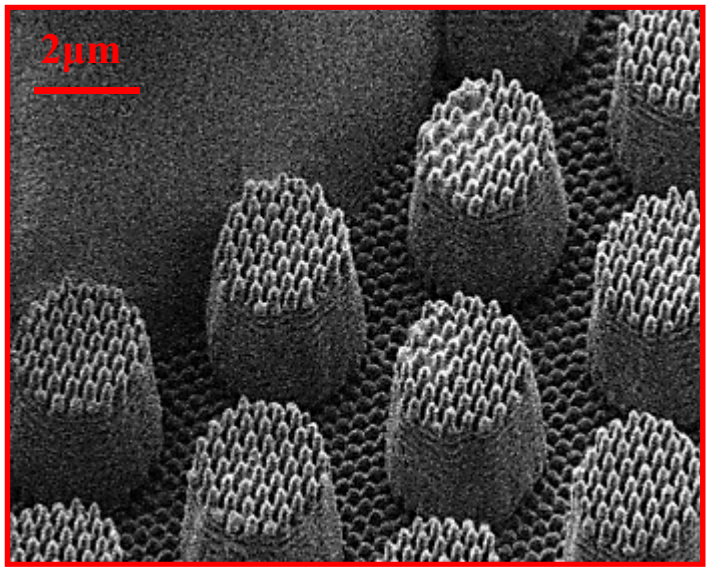
PFPE - Perfluoropolyether
PMMA - Poly(methyl methacrylate)
PDMS - Polydimethylsiloxane

Multilevel Hierarchical Structures

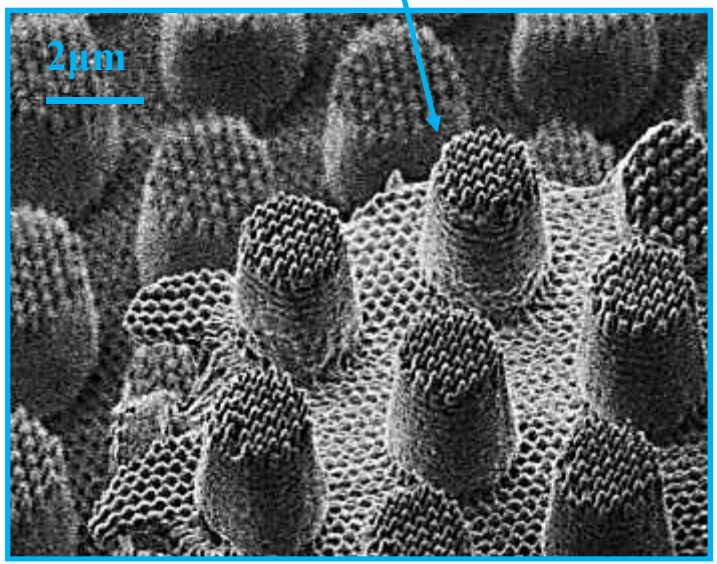


Multilevel Hierarchical Structures

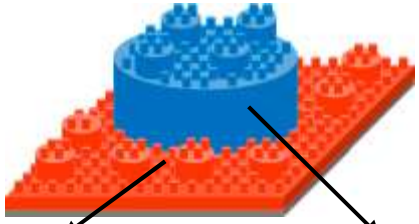




BOTTOM



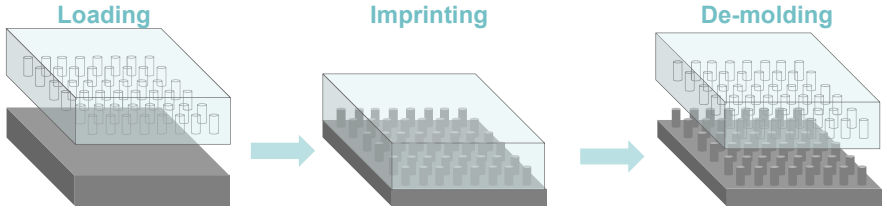
TOP



EpoTek
$T_{UV\text{-}NIL}$=60ºC
P_{NIL}=30bar
t=5min

AZ 9260
S.C: 2000rpm, 1min
T_{NIL}=110ºC
P_{NIL}=30bar
t=5min

Nanoimprint Lithography (NIL) Tools



3” wafers - UV & Thermal NIL
Thermosets & thermoplastic materials

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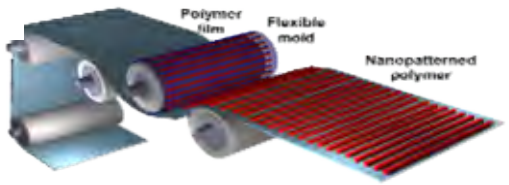
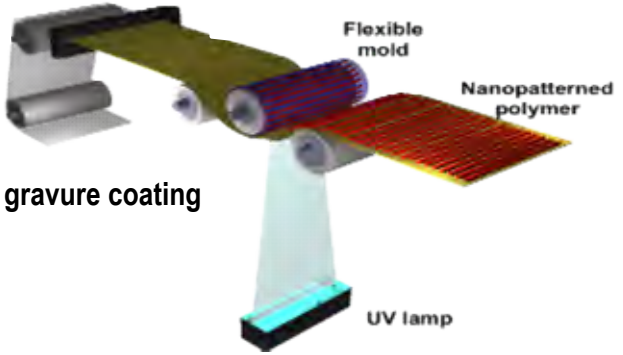
Thermal -NIL



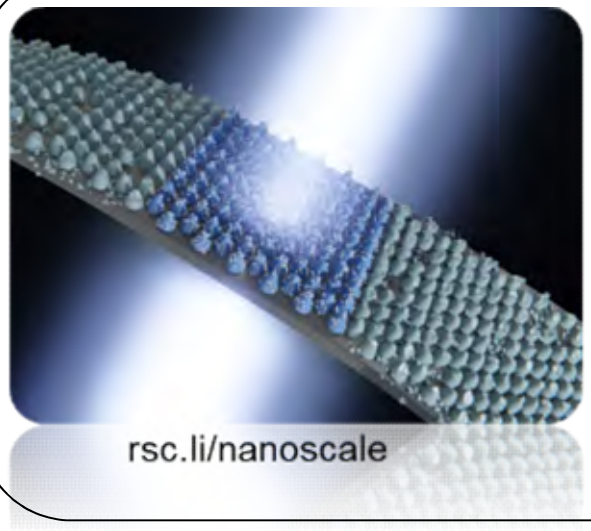
UV-NIL



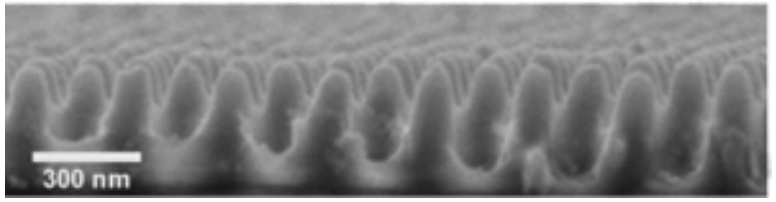
Reverse gravure coating

Roll to Roll NIL - Pilot facility for scale-up processing (5 m/min)

Moth-eye biomimetic anti- reflective surfaces



Moth-eye TiO_2 reinforced surface nanocomposite nano patterns



Nanoscale

PAPER

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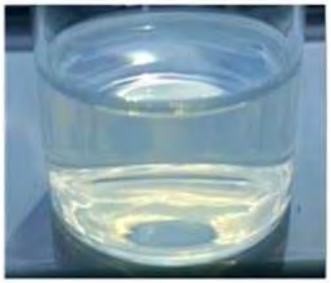
Single-imprint moth-eye anti-reflective and self-cleaning film with enhanced resistance†

Iván Navarro-Baena, Alejandra Jacobo-Martín, Jaime J. Hernández, Jose R. Castro Smirnov, Felipe Viela, Miguel A. Monclús, Manuel R. Osorio, Jon M. Molina-Aldareguia and Isabel Rodriguez

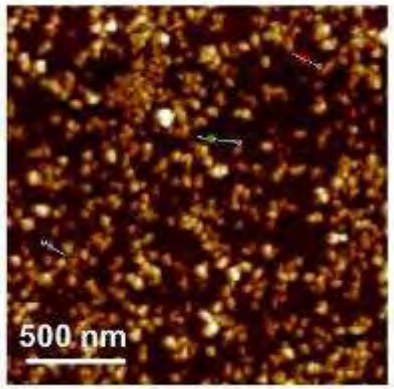
TiO_2 Surface Nanocomposite

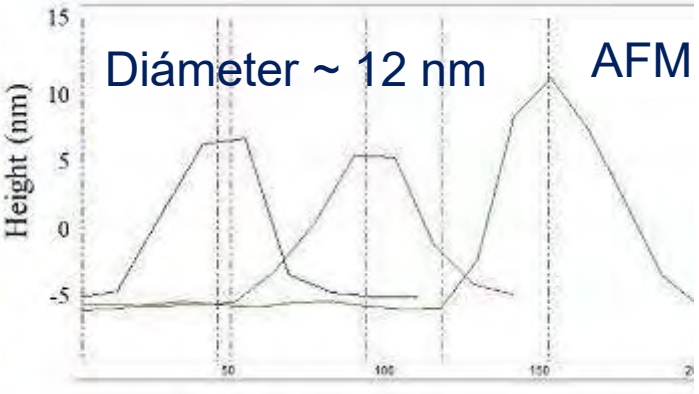
Preparation of polymer nanocomposite

TiO_2 nanoparticles: Hydrothermal synthesis

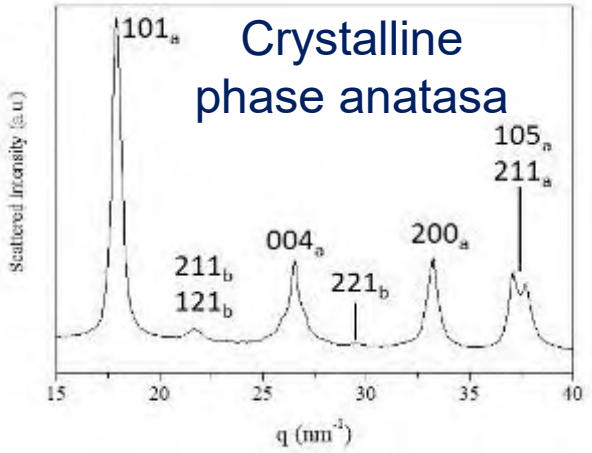


Coloidal suspensión

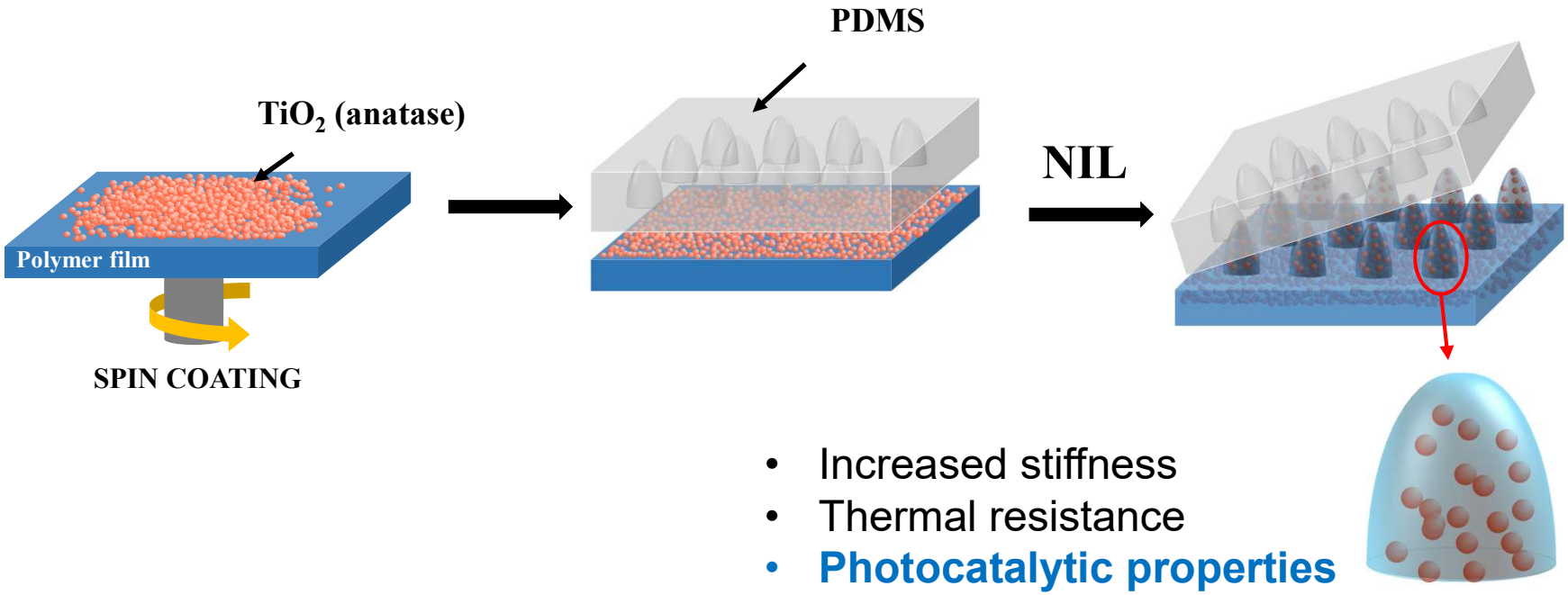




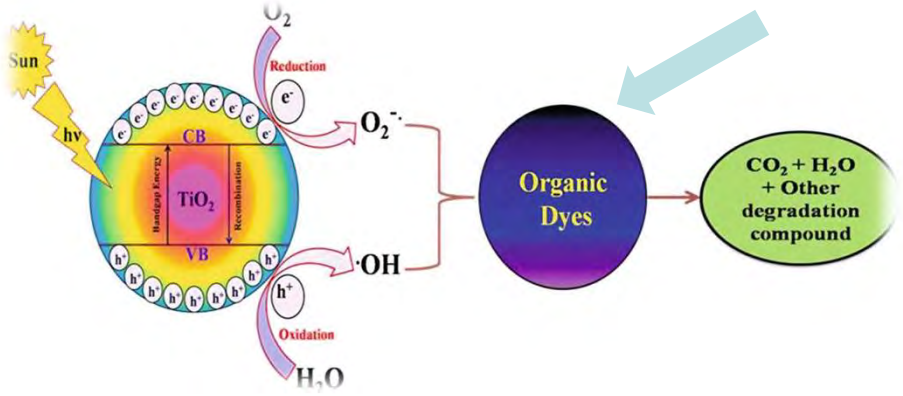
WAXS



Anti-reflective surface nanocomposite

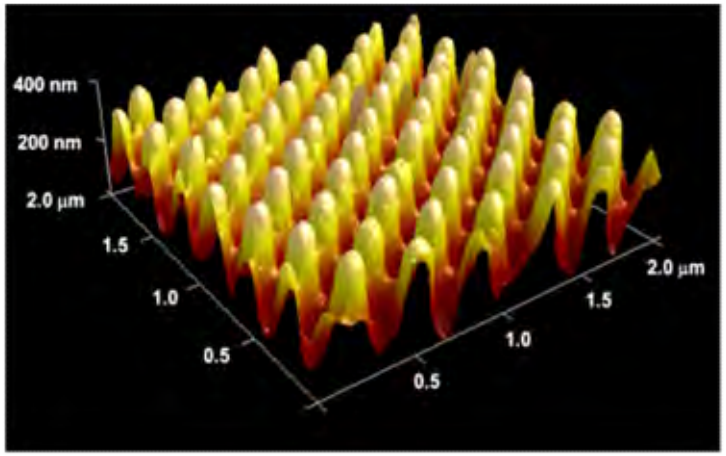


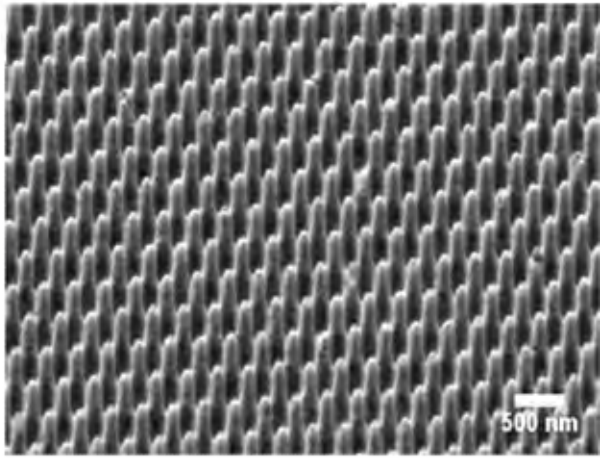
- Increased stiffness
- Thermal resistance
- **Photocatalytic properties**



Moth-eye imprinted topography

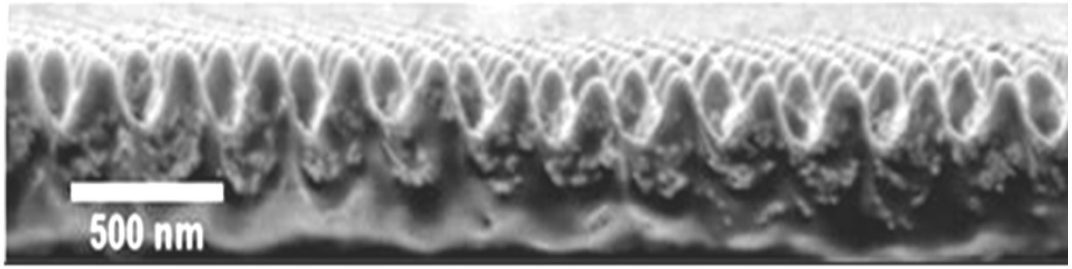
Morphology: SEM & AFM images





Moth-eye Nano-patterns

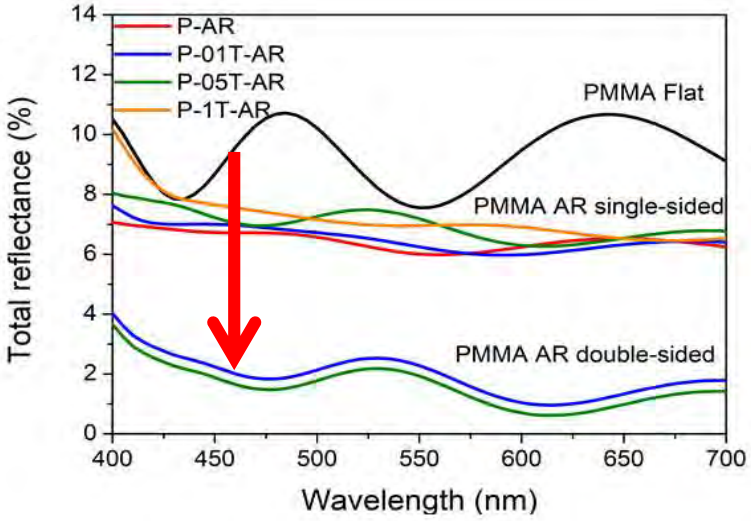
Nano-particles



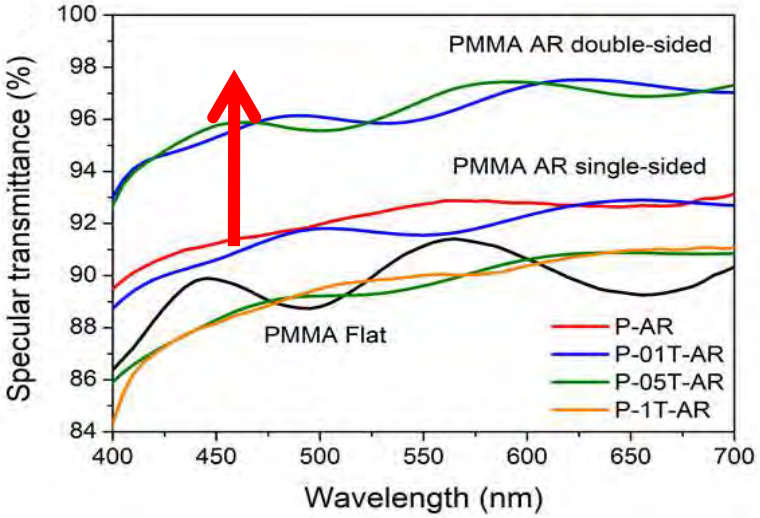
- High fidelity replication of the master mold on PMMA Nanocomposites
- Good nanoparticles dispersion
- Embedded particles within the polymer

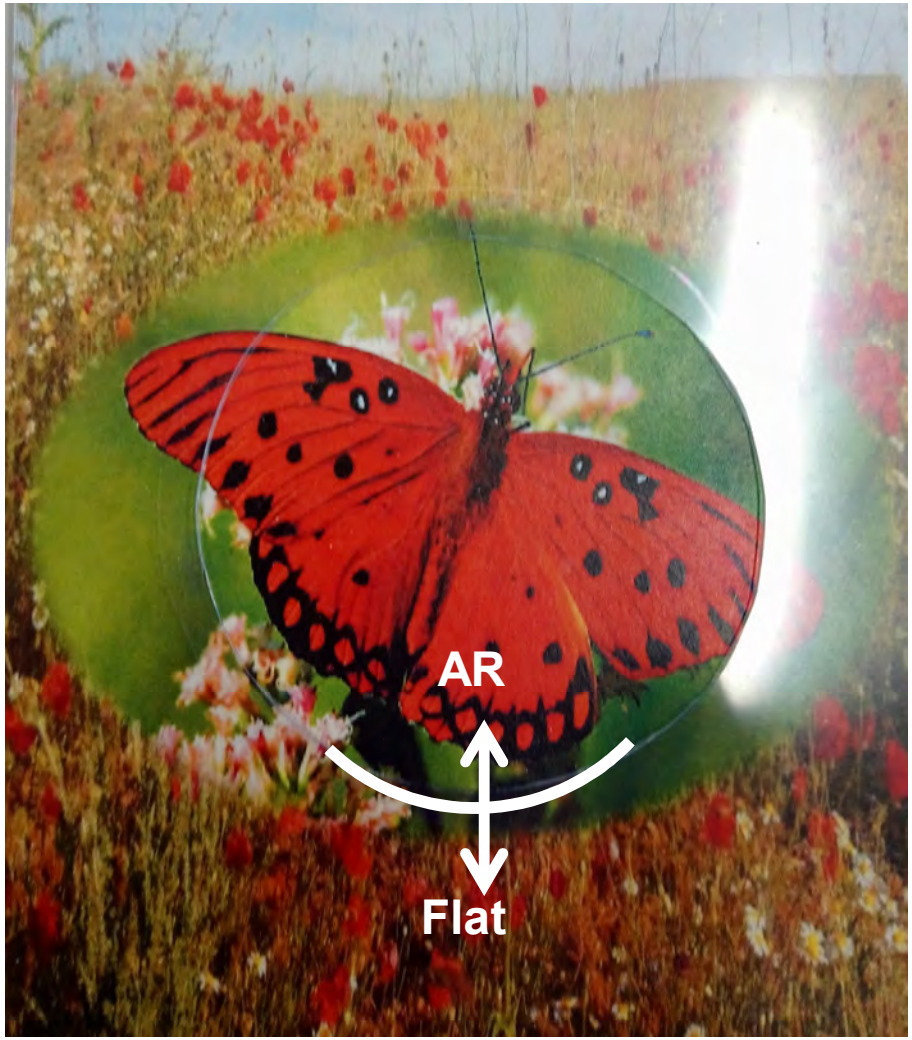
Moth-eye optical characterization

Total Reflectance

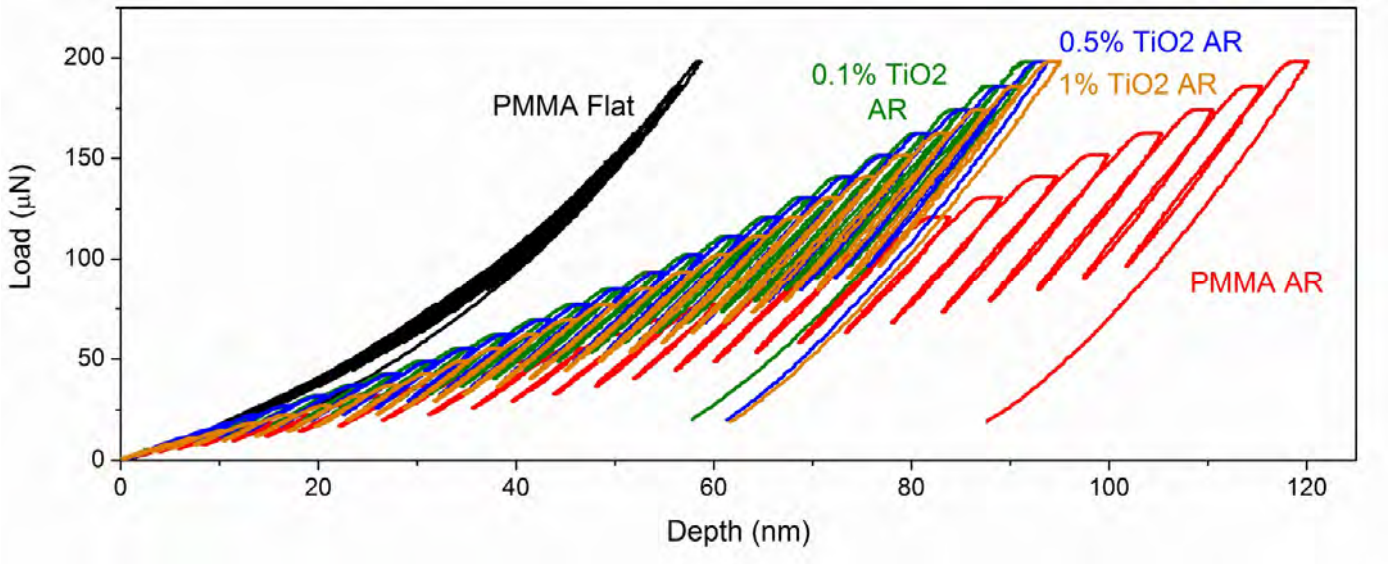


Transmittance





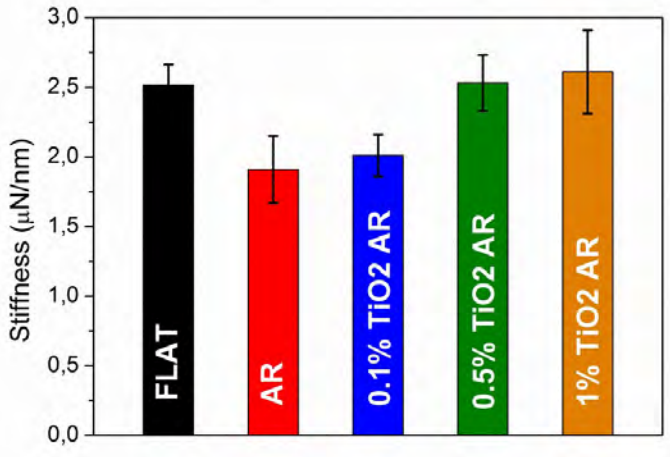
Mechanical behavior:
Nanoindentation

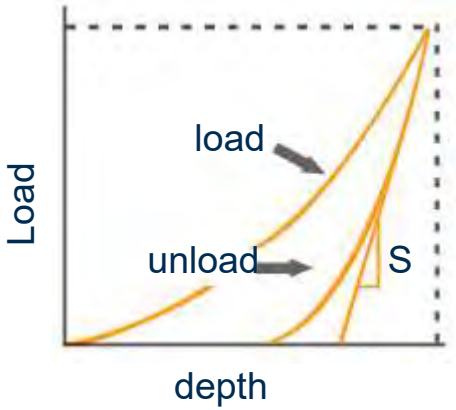


20 load/unload cycles until maximum load of 200 µN

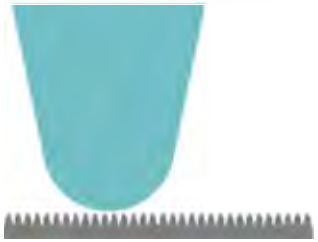
Stiffness

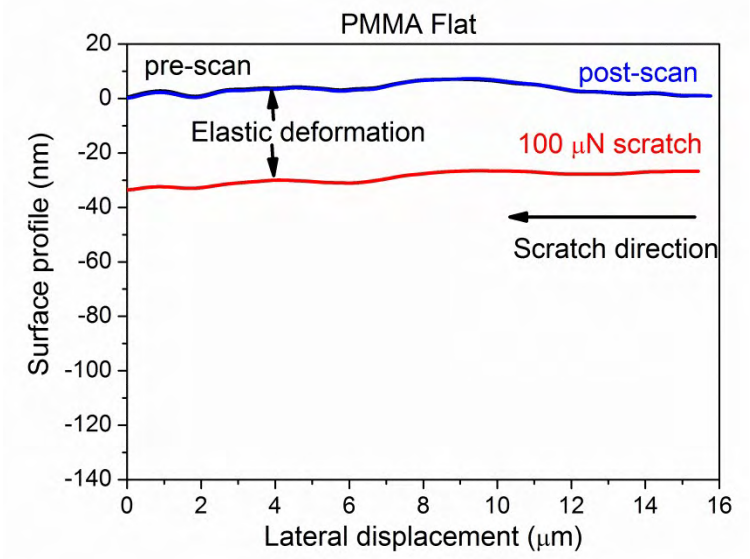
Slope of the curves in the elastic region (maximum of 10 nm)



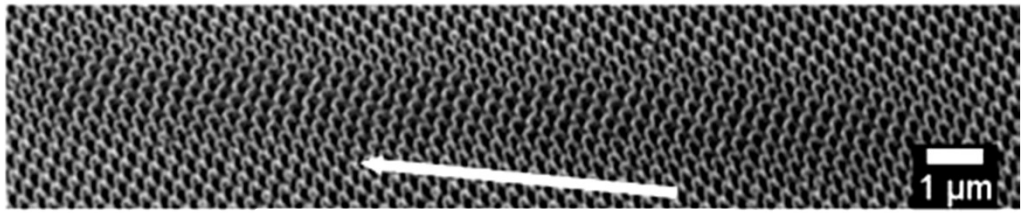


Mechanical resistance : Nanoscratch

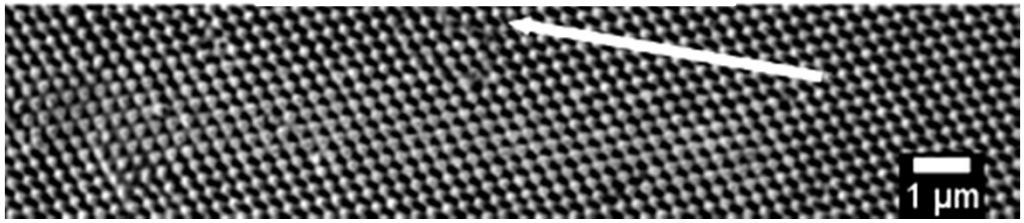


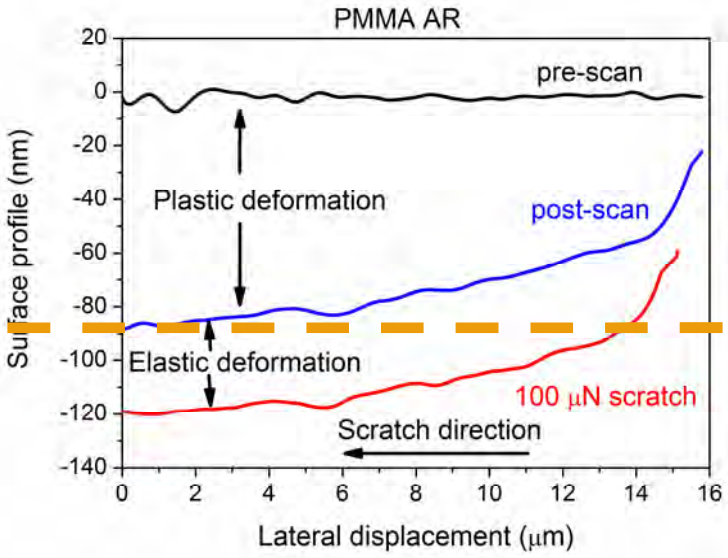


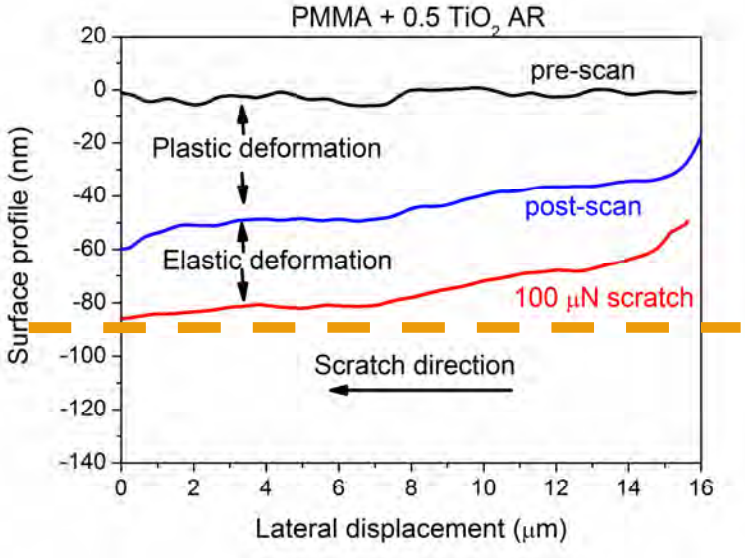
PMMA



PMMA + 0.1 wt% TiO_2

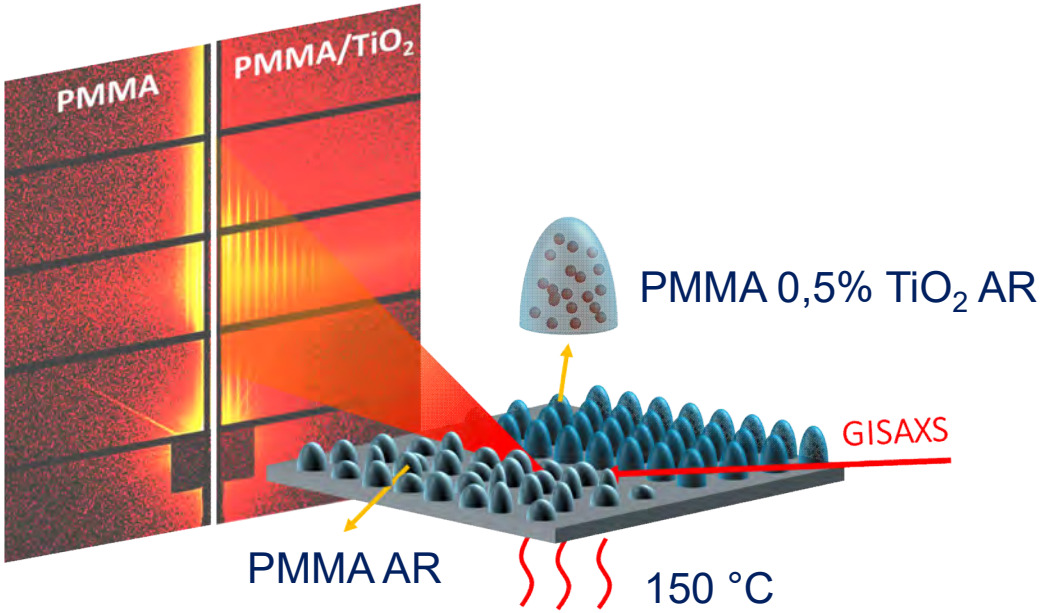






Thermal stability of AR patterns

Grazing Incidence Small Angle X-ray Scattering: GISAXS



IOP Publishing Nanotechnology
Nanotechnology 32 (2021) 385302 (9pp)

Improved thermal stability of antireflective moth-eye topography imprinted on PMMA/TiO_2 surface nanocomposites

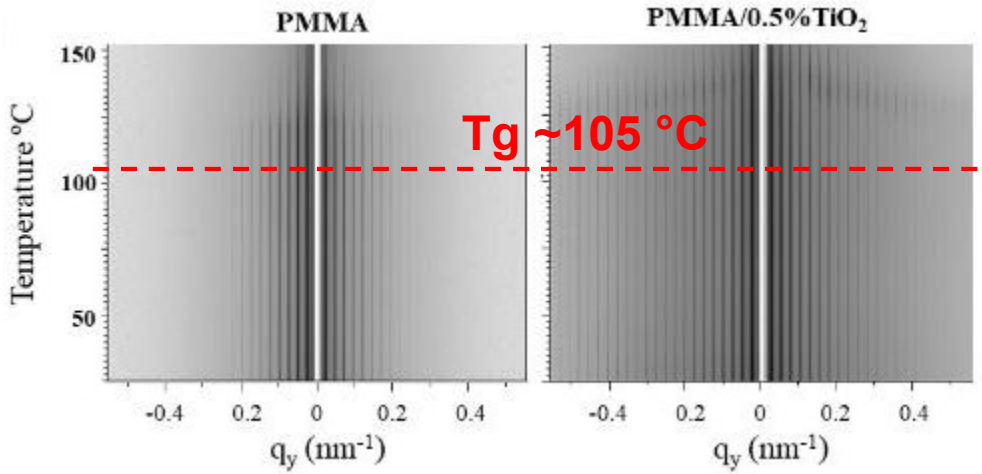
Alejandra Jacobo-Martín, Jaime J Hernández, Patricia Pedraz, Eduardo Solano, Iván Navarro-Baena and Isabel Rodríguez

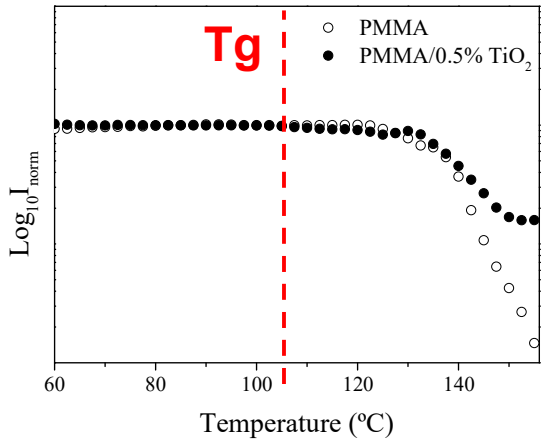
Distortions on the AR pattern

↓

Changes on the diffraction pattern

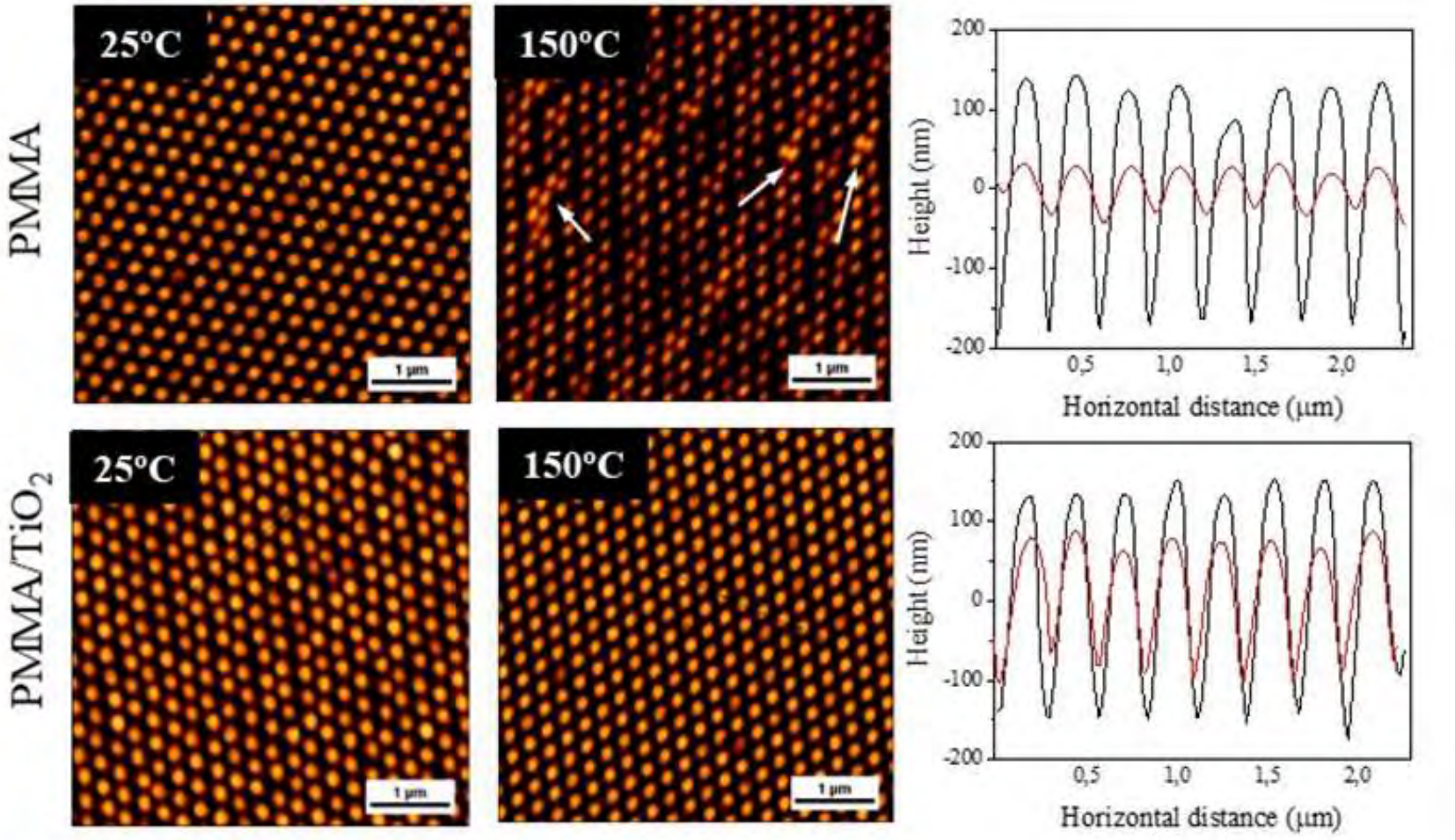
Integrated scattered intensity plots





Improved thermal stability

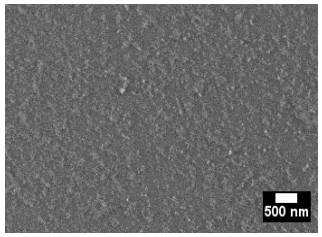
Moth-eye PMMA/TiO2 surface nanocomposites

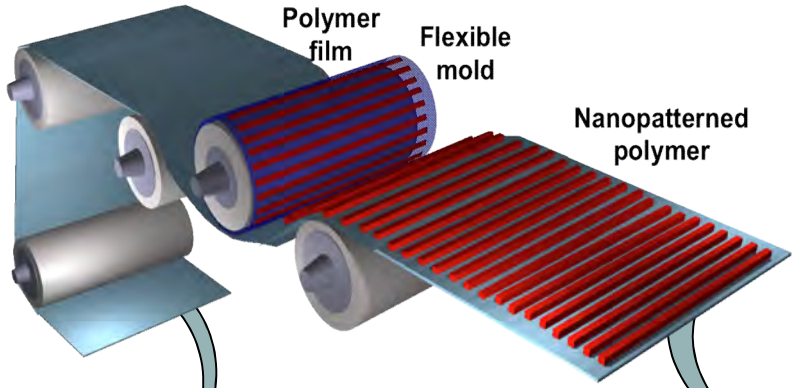


"anchoring effect of the TiO_2 NP to the polymer chain mobility" – pattern stability

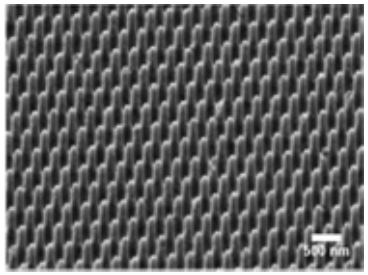
Continuous R2R process

2. Surface nanoimprinting



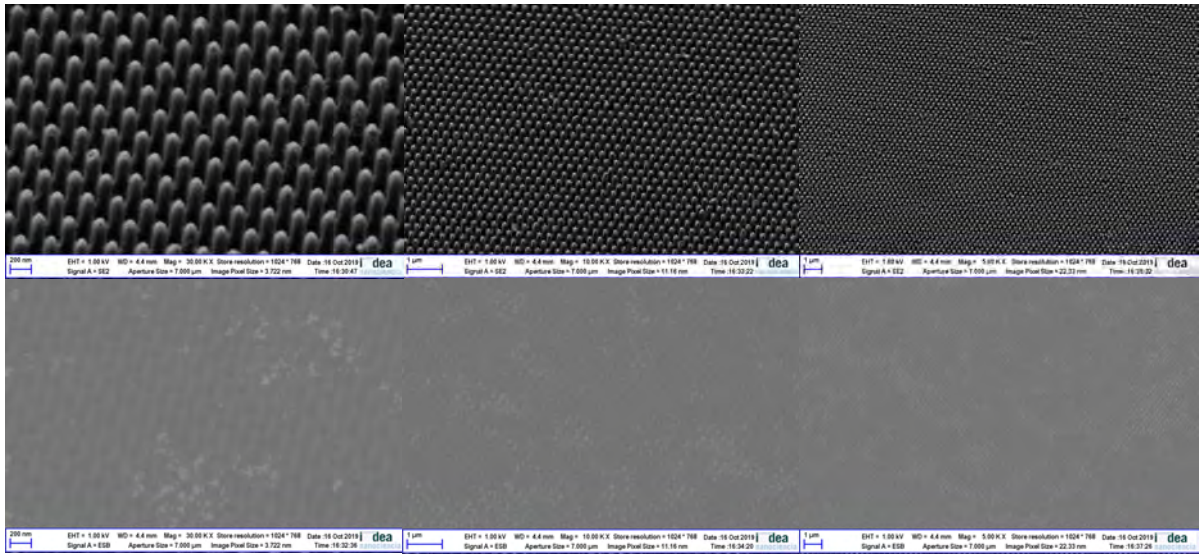


➢ Nanopatterned nanocomposite



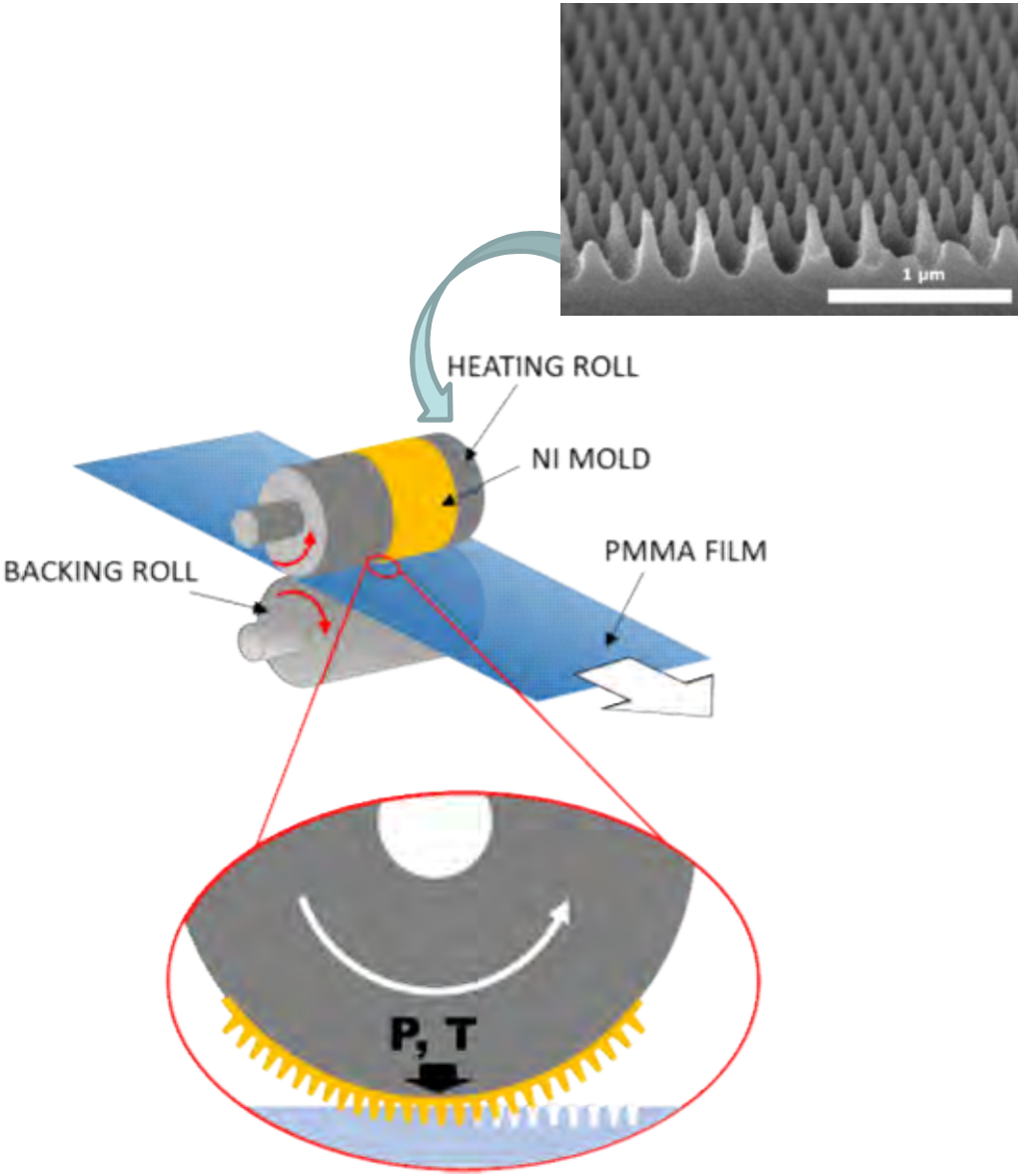
1. Nanoparticle coating

PET + PMMA + 025 % TIO2 ARC (R2R)



Patent : Polymeric composites with functional surfaces EP3436233B1 0000000

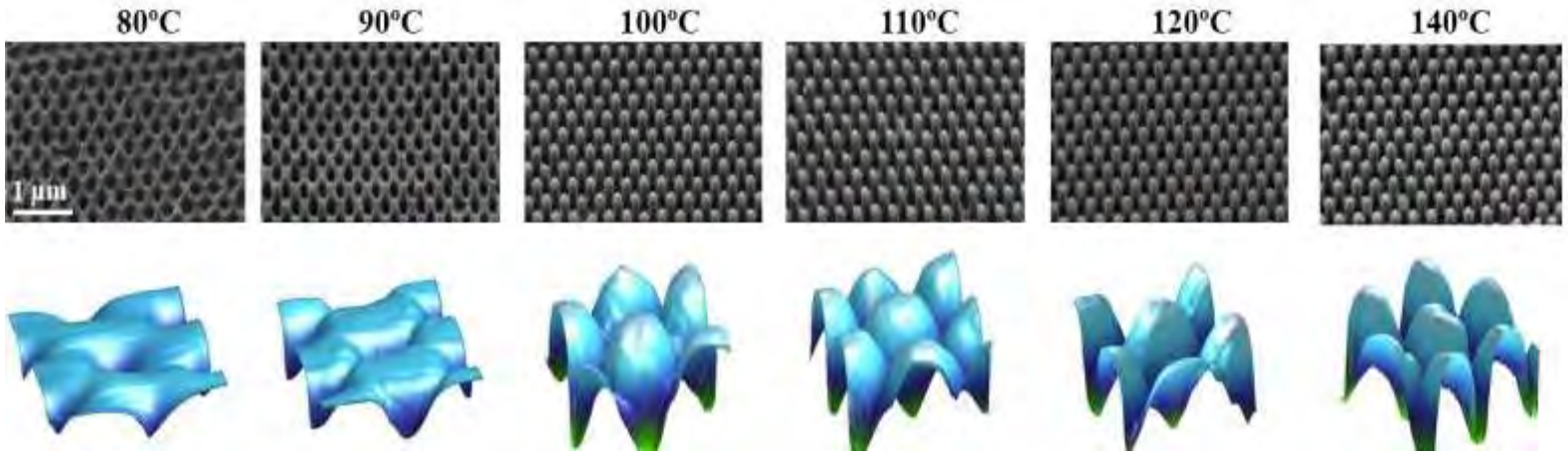
Thermal R2R imprinting of moth-eye non-reflective films



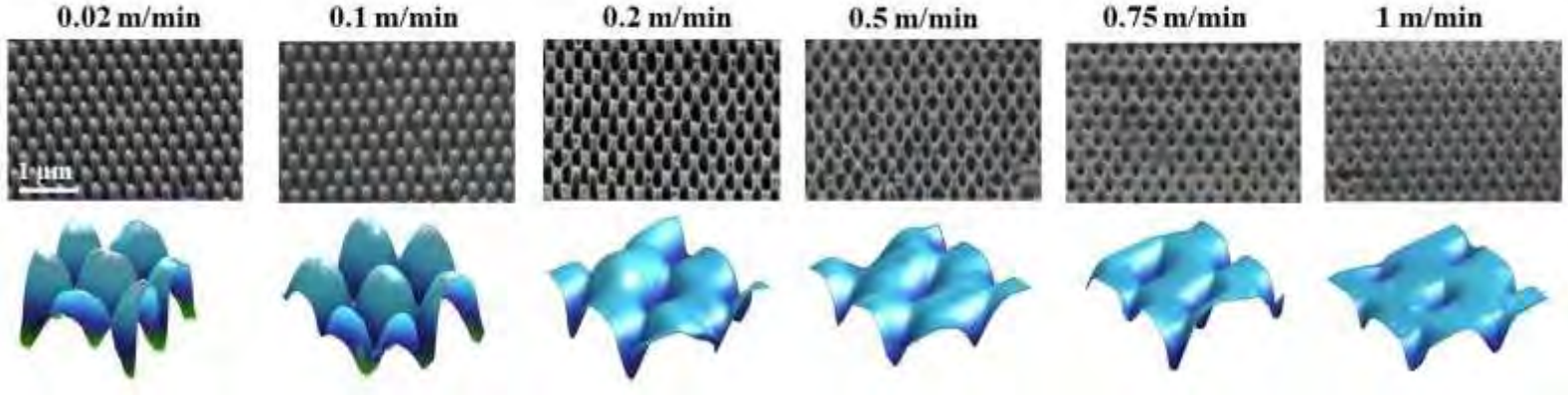
Scientific Reports, 2021 , 11(1), 1-15.

Thermal R2R imprinting of moth-eye non-reflective films

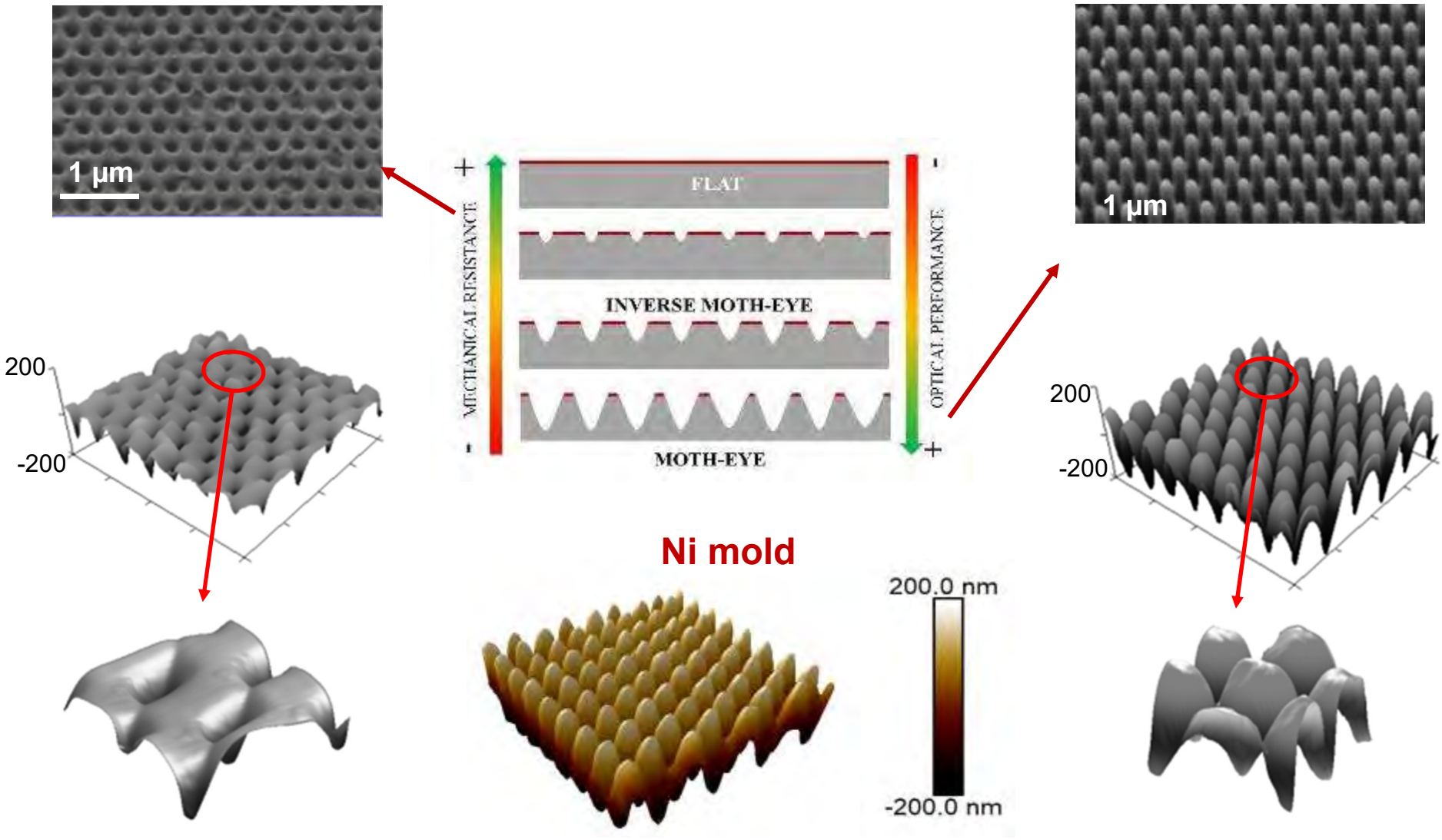
@ Variable temperature



@ Variable speed



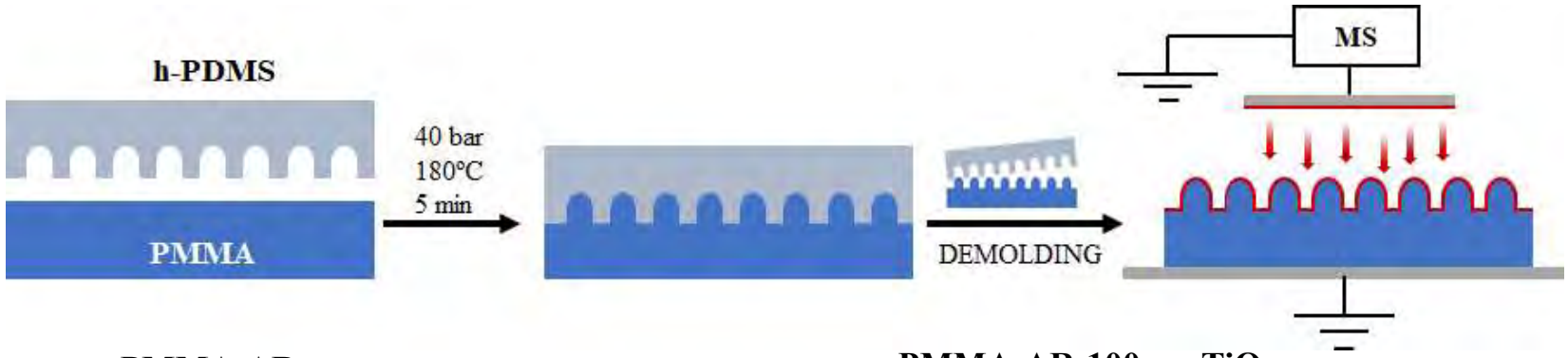
Thermal R2R imprinting of moth-eye non-reflective films



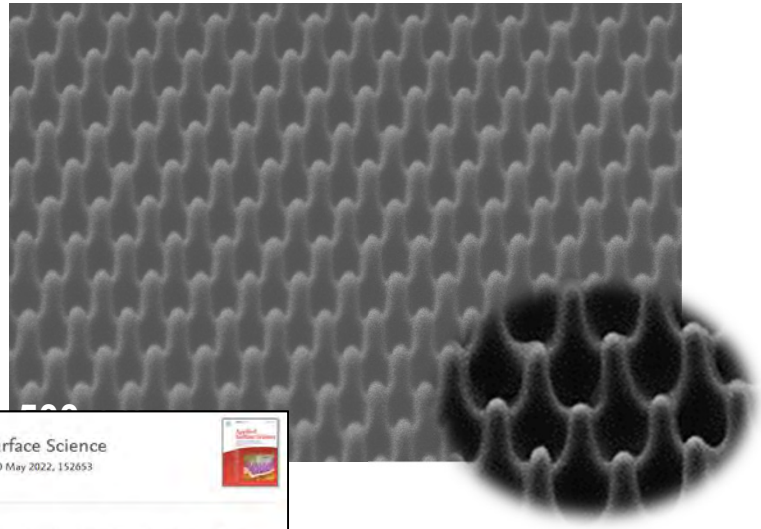
TiO_2-PMMA Moth-eye MS coating

NANOIMPRINT LITHOGRAPHY

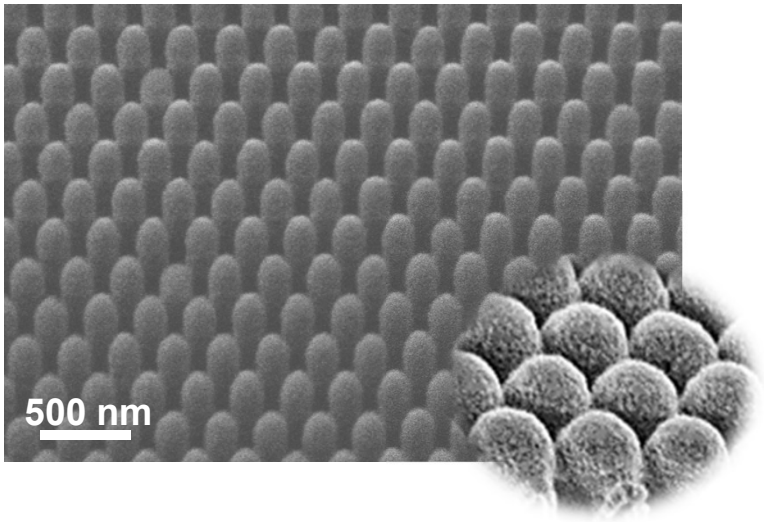
MAGNETRON SPUTTERING

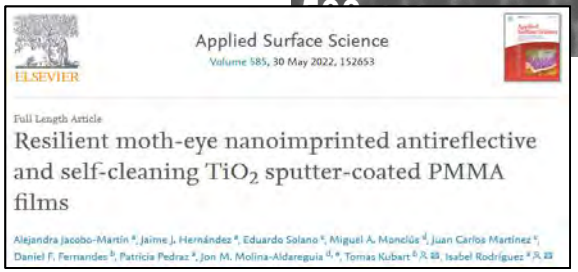


PMMA-AR



PMMA-AR-100nm-TiO_2





Applied Surface Science
Volume 585, 30 May 2022, 152653

Full Length Article

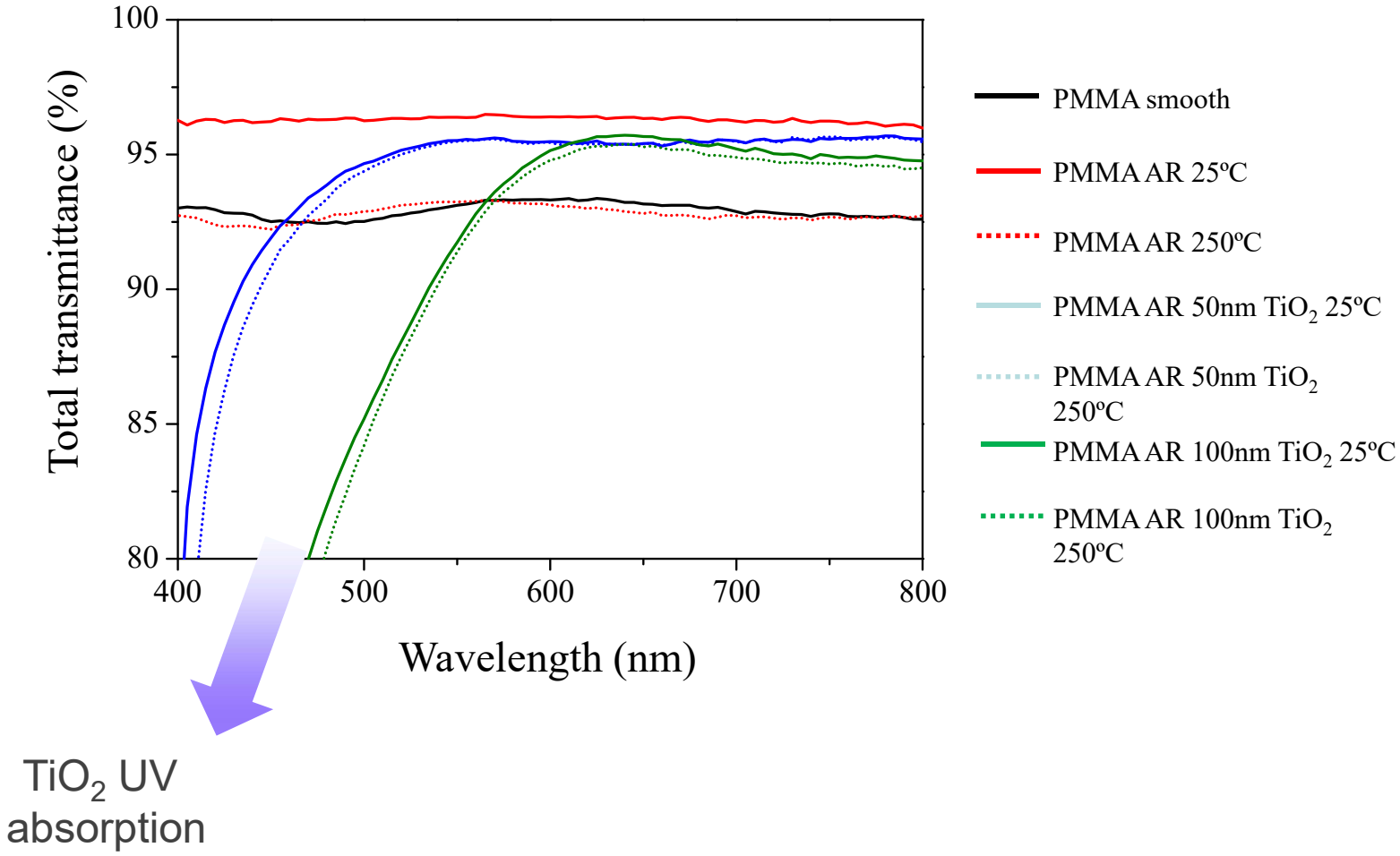
Resilient moth-eye nanoimprinted antireflective and self-cleaning TiO_2 sputter-coated PMMA films

Alejandra Jacobo-Martín [a], Jaime J. Hernández [a], Eduardo Solano [b], Miguel A. Monclús [d], Juan Carlos Martínez [c], Daniel F. Fernandes [b], Patricia Pedraz [a], Jon M. Molina-Aldareguía [d, *], Tomas Kubart [b, **], Isabel Rodríguez [a, **]

UPPSALA UNIVERSITET

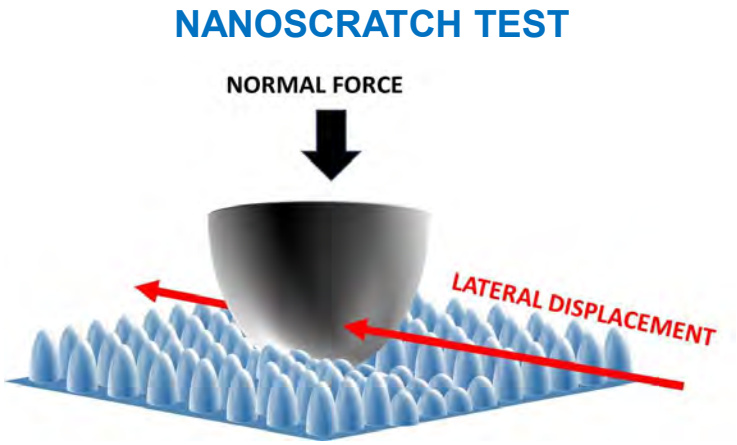
Daniel Fernandes
Tomas Kubart

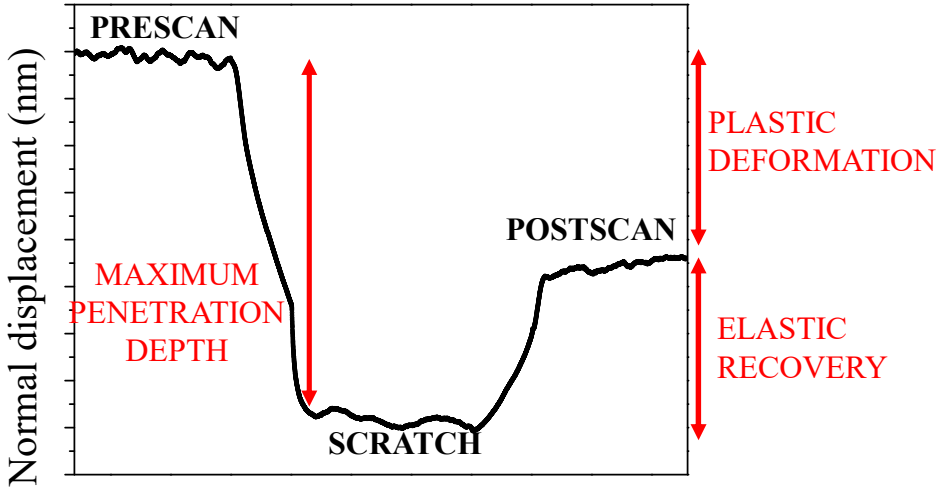
Optical transmittance

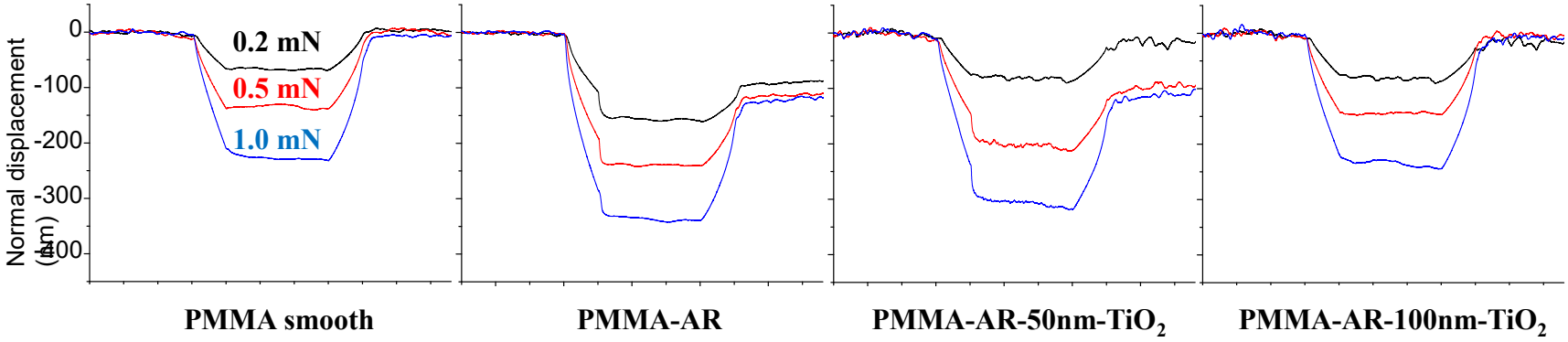


ANTIREFLECTIVE FUNCTIONALITY IS PRESERVED AT T>>Tg

Mechanical resistance

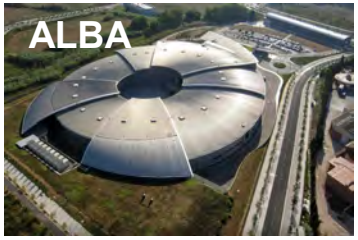




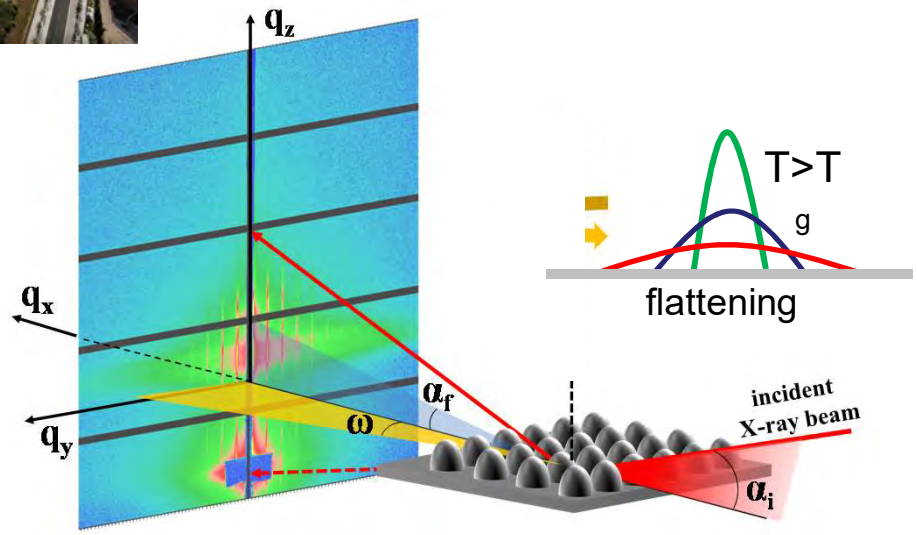


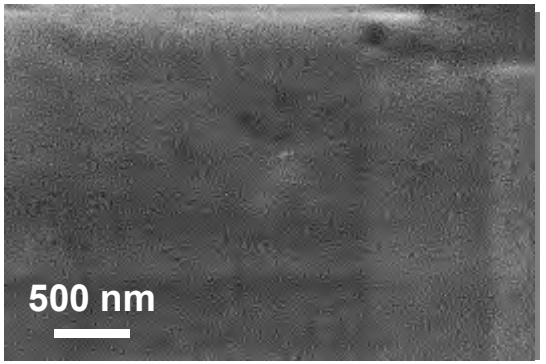
TIO_2 ENCAPSULATION IMPROVES SCRATCH RESISTANCE OF AR NANOSTRUCTURES

Thermal resistance

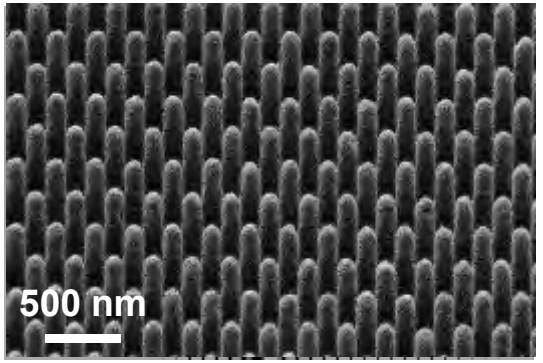


Grazing Incidence Small Angle X-ray Scattering (GISAXS)

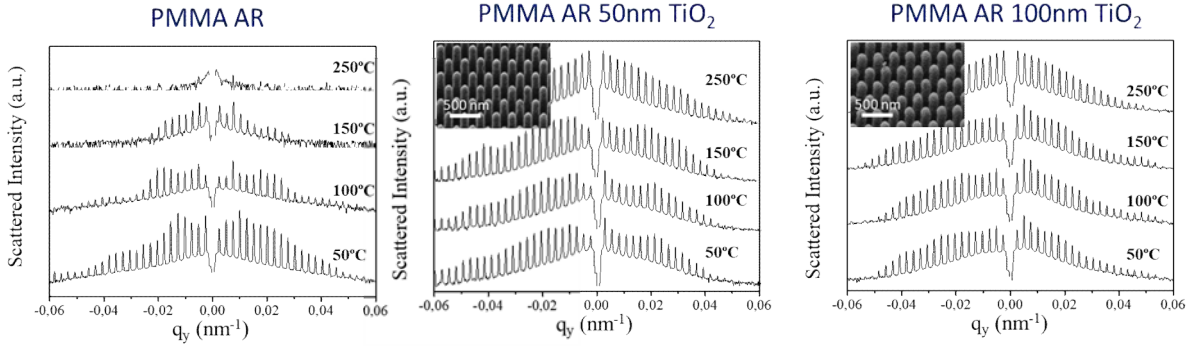




PMMA-AR



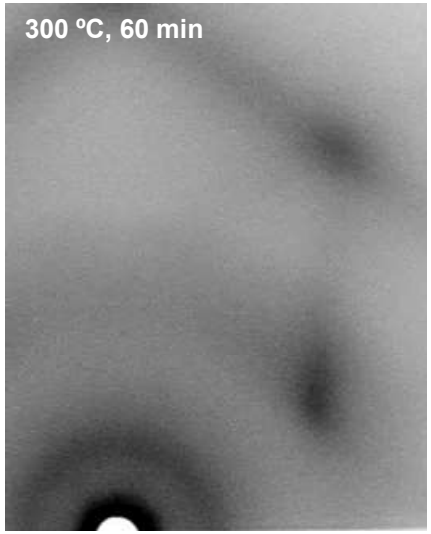
PMMA-50nm TiO_2-AR



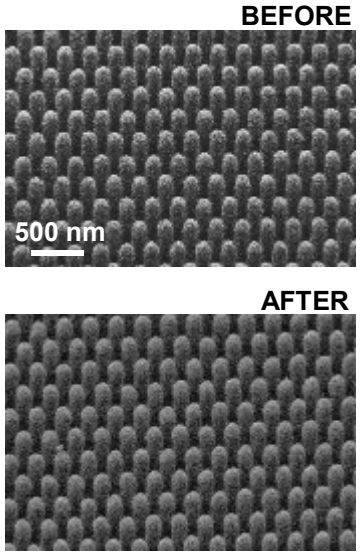
TIO_2 ENCAPSULATION PREVENTS REFLOW AND DISTORTION EFFECTS up to 250 C

Photoinduced self-cleaning

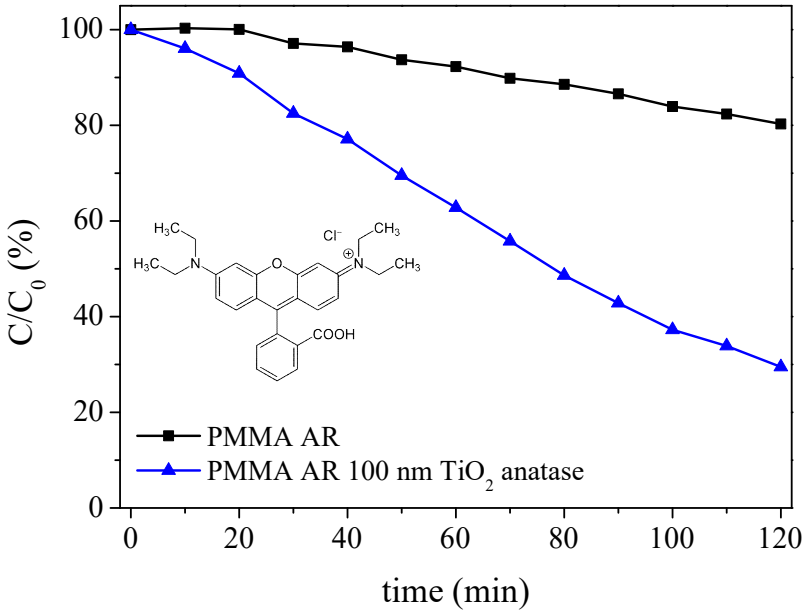
Amorphous TiO_2 → anatase

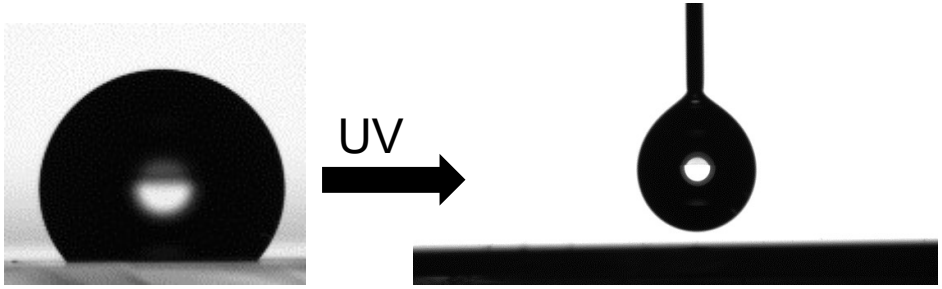


GIWAXS



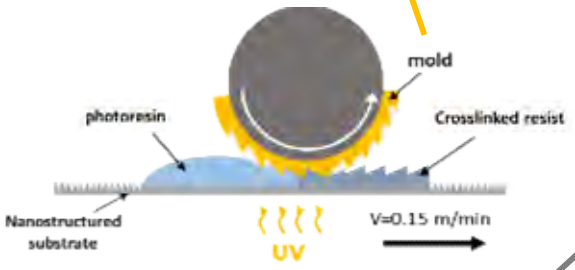
RhB UV degradation assay

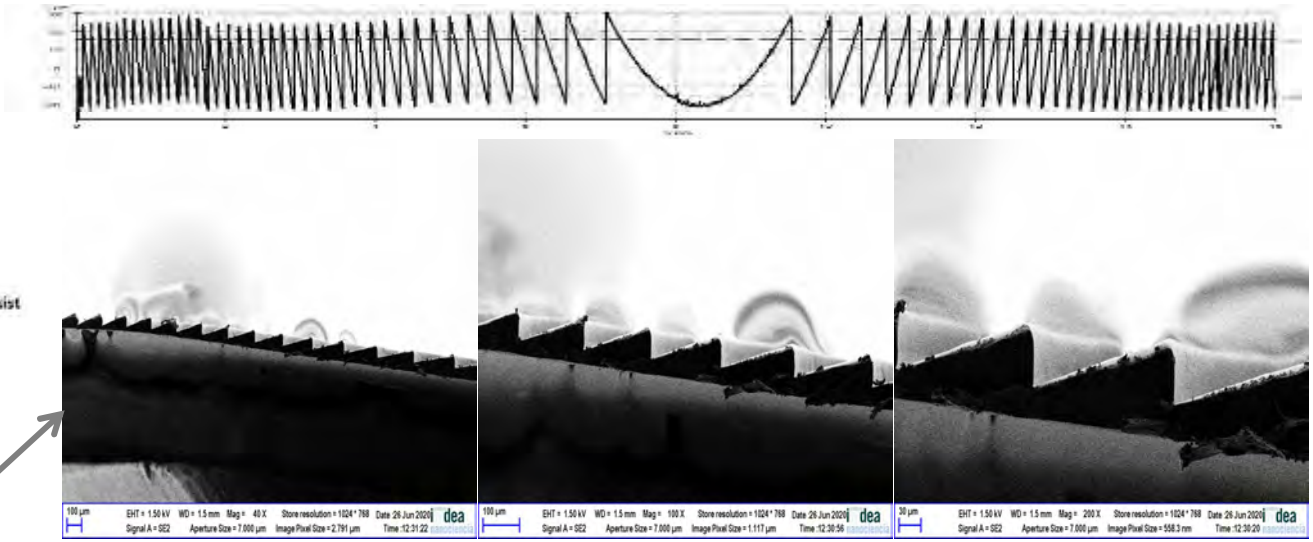


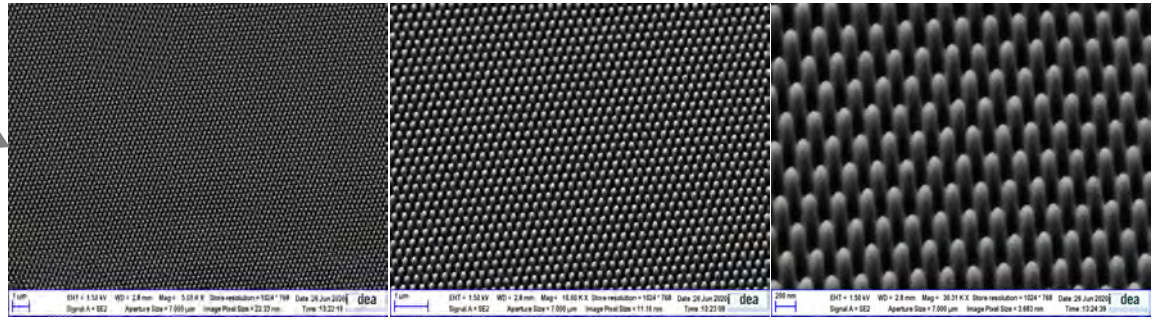


POLLUTANT DEGRADATION + SUPERHYDROPHYLICITY = SELF-CLEANING

UV R2R NIL Fresnel lens with anti- reflective moth eye







Acknowledgements

Group members

Jaime Hernandez
Alberto Martín Asensio
Miguel Esteban Lucía
Sergio Dávila
Maria Teresa Alameda

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Jean Cacheux
Ivan Navarro Baena

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Ignacio Antón

Eduardo Solano
Juan Carlos Martínez

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Thank you for you attention !