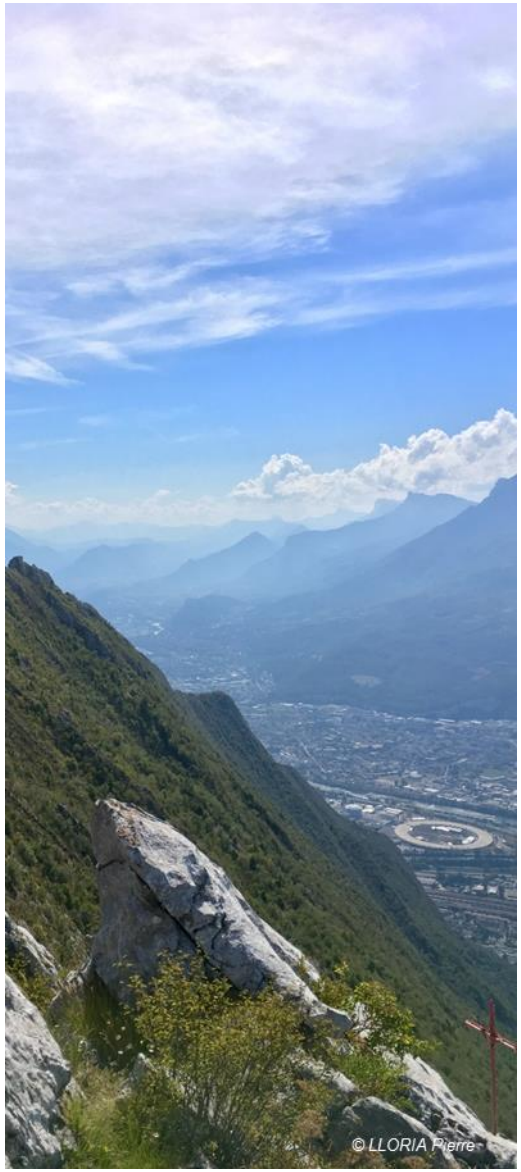
PARTNERSHIP FOR SOFT CONDENSED MATTER

ESRF | The European Synchrotron

User meeting – 4th February 2019 - Tutorial 1

The PSCM: Instruments available for off line pre/post characterization

LLORIA Pierre



1. Presentation of the PSCM
2. The journey of an ESRF user
3. Perspective

Partnership for Soft Condensed Matter

- **Definitions and Missions**
- A joint ESRF-ILL initiative for advanced Soft Matter Research.
- New platform allowing enhanced exploitation of X-rays and Neutrons.

- **Actions and Goal**
- Combine the ESRF-ILL strengths and resources with new expertise and capacities contributed by Novel PSCM partners.
- Enhanced user support: sample preparation, complementary techniques and implement new sample environment.

Location and laboratories:

- **Users** receive personally assigned badges to access the PSCM Labs
- **Onsite Staff** Members can ask permanent access to the PSCM Labs
- **Scientific Visitors** can use the specifically reserved PSCM Corner Office
- **Partners** receive PSCM office space assigned to their staff members

Laboratories and Instruments

Sample preparation laboratory

- Principal tools for sample preparation
- Lab-bench dedicated for the users
- Maintain stock of solvents

Instrumentation

Surfaces
Langmuir Trough NIMA 611
Spin Coater Delta6 MicroTec
Harrick Plasma Cleaner
Kruss DS114 Contact Angle
Kruss K11 Tensiometer
Q-Sense E4 QCM

Spectroscopy
Jasco V-630 UV-vis
Jenway 6705 UV-vis
Jasco FTIR
OceanOptics Raman

Microscopy
Olympus BX61
Olympus BX50
Accurion EP^2 BAM
Accurion EP^3 BAM

Light Scattering
ALV CGS-3 SLS/DLS
Malvern Zeta-Sizer
Beaglehole Ellipsometer

AFM Platform
Asylum CYPHER
Asylum MFP-3D
DI Dimension 3100

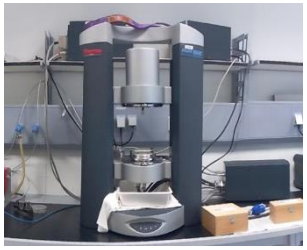
Lipid Extraction
HPLC Setup
Electrophysiology

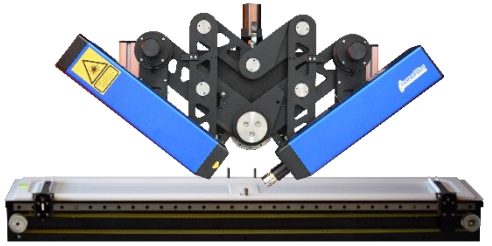
Rheology
Haake MarsII
Anton Paar MCR 501

Aqueous Chemistry
pH-meter, Sonicators, Rotovap,…
Ovens, Baths, DI water, Balances

Calorimetry
Setaram DSC-131
Setaram μDSC-III

Microfluidics
3D-printer ASIGA Pico2 HD
Olympus SZ61





Sample preparation laboratory

- Principal tools for sample preparation
- Lab-bench dedicated for the users
- Maintain stock of solvents

Instrumentation

Surfaces
Langmuir Trough NIMA 611
Spin Coater Delta6 MicroTec
Harrick Plasma Cleaner
Kruss DS114 Contact Angle
Kruss K11 Tensiometer
Q-Sense E4 QCM

Spectroscopy
Jasco V-630 UV-vis
Jenway 6705 UV-vis
Jasco FTIR
OceanOptics Raman

Microscopy
Olympus BX61
Olympus BX50
Accurion EP^2 BAM
Accurion EP^3 BAM

Light Scattering
ALV CGS-3 SLS/DLS
Malvern Zeta-Sizer
Beaglehole Ellipsometer

AFM Platform
Asylum CYPHER
Asylum MFP-3D
DI Dimension 3100

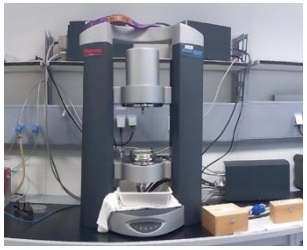
Lipid Extraction
HPLC Setup
Electrophysiology

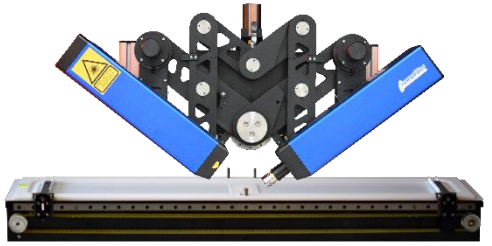
Rheology
Haake MarsII
Anton Paar MCR 501

Aqueous Chemistry
pH-meter, Sonicators, Rotovap,…
Ovens, Baths, DI water, Balances

Calorimetry
Setaram DSC-131
Setaram μDSC-III

Microfluidics
3D-printer ASIGA Pico2 HD
Olympus SZ61





PSCM Staff:

PSCM Coordinator Diego PONTONI (Scientist)

General support & Complementary techniques Pierre LLORIA (Technician)

- Principal tools for sample preparation
- Five main instruments
- Managing the Lab-bench use

Micro fluids laboratory: Peter Van Der Linden (Engineer)

- Microfluidic development
- Major sample environment cell

AFM laboratory: Alain Panzarella (Engineer) & Marie Capron (Postdoc)

- Three different commercial AFMs
- Help the users and staff

ESRF user: SC/4644 @ID03, SC4784 @BM26B & SC/4699 @ID03

- Student: 3rd year PhD Fabrizio Corrado Adamo
- Institute: Università Politecnica delle Marche, Ancona, Italy
- Project: Surface Anchoring of Bent-Core Mesogens (BCMs)
- Materials: a specific bent-core mesogen (*Figure 1*)
- PSCM techniques: **Substrate preparation**, **Langmuir Blodgett**, **Brewster Angle Microscope**, **Atomic Force Microscope**.
- ESRF techniques: GIWAXS (*Figure 2*), XRR, GISAXS, GIXRD,…

PhD Student
Liquid Crystals

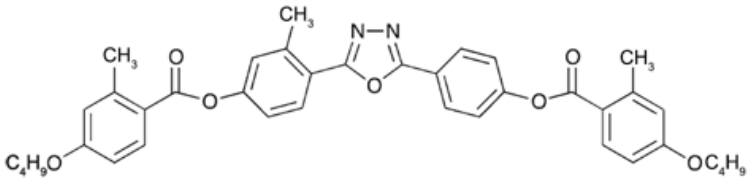


Figure 1: Molecular structures of Bent-core Mesogens (BCM)

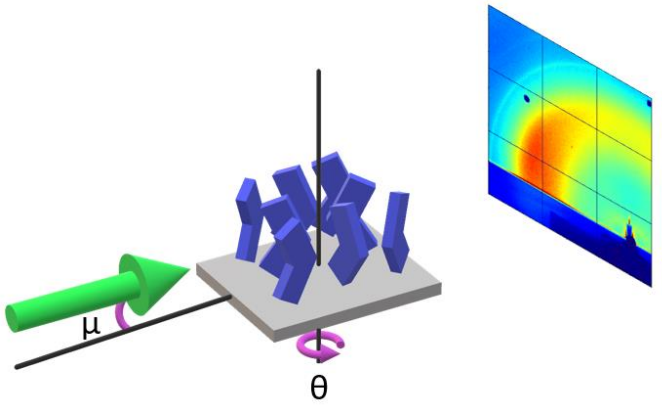
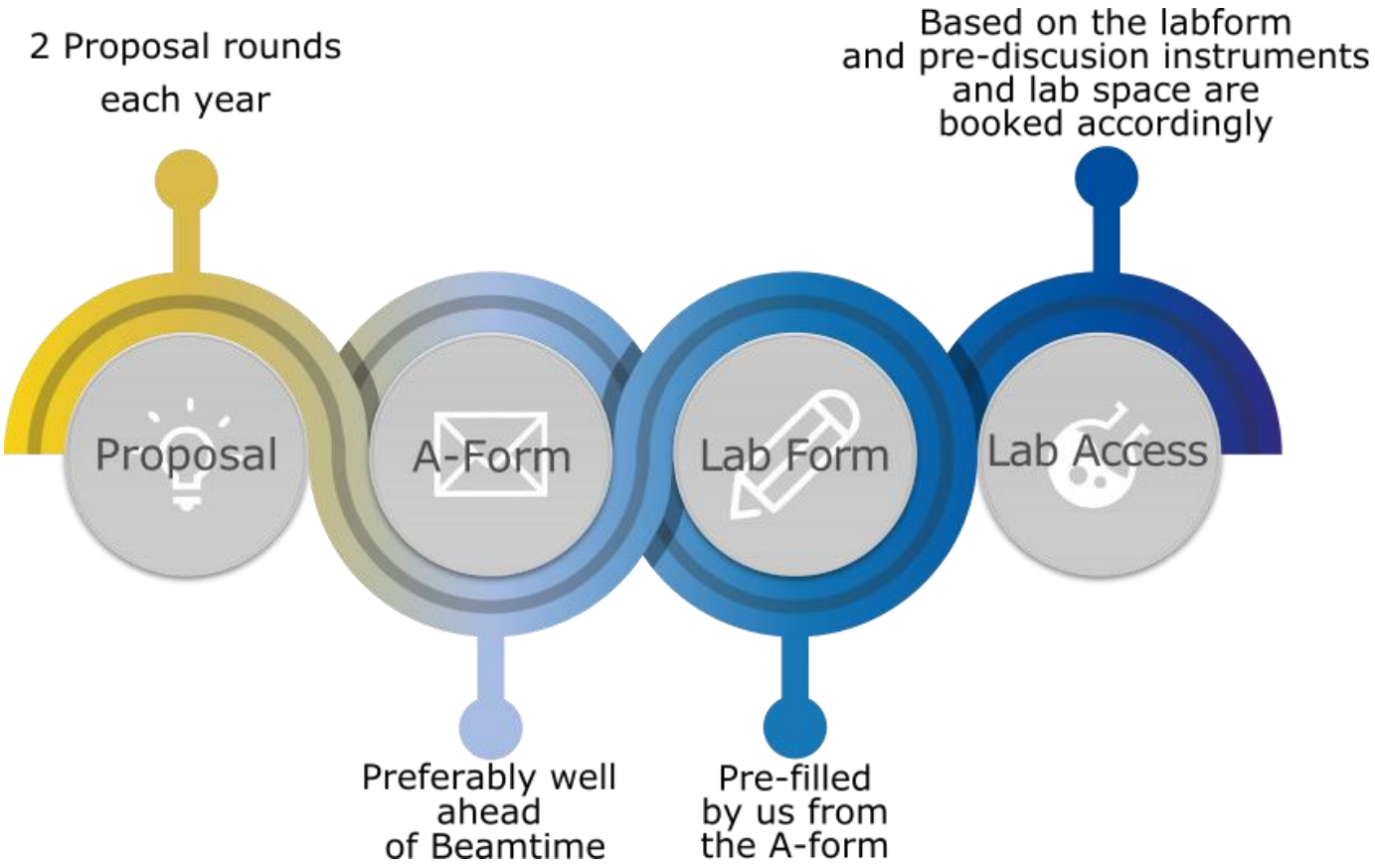


Figure 2: GIWAXS measurement on BCM

Vita et al. – *Polar order in bent-core nematics: An overview*
Journal of Molecular Liquids 267 (2018) 564-573

PSCM access channel



pscm-support@esrf.fr

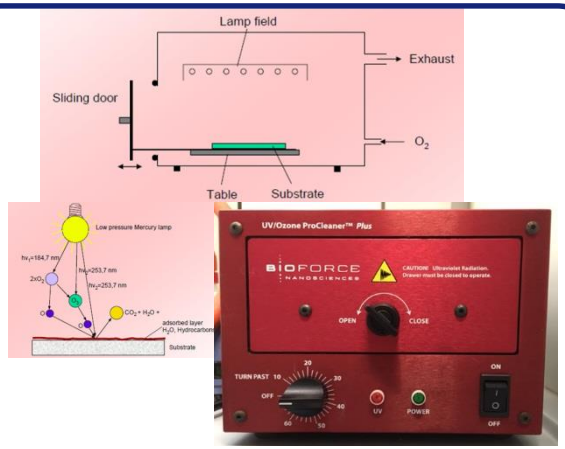
Substrate preparation

60 m² sample preparation lab with dedicated lab bench for user.

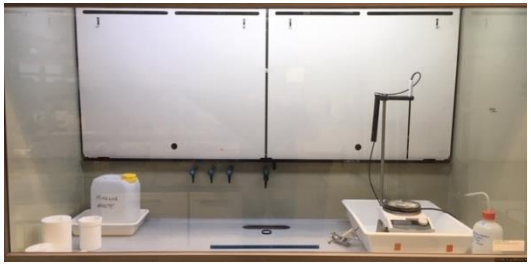
Prepared lab space with mains solvent for, crystals disillusion, substrates cleaning and provide clean glassware.

3 main tools for surface cleaning and preparation: Plasma cleaner, UV Ozone cleaner and Piranha etching.

Ion bombardment plasma discharge



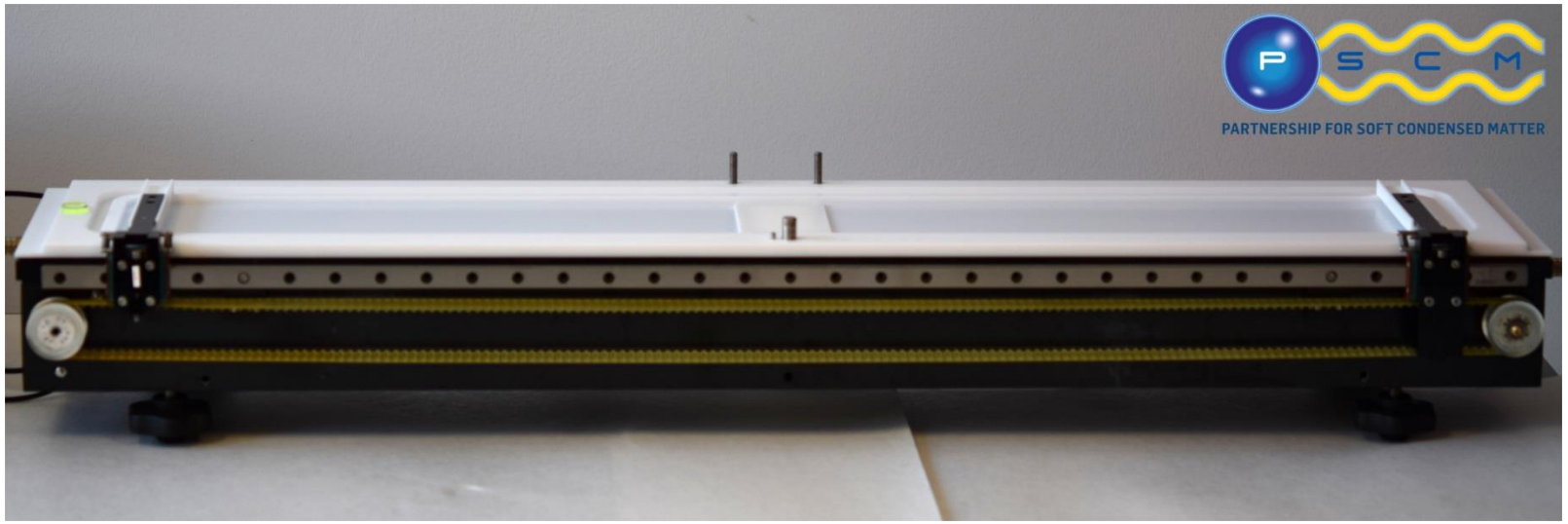
Dry oxidation cleaning



$H_2SO_4 + H_2O_2 \rightarrow H_3O+ + HSO_4^- + O$

Wet oxidation cleaning

Langmuir Trough Nima

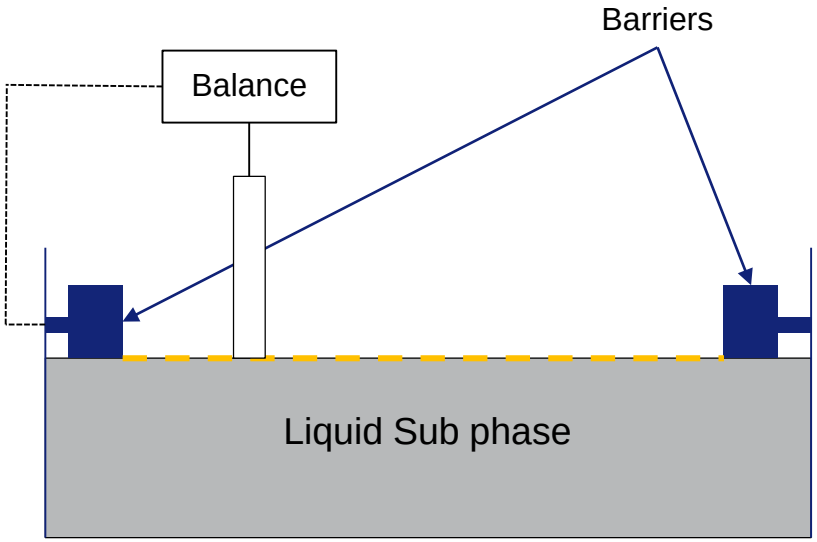


Preparation of molecular layers on liquid surfaces from insoluble amphiphilic molecules (Liquid crystal, fatty acid, lipids, surfactant…)

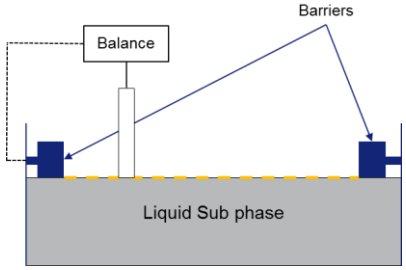
Langmuir trough characteristics:

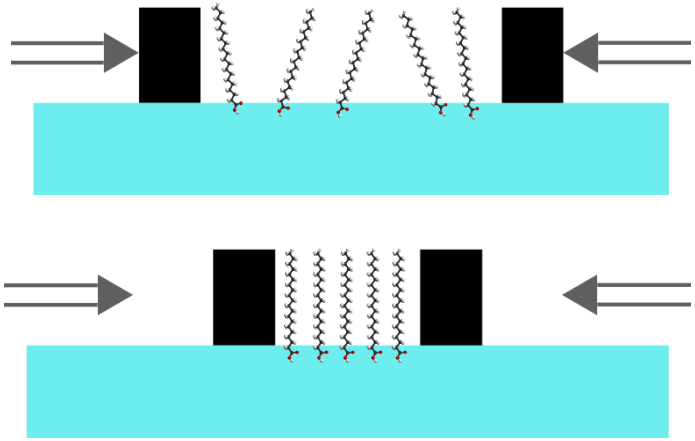
- Maximum area: 690 cm^2
- Minimum area: 80 cm^2
- Barrier speed max: 654.2 $cm^2.min^{-1}$
- Barrier speed min: 5.2$cm^2.min^{-1}$

Langmuir Blodgett monolayer



Langmuir Blodgett monolayer





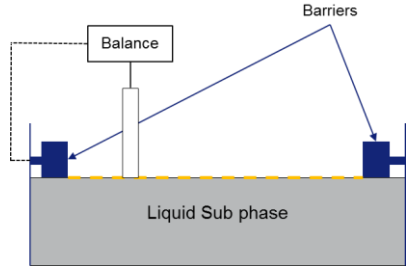
Molecular Area

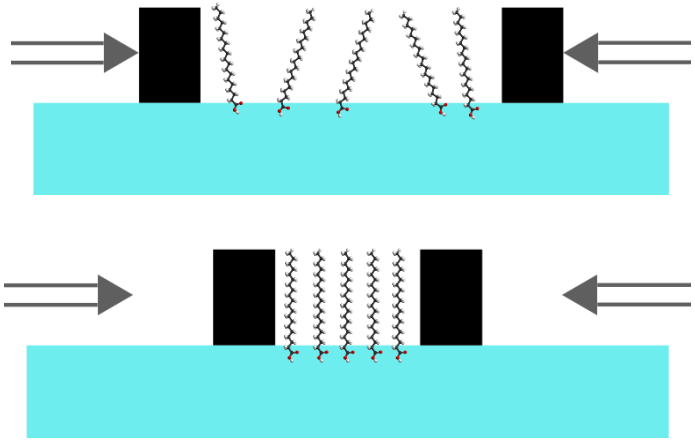
Molecular Area ($A°^2$/molecule) → $A_m = \frac{A_t}{N}$

A_t ← Effective area of the trough ($A°^2$)

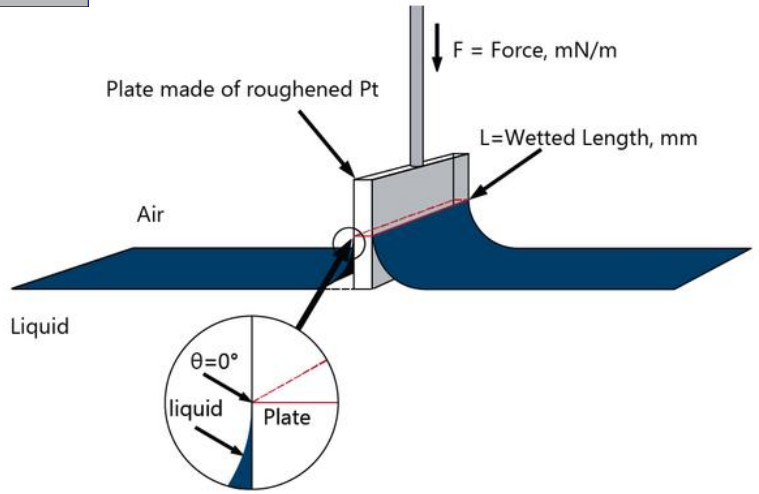
N ← Number of molecules spread

Langmuir Blodgett monolayer





Molecular Area



Surface tension

Molecular Area ($A^{\circ 2}$/molecule) → $A_m = \frac{A_t}{N}$

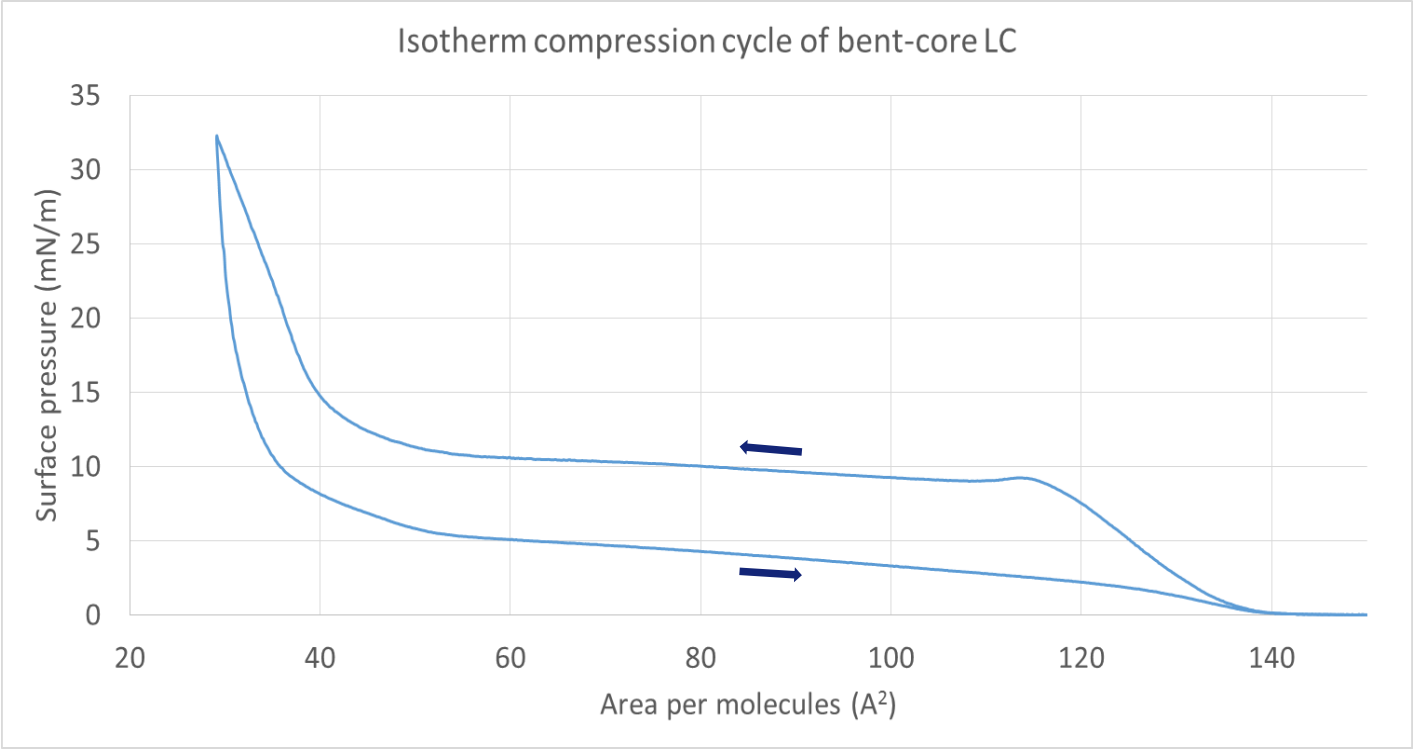
A_t ← Effective area of the trough ($A^{\circ 2}$)

N ← Number of molecules spread

$$\gamma = \frac{F}{P.\cos(\theta)}$$

γ = Surface tension (mN/m)
F = Vertical Force (mN)
P = Wetted perimeter (m)
θ = Contact angle

Langmuir Blodgett monolayer



Molecular Area

Surface tension

Molecular Area (A°²/molecule) → $A_m = \frac{A_t}{N}$

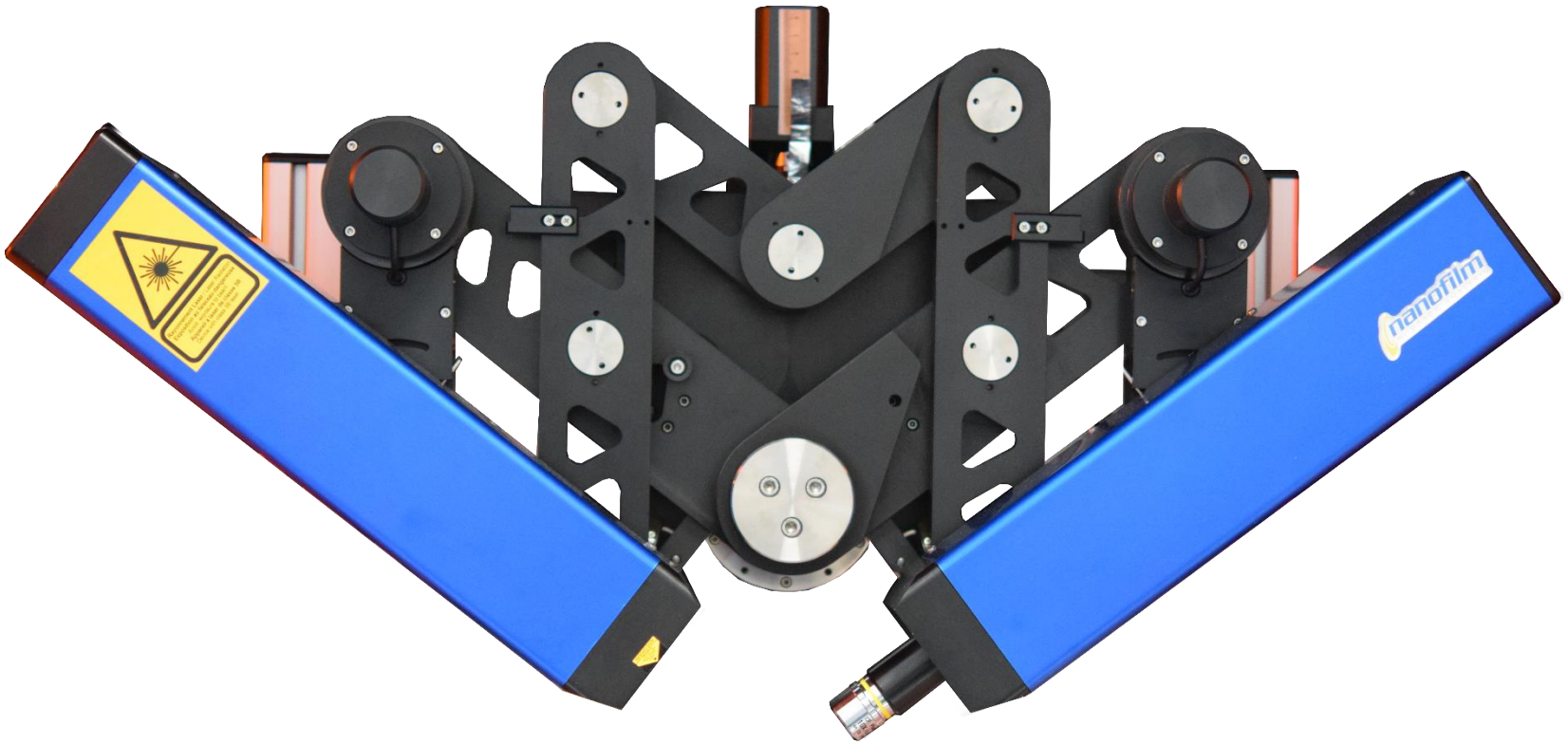
A_t ← Effective area of the trough (A°²)

N ← Number of molecules spread

$$\gamma = \frac{F}{P.\cos(\theta)}$$

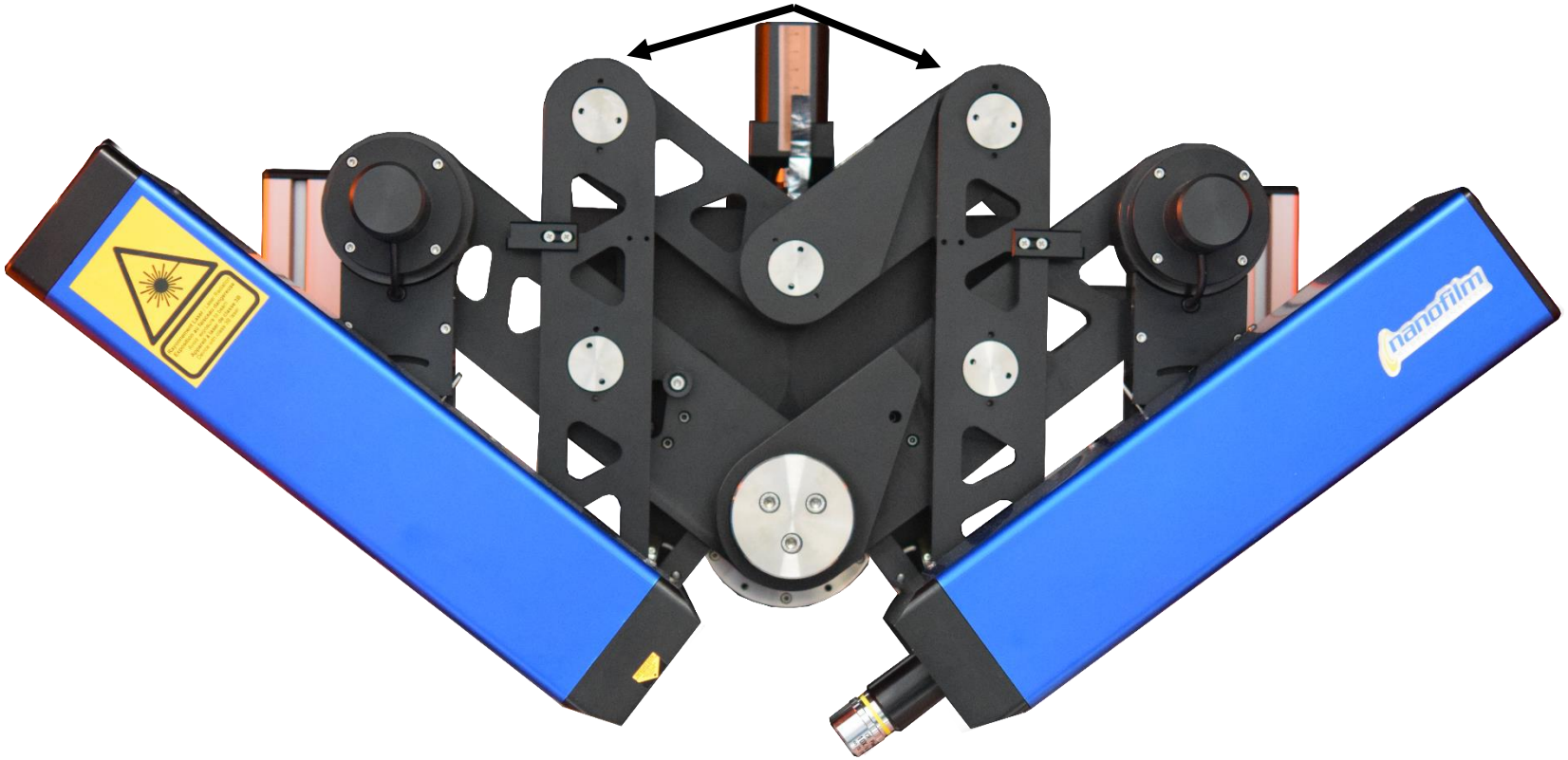
γ = Surface tension (mN/m)
F = Vertical Force (mN)
P = Wetted perimeter (m)
θ = Contact angle

Brewster's angle Microscope EP3



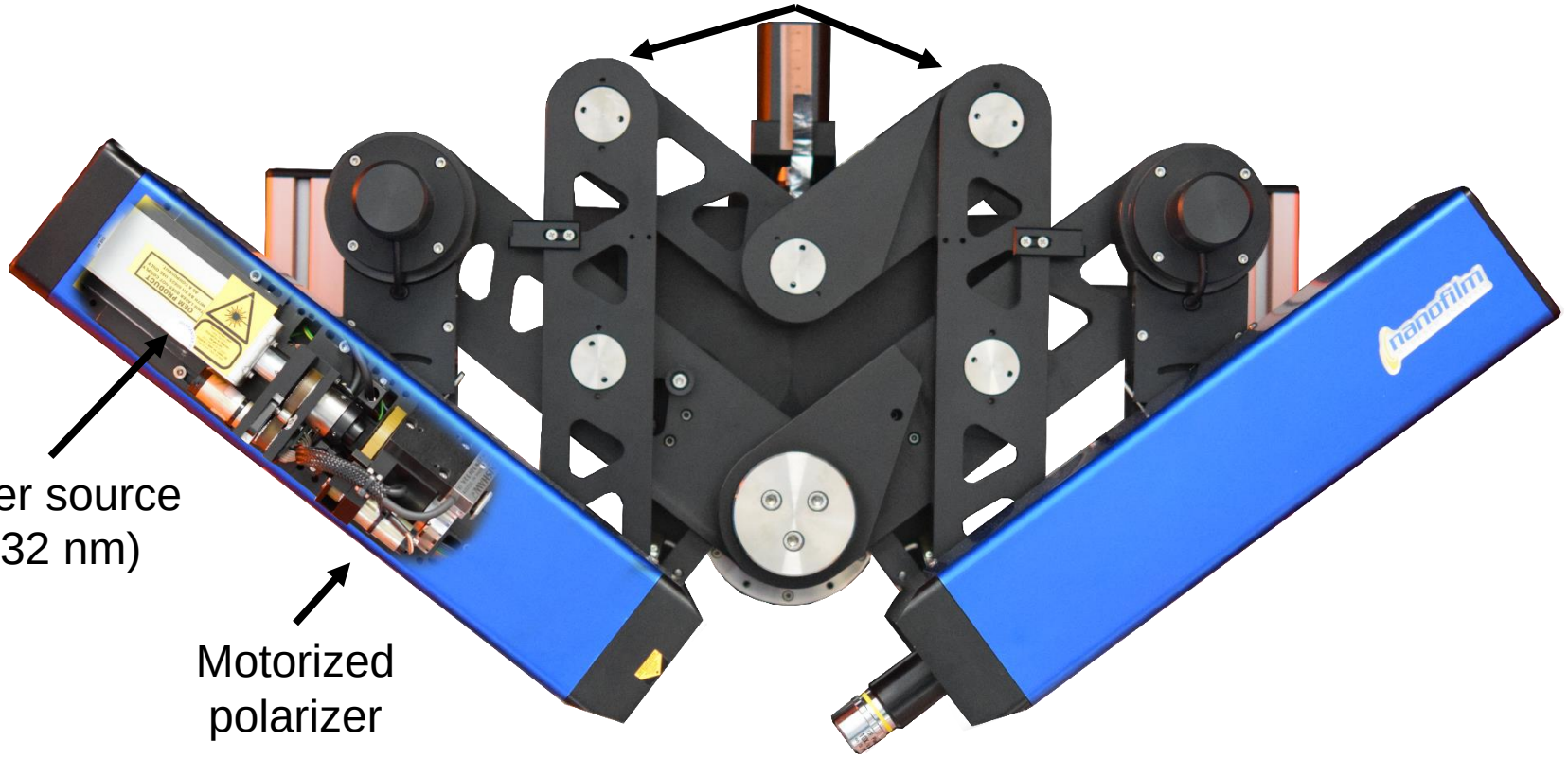
Brewster's angle Microscope EP3

Motorized goniometer:
Angle of incidence (AOI) range: 40-90°
Absolute angle accuracy: 0.01°



Brewster's angle Microscope EP3

Motorized goniometer:
Angle of incidence (AOI) range: 40-90°
Absolute angle accuracy: 0.01°

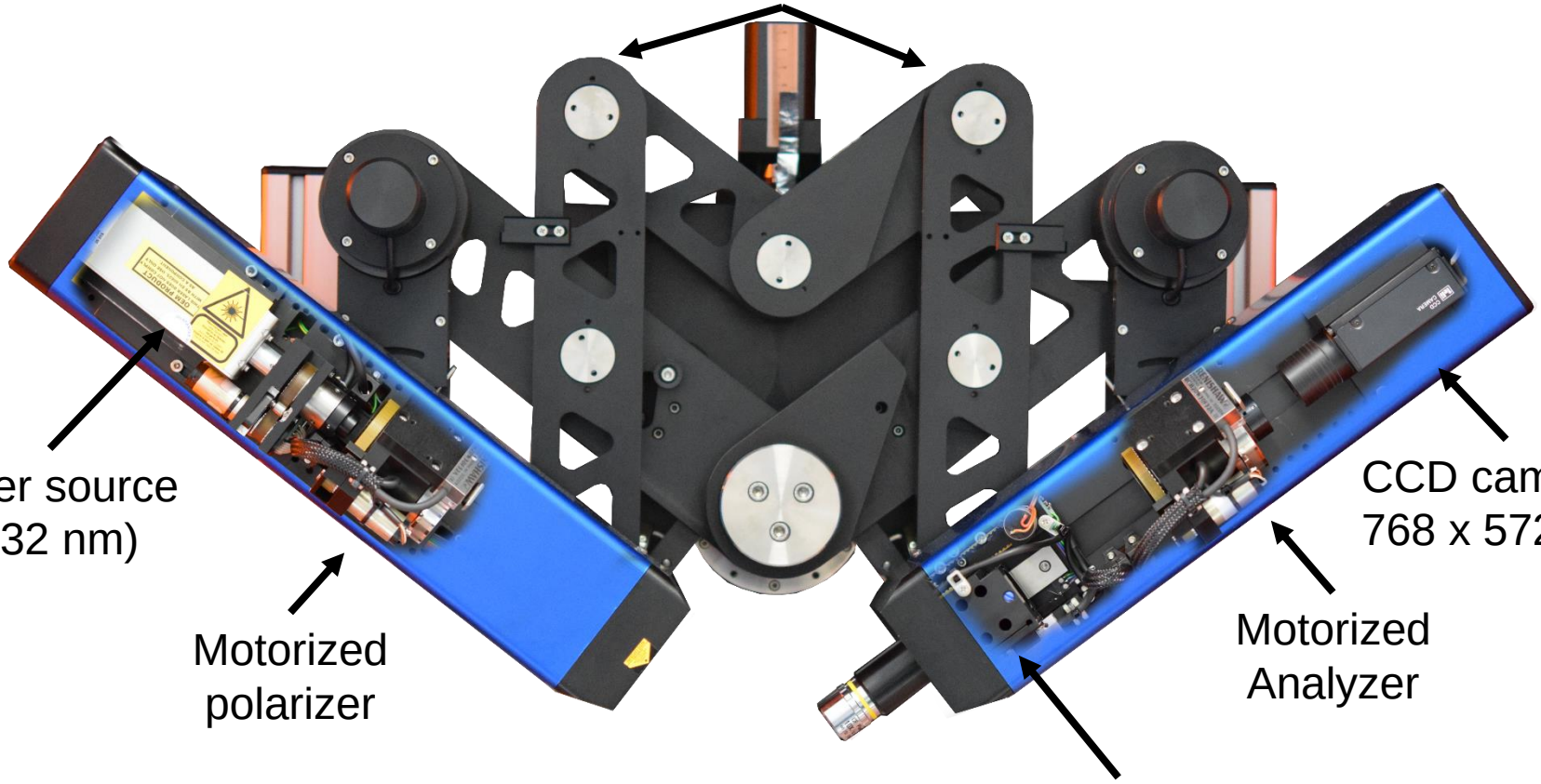


Laser source
(532 nm)

Motorized
polarizer

Brewster's angle Microscope EP3

Motorized goniometer:
Angle of incidence (AOI) range: 40-90°
Absolute angle accuracy: 0.01°



Laser source
(532 nm)

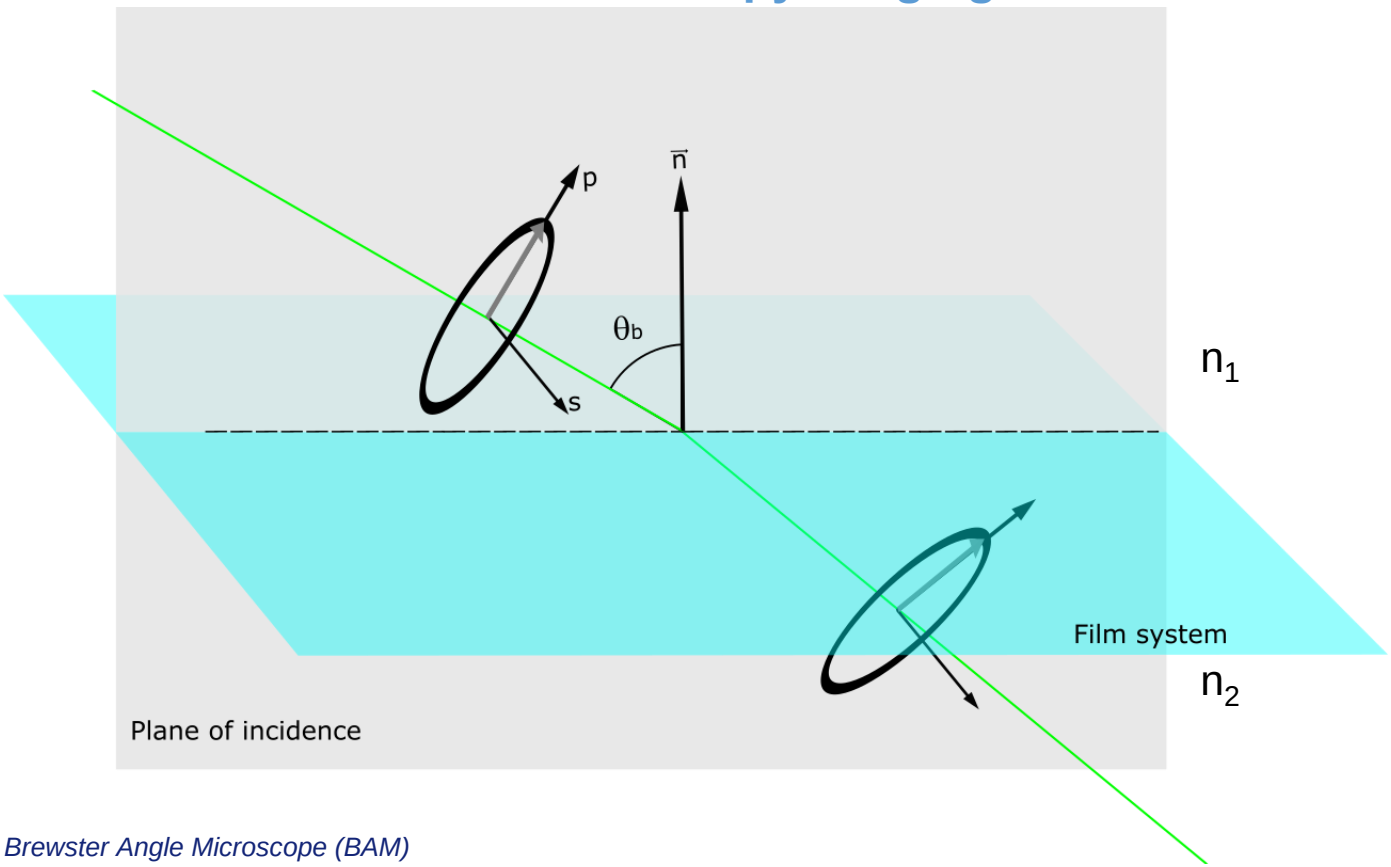
Motorized
polarizer

CCD camera
768 x 572px

Motorized
Analyzer

Focus scanner with 10X or
20X CF Plan Nikon
objectives

Generated contrast in Brewster's microscopy imaging

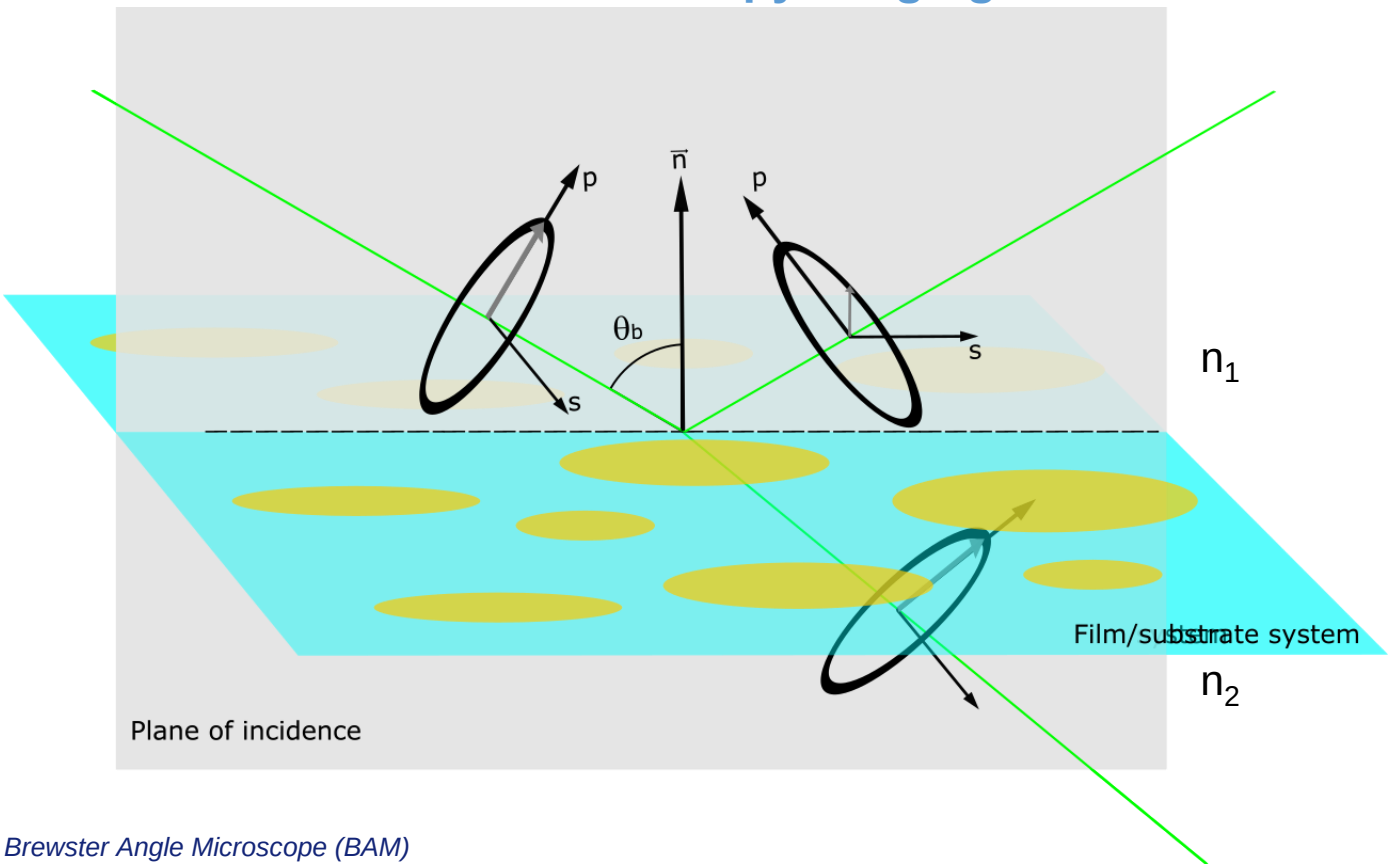


Sketch of the Brewster Angle Microscope (BAM)

- At the Brewster's angle p-polarized light is not reflected

$$\theta_b = \tan^{-1}\left(\frac{n_2}{n_1}\right)$$

Generated contrast in Brewster's microscopy imaging



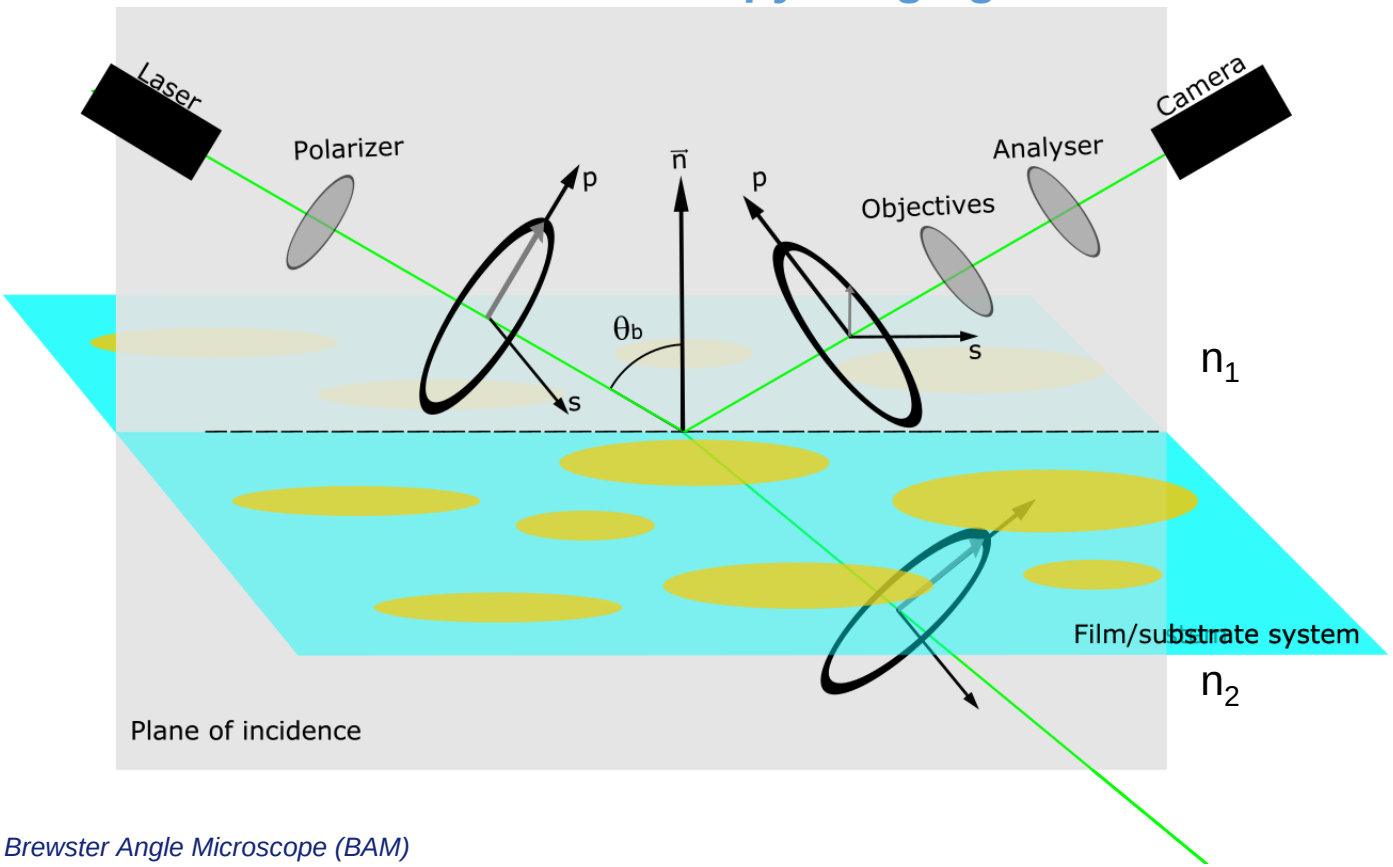
Sketch of the Brewster Angle Microscope (BAM)

- At the Brewster's angle p-polarized light is not reflected

$$\theta_b = \tan^{-1}\left(\frac{n_2}{n_1}\right)$$

- Introduction of thin film (nanoparticles, lipids…) on the surface modifies the local index of refraction and introduces local reflection and contrast on the film.

Generated contrast in Brewster's microscopy imaging



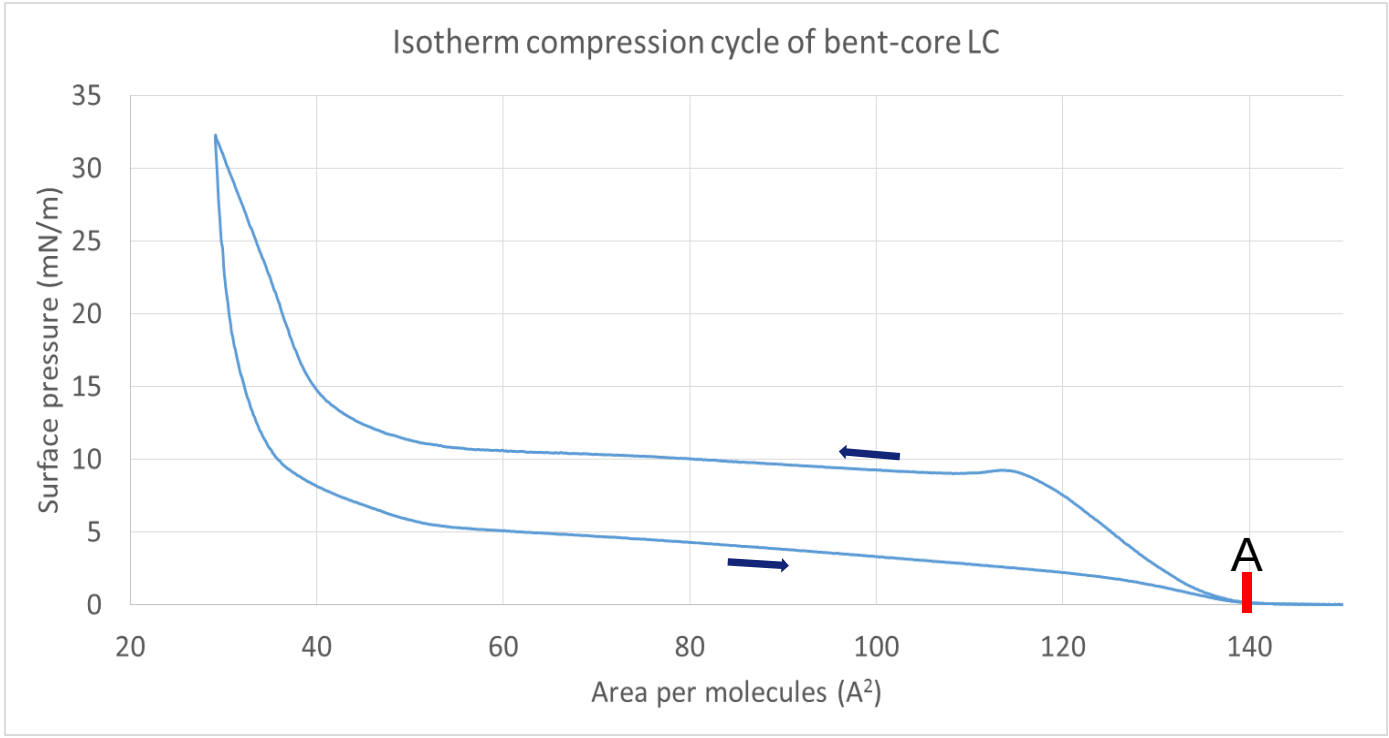
Sketch of the Brewster Angle Microscope (BAM)

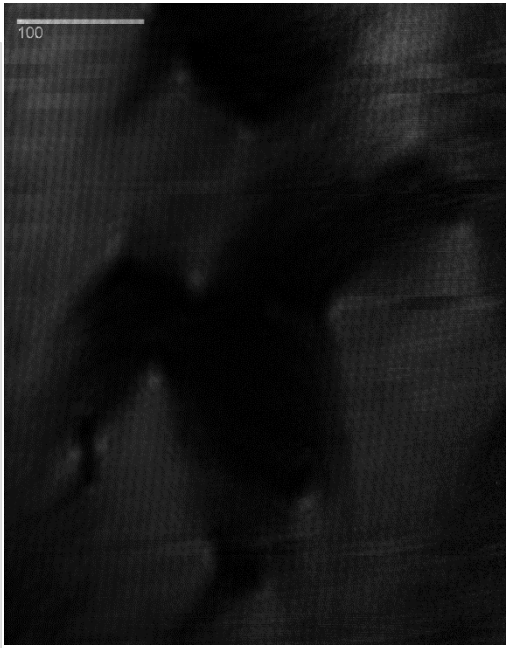
- At the Brewster's angle p-polarized light is not reflected

$$\theta_b = \tan^{-1}\left(\frac{n_2}{n_1}\right)$$

- Introduction of thin film (nanoparticles, lipids…) on the surface modifies the local index of refraction and introduces local reflection and contrast on the film.

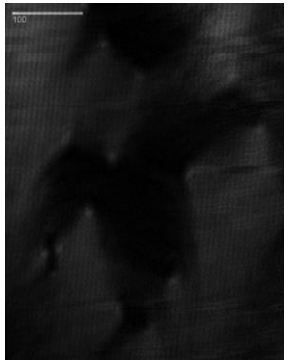
Imaging LC monolayer formation

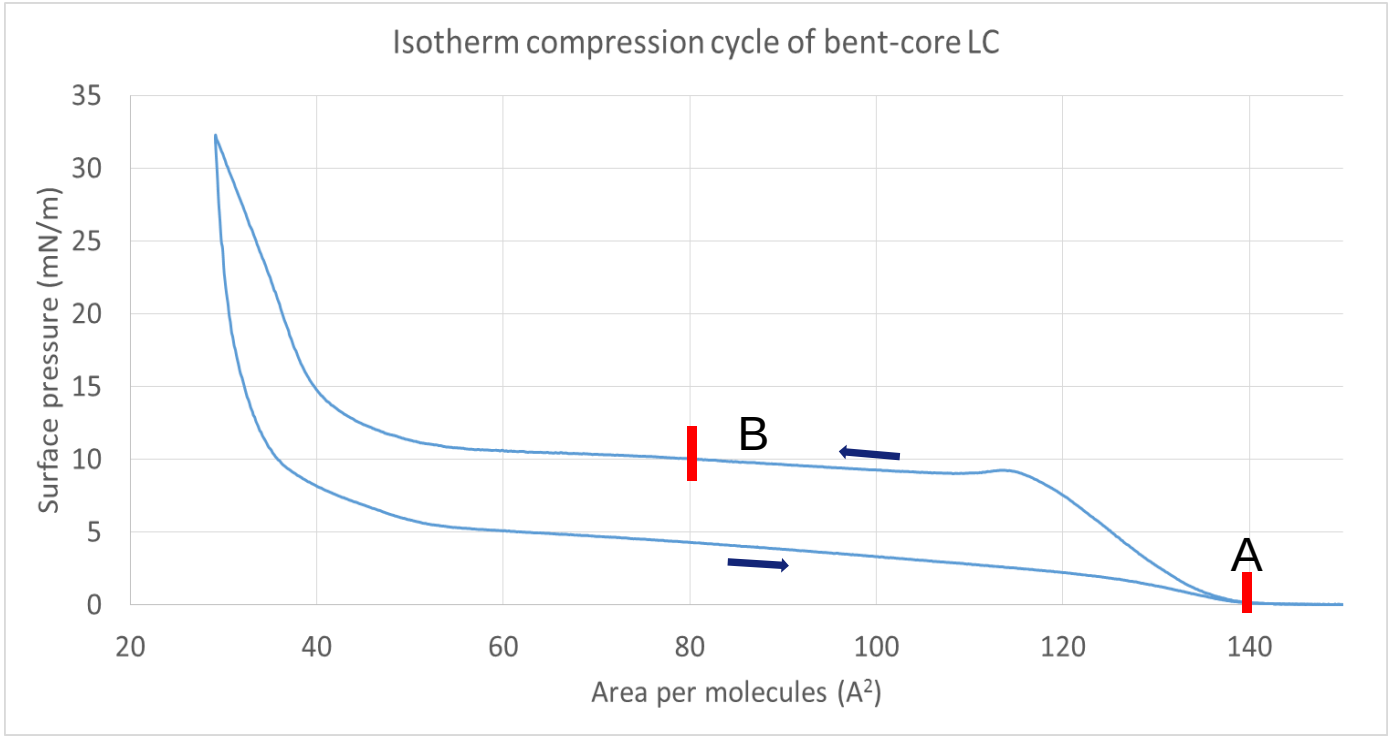


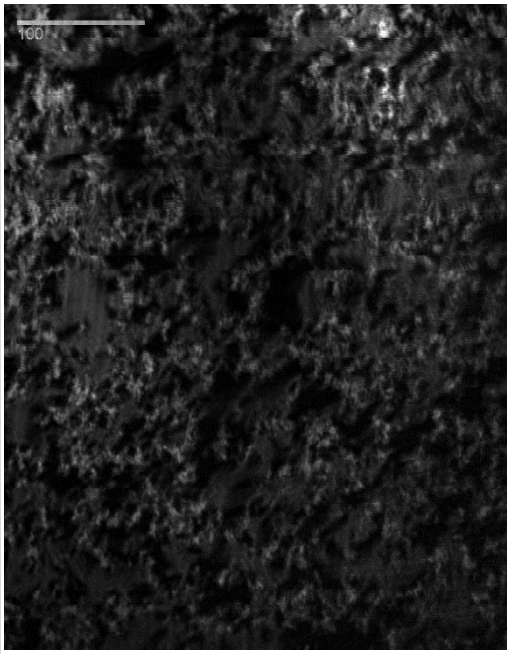


Imaging LC monolayer formation

A

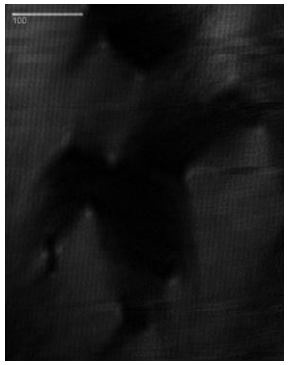




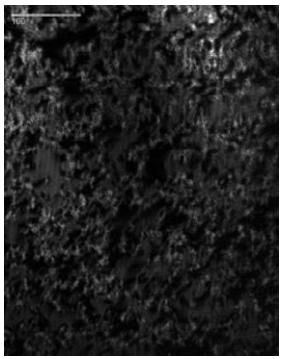


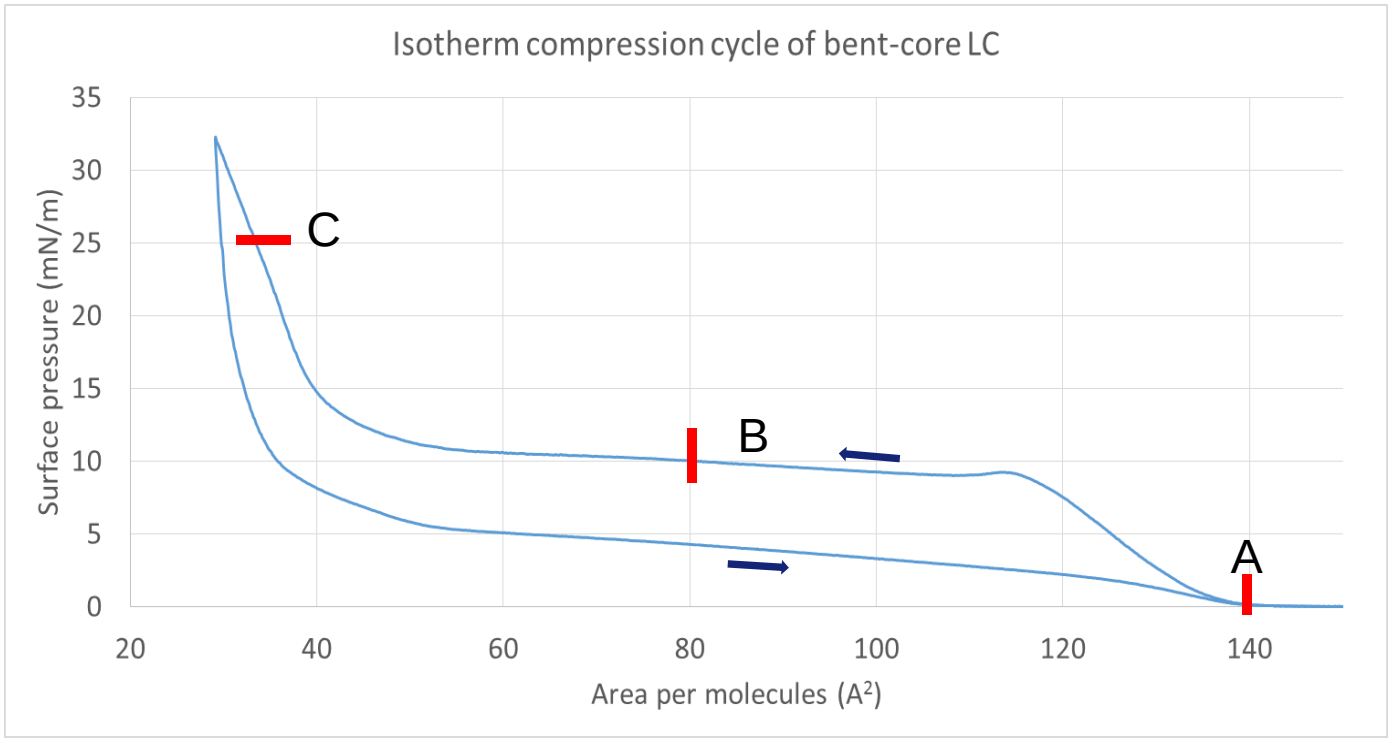
Imaging LC monolayer formation

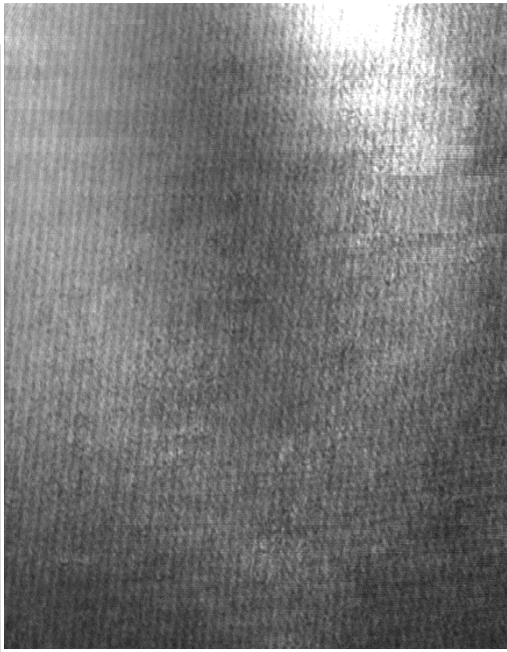
A



B

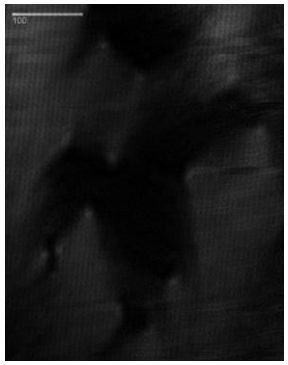




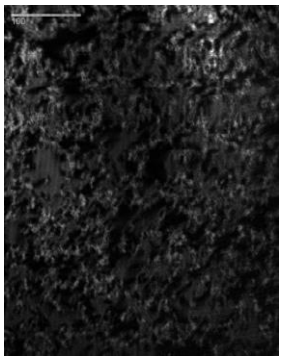


Imaging LC monolayer formation

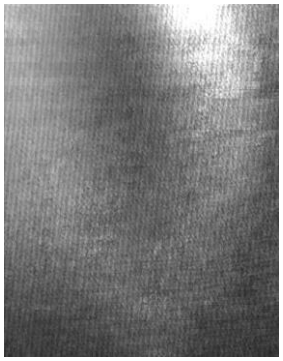
A

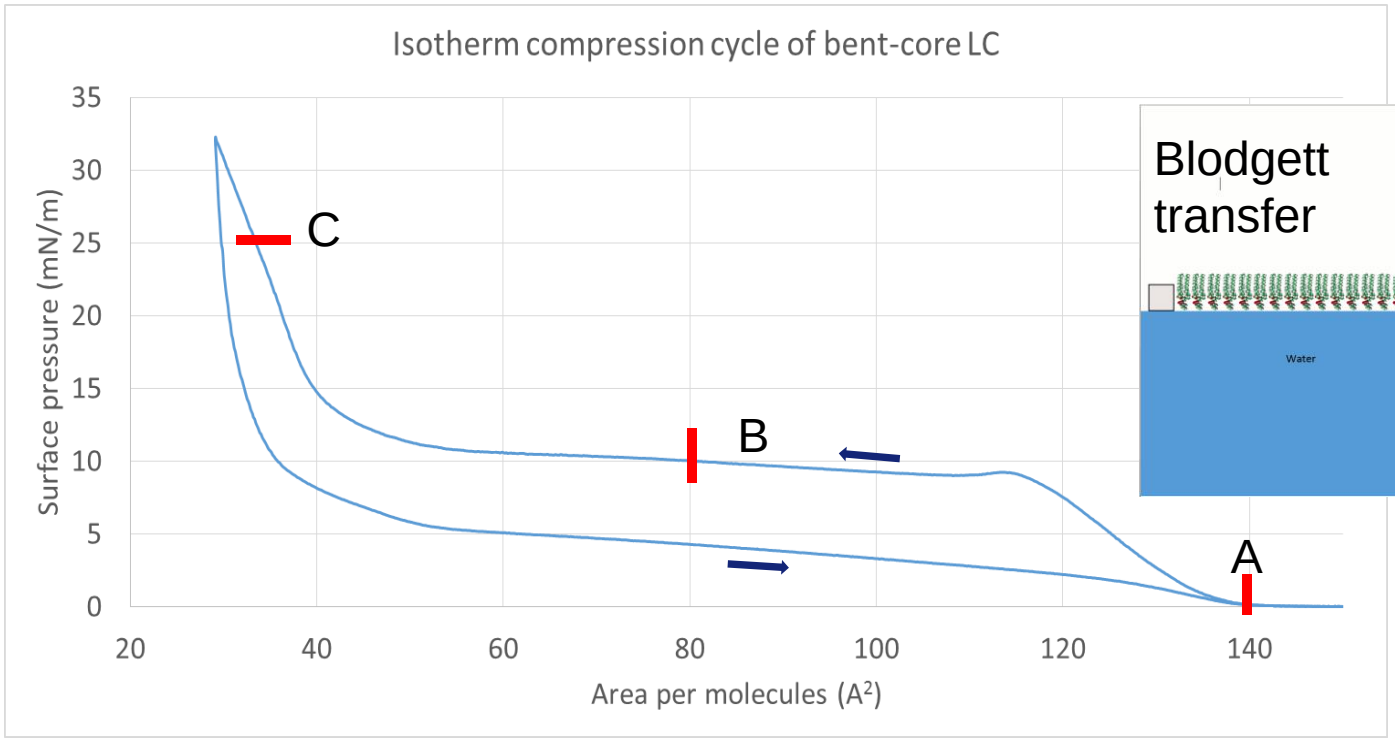


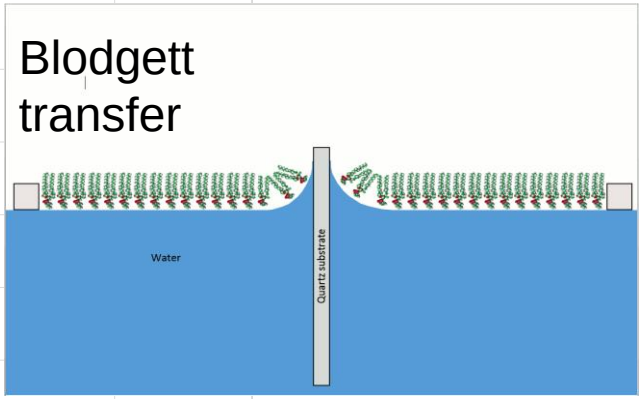
B



C







Atomic force microscope:

Asylum **R**esearch
CYPHER S
(Oxford Instruments)

Instrument parameters	
XY Scan size:	30x30 µm
Z Scan size:	5 µm
Sample Height:	5 mm
Excitation:	Piezo-Acoustic, Photo-Thermal
Operation Modes:	Contact, AC, Meca, Elec, PFM, Cond.
	Air, Droplet

Asylum **R**esearch **MFP 3D**
(Oxford Instruments)

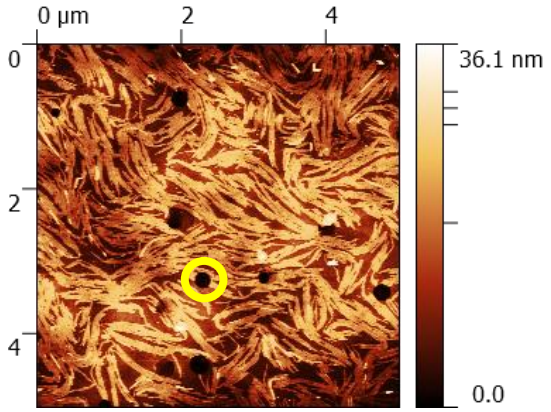
Instrument parameters	
XY Scan size:	90x90 µm
Z Scan size:	15 µm
Sample Height:	8 mm
Excitation:	Piezo-Acoustic
Operation Modes:	Contact, AC, Meca, Elec, PFM
	Air, Liquid

Digital **I**nstruments
Dimension 3100 *(Bruker)*

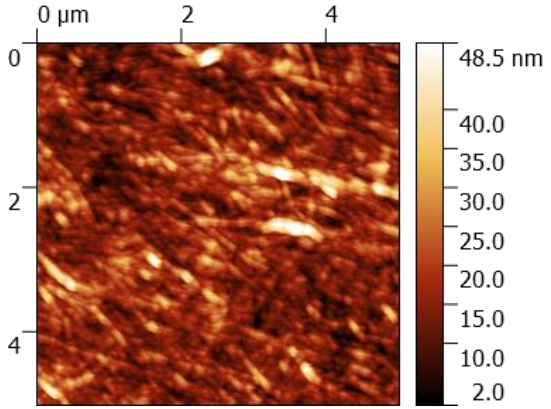
Instrument parameters	
XY Scan size:	100x100 µm
Z Scan size:	6 µm
Sample Height:	15 mm/30 mm
Excitation:	Piezo-Acoustic
Operation Modes:	Contact, Tapping
	Air, Droplet

Pre X-ray AFM imaging

- Checking the sample preparation
- Checking the sample surface
- Having a reference image before X-ray exposition



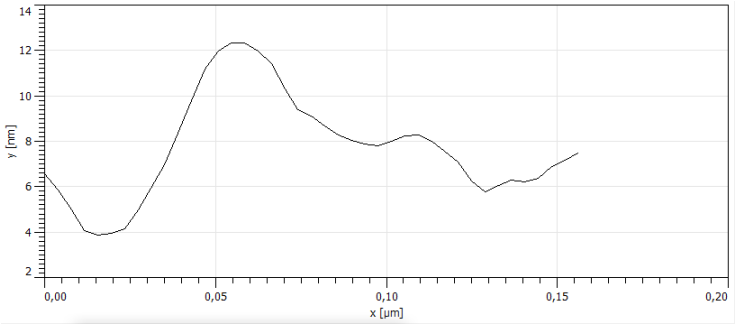
Mono-layer with holes

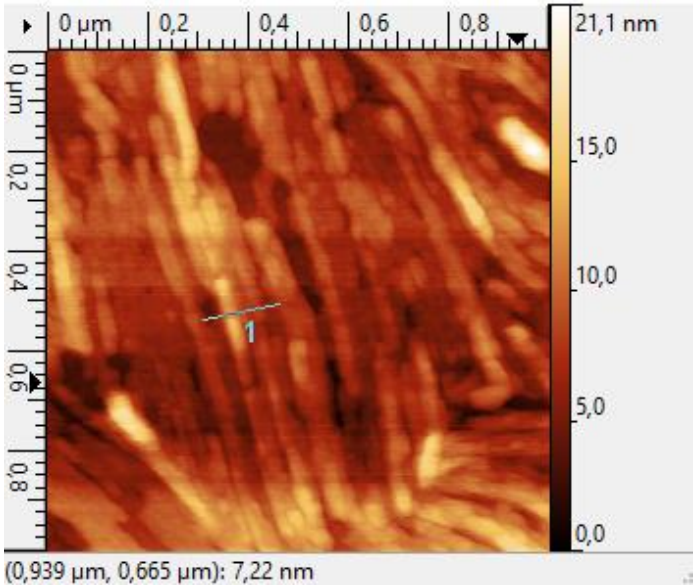


Several layers

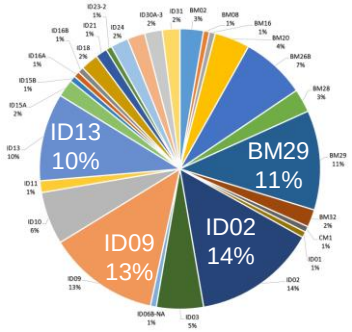
Post X-ray AFM imaging

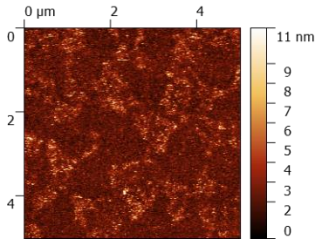
- Checking the effect of the beam on the sample
- Measuring the parameters of sample (size, roughness, …)
- Measuring properties that are not available from the X-ray data (mechanical, electrical, piezo response properties,…)





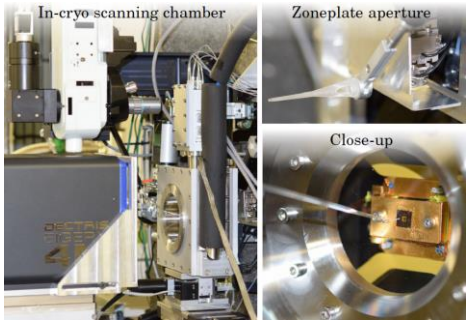
The PSCM labs were used by **75 ESRF staff** members and **300 ESRF users** related to nearly **200 ESRF beamtimes** carried out on 28 different ESRF stations



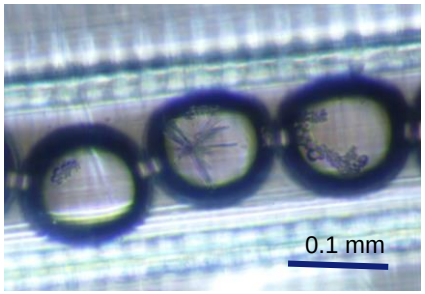


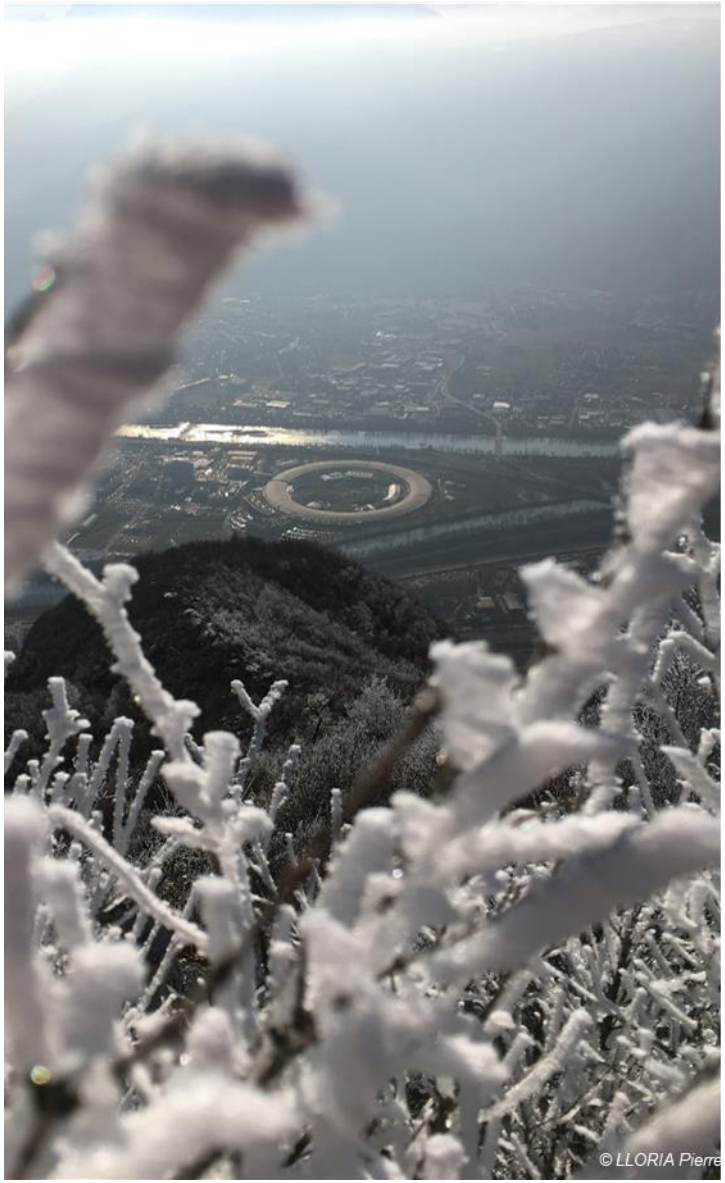
Manno et al.
BM29 (MX-1956)
Protein Aggregates

In **October 2018** the first open and competitive call for PSCM partnership programs received expressions of interest from **15** leading European Institutes.



The PSCM will deliver **advanced support services** to as many new Partners as possible during the **ESRF-EBS era from 2020 onward.**





Thank you for your attention