Liquid and material properties measurements

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Workshop on standardization of test methods in microfluidics

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Outline

Introduction: Material properties and liquid properties

Wettability

Surface roughness

Density

Viscosity

Refractive index

Summary and conclusion

Introduction: Material properties

MFMET
Establishing Metrology Standards in Microfluidic Devices

Properties of the materials used for microfluidic devices

Wettability

The degree to which a solid and a liquid maintains adhesive contact [ISO 19403-1]

Wettability can affect the behaviour of fluid flow in microfluidic devices.

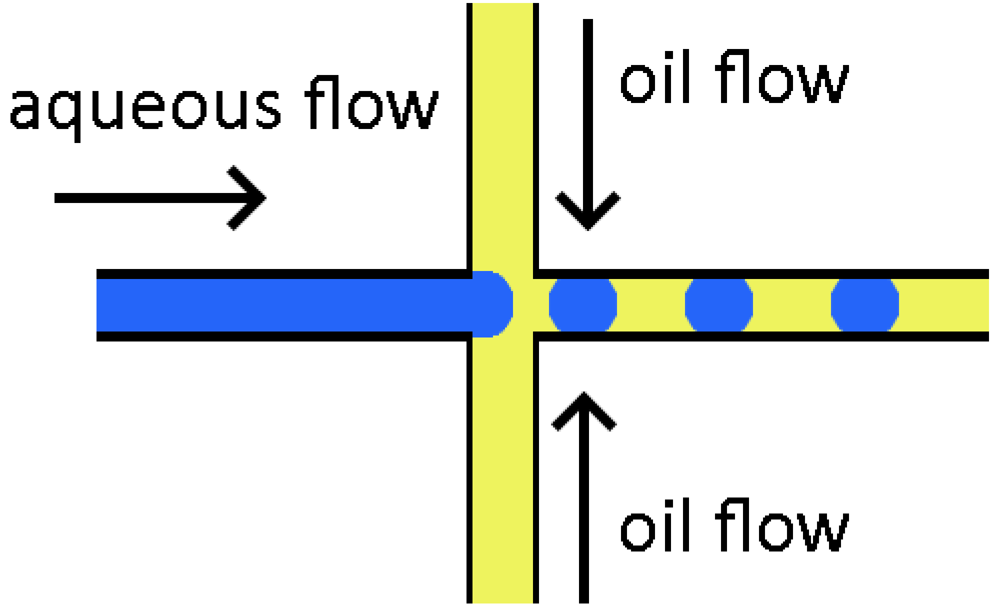
Special mechanisms:
Droplet formation and evolution
Spreading of a fluid on a surface

Surface roughness

Related to the quality of a surface of not being smooth
[ISO 25179-2, ISO 21920-2]

Higher surface roughness can contribute to extra friction for flow

Diagram of droplet formation in a microfluidic device



Credit: Wikimedia commons, Wongbria, 2020

Introduction: Liquid properties

Properties of a liquids used in microfluidic devices

Density

A substance's mass per unit of volume
May influence flow associated with height differences

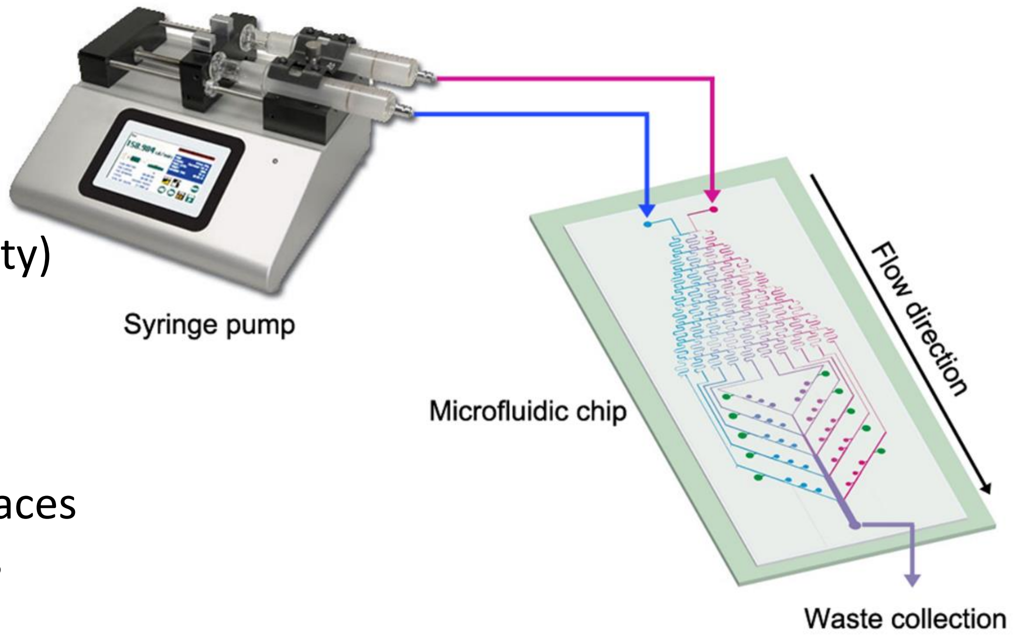
Viscosity

A fluid's resistance to deformation, or "thickness" (here dynamic viscosity)
May influence the flow by contributing to the friction of the flow

Refractive index

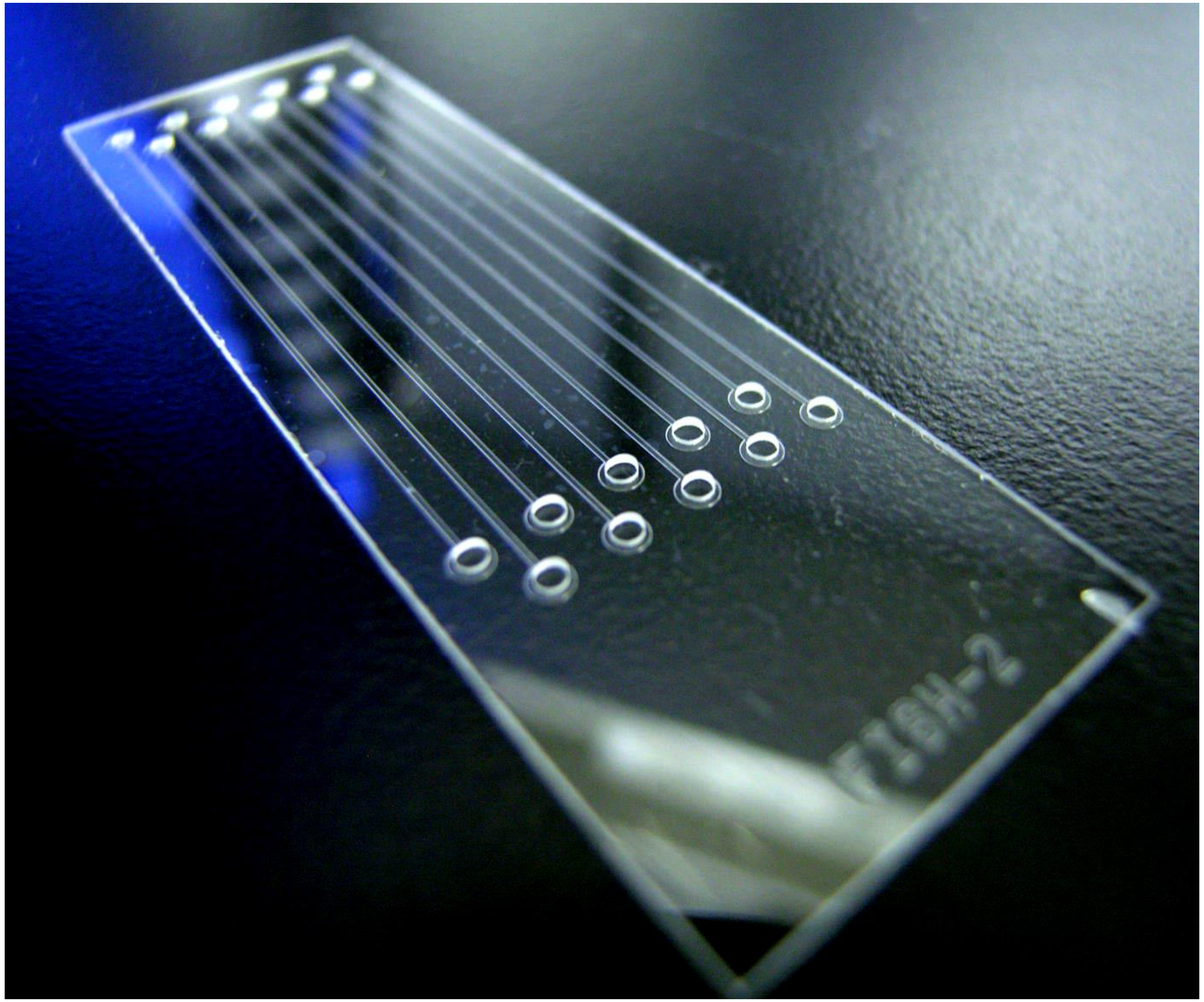
A measure of a materials ability to refract (bend) light at material interfaces
May influence optical detection techniques for microfluidic applications

Illustration of a microfluidic device. A more viscous fluid would exert a greater friction to be pumped through microchannels



Credit: Wikimedia commons, Xiliang Tian, 2012

Material properties

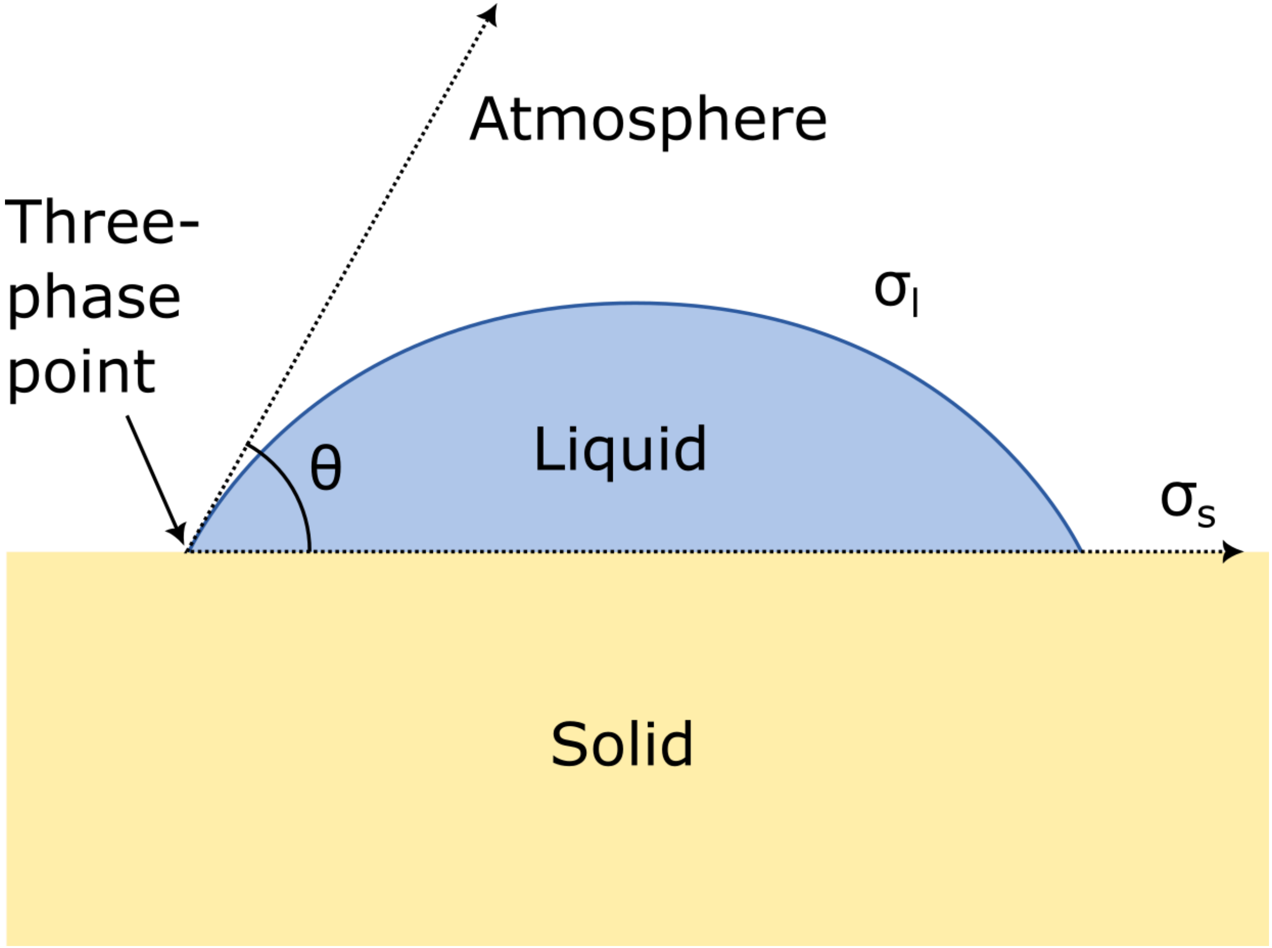


A microfluidic chip made of Polydimethylsiloxane (a polymer called PDMS) and mounted on a glass slide

Credit: Wikimedia commons, Vincent J. Sieben et al., 2008

Wettability: Contact angle

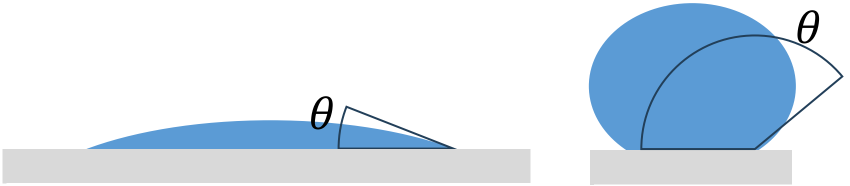
The contact angle θ is the angle between the drop base and the drop tangent at the three-phase point.



Credit: Daugbjerg et al. unpublished

The contact angle θ depends on:

σ_l - the surface tension of the liquid
σ_s - the surface energy of the surface



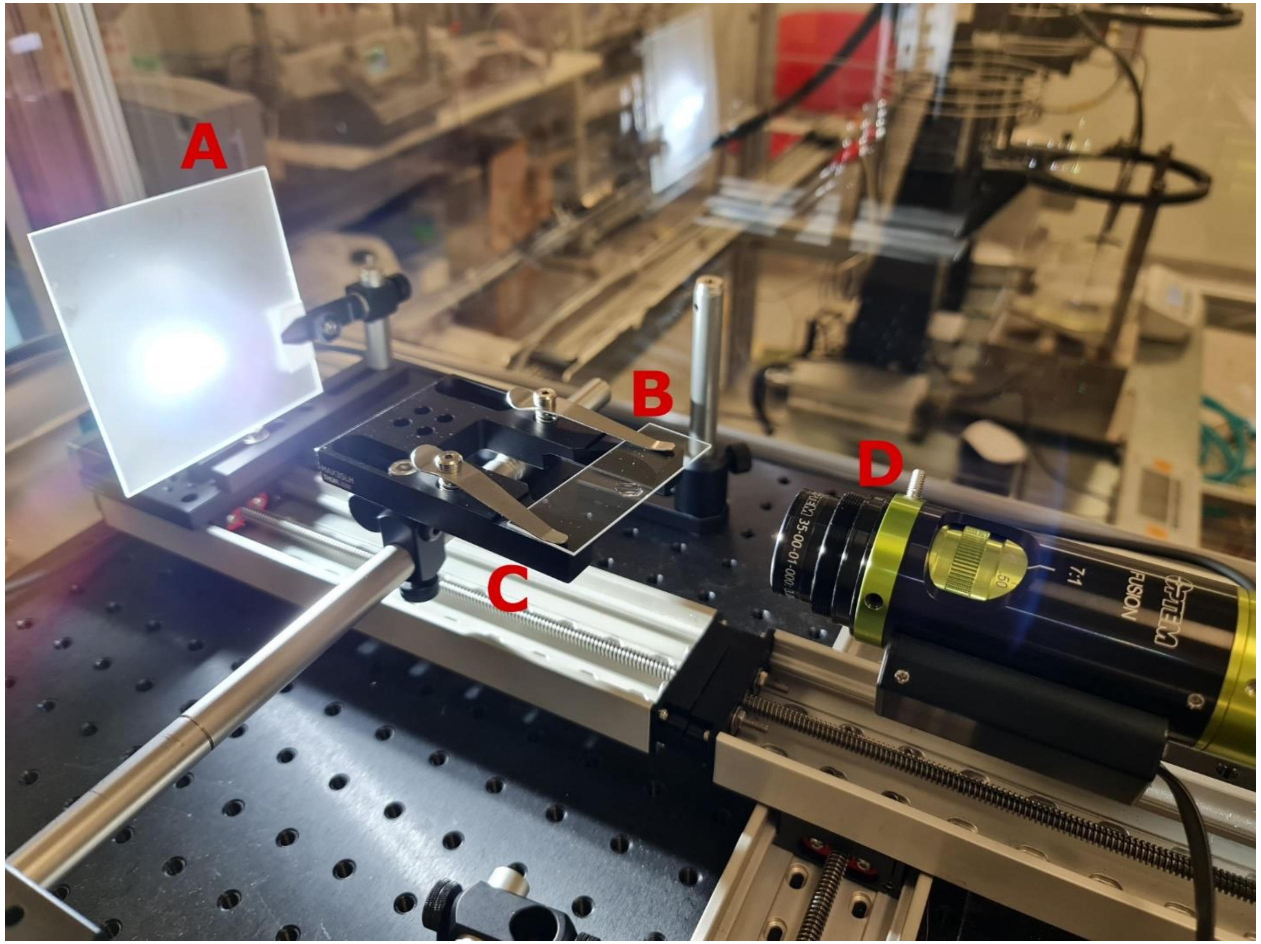
Hydrophilic
Low σ_l
High σ_s

Hydrophobic
High σ_l
Low σ_s

Credit: CETIAT

Wettability: Contact angle

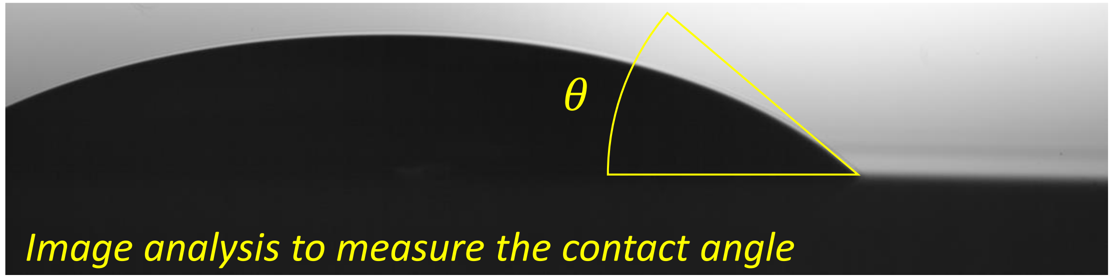
Measurement of contact angle is described in ISO 19403-2.



Credit: CETIAT

The optical setup at CETIAT in Villeurbanne, France

A) White diffused lighting
B) Slide under test
C) Slide holder
D) High resolution microscope camera.



Credit: CETIAT

MFMET Establishing Metrology Standards in Microfluidic Devices

Wettability: Surface energy

The Owens-Wendt-Rabel-Kaelble (OWRK) model considers surface energy σ_s and surface tension σ_l to be sums molecular interactions from a polar (p) component and a dispersive (d) component (ISO 19403-1):

$$\sigma_l = {\sigma_l}^p + {\sigma_l}^d$$

$$\sigma_s = {\sigma_s}^p + {\sigma_s}^d$$

…and formulates a linear equation relating contact angle θ, surface energy σ_s, and surface tension σ_l.

$$y = c + m \cdot x$$

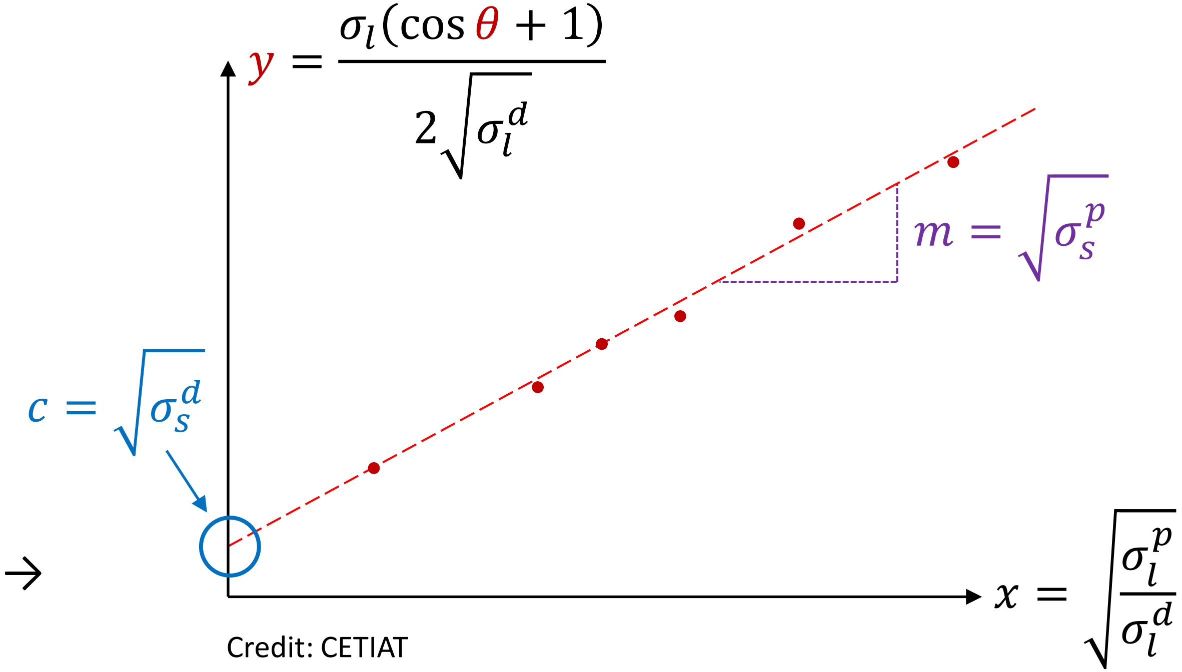
$$\frac{\sigma_l(\cos\theta + 1)}{2\sqrt{\sigma_l^d}} = \underbrace{\sqrt{\sigma_s^d} + \sqrt{\sigma_s^p}}_{\text{Surface energy}} \cdot \sqrt{\frac{\sigma_l^p}{\sigma_l^d}}$$

Measured contact angle

Surface energy

Liquid properties from literature

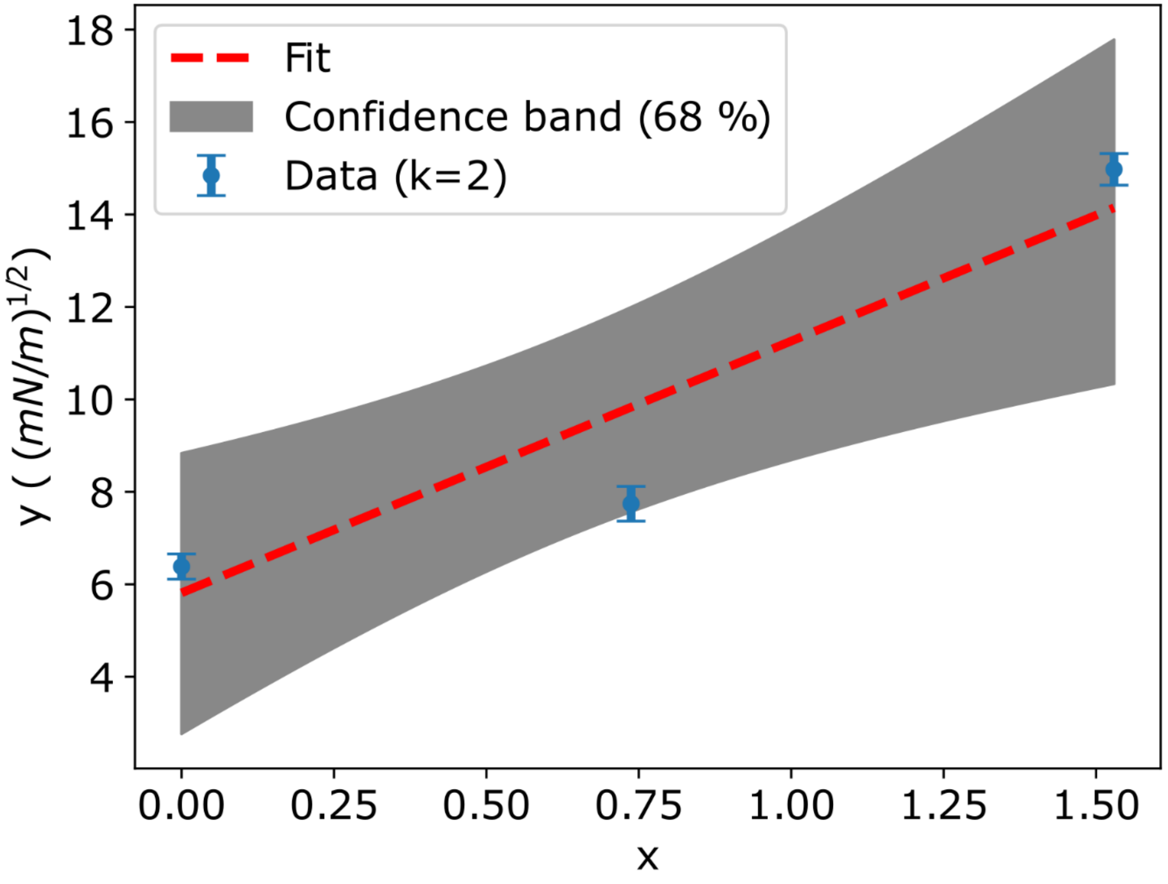
Several contact angle measurements with liquids of known surface tension may determine the surface energy of a sample→



Credit: CETIAT

Wettability: Surface energy

OWRK model with three liquids (water, di-iodomethane, ethylene glycol) on borosilicate glass slide (D263® bio)



Credit: Daugbjerg et al. unpublished

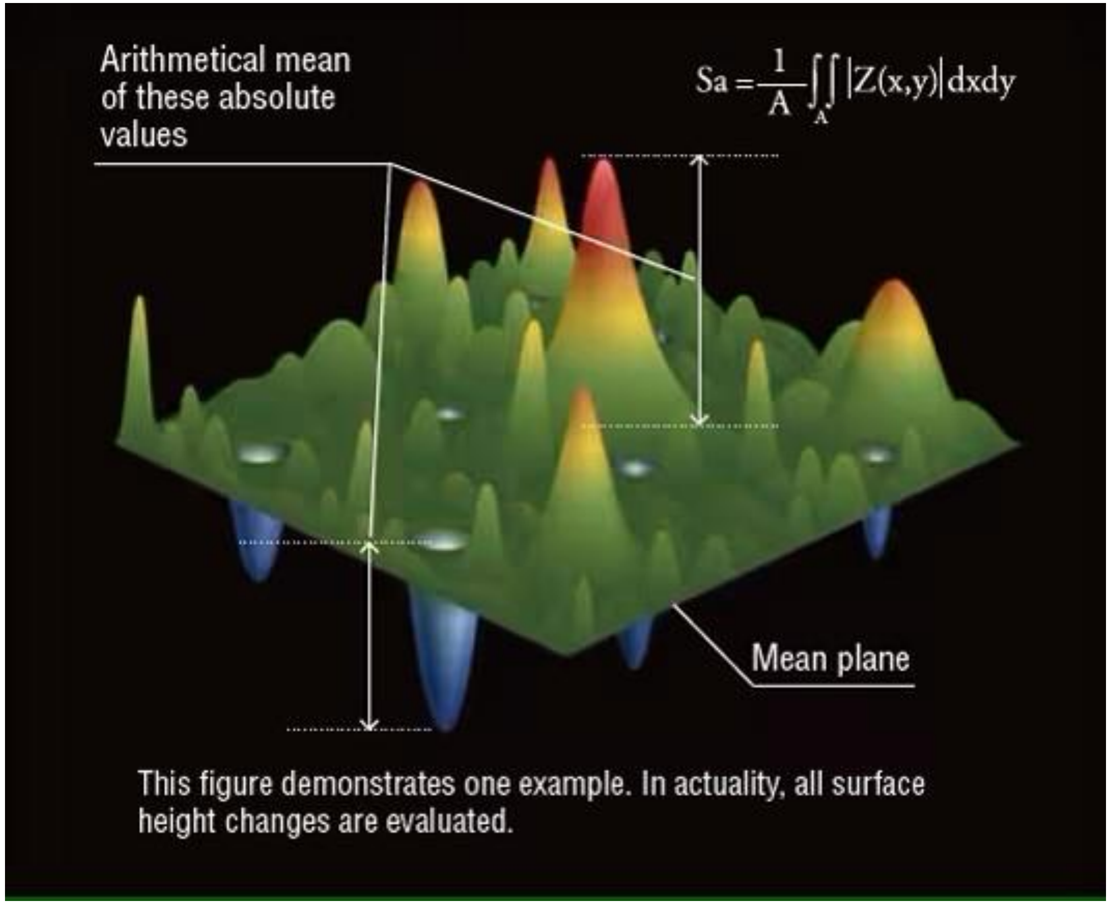
Values for σ_s^p, σ_s^d, and σ_s from the fit (k=2)

Parameter	Value ± unc. (mN/m)
σ_s^p	30 ± 40
σ_s^d	34 ± 38
σ_s	63 ± 55

Mitigation of difference between data and fit: Fitting with LMFIT with scale_covar=True (uncertainty of fit parameters were scaled according to this difference)

Surface roughness

Surface roughness is related to irregularities of a surface that makes a surface not-smooth.



Surface roughness is quantified by surface roughness parameters, e.g. area surface roughness parameter S_a

$$S_a = \frac{1}{A}\iint_A |z(x,y)|dxdy$$

(average of the absolute height, notice that valleys and peaks don't cancel each other)

Reference: ISO 25178-2:2021

Illustration of a 2D surface with surface roughness. Credit: keyence.com

Atomic force microscopy (AFM)

AFM works by scanning a surface with a sharp probe, while measuring the interaction of probe and surface.

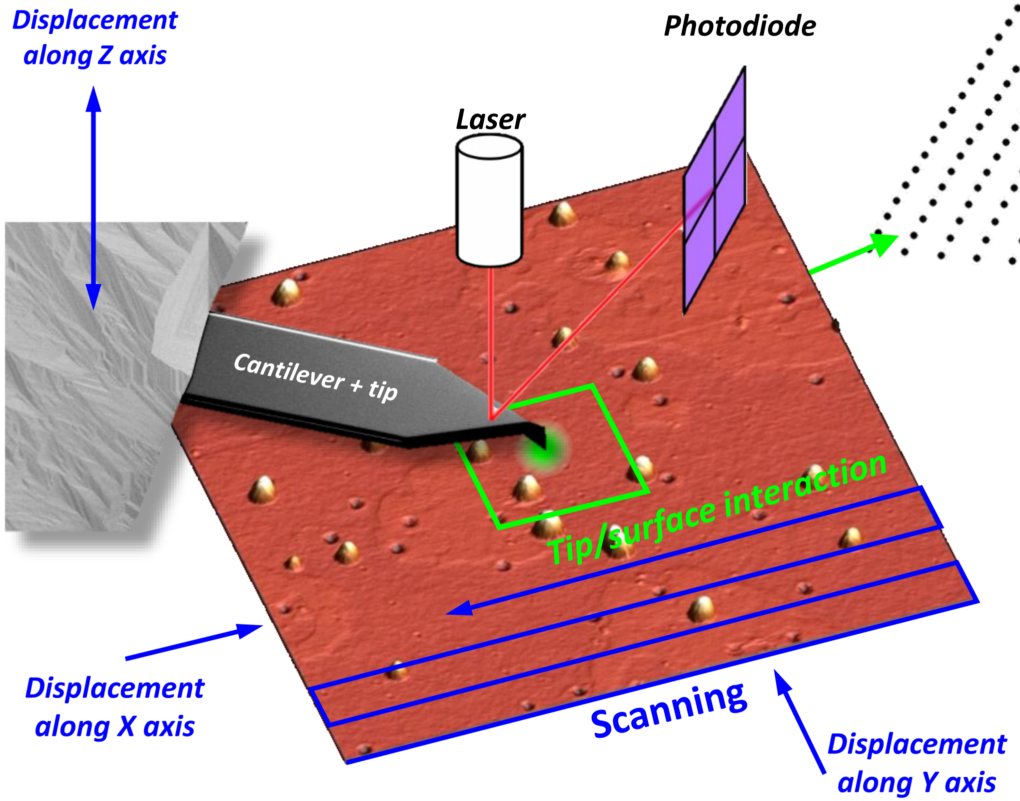
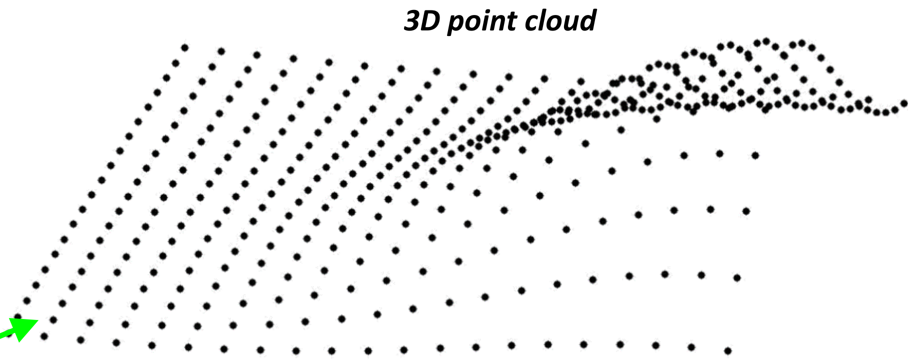
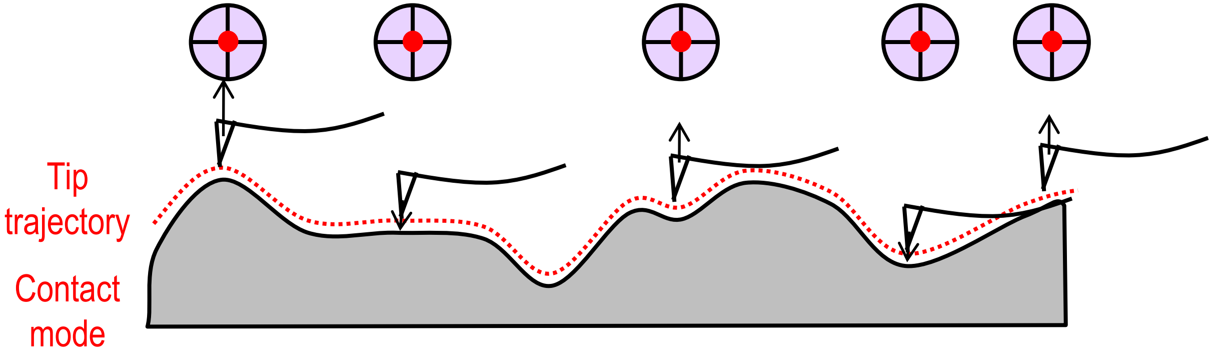


Figure credit: LNE



The instrument produces an image from the 3D point cloud acquired during the scan. Each pixel has XYZ coordinates, ideally acquired by position sensors.

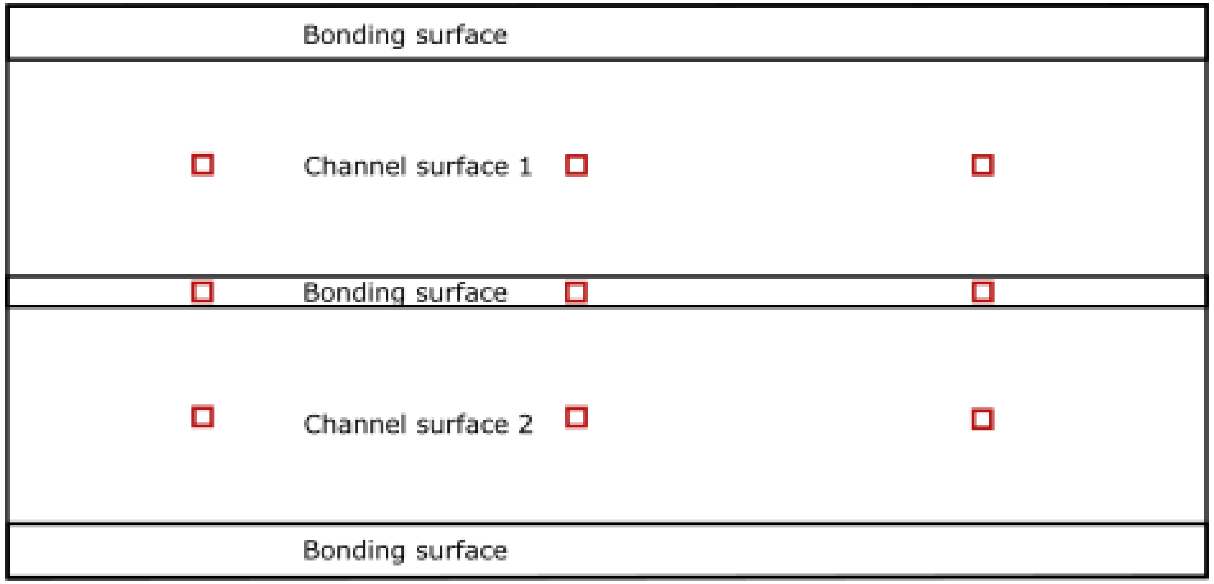


Instrument of this project: A Veeco Dimension 3100 in "tapping mode" at LNE at Trappes, France.

AFM results

Surface roughness measurements with AFM at nine locations for three samples of glass substrates with open channels

- Atomic force microscopy measurement



Credit: Daugbjerg et al. (unpublished)

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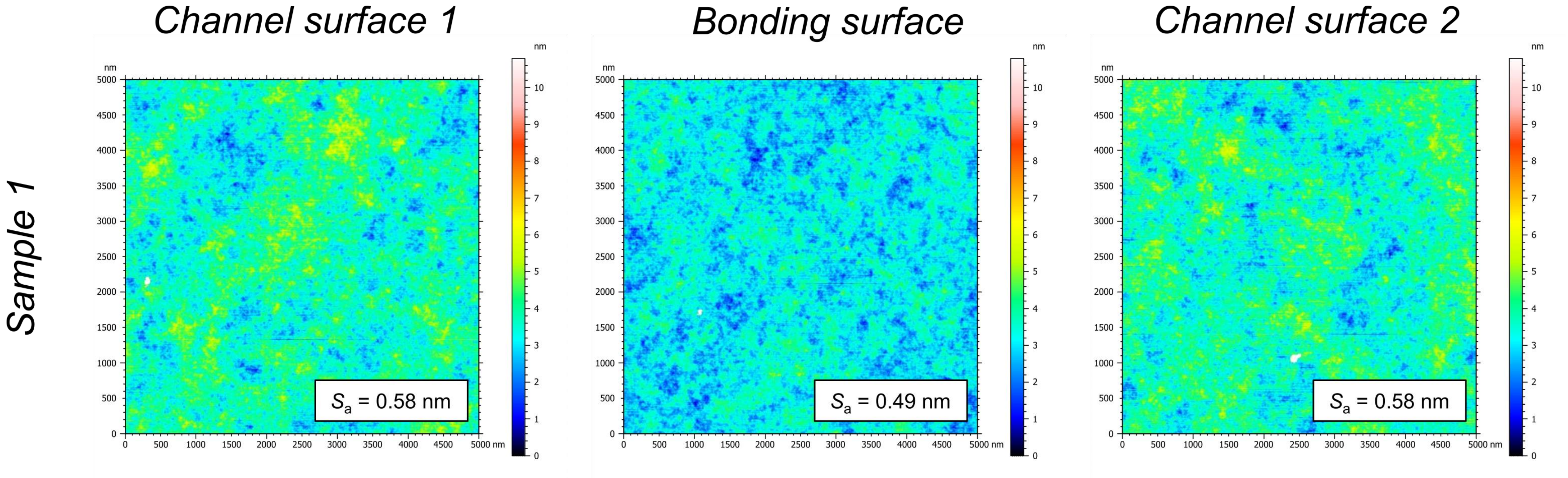
		AFM (k=2)
Sample	Location	S_a (nm)
2	Channel surface 1	0.60 ± 0.04
	Channel surface 2	0.60 ± 0.06
	Bonding surface	0.51 ± 0.04
3	Channel surface 1	0.56 ± 0.06
	Channel surface 2	0.56 ± 0.02
	Bonding surface	0.49 ± 0.04

Channel surfaces are slightly rougher than bonding surfaces

AFM results

Surface roughness measurements with AFM shown as topographic maps



Channel surfaces were manufactured using wet etching, and appear more homogeneous than the bonding surface

Credit: Daugbjerg et al. (unpublished)

Liquid properties

Impact of a water drop on a surface of water

Credit: Wikimedia commons, José Manuel Suárez, 2008

Density measurements

Density measurements were made using an Anton Paar DMA 5000 instrument at IPQ in Caparica, Portugal.

Credit: Batista et al. 2024

Credit: Batista et al. 2024

(20 ˚C)	Water	PBS	SBF	U
Density (kg/m^3)	998.203*	1005.225	1007.523	0.033

PBS = Phosphate Buffered Saline (pH and osmolarity ≈ human)
SBF = Simulated Body Fluid (ion conc. ≈ human blood plasma)

Measurements of density vs temperature is a possibility.

Credit: Anton Paar

Some digital density meters use an oscillating U-shaped tube to determine density. The oscillation frequency depends on the density of the liquid filling the tube.

*Spieweck, F. & Bettin, H.: Review: Solid and liquid density determination. Technisches Messen 59 (1992), pp. 285-292.

Viscosity measurements

Viscosity measurements were made using an Ubbelohde viscometer (glass capillary) at IPQ in Caparica, Portugal.

Markings for determining the volume

Glass capillary

Credit: Batista et al. 2024

Brief note on the concept:

- Measure the time it takes for the volume to flow through the glass capillary → flow rate
- Assumes Poiseuille flow in the glass capillary → Determines viscosity from flow rate

Credit: Batista et al. 2024

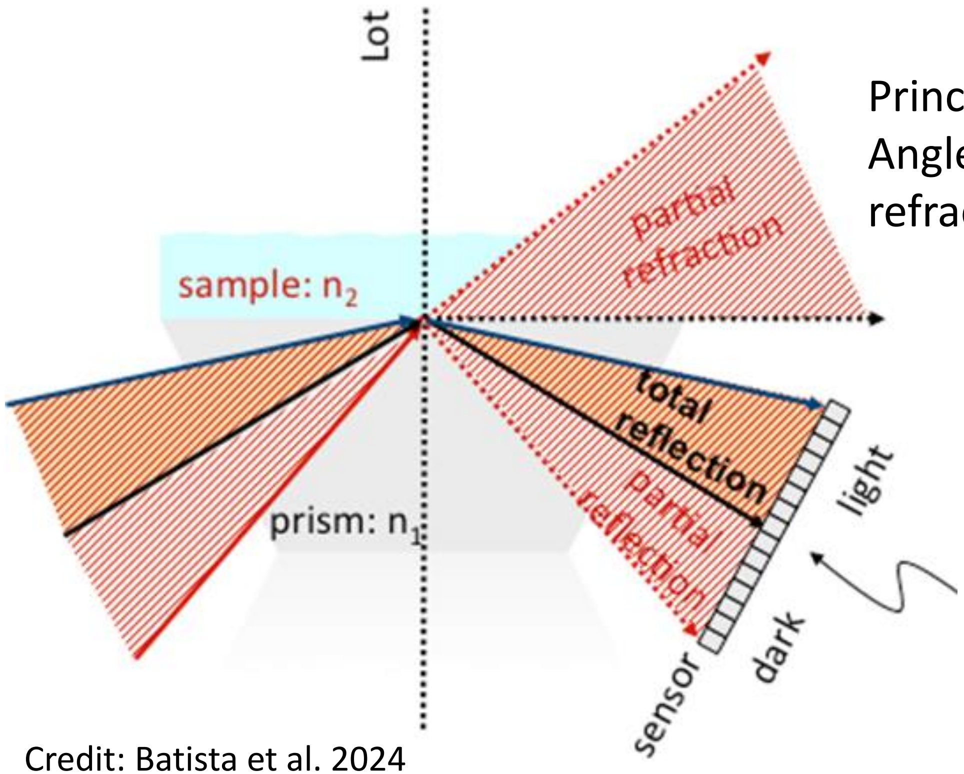
20 ˚C	Viscosity, μ (mm^2/s)	U (mm^2/s)
Water	1.0034*	0.0017*
PBS	1.0386	0.0055

Viscosity not measured for non-Newtonian SBF

* ISO/TR 3666:1998 (E) Viscosity of water

Refractive index measurements

Refractive index measurements were made using an Anton Paar Abbemat 550 refractometer at IPQ in Caparica, Portugal.



Credit: Batista et al. 2024

Principle of refractometer
Angle of total reflection → refractive index

Credit: Batista et al. 2024

20 ˚C	Water	PBS	SBF	U
Refractive index	1.332986*	1.334656	1.335886	0.000009

* OIML R 124, edition 1997 (E)

Credit: Batista et al. 2024

Summary and conclusion

We saw test methods for material properties and liquid properties relevant for microfluidics:

Wettability

Surface roughness

Density

Viscosity

Refractive index

The test methods and their quantities may be used in characterization and standardization of microfluidics, and contribute to a common language and harmonization in microfluidics.

Project Team

THANK YOU

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