



Morphologie et rugosité des surfaces de verres



Damien Vandembroucq

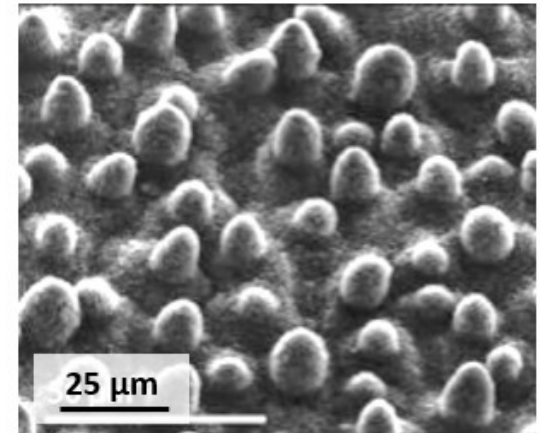
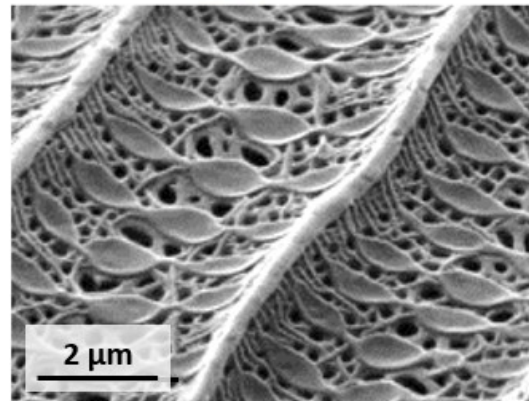
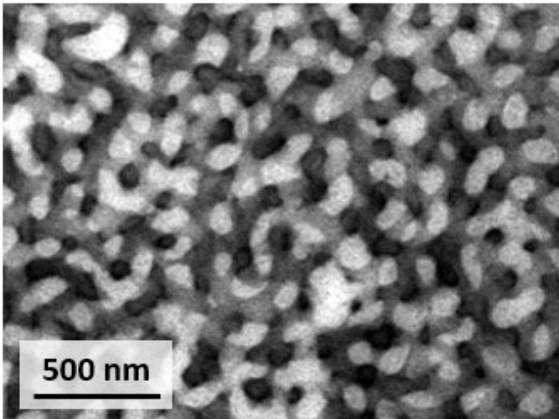
Laboratoire PMMH, ESPCI Paris PSL, CNRS,
Sorbonne Université, Université de Paris France
(Laboratoire SVI, CNRS/Saint-Gobain)

Ecole Surface des Verres, Oléron 16-20 octobre 2023





Surfaces texturées : bio-inspiration



Float glass process



Verres imprimés



SGG PIXARENA



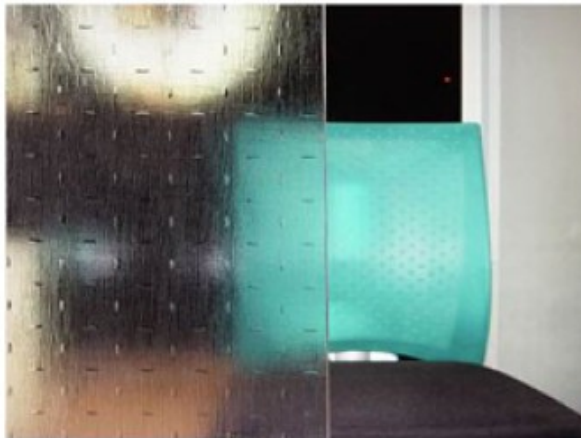
SGG LISTRAL M



SGG WATERDROP



SGG ENTRELAZADO



SGG MARIS

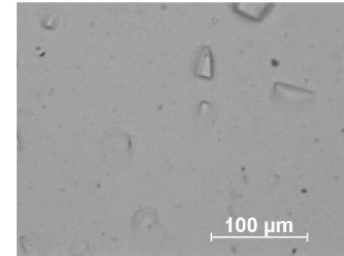


SGG THELA

Verres dépolis

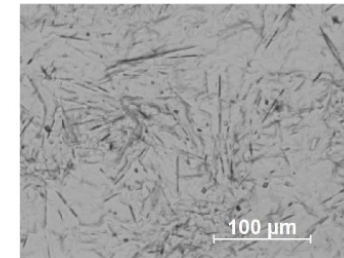
Planilux

G1



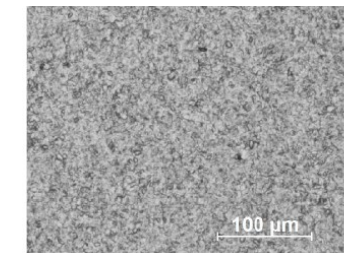
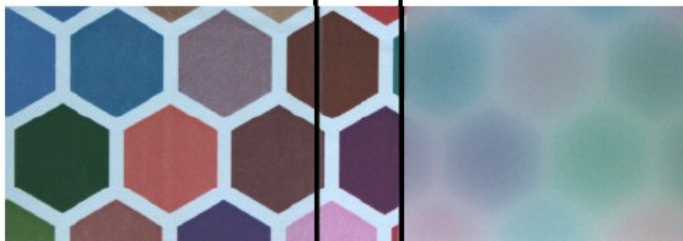
Planilux

G2



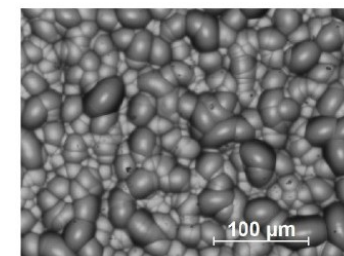
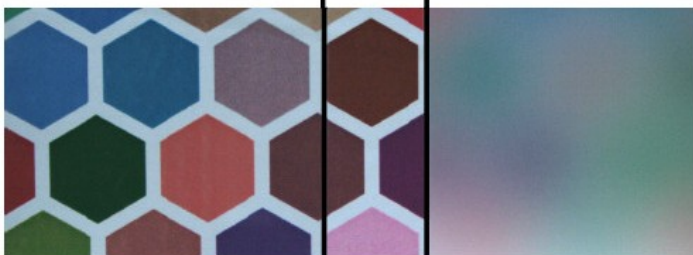
Planilux

G3



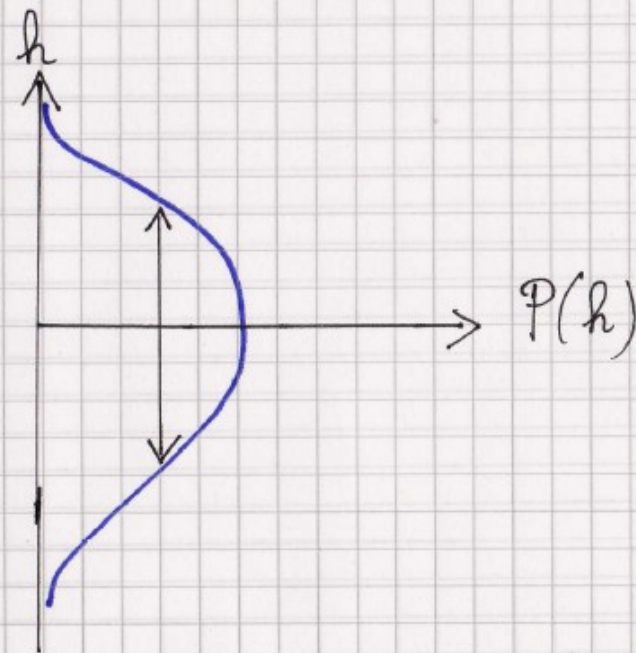
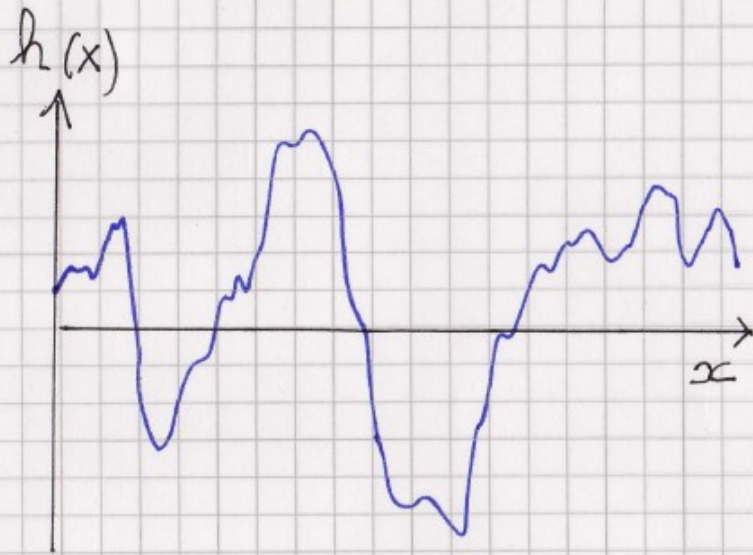
Planilux

G4



Interfaces rugueuses

Rugosité : distribution des hauteurs



▫ Approche probabiliste

▫ Un paramètre essentiel : la LARGEUR de la distribution

- plusieurs estimateurs pour une même quantité

$$R_q = \text{RMS} = \text{écart-type de la distribution} = \left(\frac{\sum h_i^2}{N} \right)^{\frac{1}{2}} = \left(\int h^2 P(h) dh \right)^{\frac{1}{2}}$$

$$R_a = \frac{1}{N} \sum_i |h_i| = \int |h| P(h) dh$$

Rugosité : variance, skewness, kurtosis

- De façon générale la distribution des hauteurs est caractérisée par la donnée de l'ensemble de ses moments

$$M_m = \int h^m P(h) dh$$

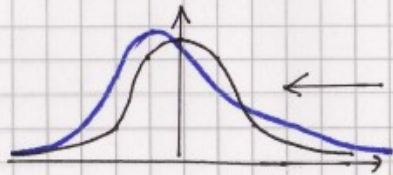
$$M_1 = \text{moyenne} = 0$$

$$M_2 = \text{variance} = \text{RMS}^2 \rightarrow \text{largeur de la distribution}$$

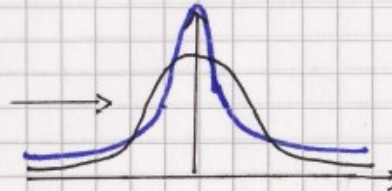
$$M_3 = \text{skewness } s \rightarrow \text{symétrie de la distribution}$$

$$M_4 = \text{kurtosis } k \rightarrow \text{forme "piquée" ou aplatie de la distribution}$$

asymétrie
 $|s| > 0$



Gaussienne



écart à la Gaussienne

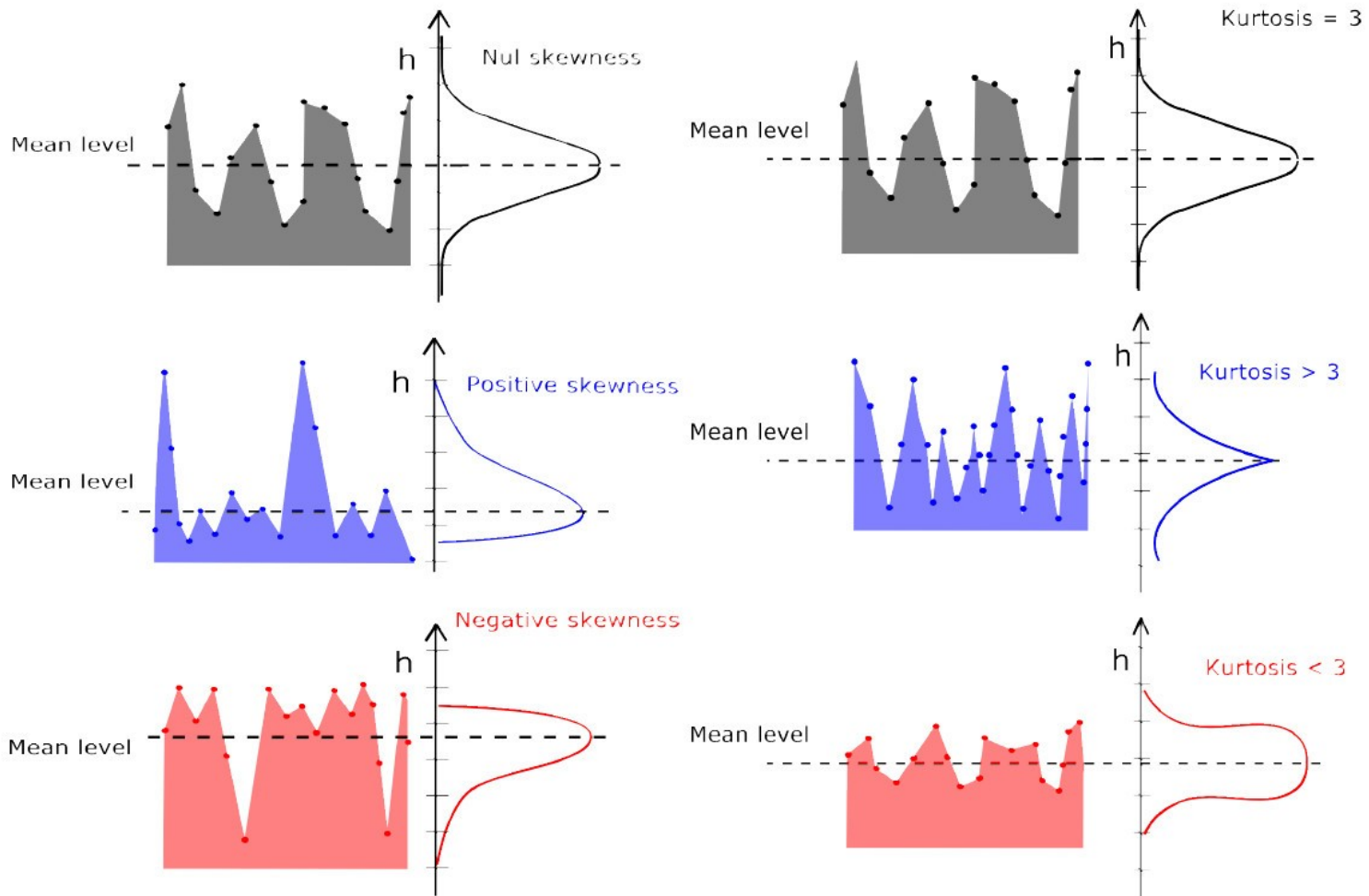
$$k-3 \neq 0$$

- En pratique, lorsque la surface est Gaussienne, la RMS suffit à caractériser la distribution

$$P(h) = \frac{1}{\sigma \sqrt{2\pi}} \exp\left(-\frac{h^2}{2\sigma^2}\right)$$

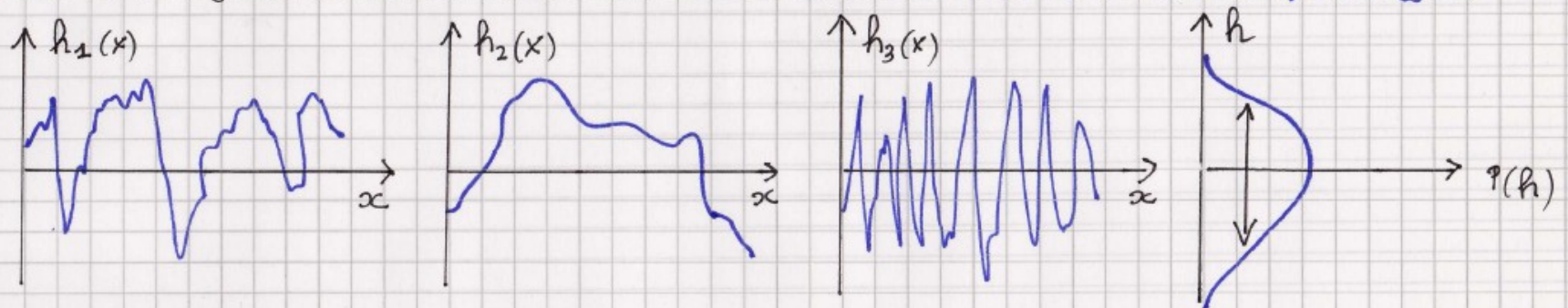
$$\sigma = \text{RMS}$$

Skewness et Kurtosis



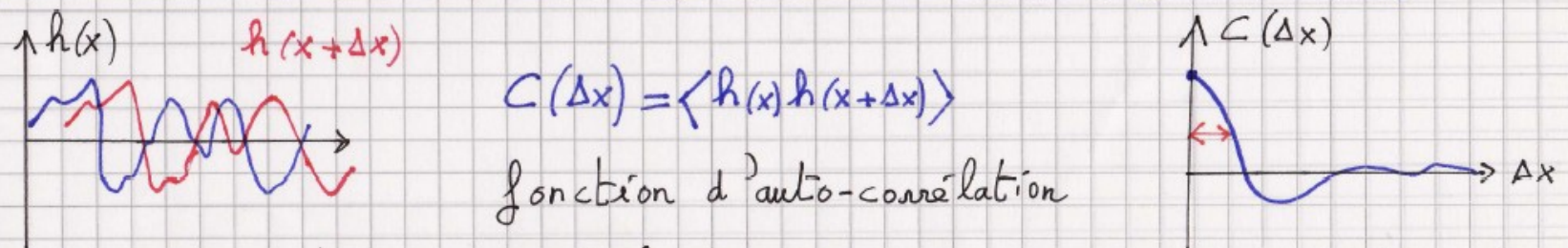
Corrélations spatiales

□ RMS et a fortiori distribution des hauteurs = essentielle mais pas suffisante



la distribution des hauteurs fournit une information sur les fluctuations **verticales** - Une même RMS, une même distribution peut correspondre à des surfaces très différentes

□ Corrélation des hauteurs → information sur les motifs **horizontaux**



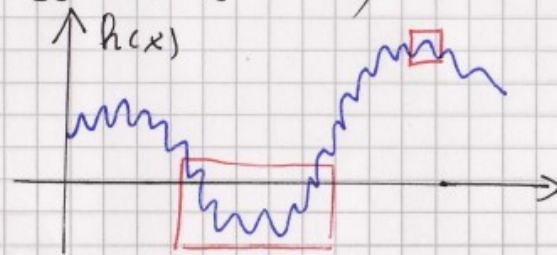
Un paramètre essentiel: la LONGUEUR de CORRÉLATION \approx taille motifs h^{aux}

Longueurs de corrélations

- Dans les cas les plus simples la fonction d'auto-corrélation $C(\Delta x)$ est très rapidement décroissante, exponentielle, Gaussienne

→ définition d'1 longueur de corrélation

- Parfois la surface est caractérisée par plusieurs longueurs de corrélation, voire par un "continuum" de longueurs de corrélations (surfaces de type "fractal" ou "auto-affines", cf + loin)

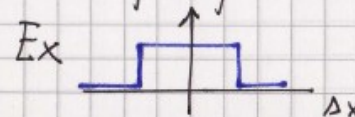


2 motifs caractéristiques à petite et grande échelle

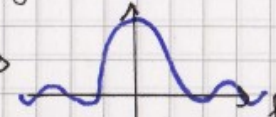
- Sur le plan mathématique, la fonction d'autocorrélation $C(\Delta x)$ est la transformée de Fourier de la densité spectrale de puissance PSD(f)

$$\text{PSD}(f) = |\tilde{h}|^2$$

→ Toute fonction réelle paire ne peut pas être une fonction d'auto-corrélation, sa TF doit être positive!



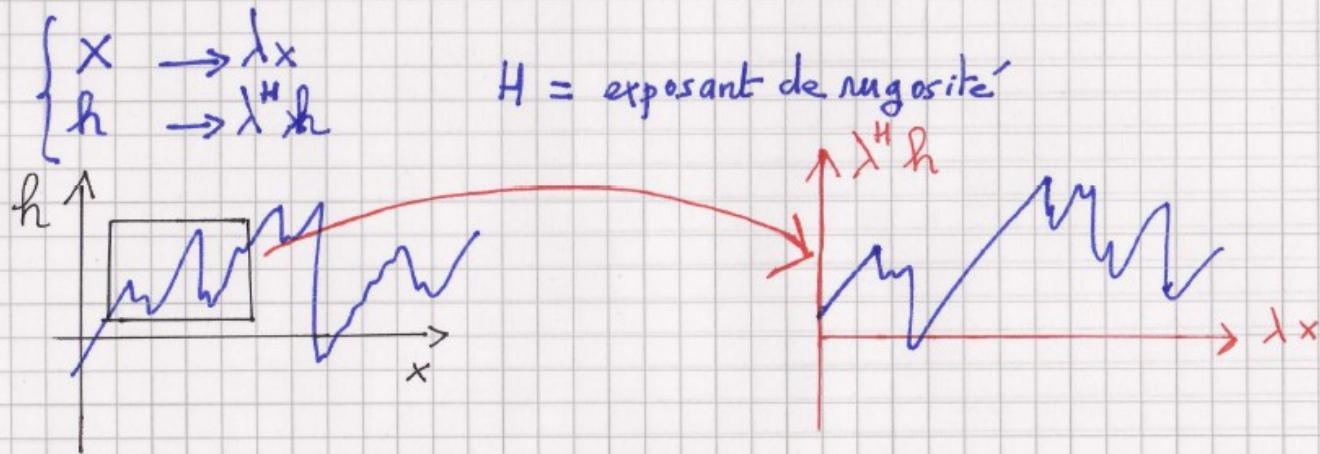
TP →



≠ fonction autocorrélation

Echelles de mesure vs longueurs de corrélations

- Dans certains cas la surface n'est pas caractérisée par λ mais par un continuum de longueurs de corrélation
- Une symétrie particulière: la symétrie auto-affine
la surface reste statistiquement invariante par des "zooms" anisotropes

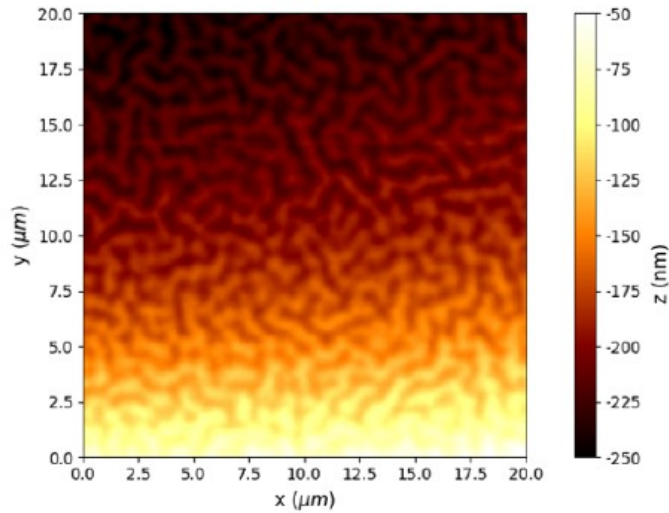


- Sur le plan pratique, cette invariance a une conséquence importante:
la rugosité RMS dépend de l'échelle sur laquelle on la mesure!

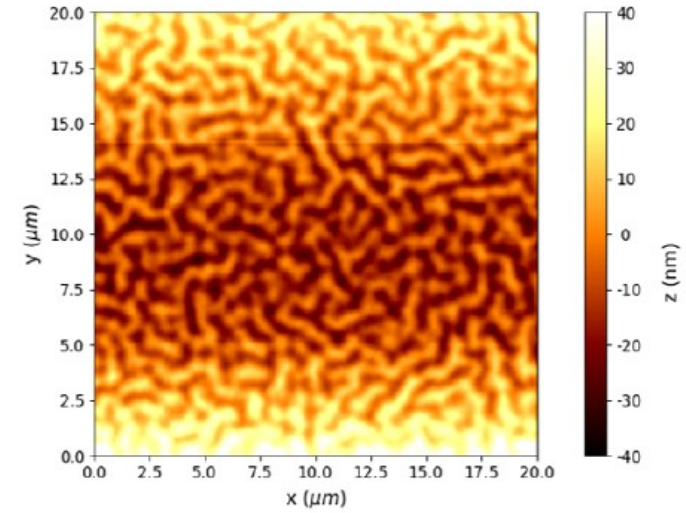
$$\sigma(\Delta x) \propto \Delta x^H$$

Rugosité : pré-traitement des données

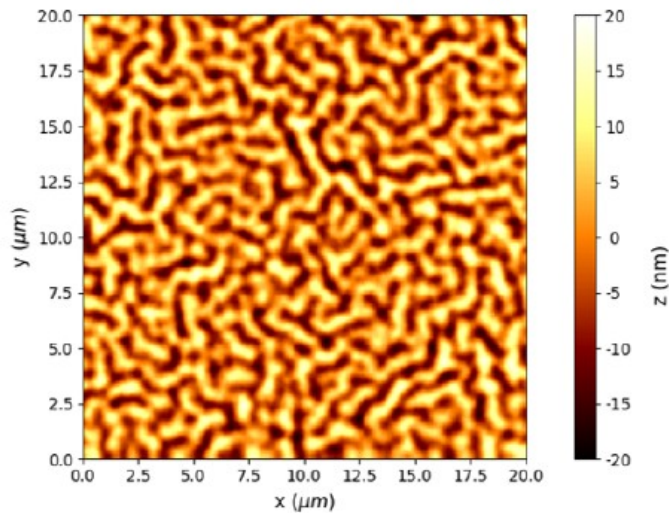
a Image brute



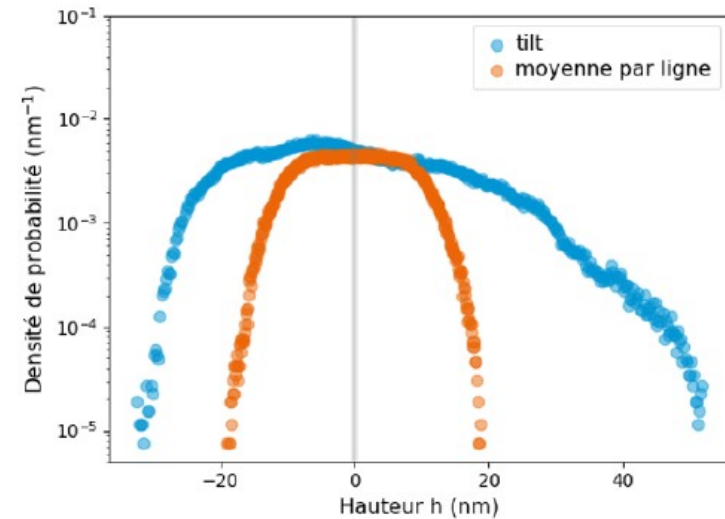
b Correction par un plan - tilt



c Correction moyenne par ligne



d Distribution des hauteurs



More about roughness, local slopes and curvatures

IOP Publishing

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Surface Topography: Metrology and Properties



PAPER





Statistically representative estimators of multi-scale surface topography: example of aluminum blasted rough samples

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C Turbil¹, J Cabrero² , I Simonsen^{1,3} , D Vandembroucq⁴  and I Gozhyk¹ 

¹ SVI, UMR 125 CNRS/Saint-Gobain Research Paris, 39 quai Lucien Lefranc, F-93303 Aubervilliers, France

² Saint-Gobain Research Provence, 550 avenue Alphonse Jauffret, F-84306 Cavaillon, France

³ Department of Physics, NTNU—Norwegian University of Science and Technology, NO-7491 Trondheim, Norway

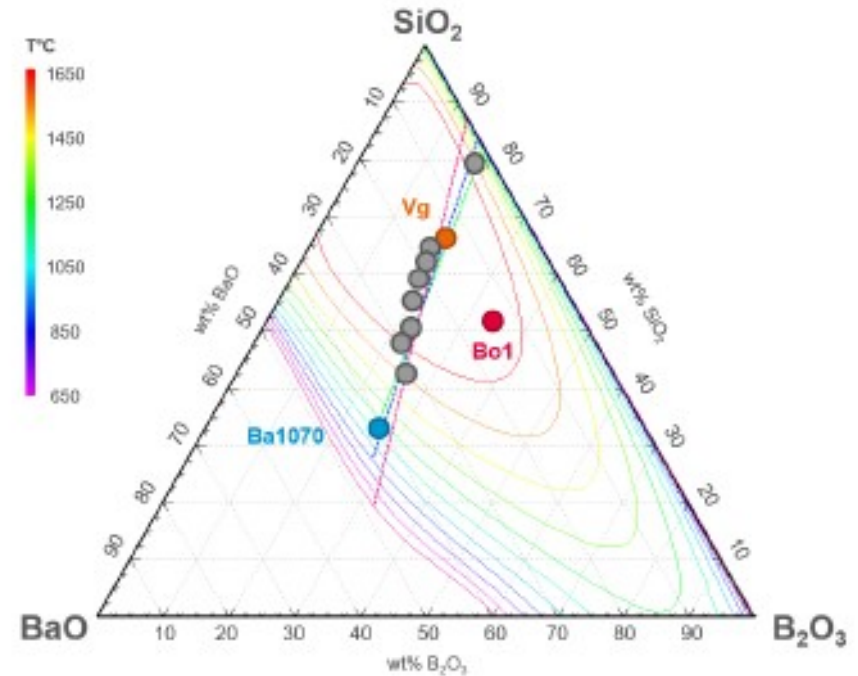
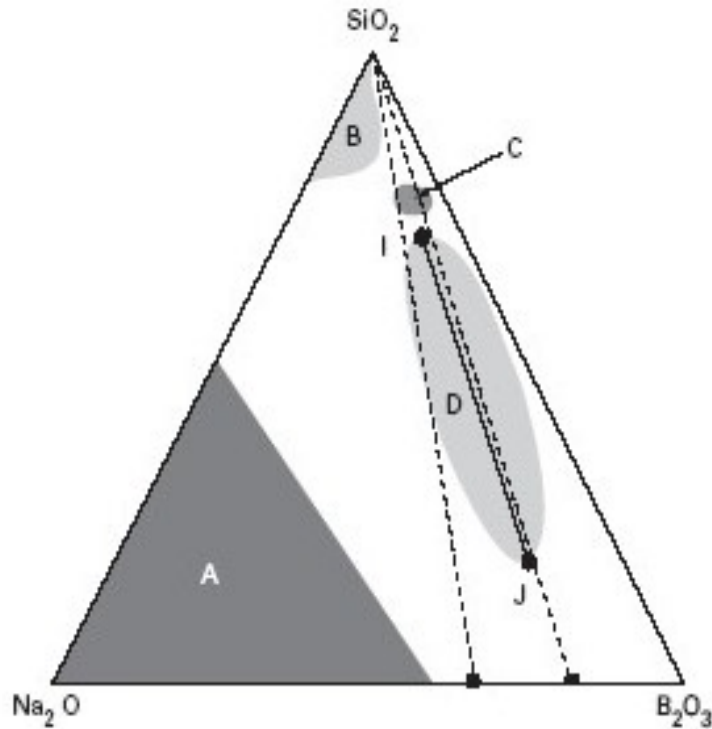
⁴ PMMH, CNRS, ESPCI Paris, PSL University, Sorbonne Université, Université de Paris, 10 rue Vauquelin, F-75231 Paris cedex 05, France

E-mail: iryna.gozhyk@saint-gobain.com

Keywords: multi-scale roughness, blasted aluminum, topography, surface slopes, mounded surface

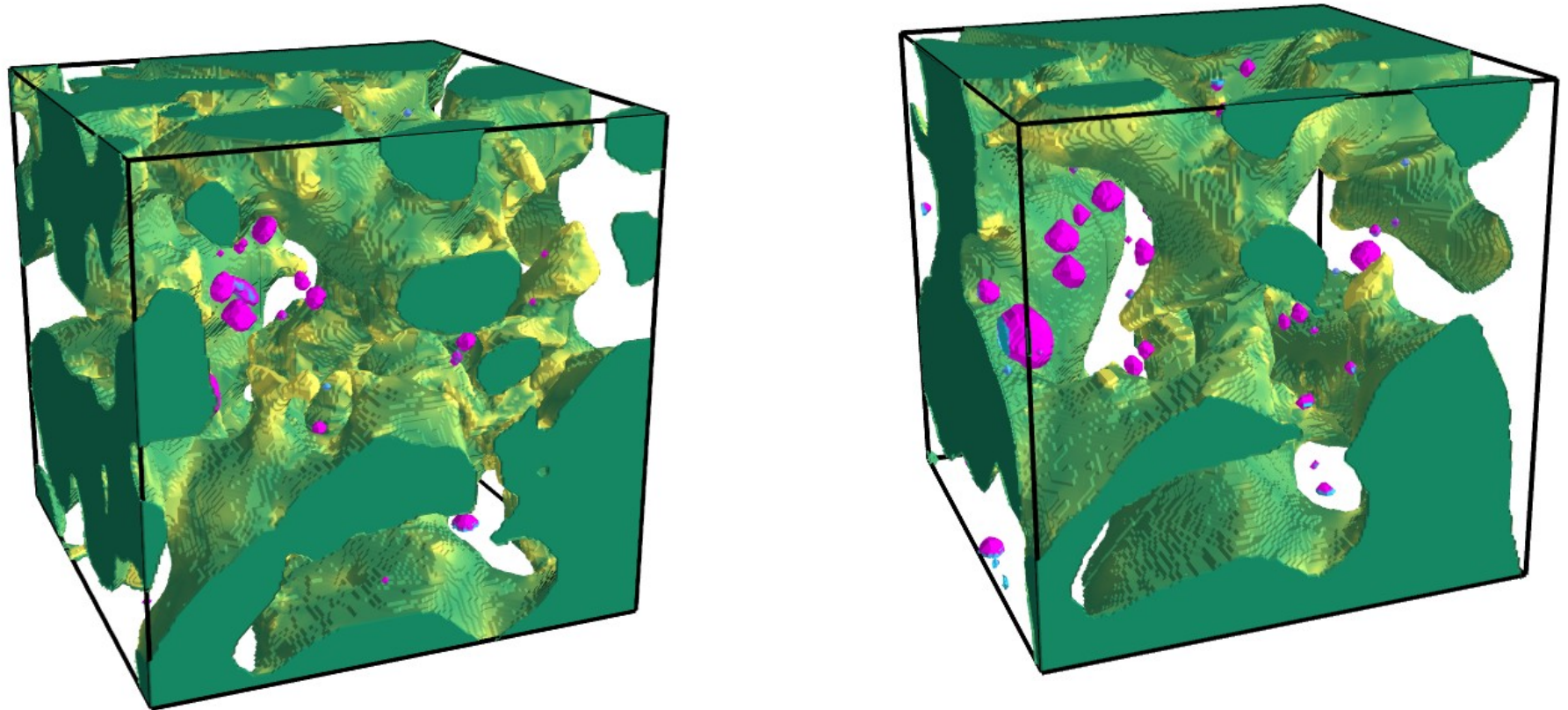
Application : étude de la séparation de phase dans les verres

Verres démixés

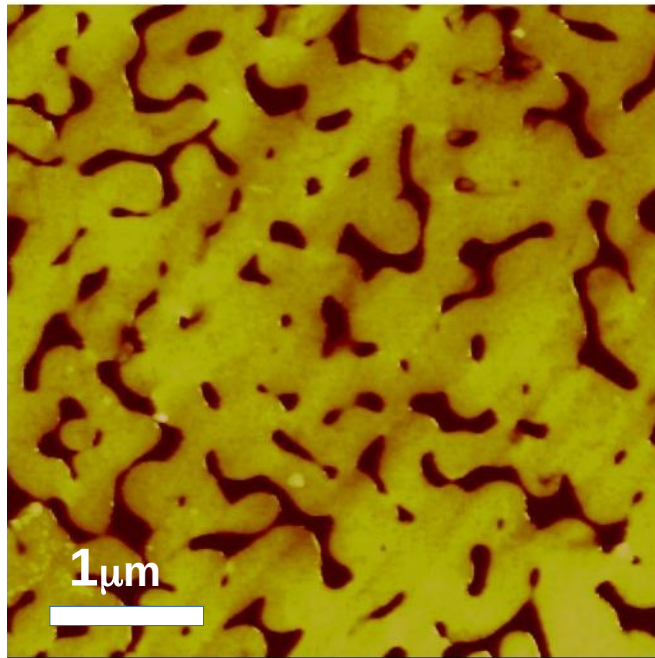


Séparation de phase dans de nombreuses compositions verrières binaires ou ternaires
Différentes tailles de domaines, morphologies variées, mûrissement hydrodynamique
ou diffusif. Applications: pyrex, vycor, etc.

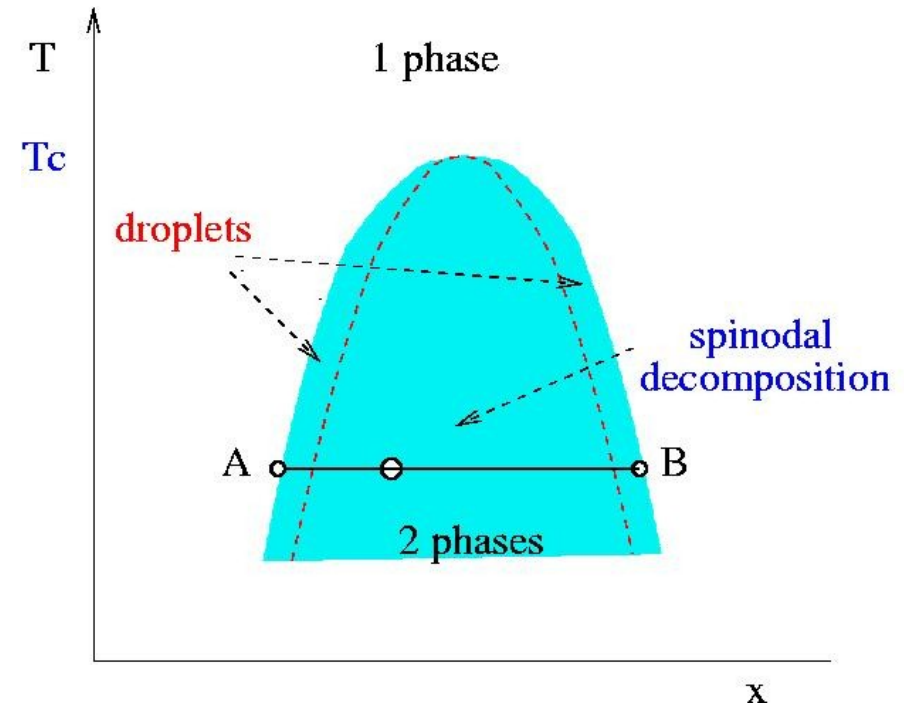
Suivi temps réel du mûrissement par tomographie X



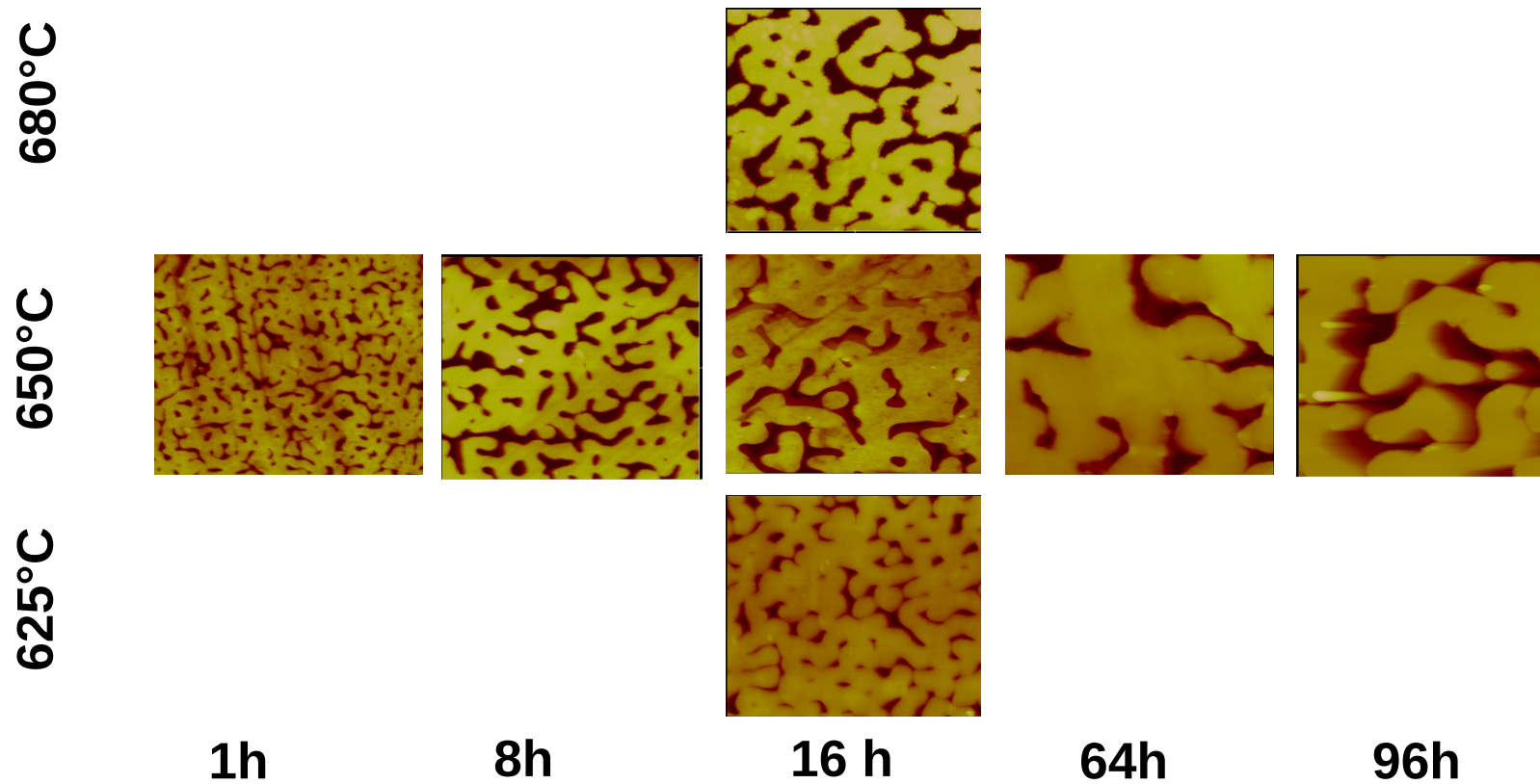
Séparation de phase dans les verres



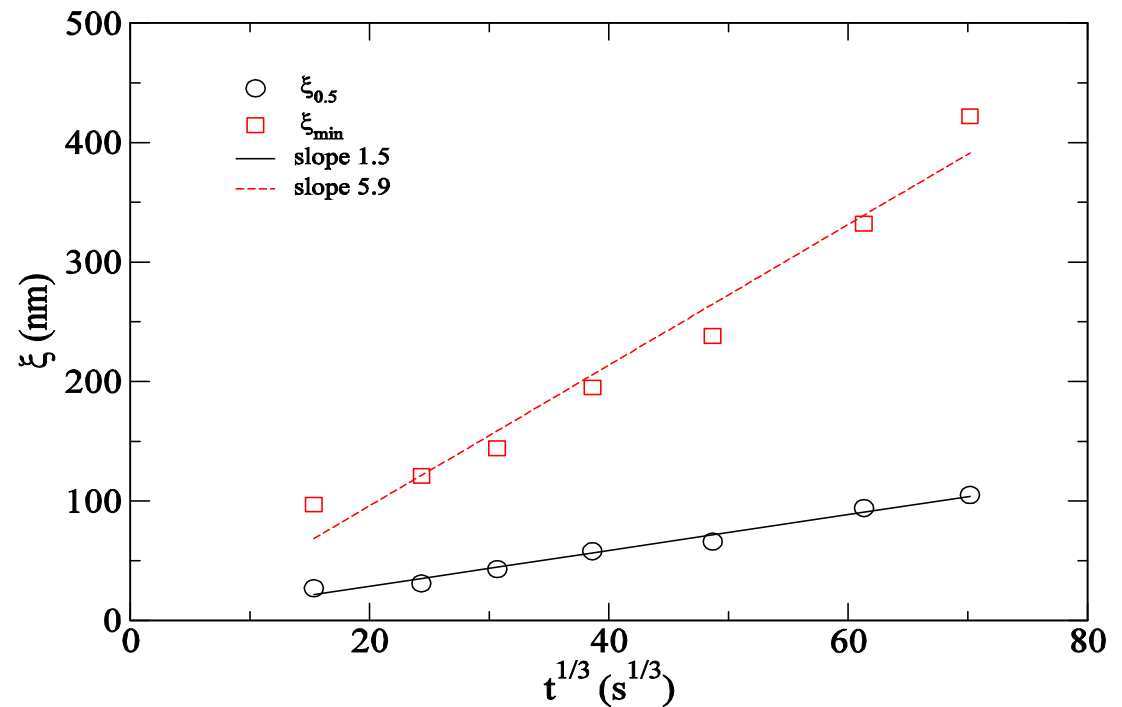
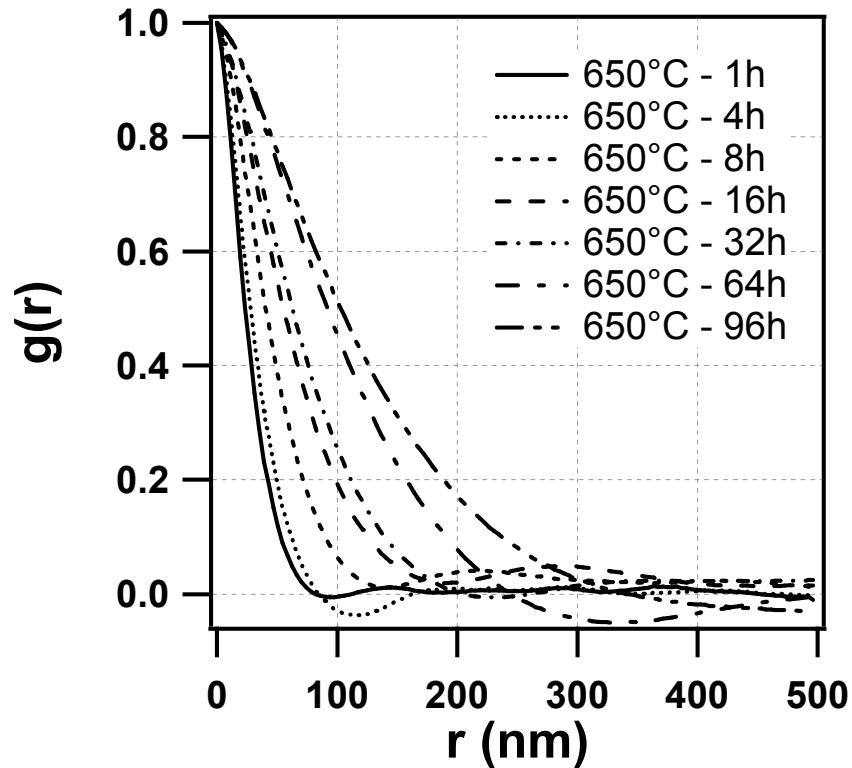
AFM image of a surface of a phase separated alkaline borosilicate glass after selective etching
Lateral size: $4\mu\text{m}$, Z-scale: [-100:+100 nm]



Cinétique de croissance des domaines



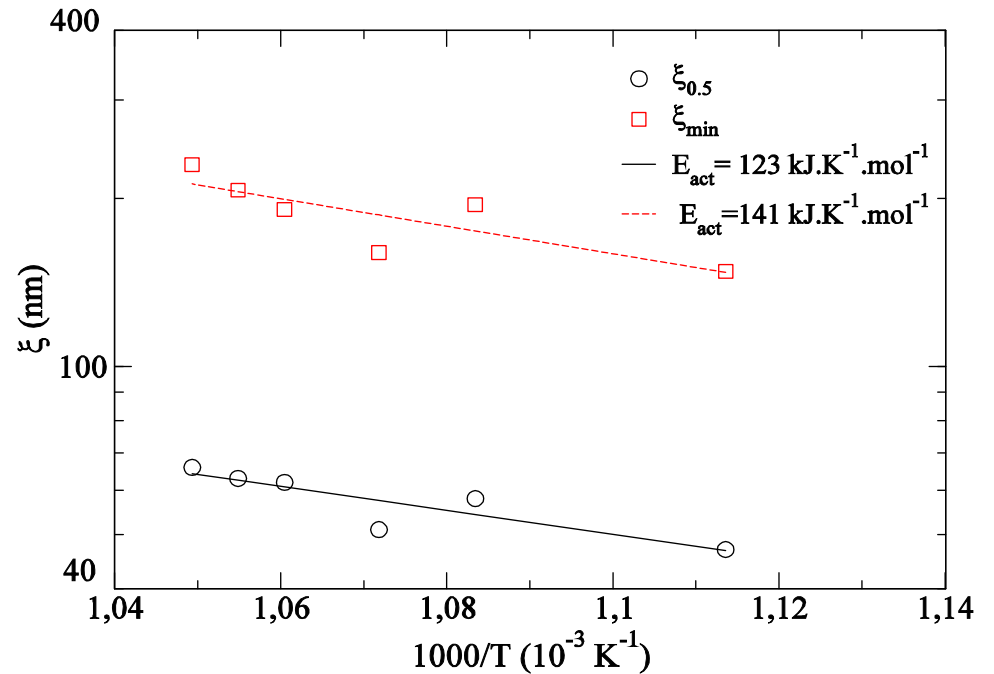
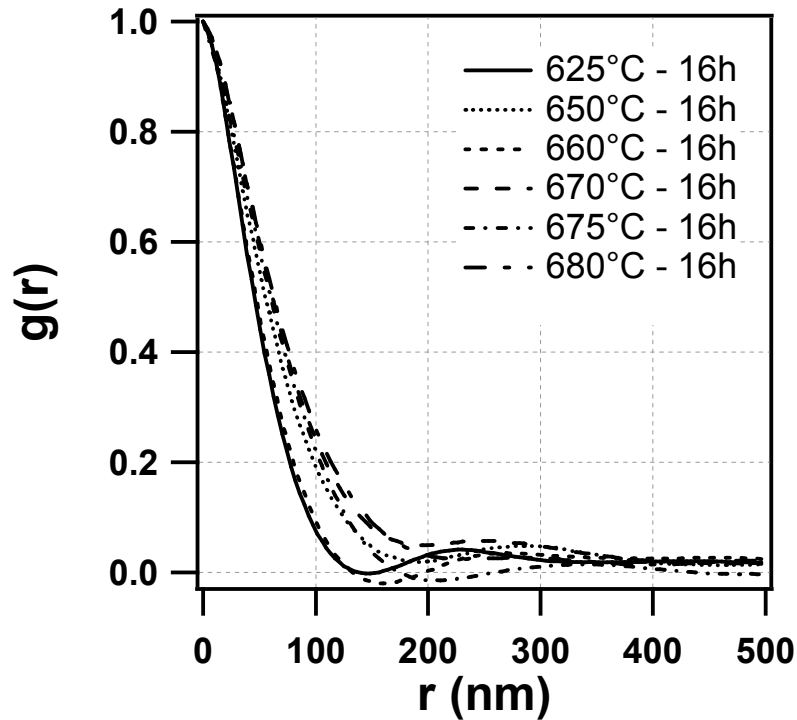
Loi d'échelle Lifshitz-Slyozhov-Wagner



$$R \approx (Kt)^{1/3}, \quad K \approx \frac{\gamma Dv}{kT}$$

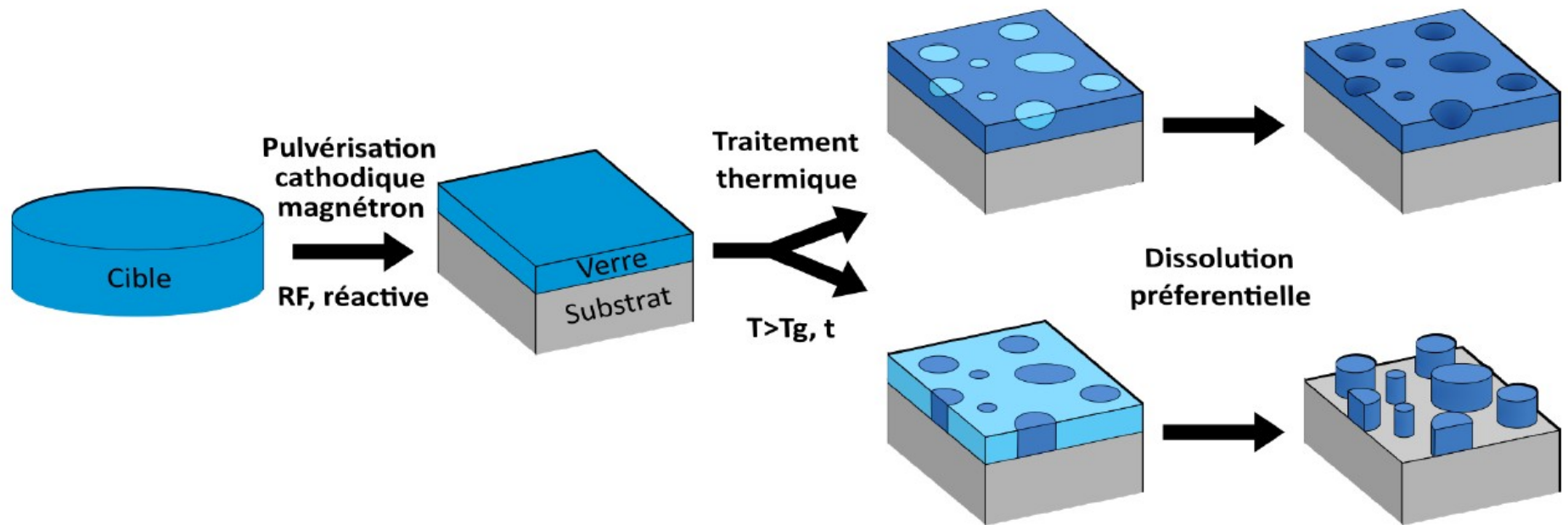
$$D \sim 10^{-16} \text{ cm s}^{-1}$$

Temperature dependence: Arrhenius behavior

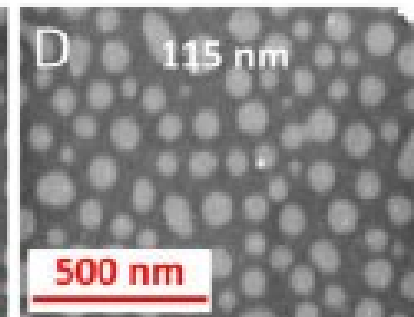
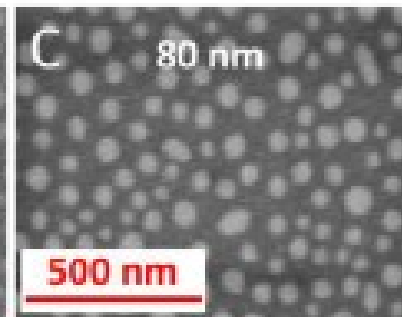
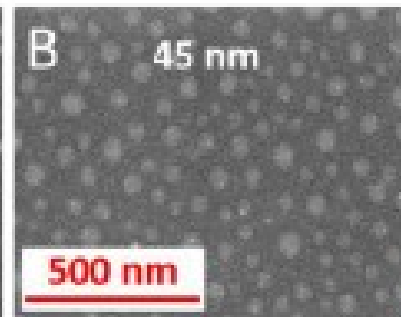
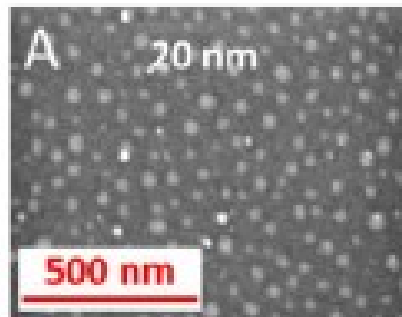
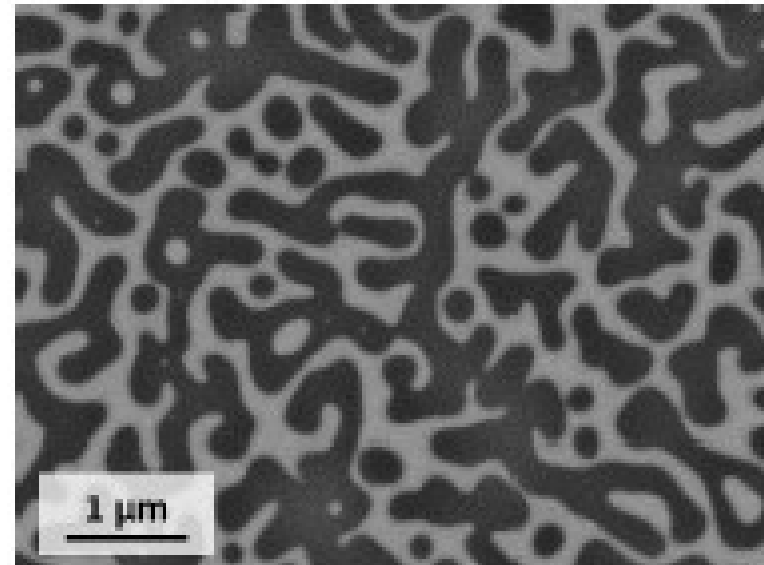
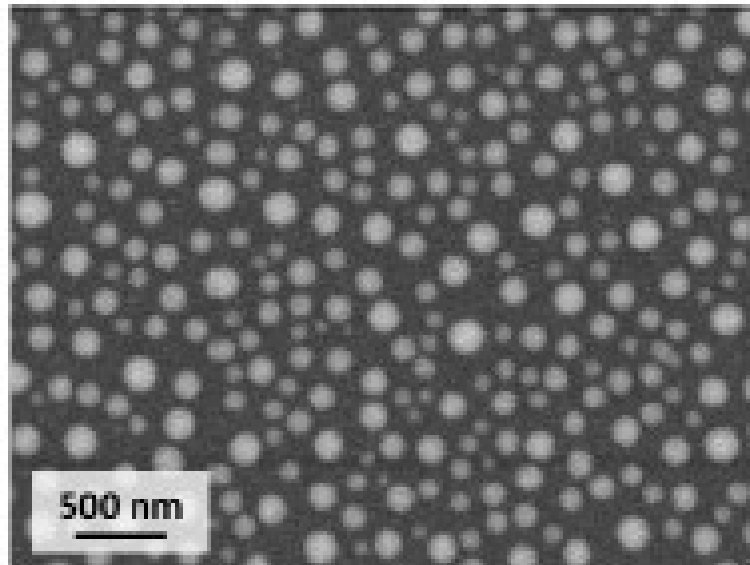


Estimate of activation energy from AFM measurements

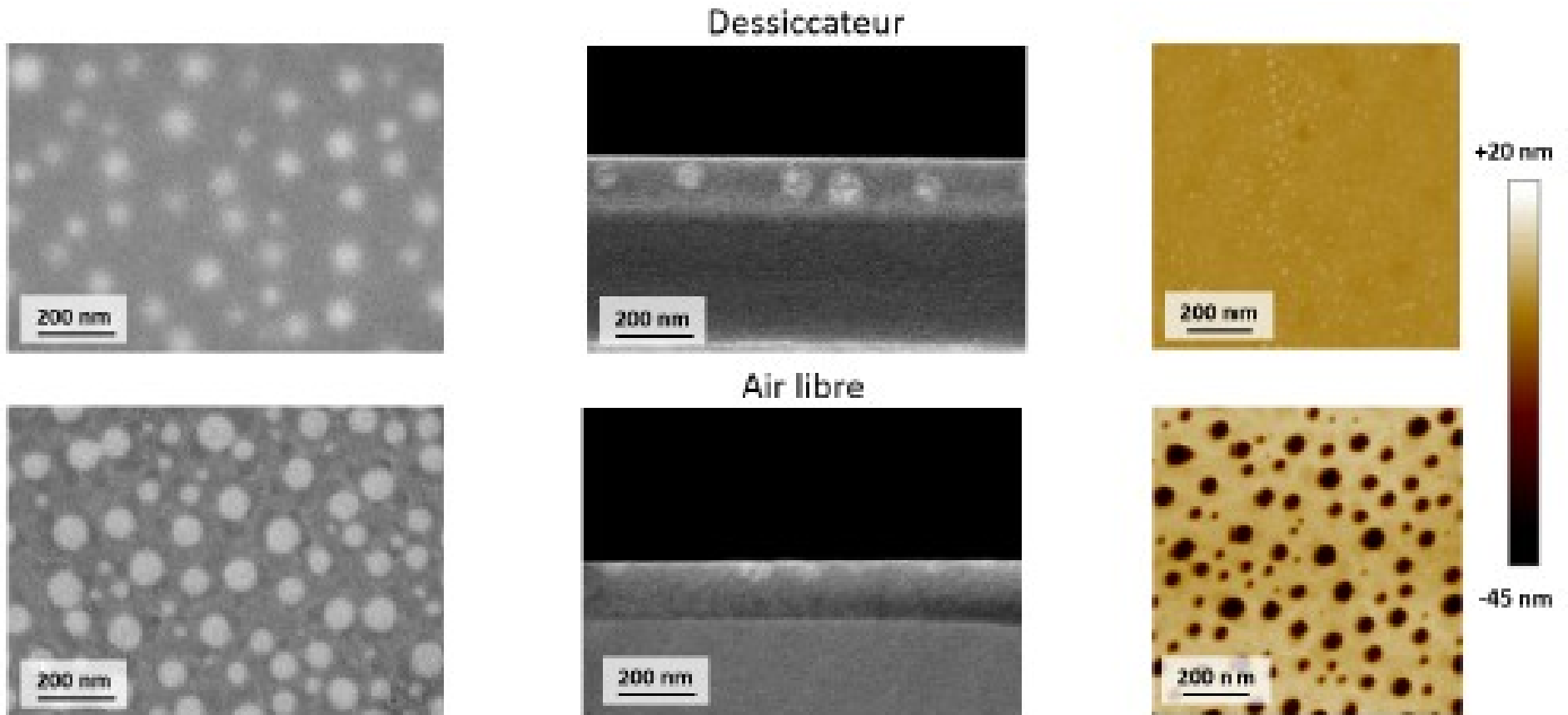
Texturation de surface par séparation de phase en couche mince



Cariétés de tailles et de morphologie des domaines

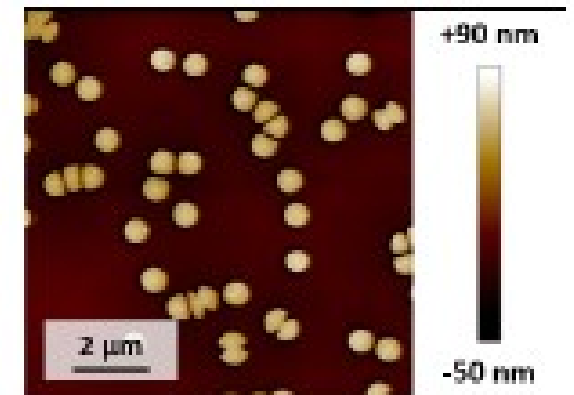
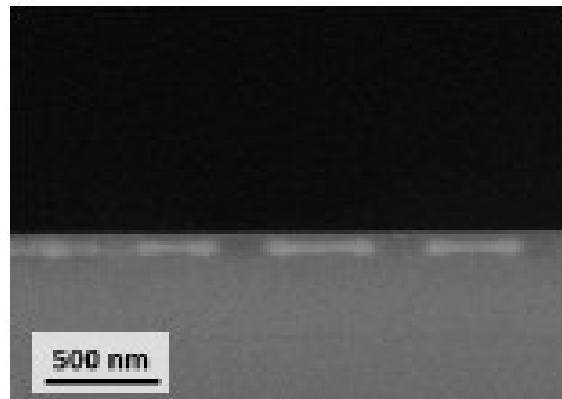
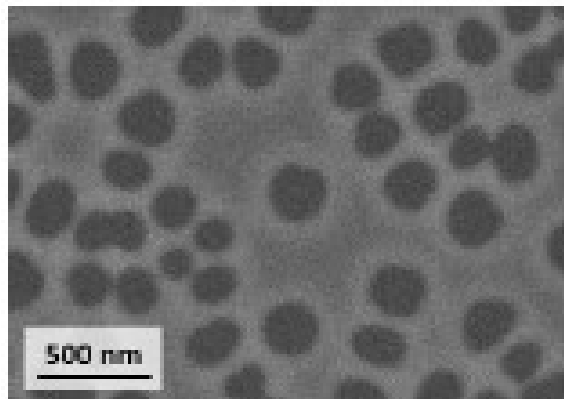
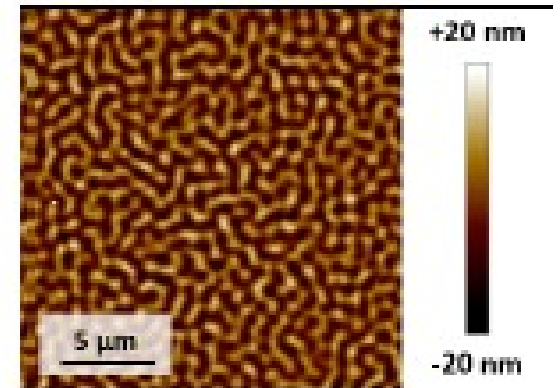
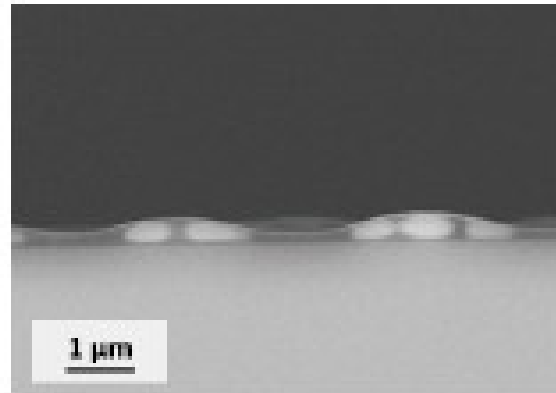
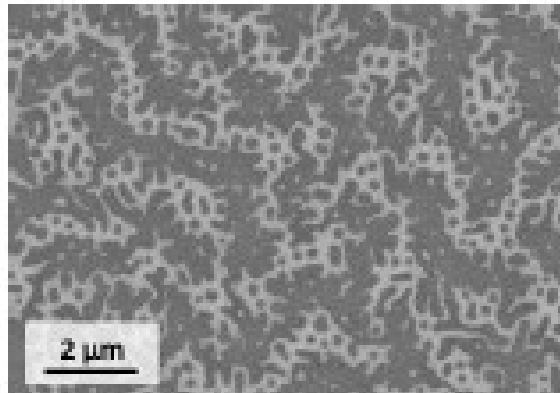


MEB vs AFM



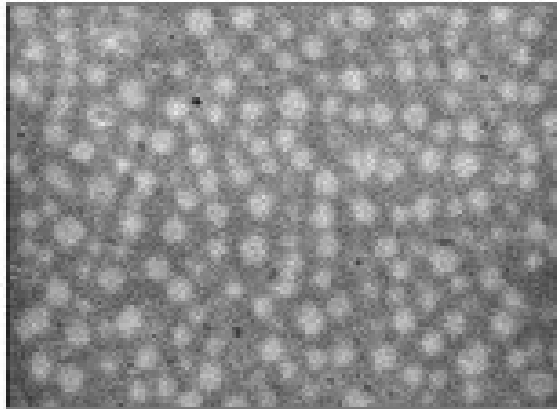
B. Bouteille Ph.D. 2017

Différentes compositions, différentes texturations

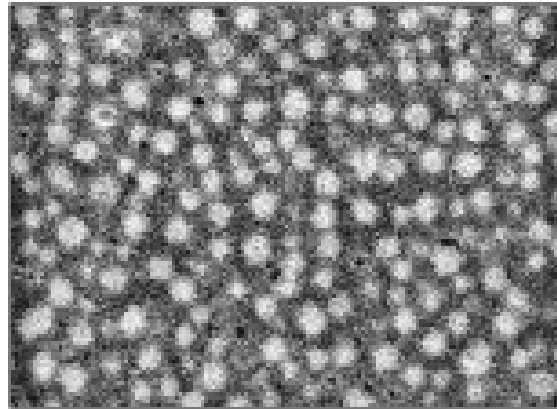


Traitement d'image

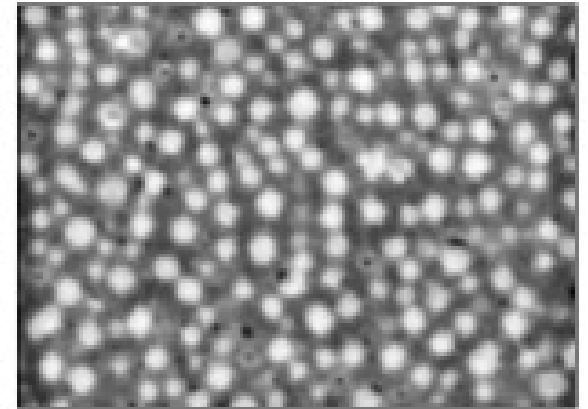
a Brut



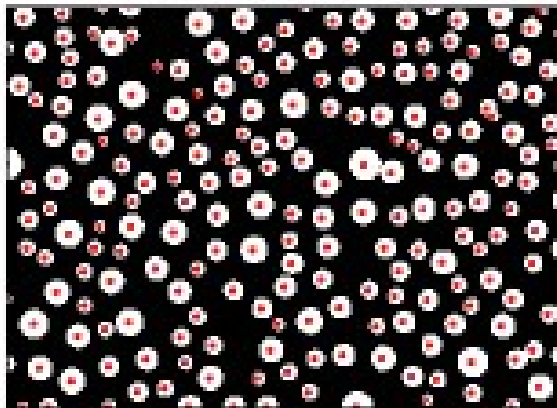
b Exposition



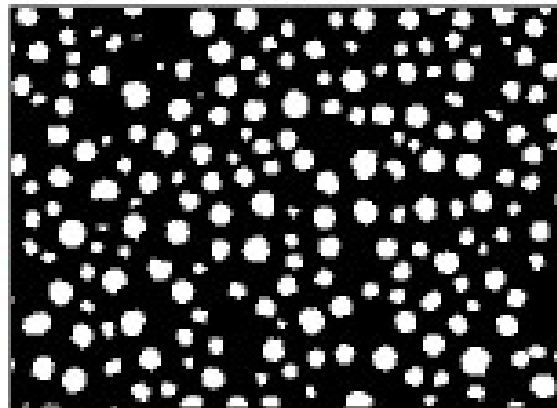
c Débruitage



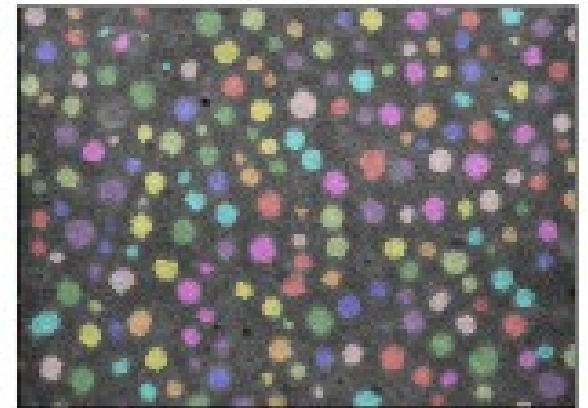
d Détection de blob



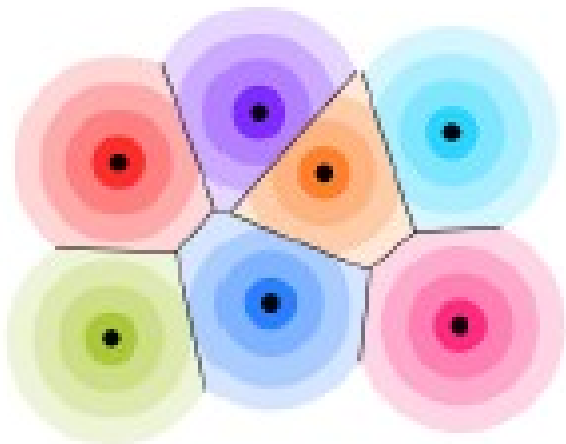
e Binarisation



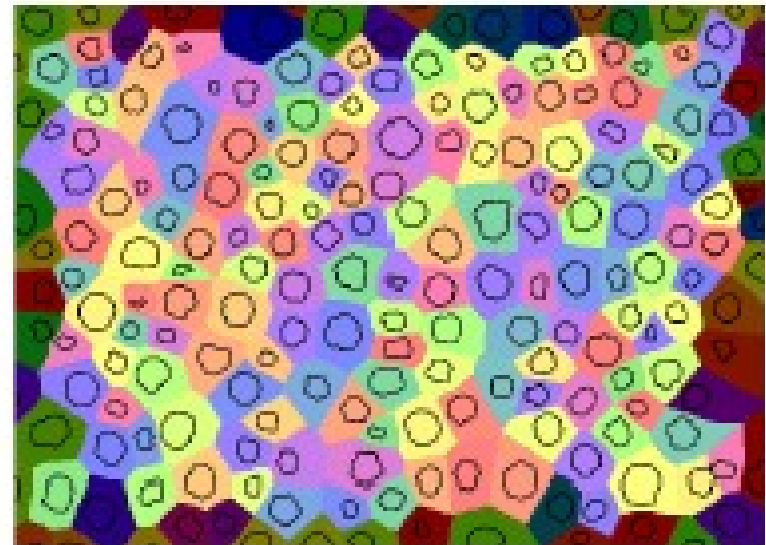
f Labélisation



Cellules de Voronoi

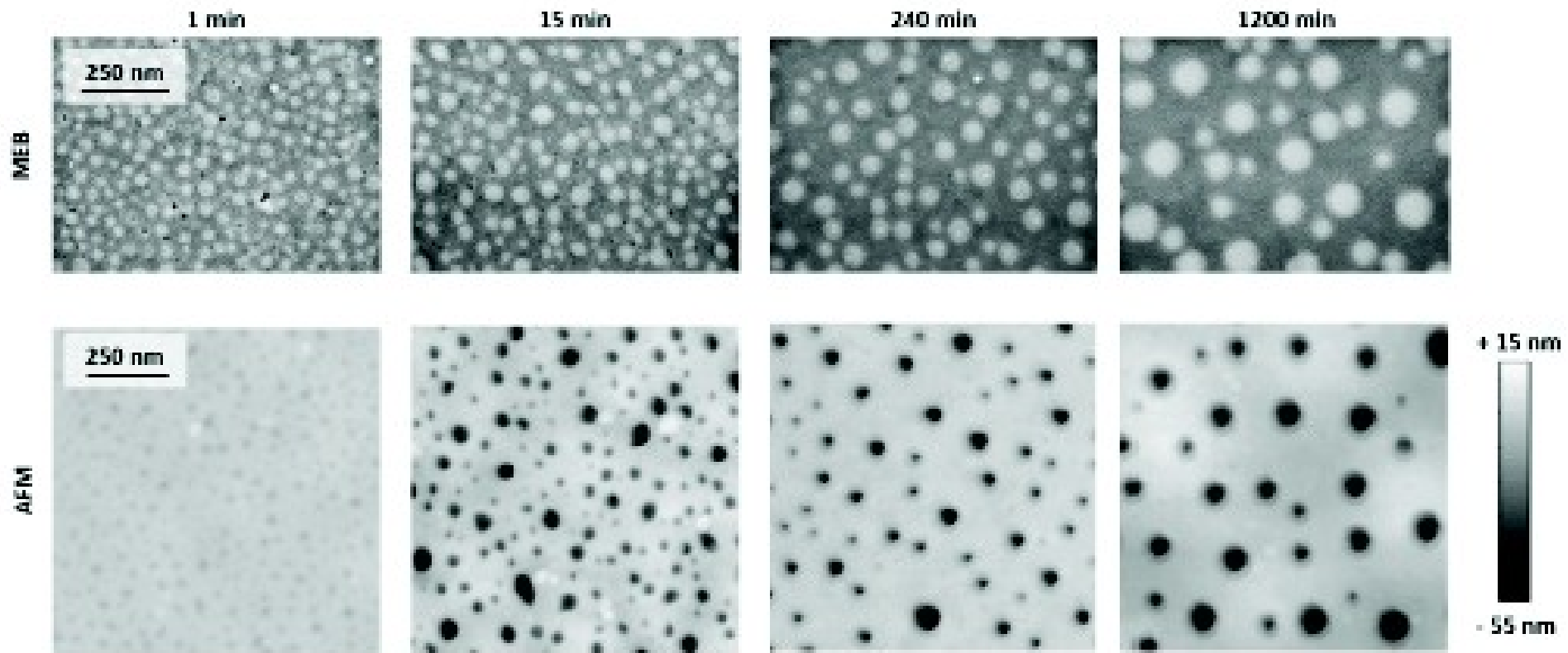


a Diagramme de Voronoi de centre à centre

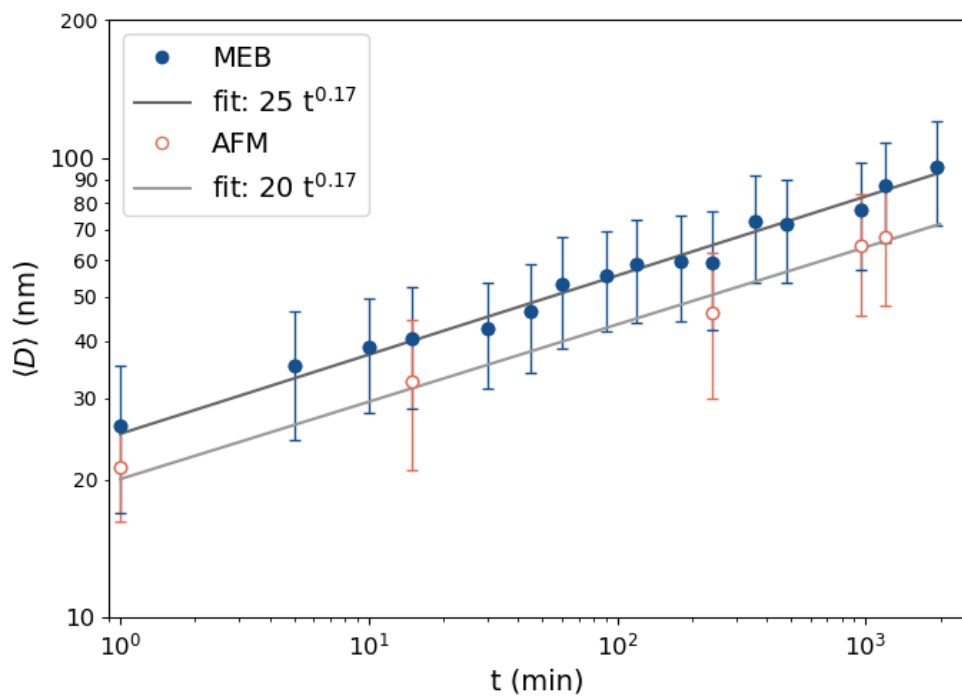


b Diagramme de Voronoi de bords à bords

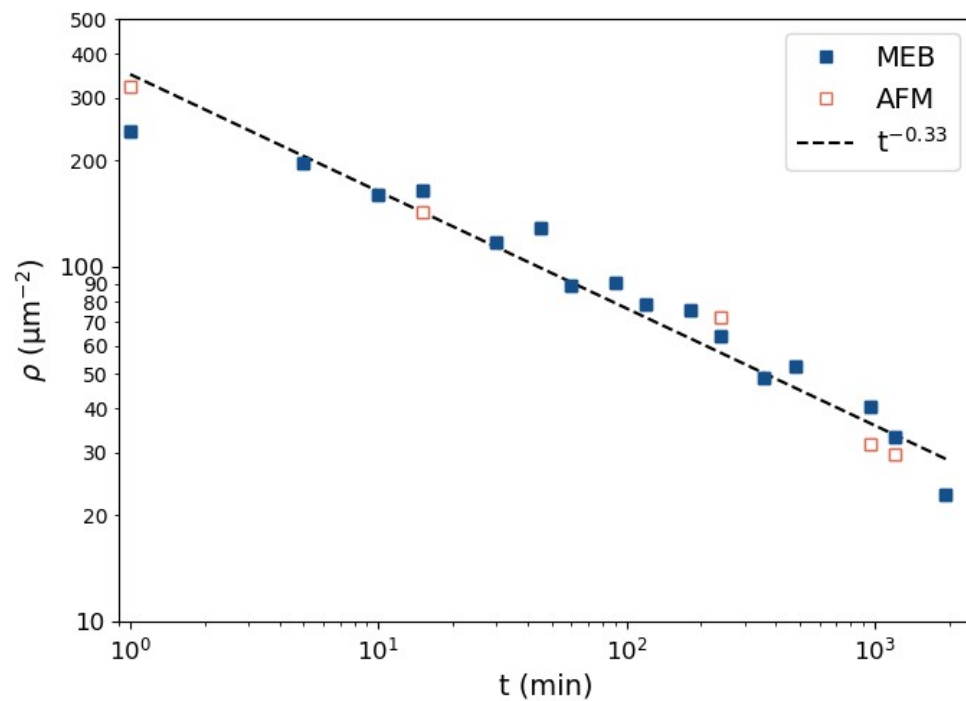
Croissance de domaines en couche mince



Cinétique (lente) de croissance

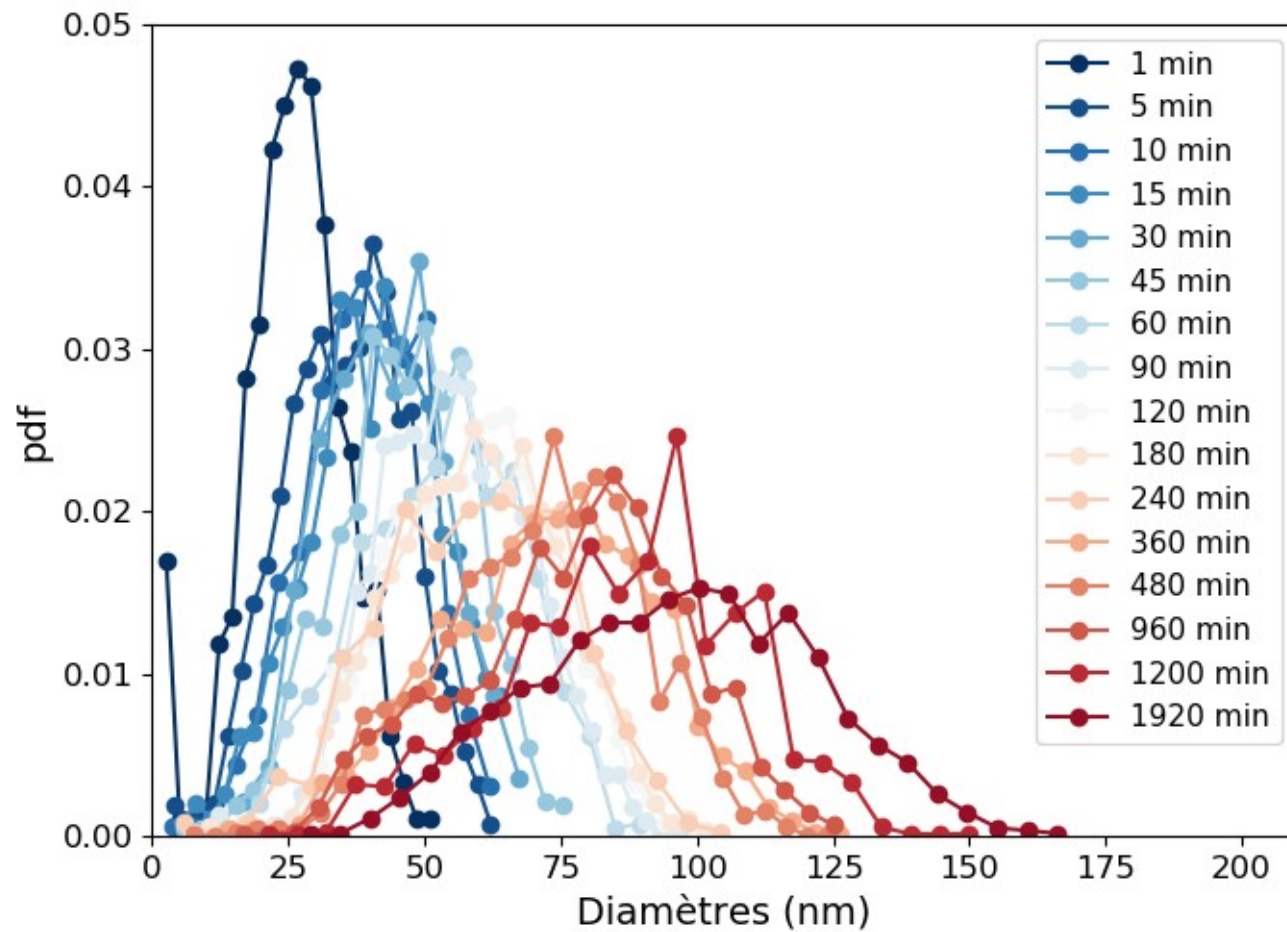


Diamètre Goutelettes

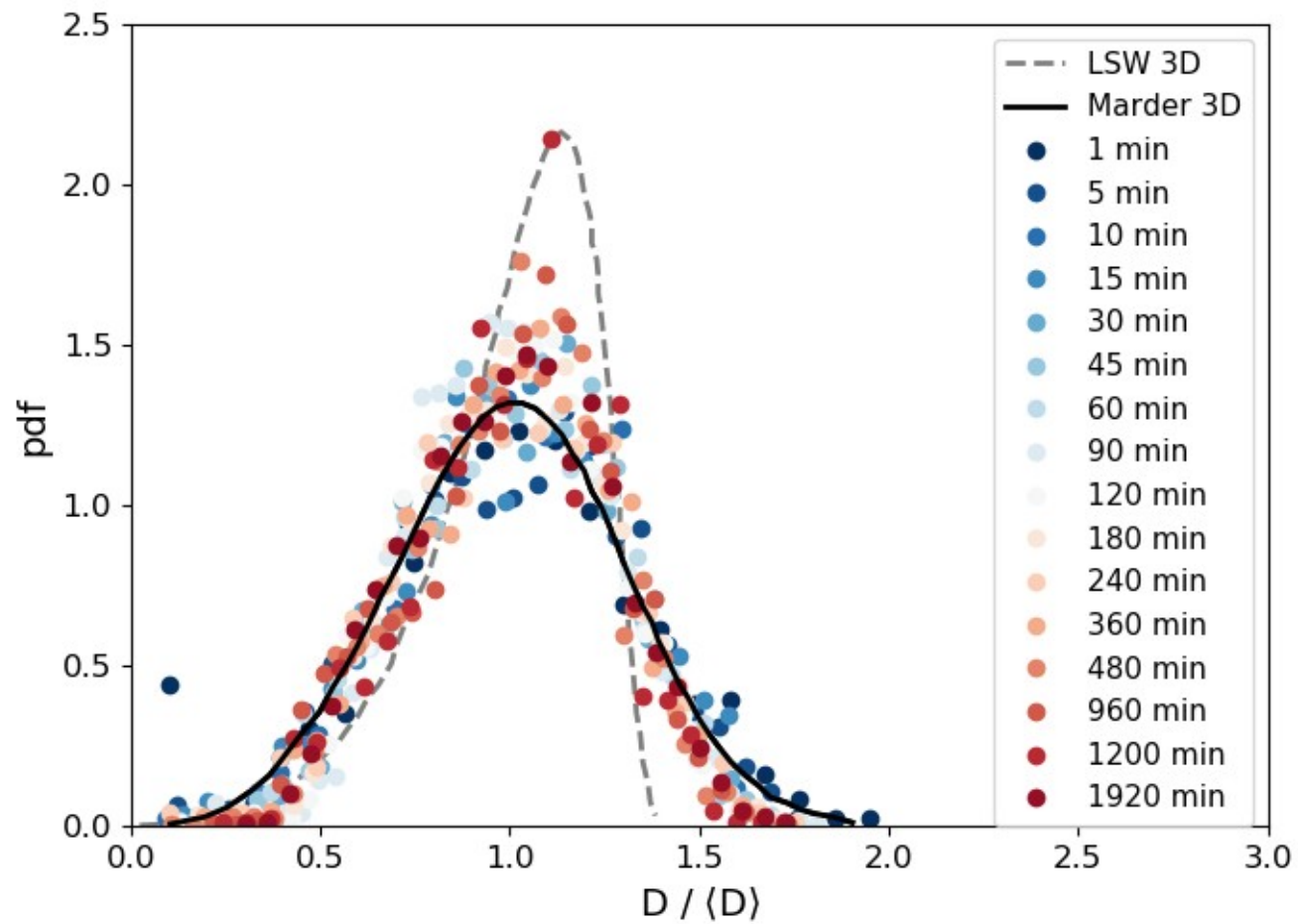


Densité Goutelettes

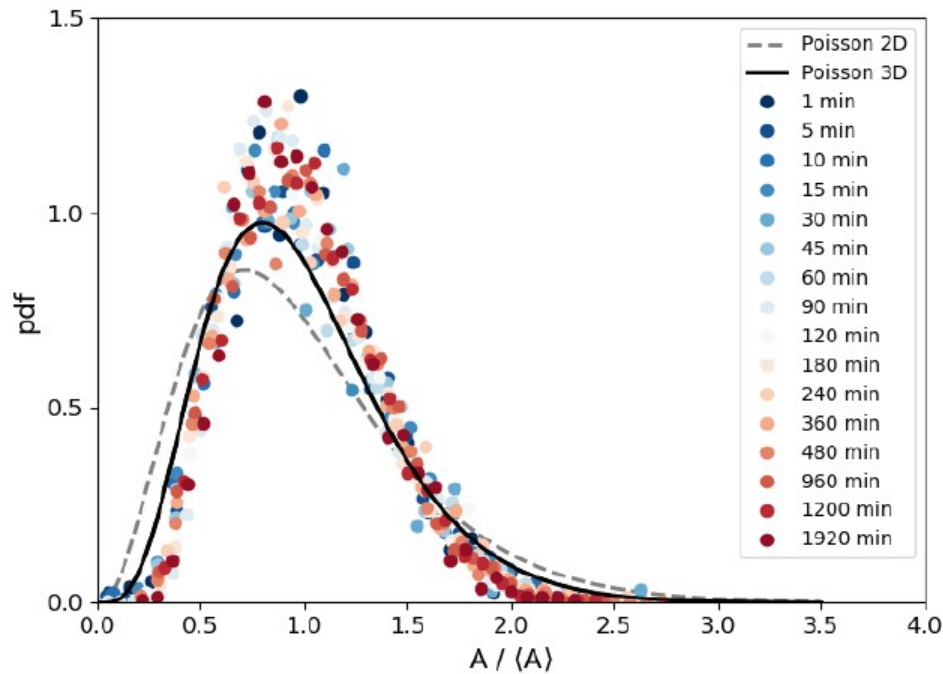
Evolution de la distribution de taille



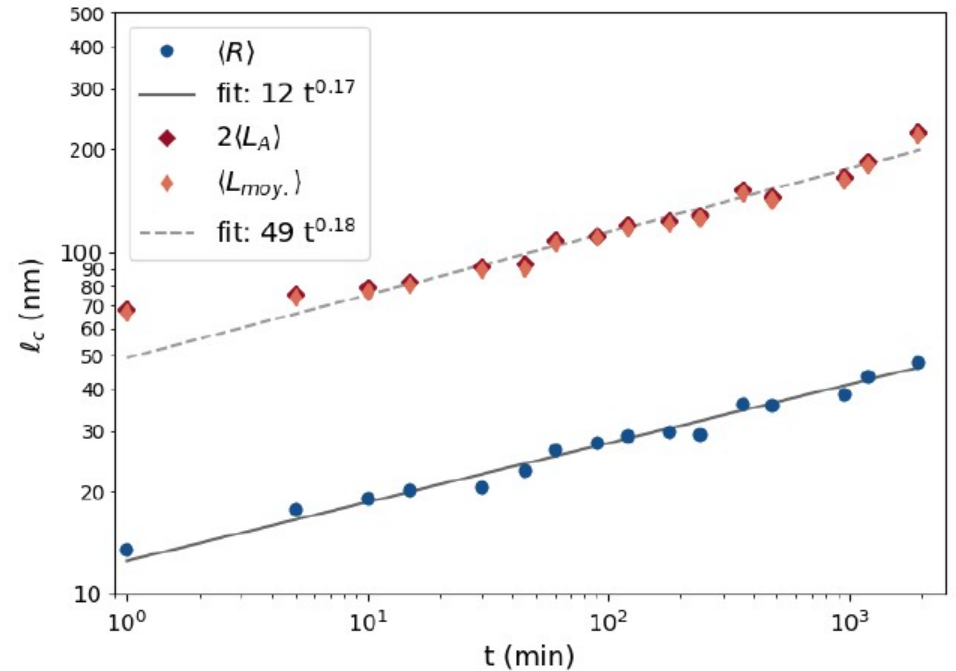
Distributions de tailles renormalisées



Cellules de Voronoi



Distribution renormalisée de l'aire des cellules de Voronoi



Evolution temporelle du rayon et de la distance inter-gouttelettes