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On the growth dynamics of low-density carbon foams in Pulsed Laser Deposition experiments

Alessandro Maffini
(Politecnico di Milano)

Milano, 21/05/2018

ERC-2014-CoG No.647554
ENSURE

NanoLab

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MILANO 1863

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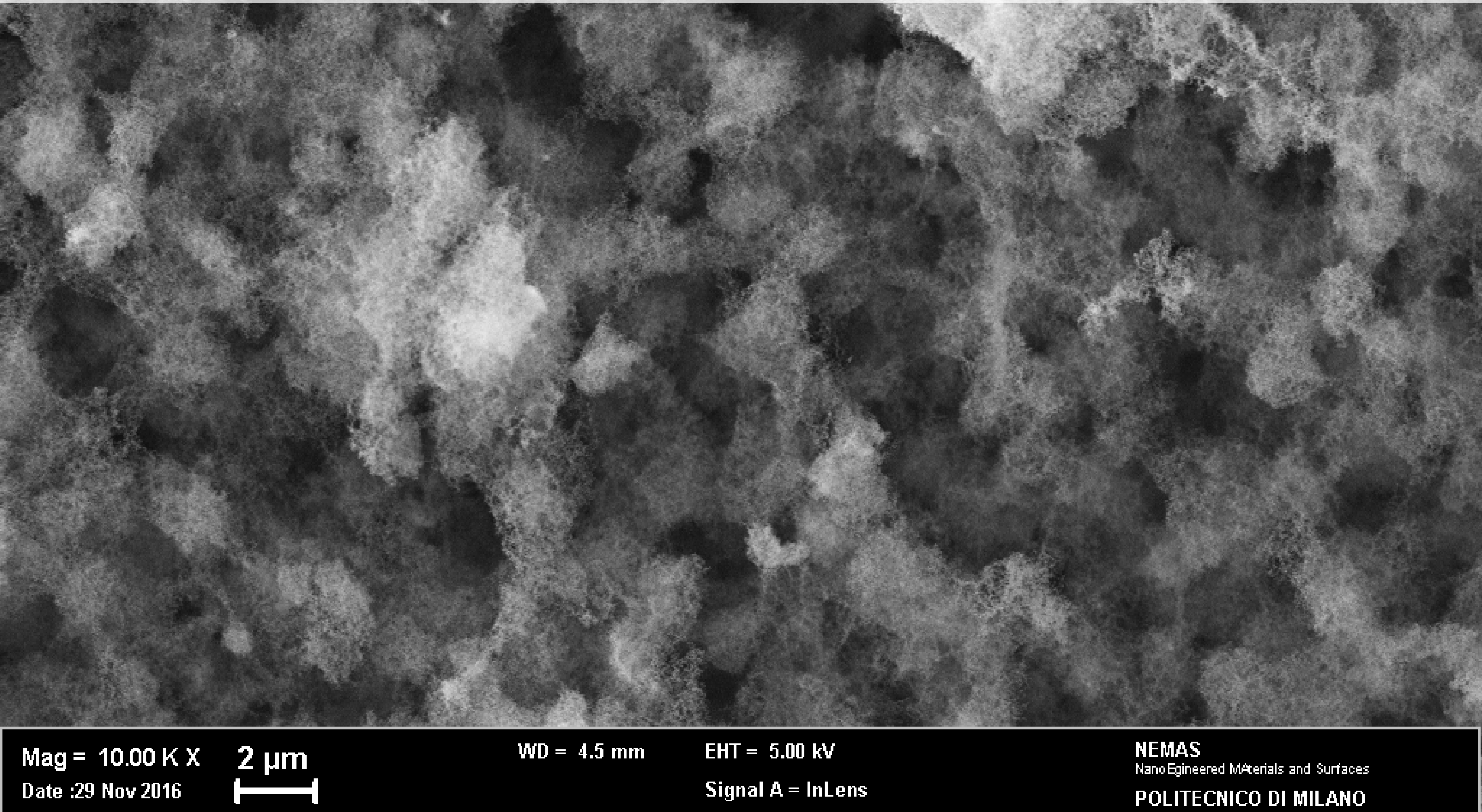

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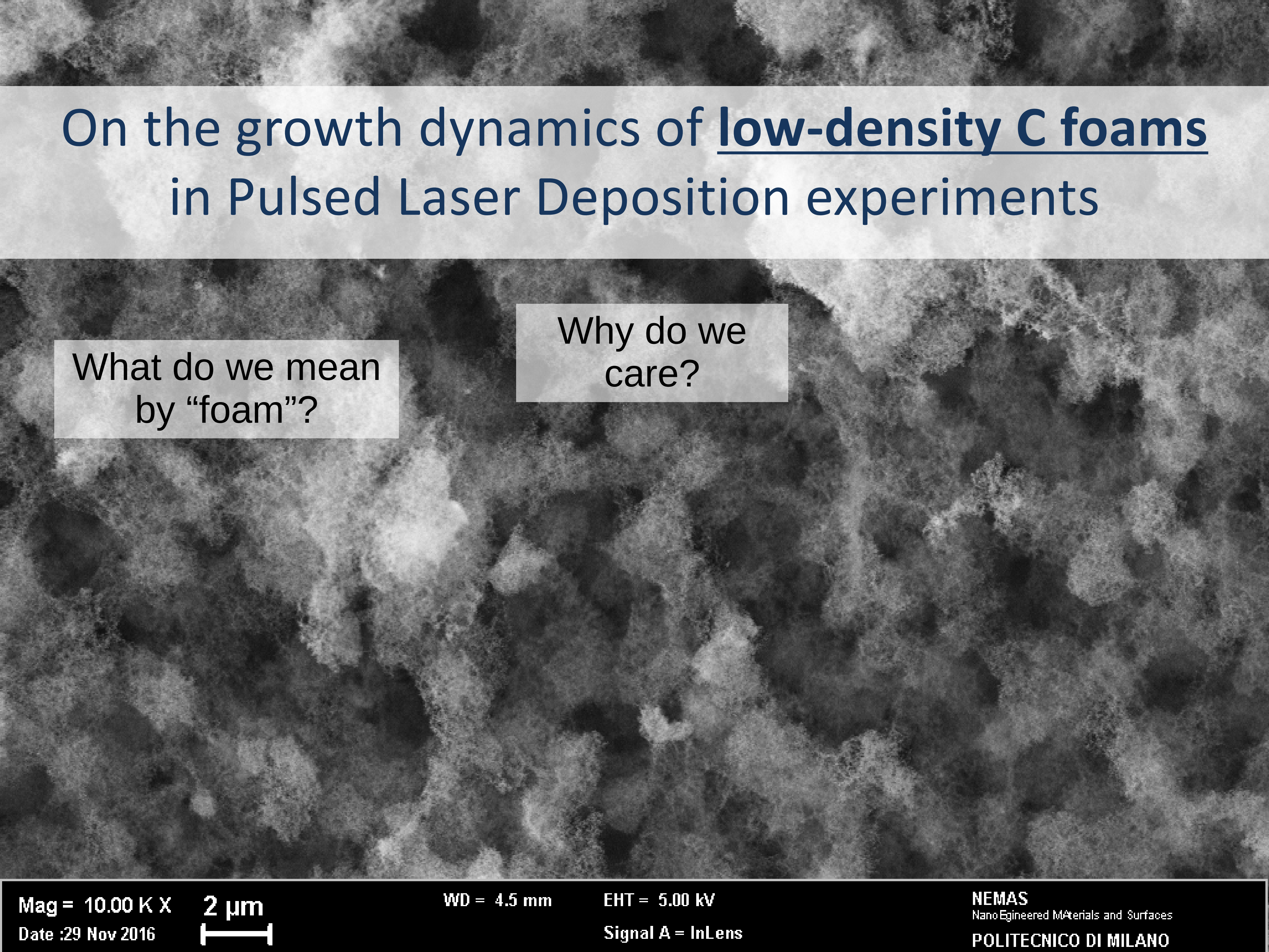
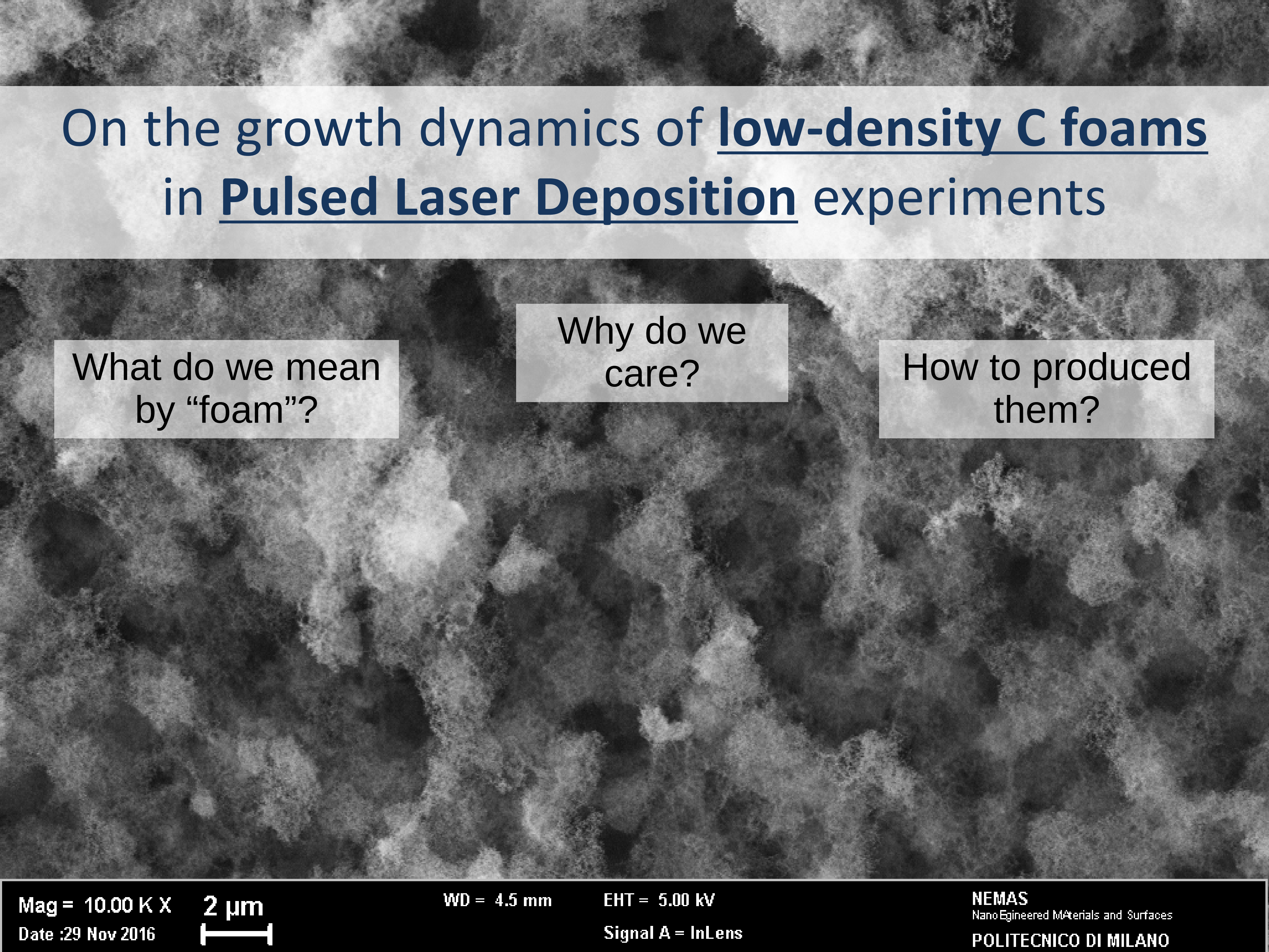


On the growth dynamics of **low-density C foams** in **Pulsed Laser Deposition** experiments

What do we mean by “foam”?

Why do we care?

How to produced them?



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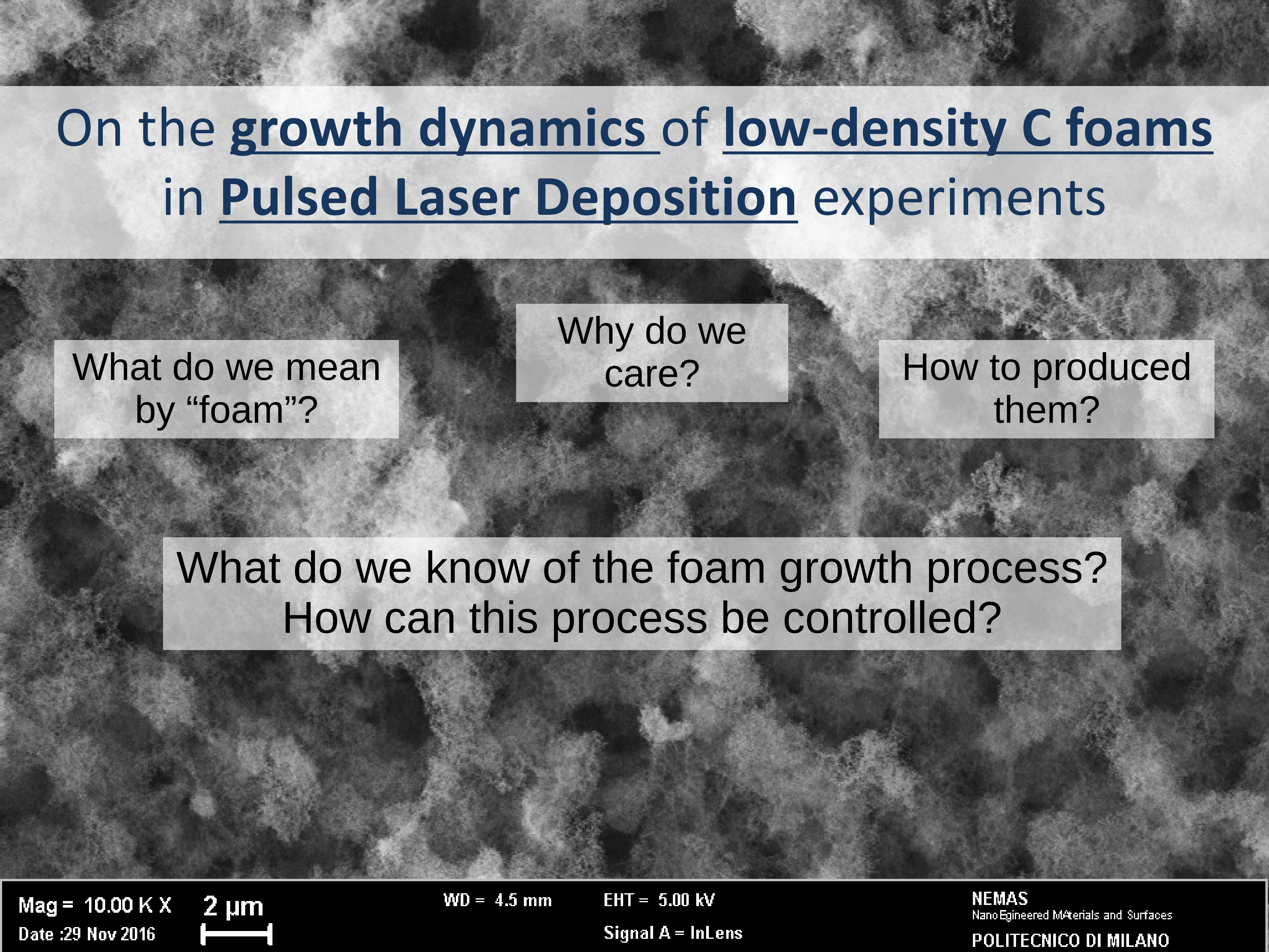

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Caveat!
Work in progress

Mag = 10.00 K X | 2 µm | WD = 4.5 mm | EHT = 5.00 kV | NEMAS Nano Egineered MAterials and Surfaces
Date :29 Nov 2016 | Signal A = InLens | POLITECNICO DI MILANO

What do we mean by “carbon foams” ?

Article Talk https://en.wikipedia.org/wiki/Carbon_nanofoam

Carbon nanofoam

From Wikipedia, the free encyclopedia

Carbon nanofoam is an allotrope of carbon discovered in 1997 by Andrei V. Rode and co-workers at the Australian National University in Canberra.[1] It consists of a cluster-assembly of carbon atoms strung together in a loose three-dimensional web. The material is extremely light, with a density of 2–10 mg/cm^3 (0.0012 lb/ft^3).[1][2] A gallon of nanofoam weighs about a quarter of an ounce.[3]

Each cluster is about 6 nanometers wide and consists of about 4000 carbon atoms linked in graphite-like sheets that are given negative curvature by the inclusion of heptagons among the regular hexagonal pattern. This is the opposite of what happens in the case of buckminsterfullerenes, in which carbon sheets are given positive curvature by the inclusion of pentagons.

The large-scale structure of carbon nanofoam is similar to that of an aerogel, but with 1% of the density of previously produced carbon aerogels—or only a few times the density of air at sea level. Unlike carbon aerogels, carbon nanofoam is a poor electrical conductor. The nanofoam contains numerous unpaired electrons, which Rode and colleagues propose is due to carbon atoms with only three bonds that are found at topological and bonding defects. This gives rise to what is perhaps carbon nanofoam's most unusual feature: it is attracted to magnets, and below −183 °C can itself be made magnetic.

A.V. Rode et al., *Formation of cluster-assembled carbon nano-foam by high-repetition-rate laser ablation*, Appl. Phys. A **70** 135 (2000)

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In this talk, I will refer to **"carbon foam"** as:

- Disordered, nanoscale structured material
- (almost) Pure carbon
- Void fraction ≈ 99% → density ≈ 10 mg/cm^3

A.V. Rode et al., *Formation of cluster-assembled carbon nano-foam by high-repetition-rate laser ablation*, Appl. Phys. A **70** 135 (2000)

Why do we care?

PHYSICAL REVIEW B **70**, 054407 (2004)

Unconventional magnetism in all-carbon nanofoam

A. V. Rode,[1,*,†] E. G. Gamaly,[1] A. G. Christy,[2] J. G. Fitz Gerald,[3] S. T. Hyde,[1] R. G. Elliman,[1] B. Luther-Davies,[1] A. I. Veinger,[4] J. Androulakis,[5] and J. Giapintzakis[5,6,*,‡]

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Journal of Biomedical Materials Research Part A / Volume 85A, Issue 3

Pore structure engineering for carbon foams as possible bone implant material

Gursel Turgut, Ayhan Eksilioglu, Nagehan Gencay, Emre Gonen, Nezih Hekim, M. F. Yardım, Damlanur Sakiz, Ekrem Ekinci

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IETEM IOP Publishing

IOP Conf. Series: Materials Science and Engineering **240** (2017) 012062 doi:10.1088/1757-899X/240/1/012062

Production of thermally conductive carbon foams and their application in automobile transport

V M Samoylov[1], E A Danilov[1], E R Galimov[2], V L Fedyaev[2,3], N Ya Galimova[2] and M A Orlov[4]

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Electrochimica Acta 270 (2018) 236–244

Contents lists available at ScienceDirect

Electrochimica Acta

journal homepage: www.elsevier.com/locate/electacta

ELSEVIER

Graphitic carbon foams as anodes for sodium-ion batteries in glyme-based electrolytes

Jorge Rodríguez-García [a], Ignacio Cameán [a,*], Alberto Ramos [b], Elena Rodríguez [a], Ana B. García [a]

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J Nanopart Res (2017) 19: 386
https://doi.org/10.1007/s11051-017-4080-7

RESEARCH PAPER

Enhanced specific surface area by hierarchical porous graphene aerogel/carbon foam for supercapacitor

Zhaopeng Xin · Weixin Li · Wei Fang · Xuan He · Lei Zhao · Hui Chen · Wanqiu Zhang · Zhimin Sun

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Plasma Physics and Controlled Fusion

Plasma Phys. Control. Fusion **58** (2016) 034019 (8pp)

doi:10.1088/0741-3335/58/3/034019

Development of foam-based layered targets for laser-driven ion beam production

I Prencipe[1,2], A Sgattoni[3,4], D Dellasega[1,5], L Fedeli[3,4], L Cialfi[1], Il Woo Choi[6,7,9], I Jong Kim[6,7,10], K A Janulewicz[6,8], K F Kakolee[6], Hwang Woon Lee[6], Jae Hee Sung[6,7], Seong Ku Lee[6,7], Chang Hee Nam[6,8] and M Passoni[1,5]

Enhanced specific surface area by hierarchical porous graphene aerogel/carbon foam for supercapacitor

Zhaopeng Xin · Weixin Li · Wei Fang · Xuan He · Lei Zhao · Hui Chen · Wanqiu Zhang · Zhimin Sun

...nodes for sodium-ion batteries in

...mean[a,*], Alberto Ramos[b], Elena Rodríguez[a],

ENSURE

Exploring the **N**ew **S**cience and engineering unveiled by **U**ltraintense ultrashort **R**adiation interaction with matt**E**r

POLITECNICO MILANO 1863

DIPARTIMENTO DI ENERGIA

ERC-2014-CoG No.647554

ERC consolidator grant: 5 year project, from September 2015 to September 2020

Goal: To **E**xplore the **N**ew **S**cience and engineering unveiled by **U**ltraintense, ultrashort **R**adiation interaction with matt**E**r

Hosted @ NanoLab, Energy department, Politecnico di Milano

Principal investigator:
Matteo Passoni, Associate professor

Team: 2 Associate Professor, 1 Assistant Professor, 3 Post-Docs, 3 PhDs + master students and support from NanoLab people

www.ensure.polimi.it

C foam for superintense laser-plasma experiments

$I_{laser}=10^{20}$ W/cm^2 ⟶ $\mathbf{E}_{laser} = 3 \times 10^{11}$ V/m = 50 X $\mathbf{E}_{atomic}$ ⟶ Full ionization ⟶ Plasma!

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Plasma critical density:

$$n_c = \frac{\pi\, m_e c^2}{e\, \lambda^2}$$

$n_c \approx 6\ \mathrm{mg/cm^3}$
(@ λ=800 nm)

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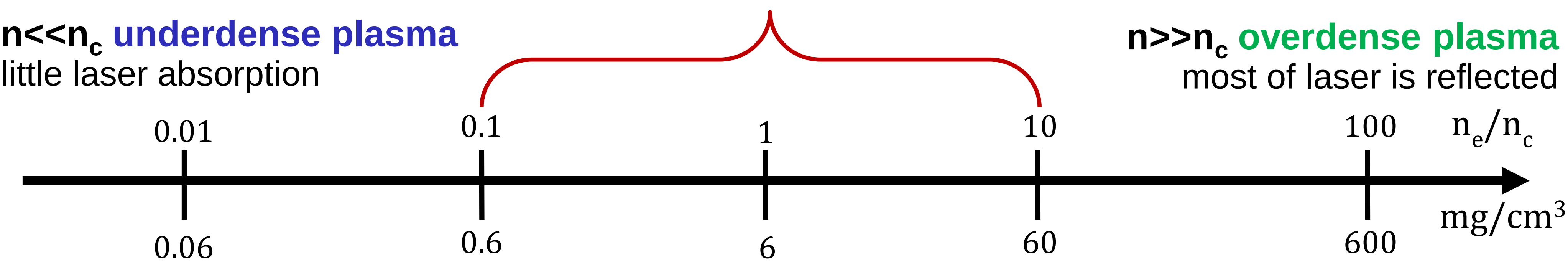
n ≈ n_c near critical plasma
strong laser-plasma coupling

n<<n_c underdense plasma
little laser absorption

n>>n_c overdense plasma
most of laser is reflected



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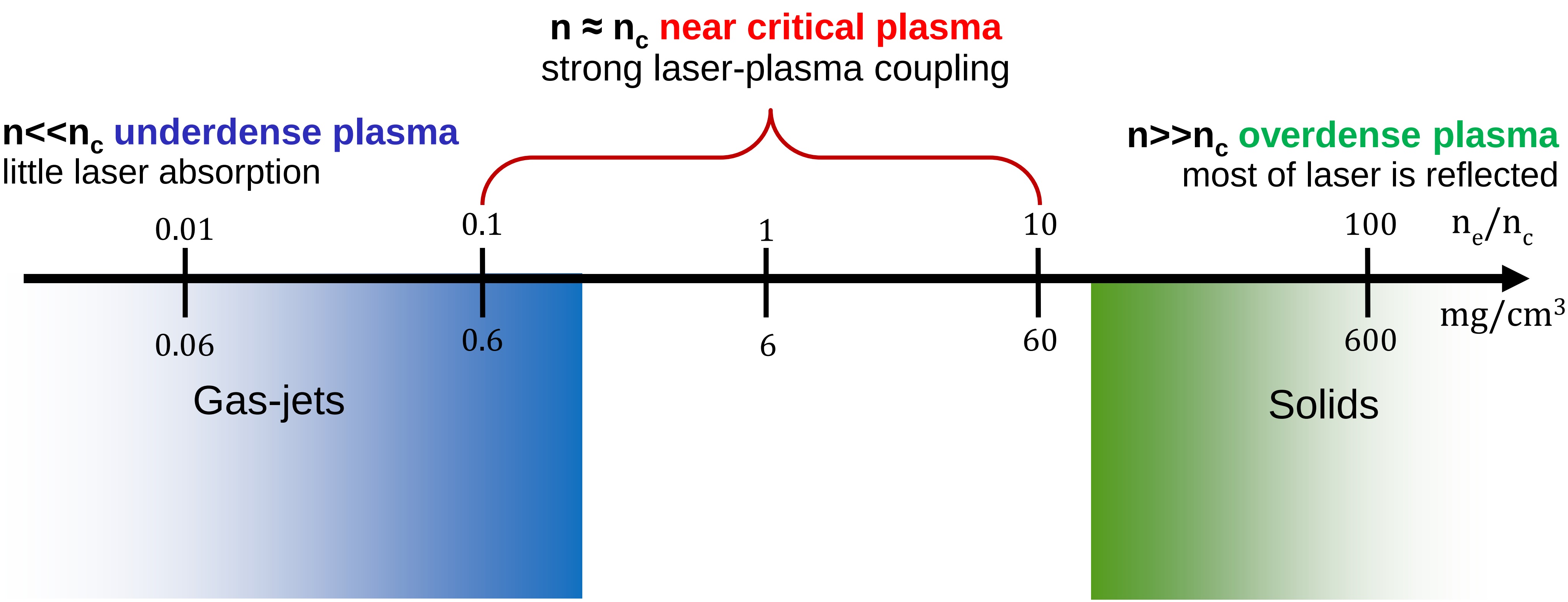

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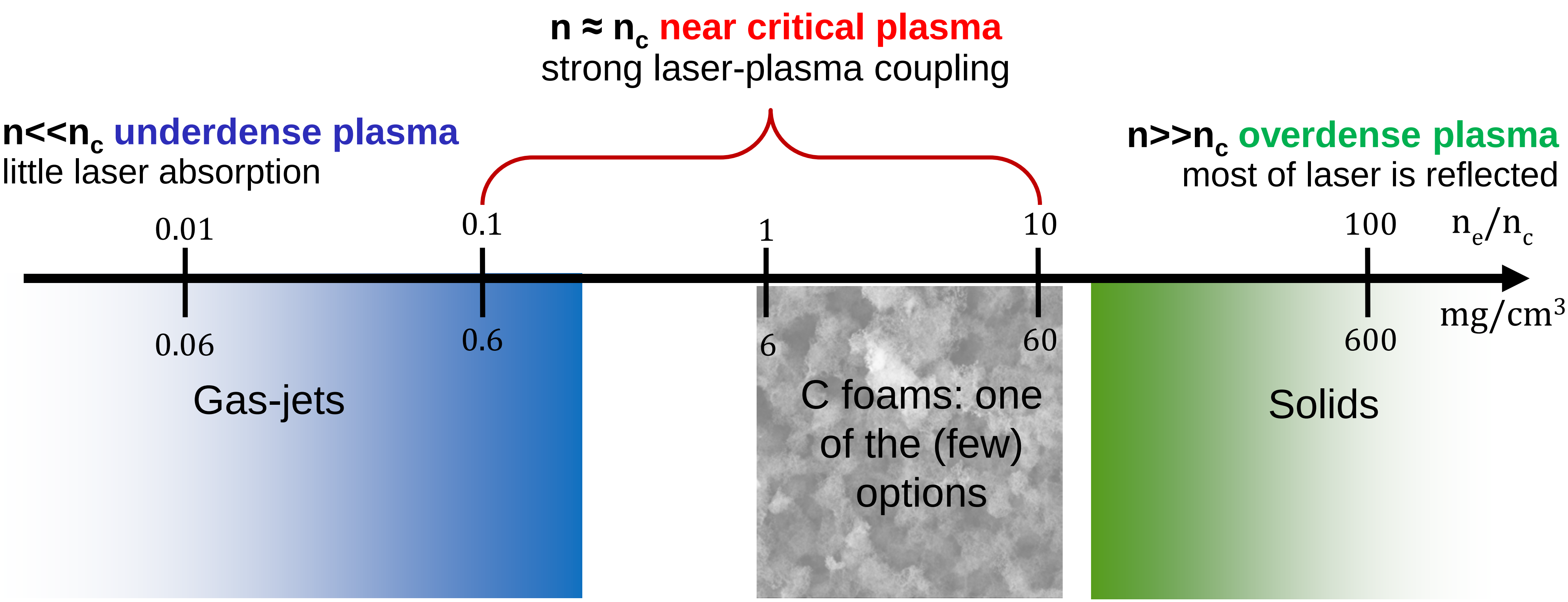

Foam-based targets for proton acceleration

ERC-2014-CoG No.647554
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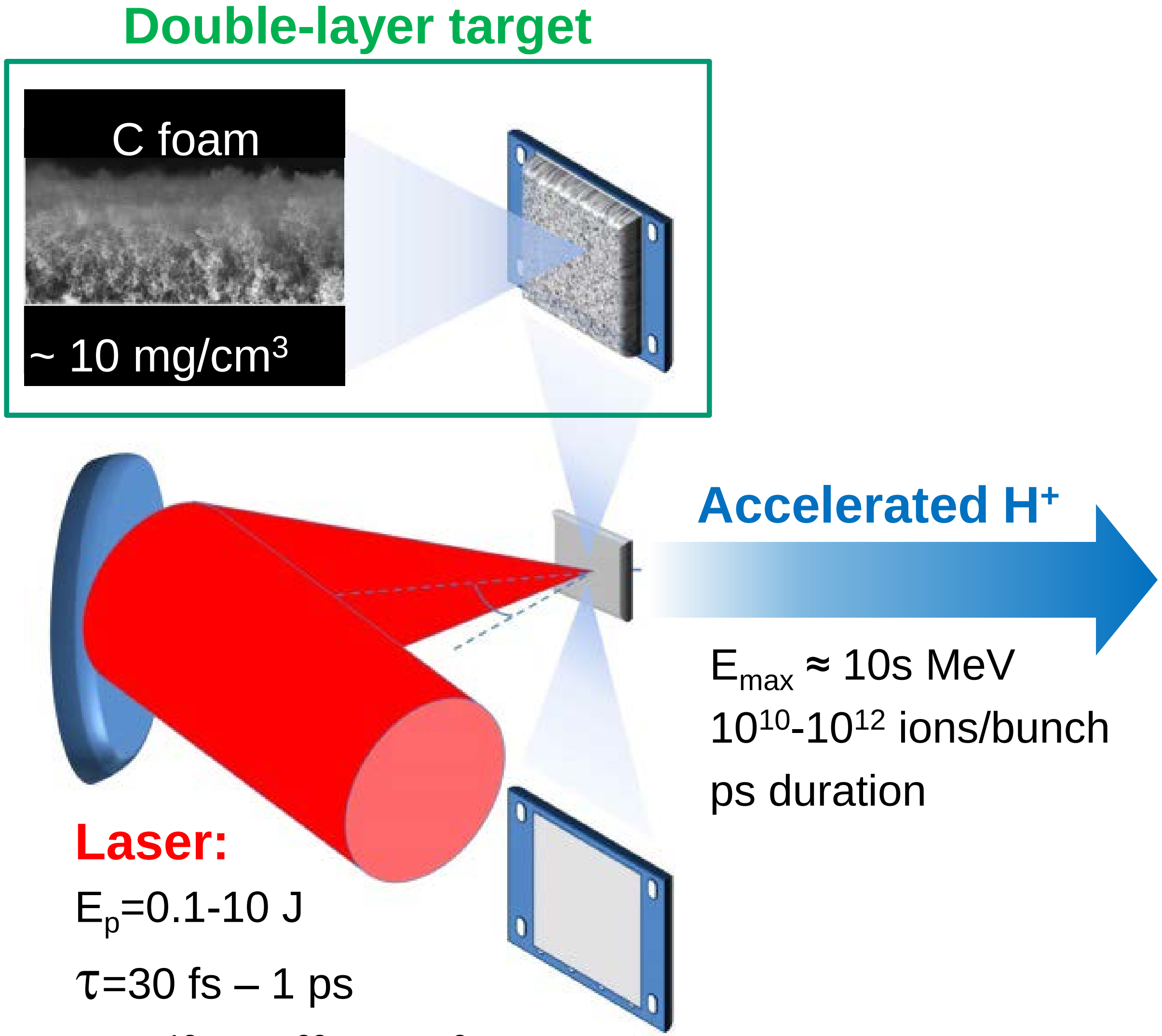

$E_{max} \approx$ 10s MeV
10^{10}-10^{12} ions/bunch
ps duration

E_p=0.1-10 J
τ=30 fs – 1 ps
$I=10^{18} – 10^{22}$ W/cm^2

M. Passoni et al., *Plasma Phys. Control. Fus.* **56** (2014)
I. Prencipe et al., *Plasma Phys. Control. Fus.* **58** (2016)
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Foam-based targets for proton acceleration

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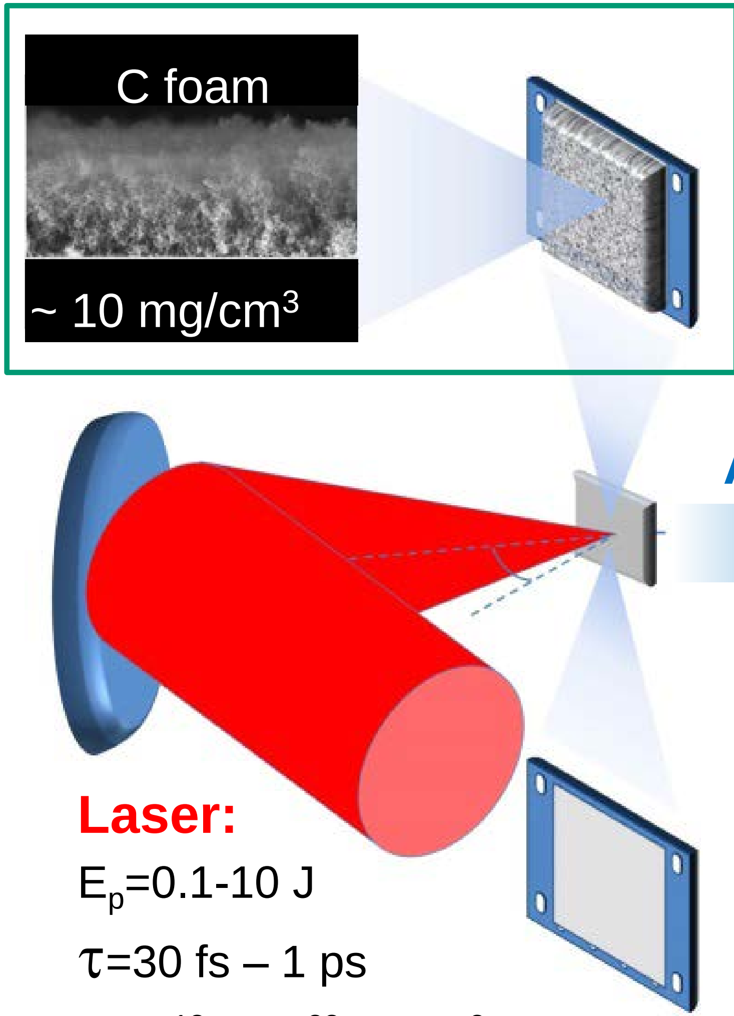
Double-layer target



Accelerated H⁺

E_{max} ≈ 10s MeV
10^{10}-10^{12} ions/bunch
ps duration

Laser:
E_p=0.1-10 J
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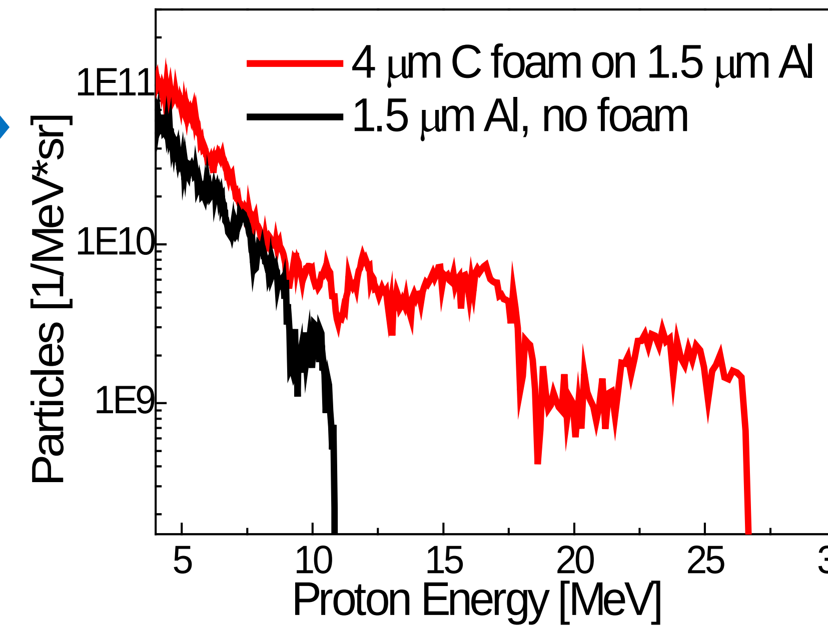
I=10^{18} – 10^{22} W/cm^2

…Foams do work!



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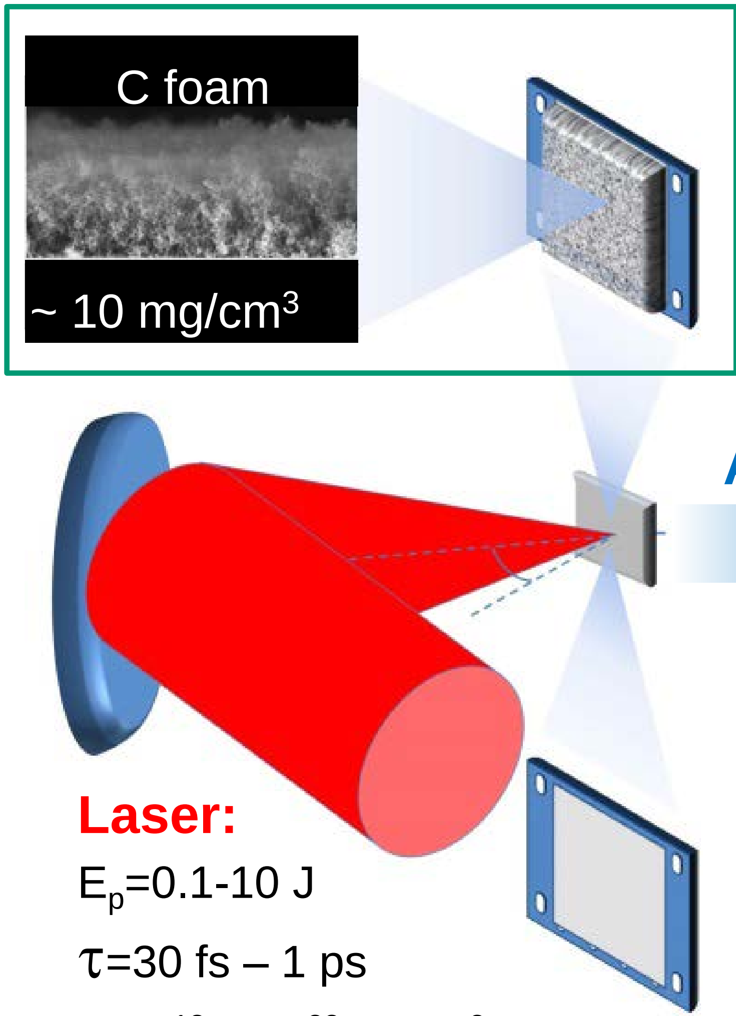

- ❑ C foam on ~μm thick foils
- ❑ It's not just a matter of density!
  (thickness, uniformity, nanostructure,…)
- ❑ "Targetry" issues
  (fragile substrate, stresses, high rep rate,..)

Accelerated H^+

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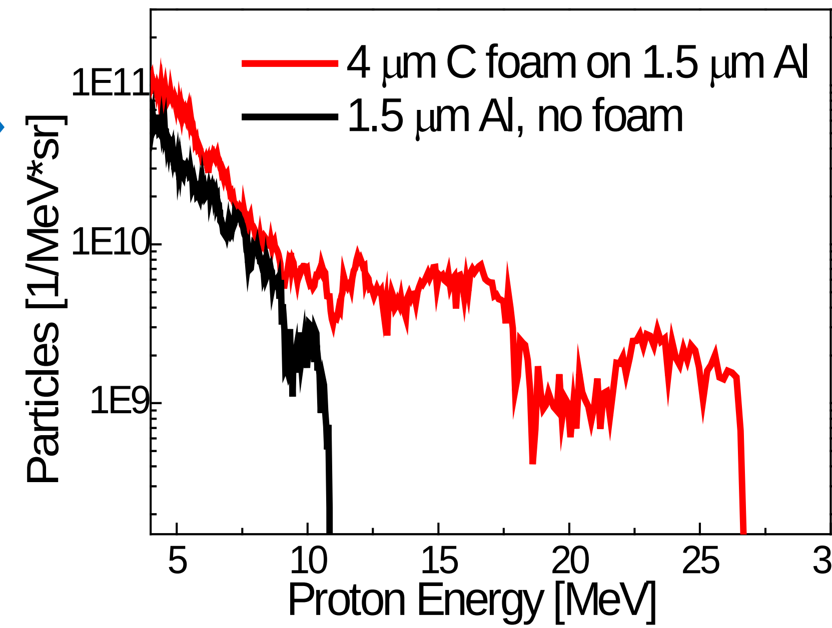

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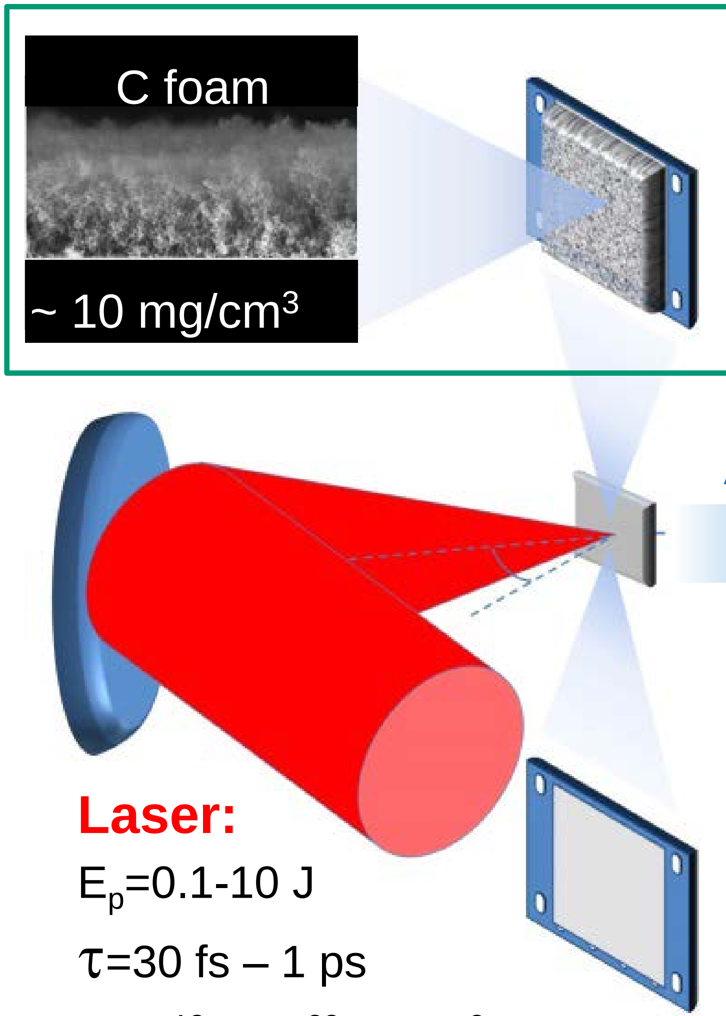

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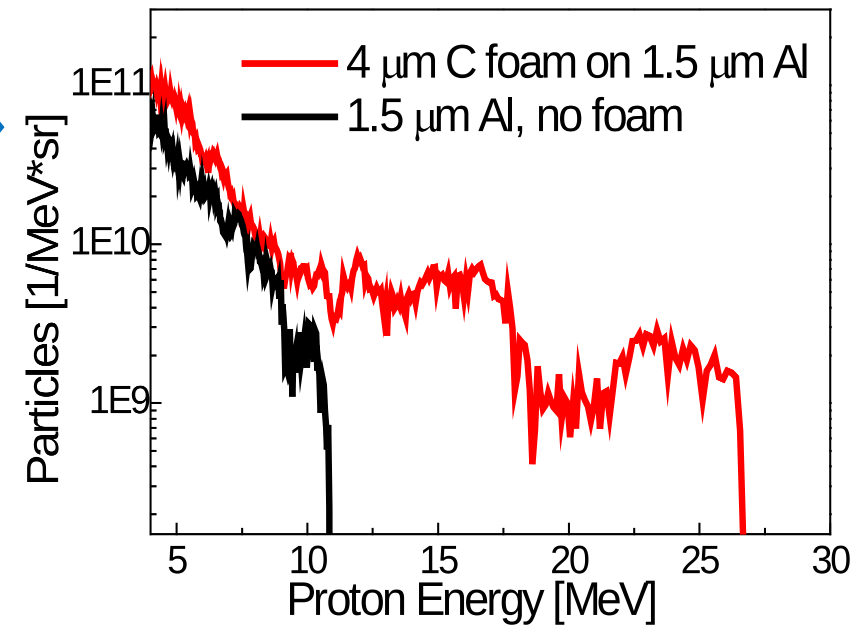

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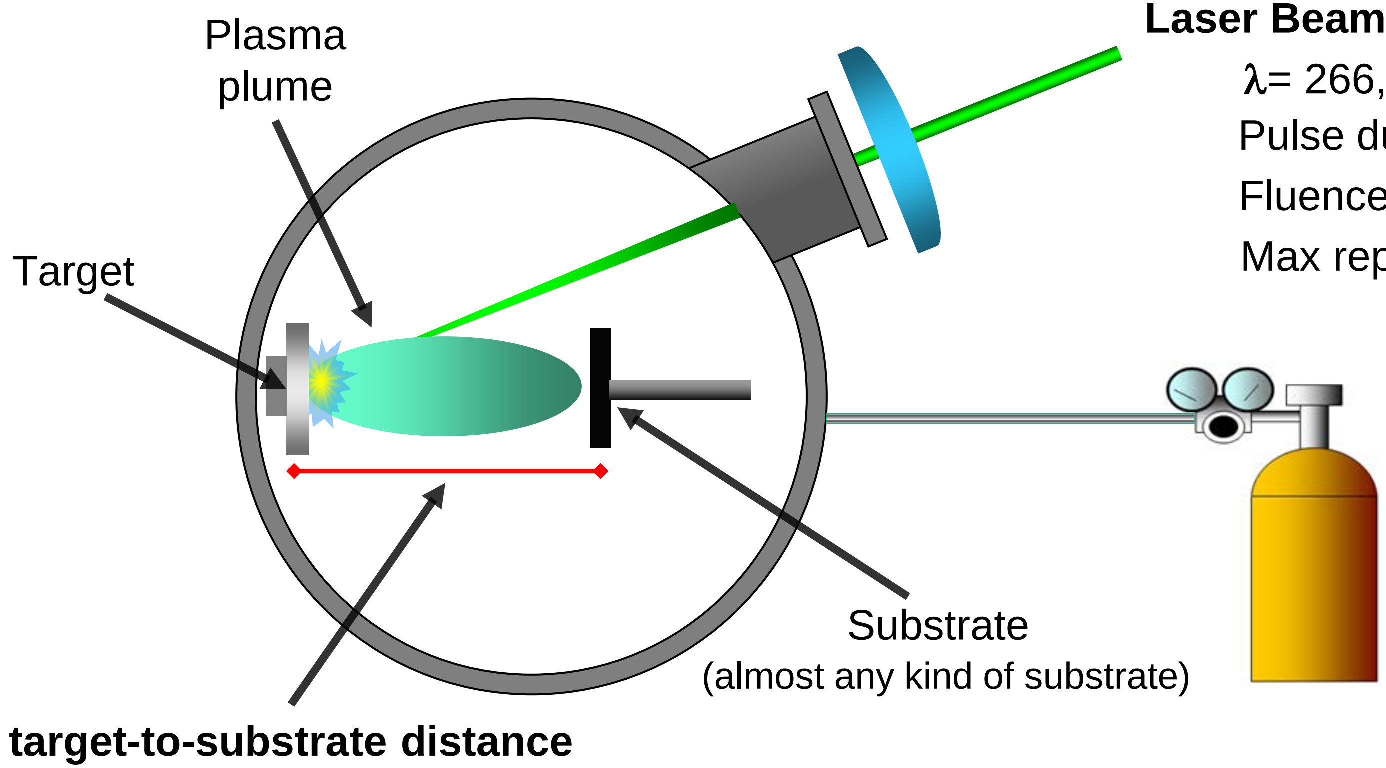
How to produce C foams : Pulsed Laser Deposition (PLD)



Laser Beam

λ= 266, 532, 1064 nm

Pulse duration= 7ns, energy= 0.1-2 J

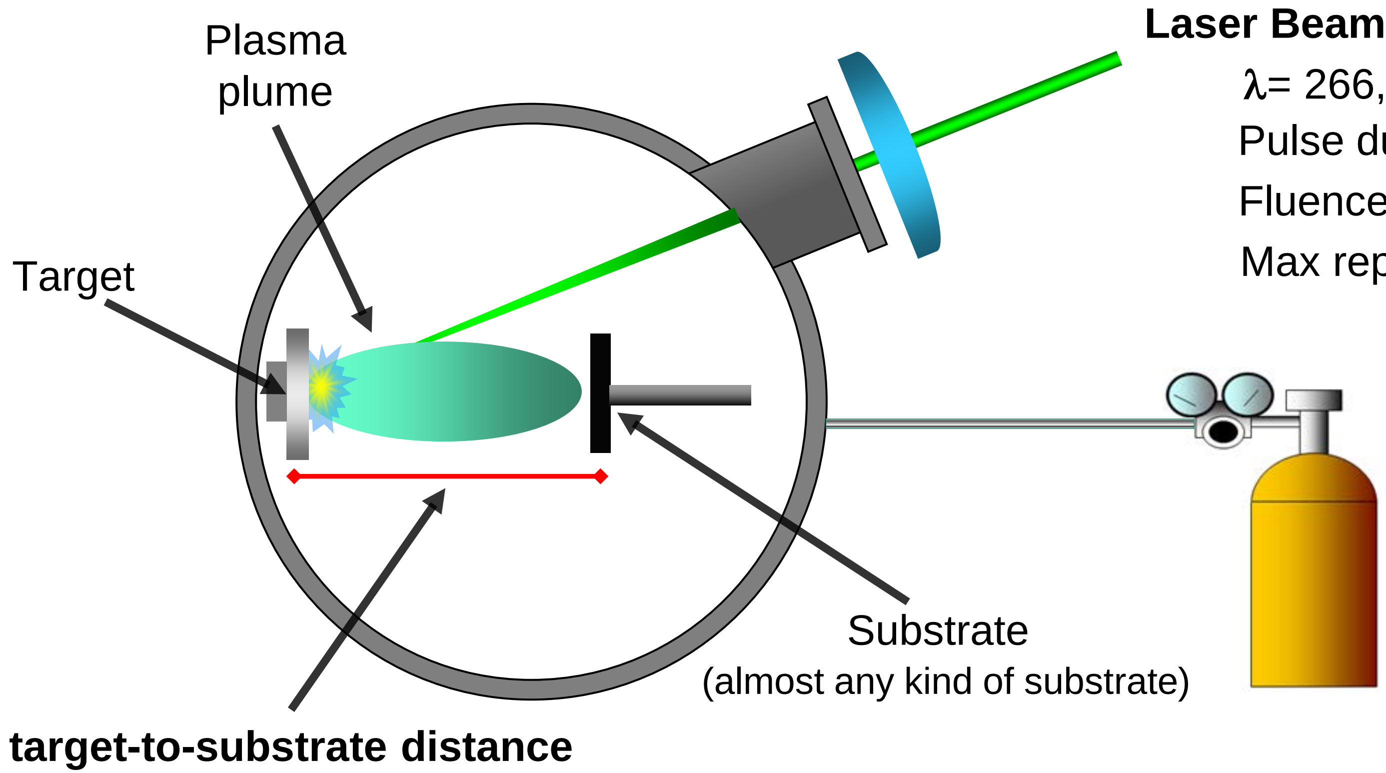
Fluence: 0.1 - 20 J/cm^2

Max rep. rate= 10 Hz

Background Gas

- Inert (He, Ar..)
- Reactive (O_2)

How to produce C foams : Pulsed Laser Deposition (PLD)



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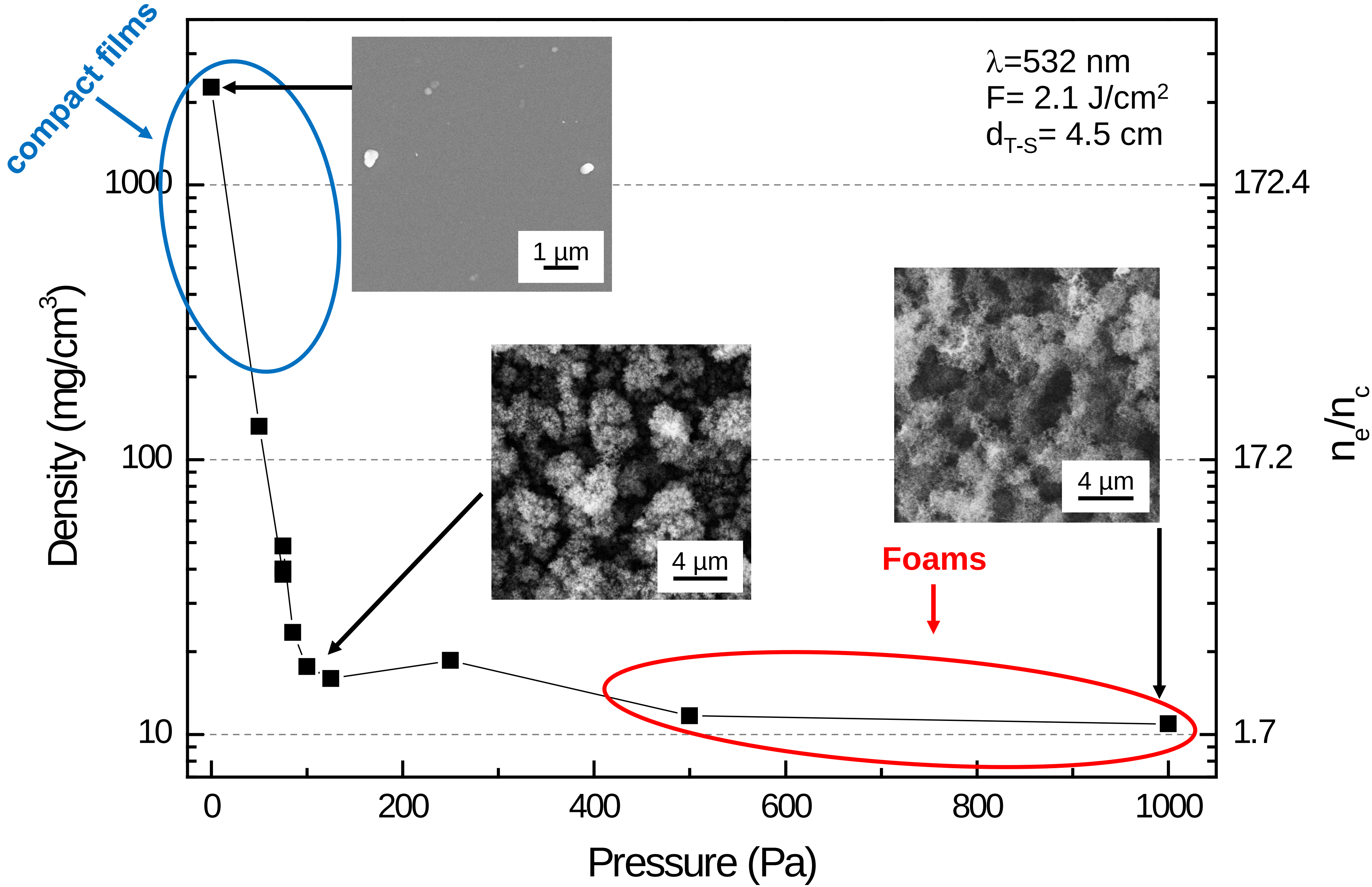
“atom by atom” deposition

“Nanoparticle” deposition

How to produce carbon foams



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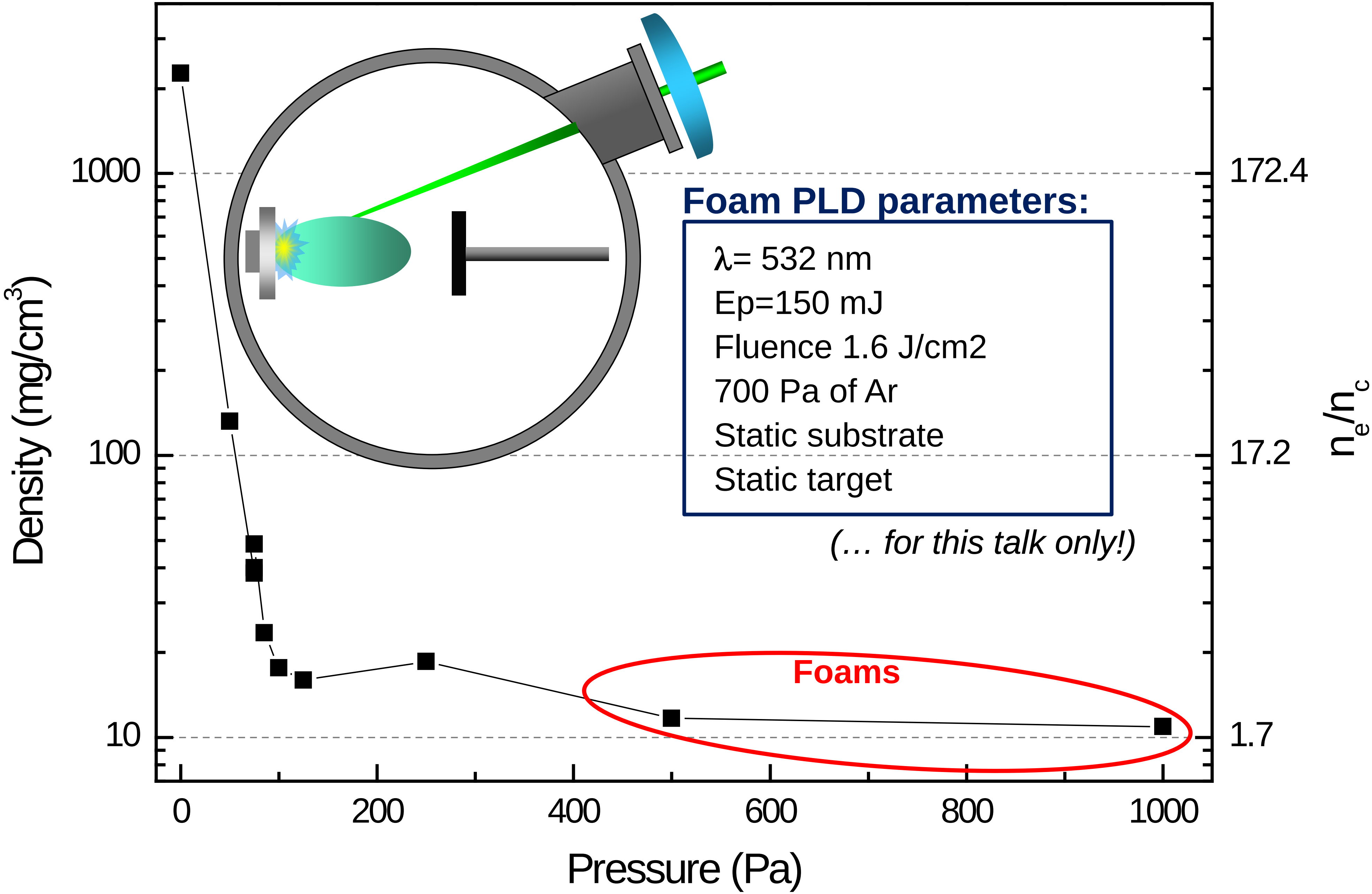
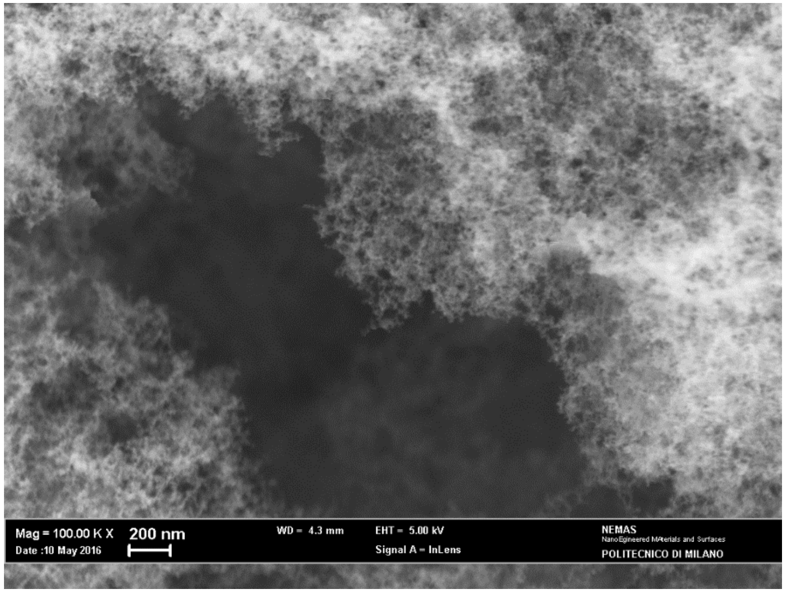


What "foams" actually are made of?



A. Zani *et al.*, Carbon, 56 358 (2013)

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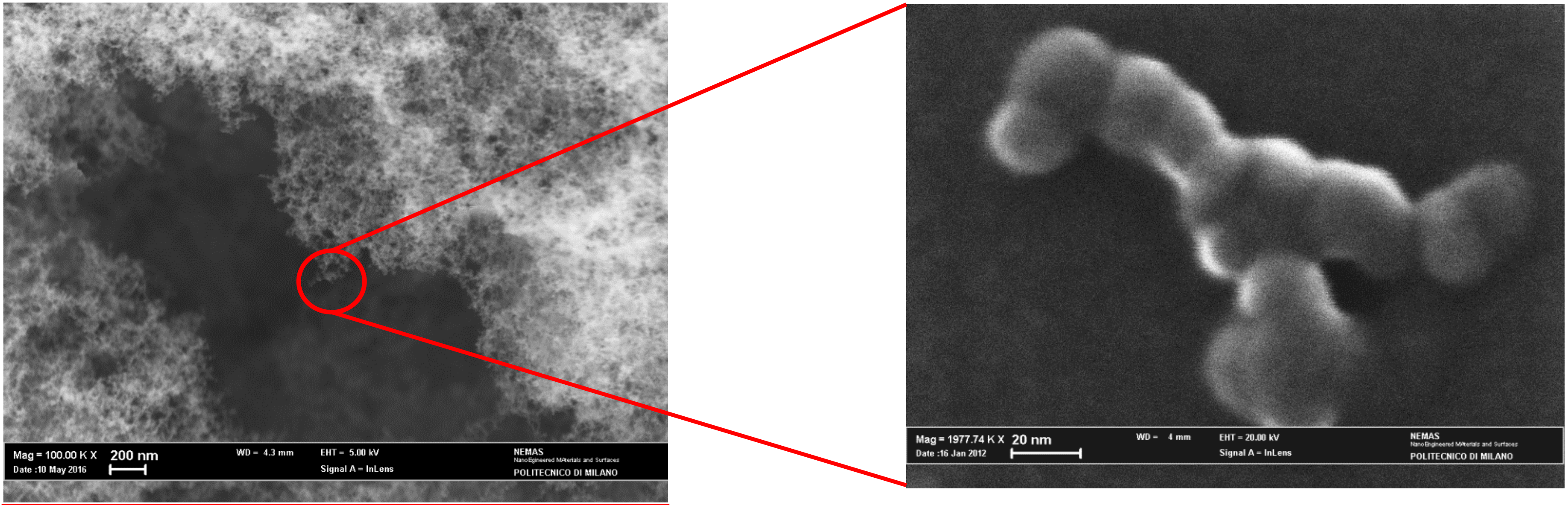

Elementary constituents:
10-20 nm C nanoparticles

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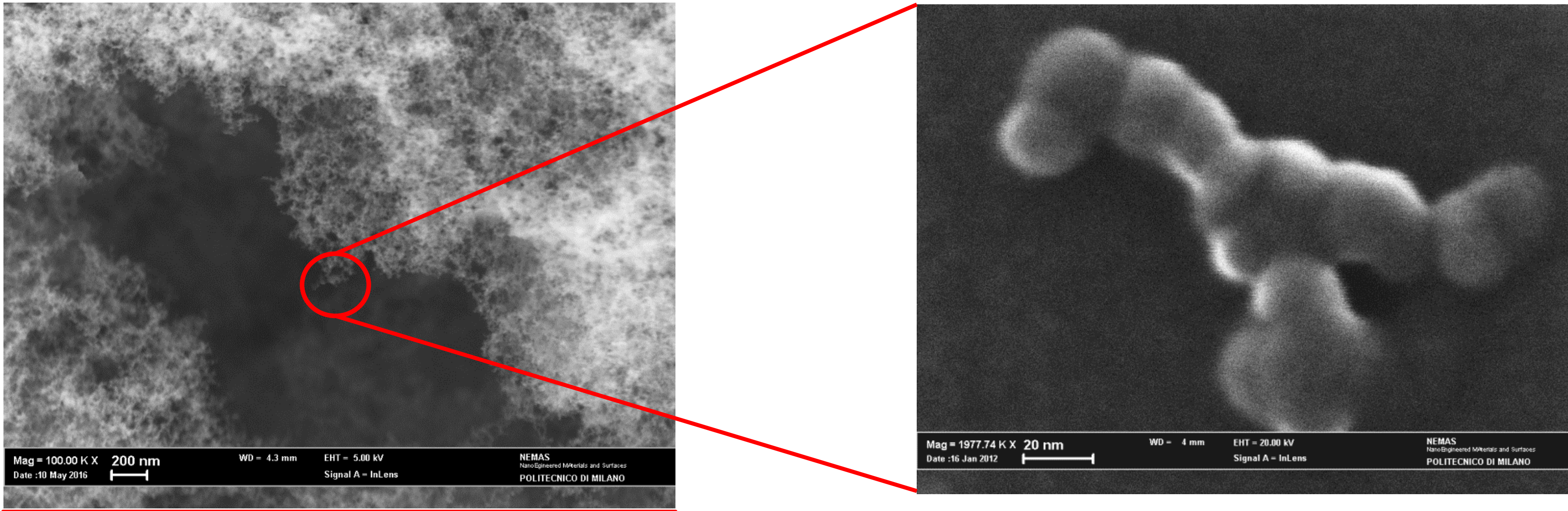
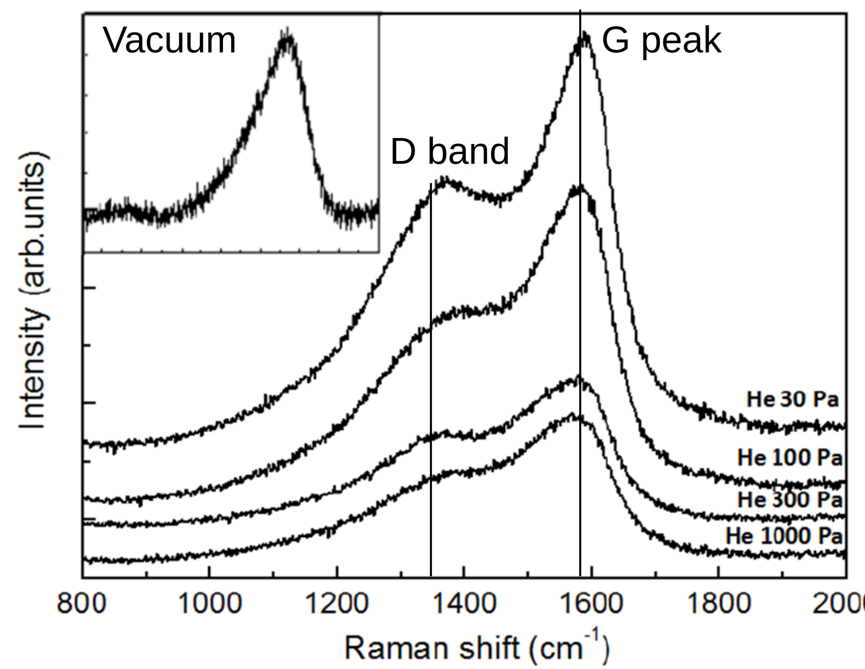


Elementary constituents:
10-20 nm C nanoparticles

C-C bonding:
Nearly pure sp^2
odd-membered rings and
few chain-like structures

Crystalline structure:
Topologically disordered domains,
Size ~ 2nm



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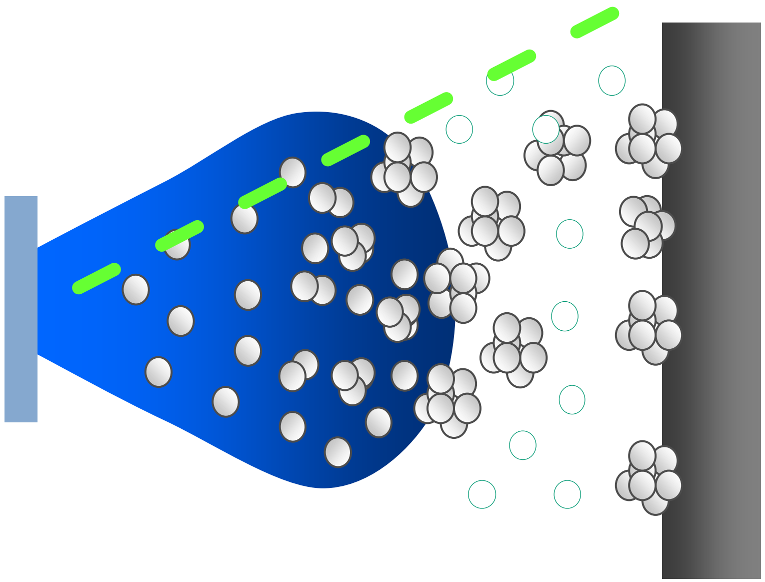
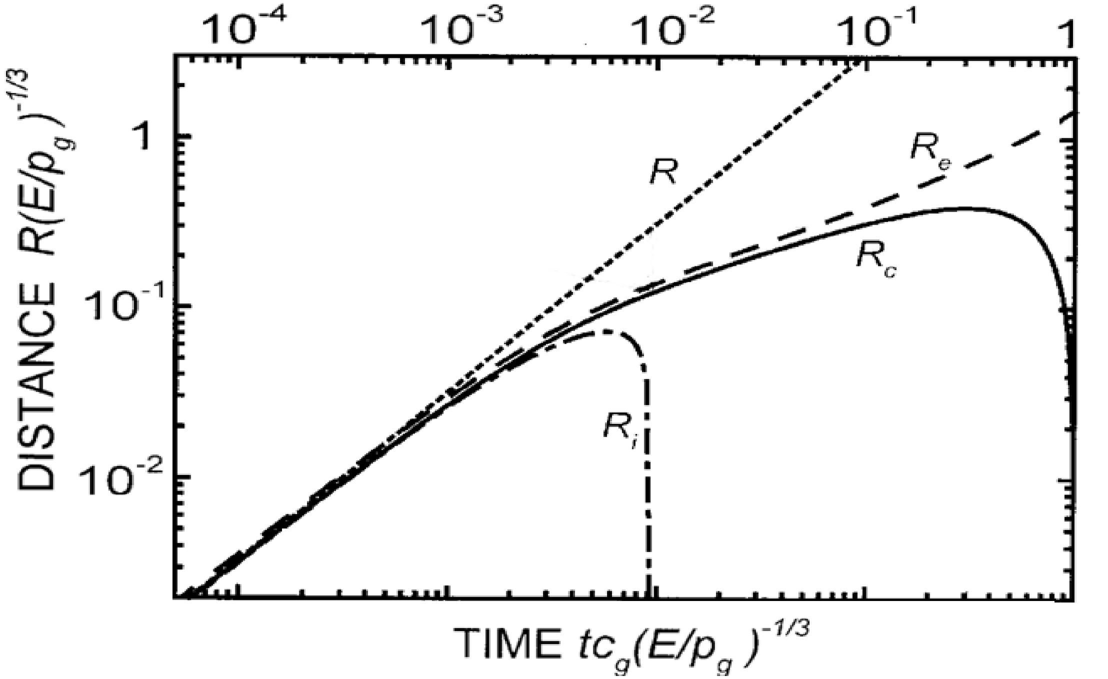
Plume expansion and NPs synthesis

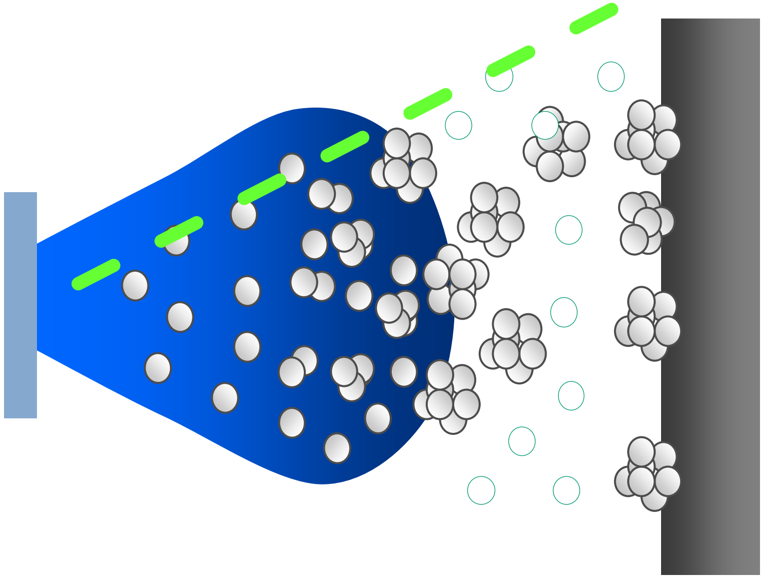




PLD plume dynamics & NP production are open research topics!

Adapted from: Arnolds et al., *Appl. Phys. A* 69 S87–S93 (1999)

Plume expansion and NPs synthesis



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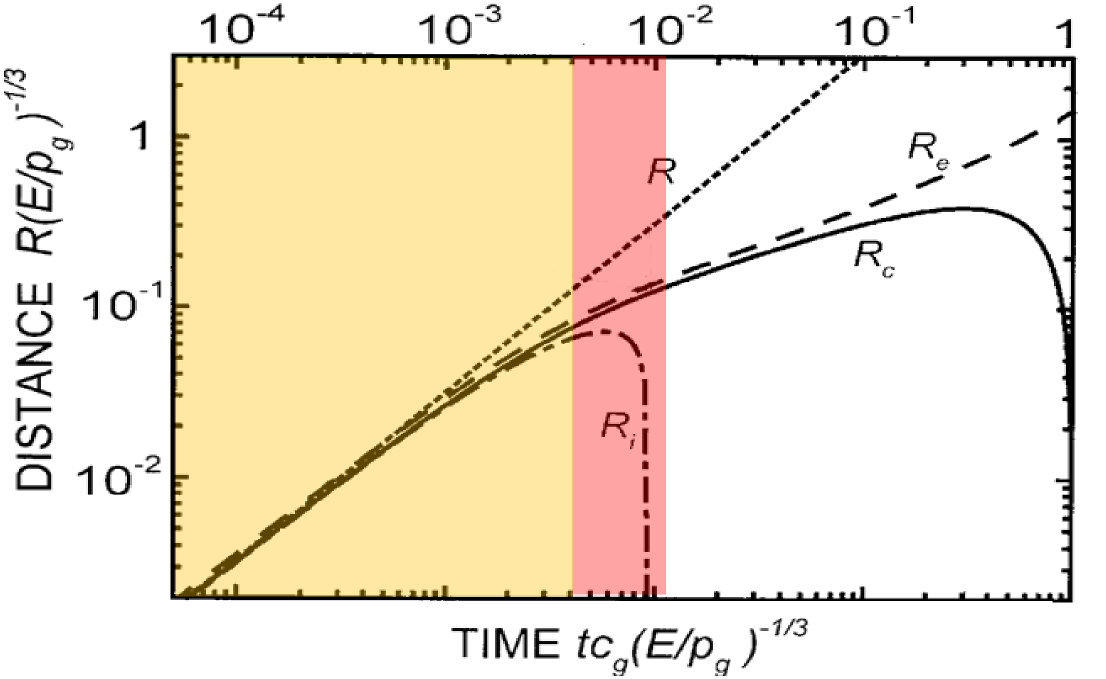

Adapted from: Arnolds et al., *Appl. Phys. A* 69 S87–S93 (1999)

A sketch of plume dynamics:

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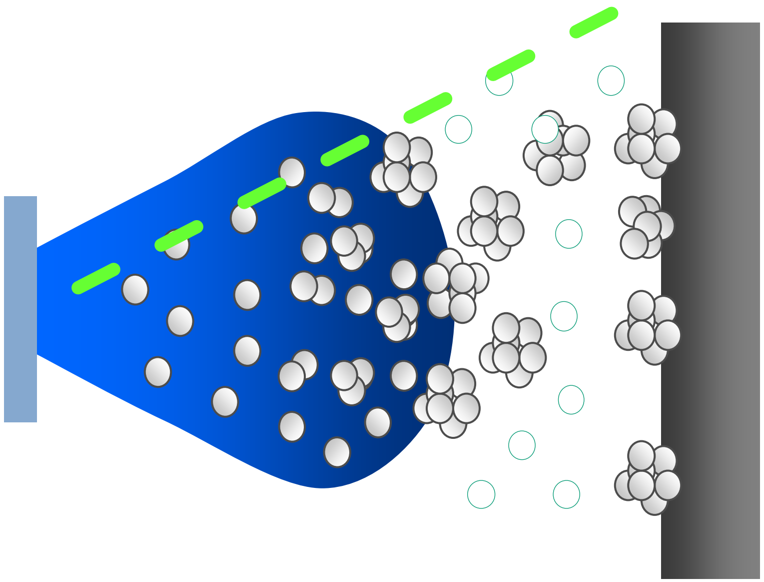
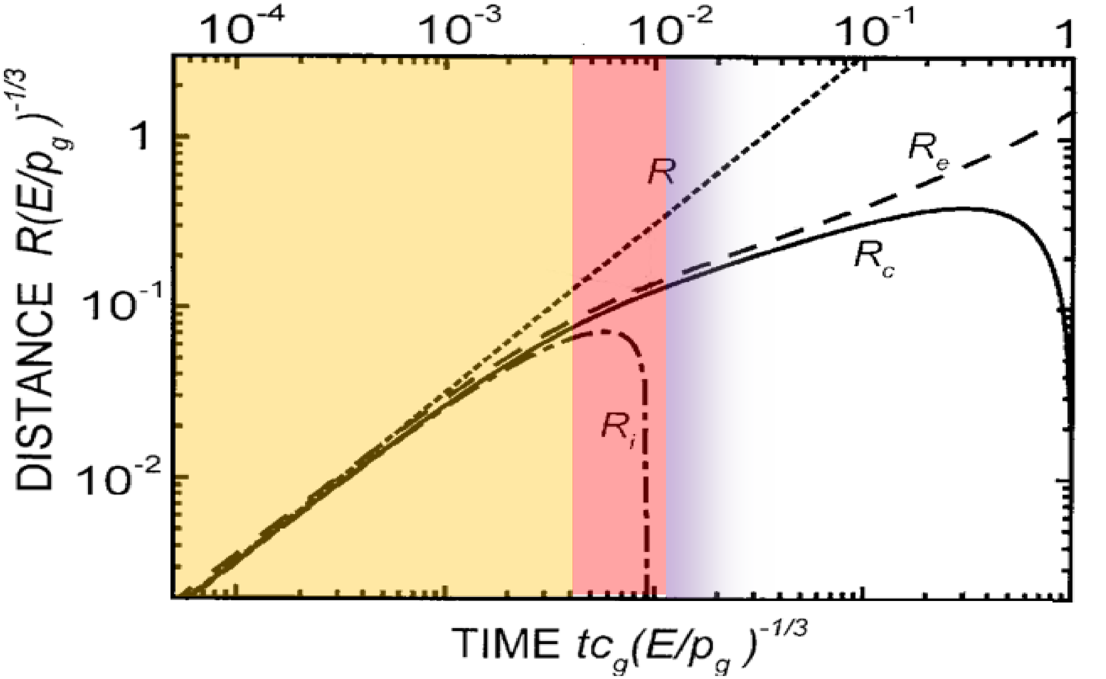
2) Shock wave formation

Plume expansion and NPs synthesis





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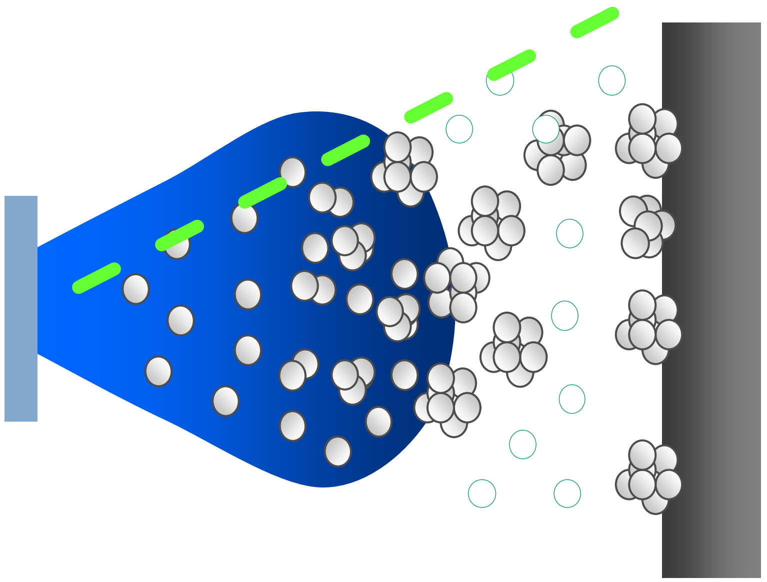

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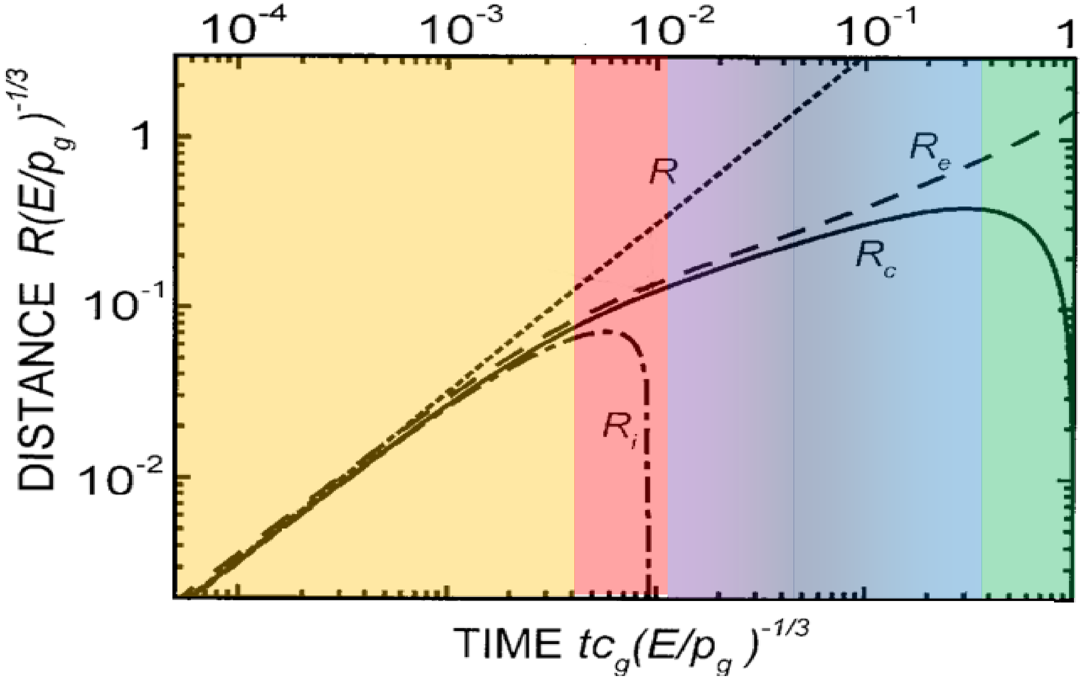

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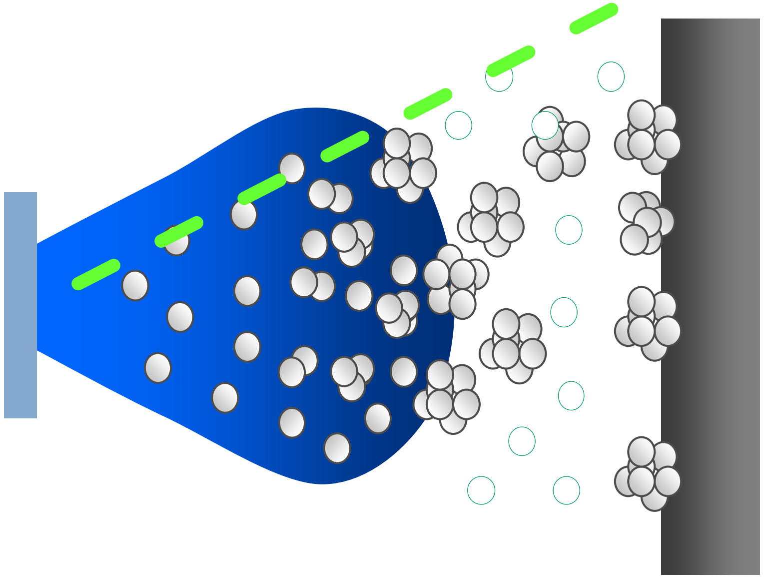

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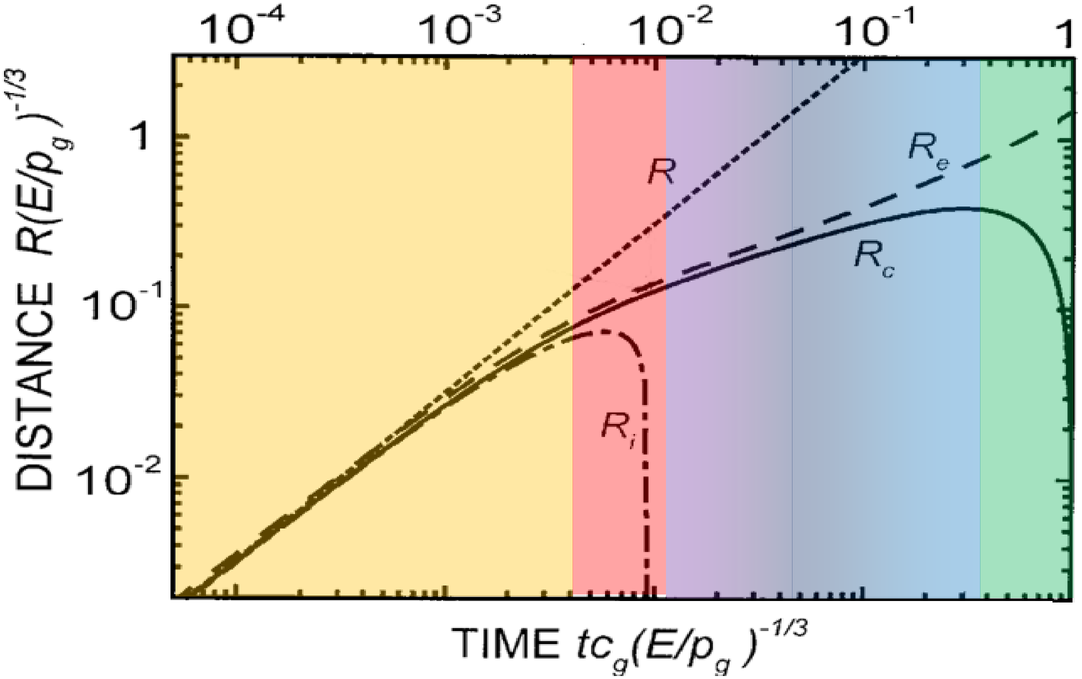

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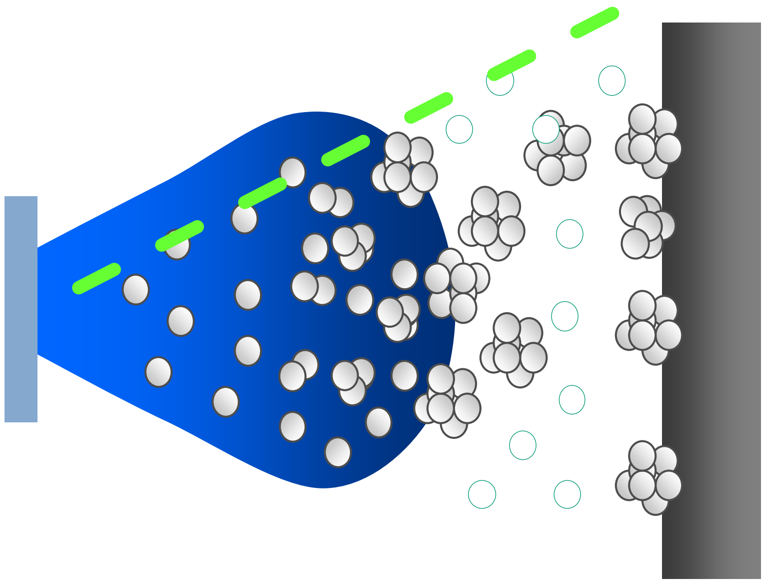

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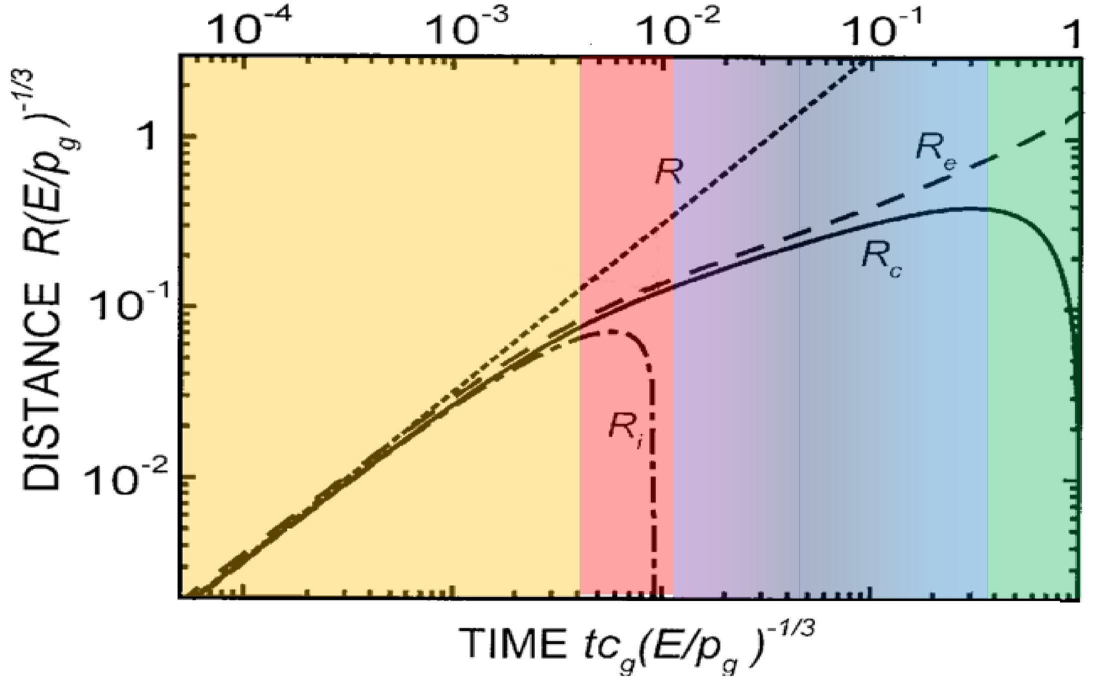

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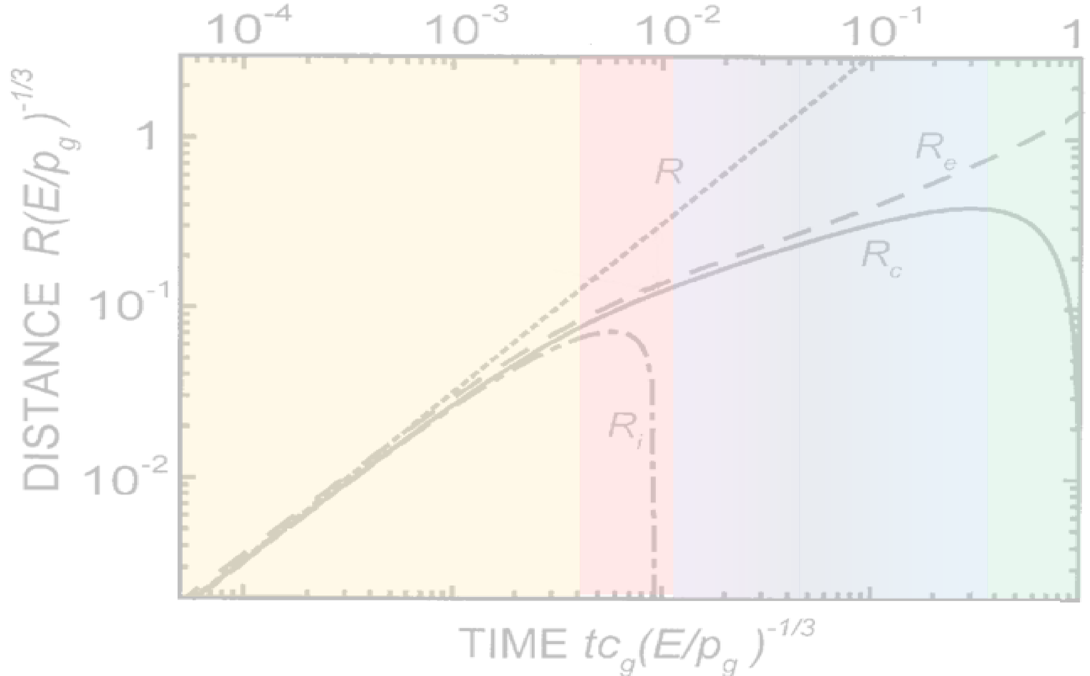

PLD plume dynamics in background gas is still an open research topic!

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REVIEWS OF MODERN PHYSICS

Growth of nanostructures by cluster deposition: Experiments and simple models

Pablo Jensen

Rev. Mod. Phys. **71**, 1695 – Published 1 October 1999 DOI: https://doi.org/10.1103/RevModPhys.71.1695

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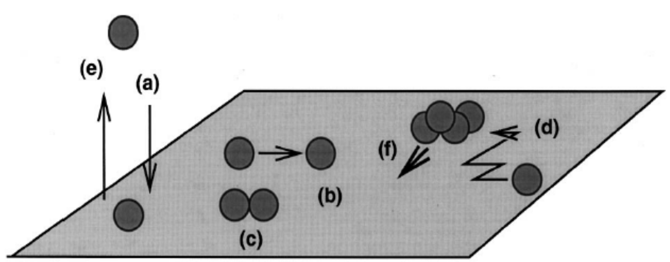

FIG. 5. Main elementary processes considered in this paper for the growth of films by cluster deposition: (a) adsorption of a cluster by deposition; (b) and (d) diffusion of the isolated clusters on the substrate; (c) formation of an island of two monomers by juxtaposition of two monomers (nucleation); (d) growth of a supported island by incorporation of a diffusing cluster; (e) evaporation of an adsorbed cluster. I also briefly consider the influence of island diffusion (f).

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Diffusion happens on substrate → 2D physics

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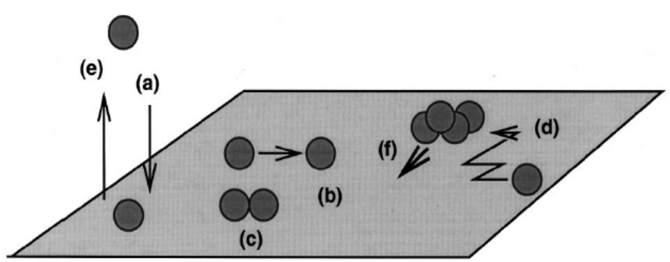

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2D Diffusion Limited Aggregation
(2D-DLA)

What is said in the literature?

IOP Publishing *Mater. Res. Express* 4 (2017) 015013 https://doi.org/10.1088/2053-1591/aa50e9

Evidence of diffusive fractal aggregation of TiO_2 nanoparticles by femtosecond laser ablation at ambient conditions

G L Celardo[1,2,3,4], D Archetti[2], G Ferrini[1,2], L Gavioli[1,2], P Pingue[5] and E Cavaliere[1,2]

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E-mail: nicedirac@gmail.com

Abstract

The specific mechanisms which lead to the formation of fractal nanostructures by pulsed laser deposition remain elusive despite intense research efforts, motivated mainly by the technological interest in obtaining tailored nanostructures with simple and scalable production methods. Here we focus on fractal nanostructures of titanium dioxide, TiO_2, a strategic material for many applications, obtained by femtosecond laser ablation at ambient conditions. We compare a theoretical model of fractal formation with experimental data. The comparison of theory and experiment confirms that fractal aggregates are formed after landing of the ablated material on the substrate surface by a simple diffusive mechanism. We model the fractal formation through extensive Monte Carlo simulations based on a set of minimal assumptions: TiO_2 nanoparticles arrive already formed on the substrate, then they diffuse in a size/mass independent way and stick irreversibly upon touching, thus forming fractal clusters. Despite its simplicity, our model explains the main features of the fractal structures arising from the complex interaction of large TiO_2 nanoparticles with different substrates. Indeed our model is able to reproduce both the fractal dimensions and the area distributions of the nanostructures for different densities of the ablated material. Finally we discuss the role of the thermal conductivity of

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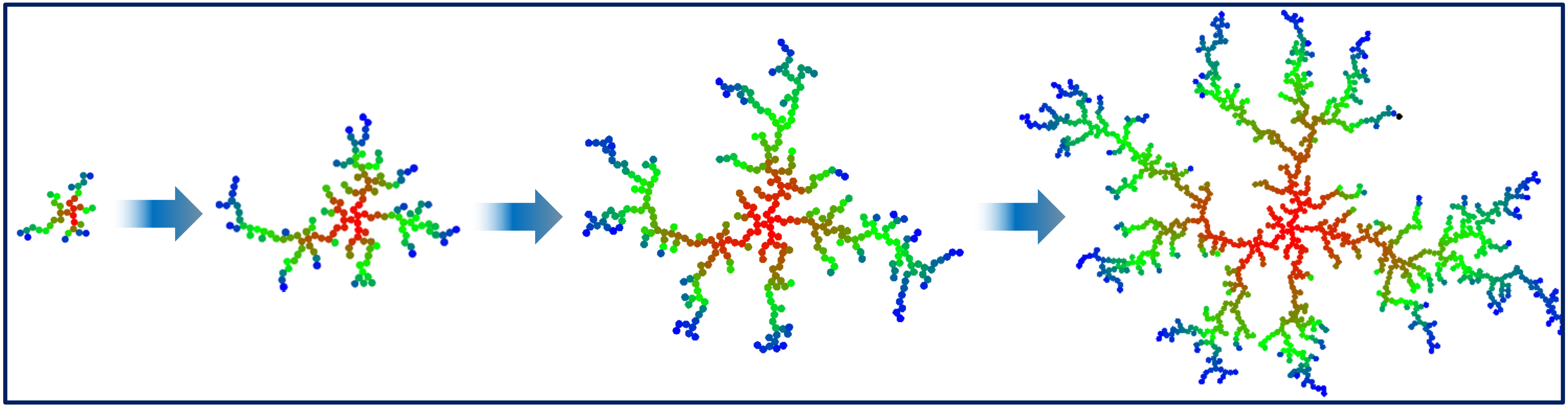
2D-DLA!

Is 2D-DLA ok to describe foam growth?

With 2D-DLA, aggregate grow like this:



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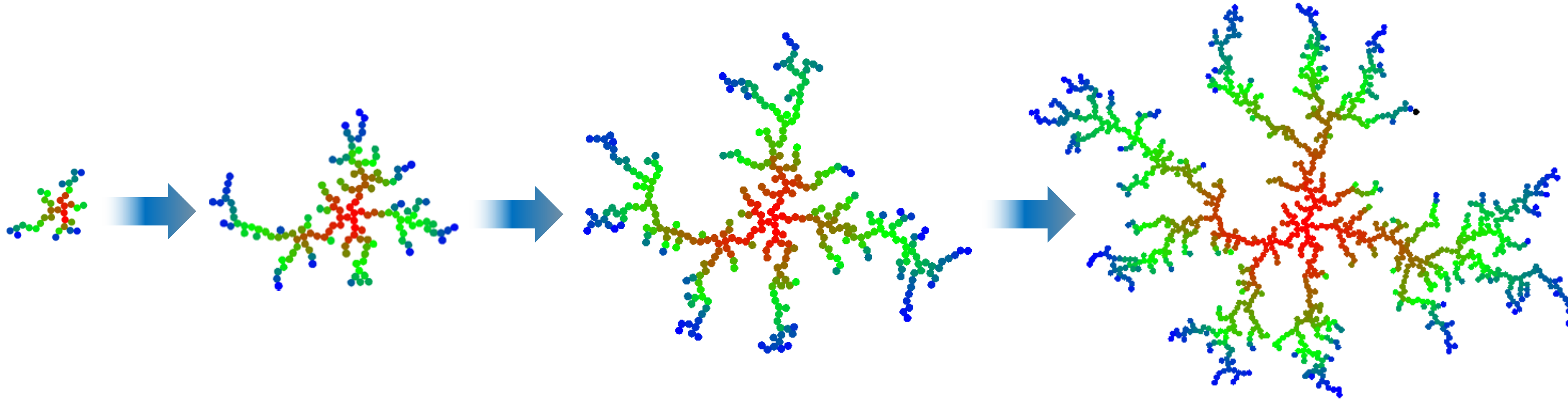

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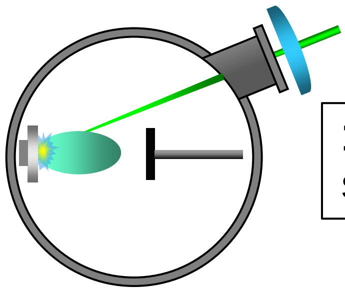

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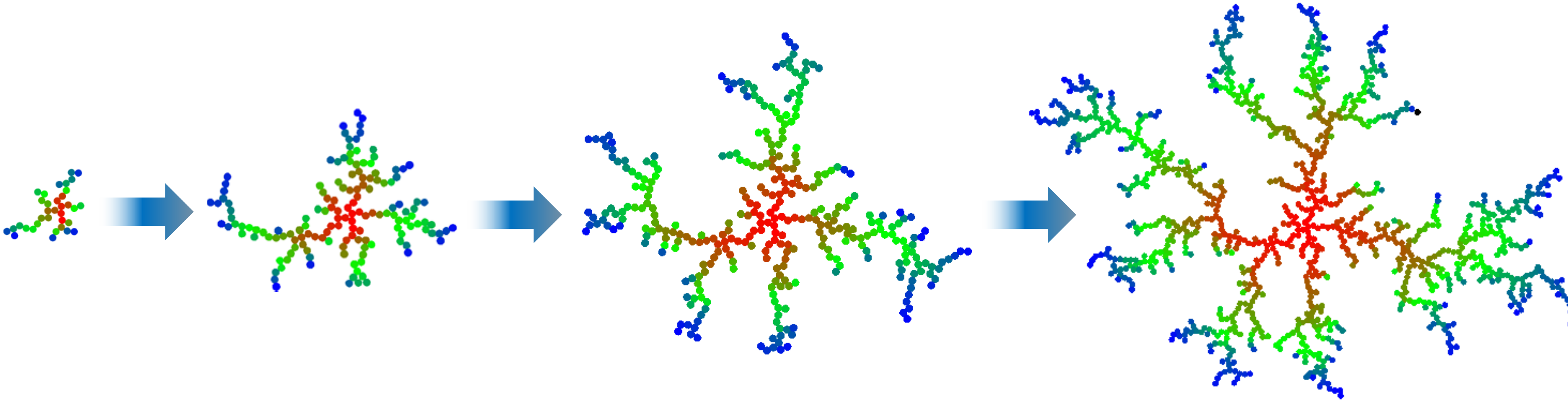

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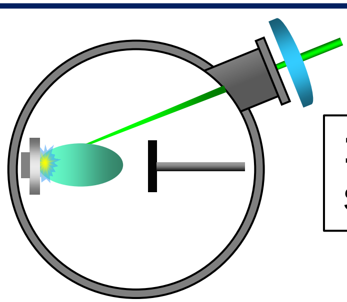

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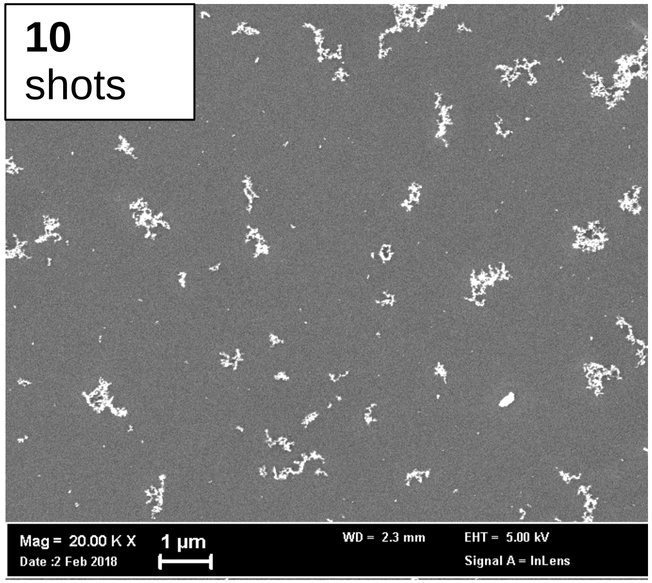
2D-DLA predicts:

1) Very small aggregates for few shots

2) Aggregate size will increase with increasing shots

10 shots



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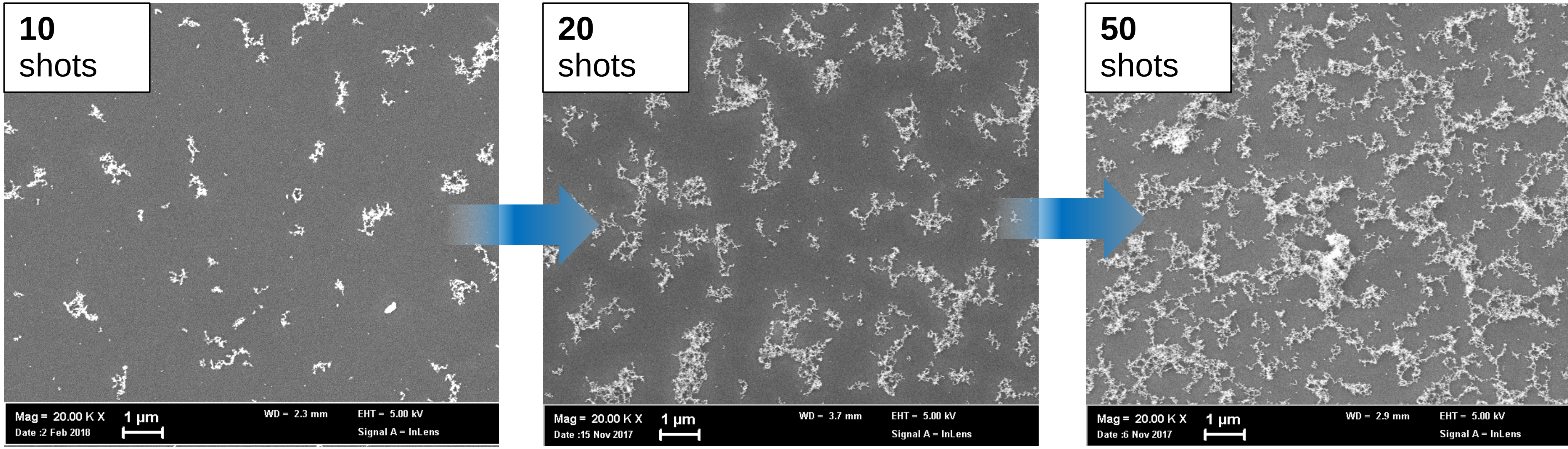

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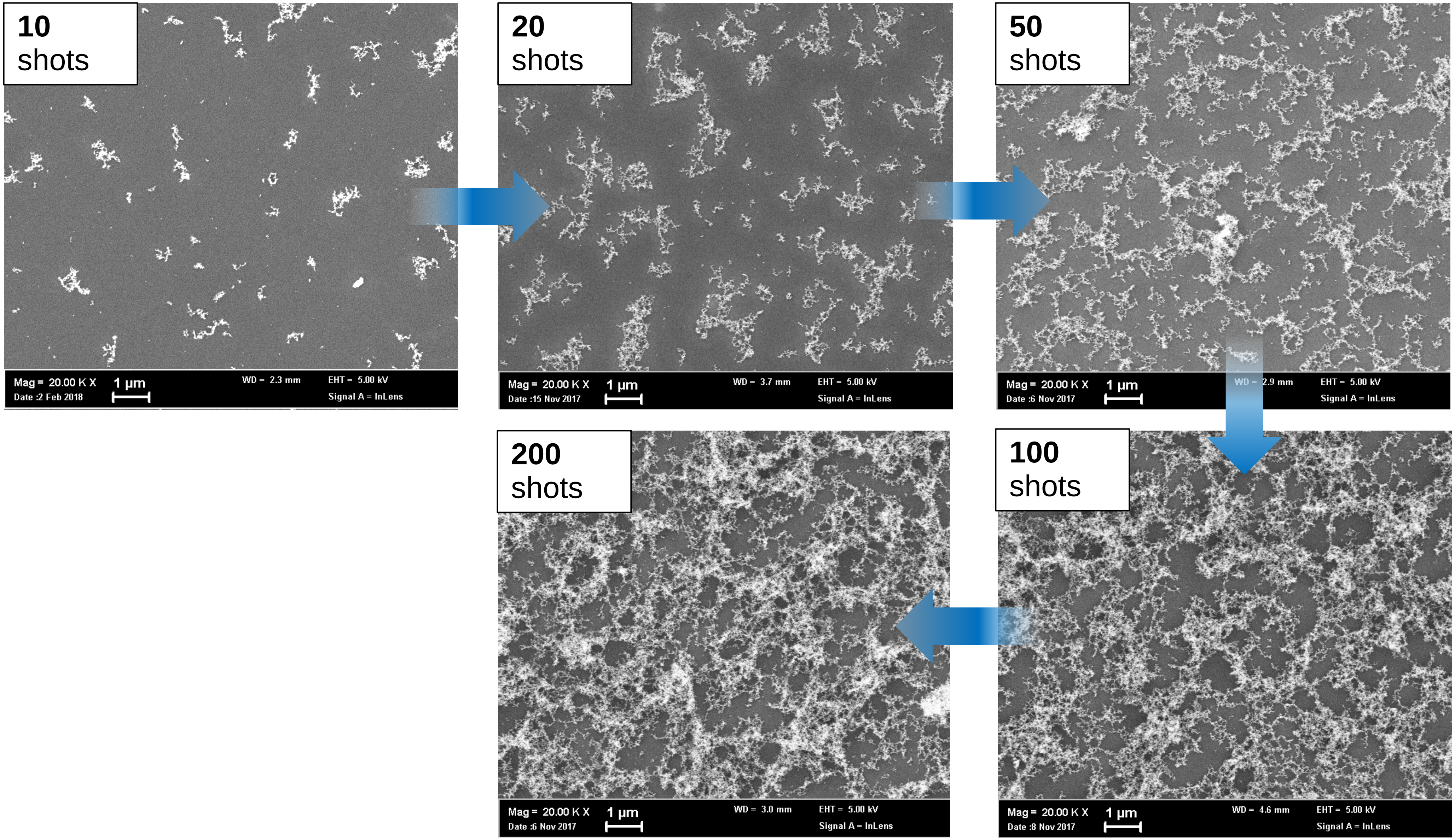

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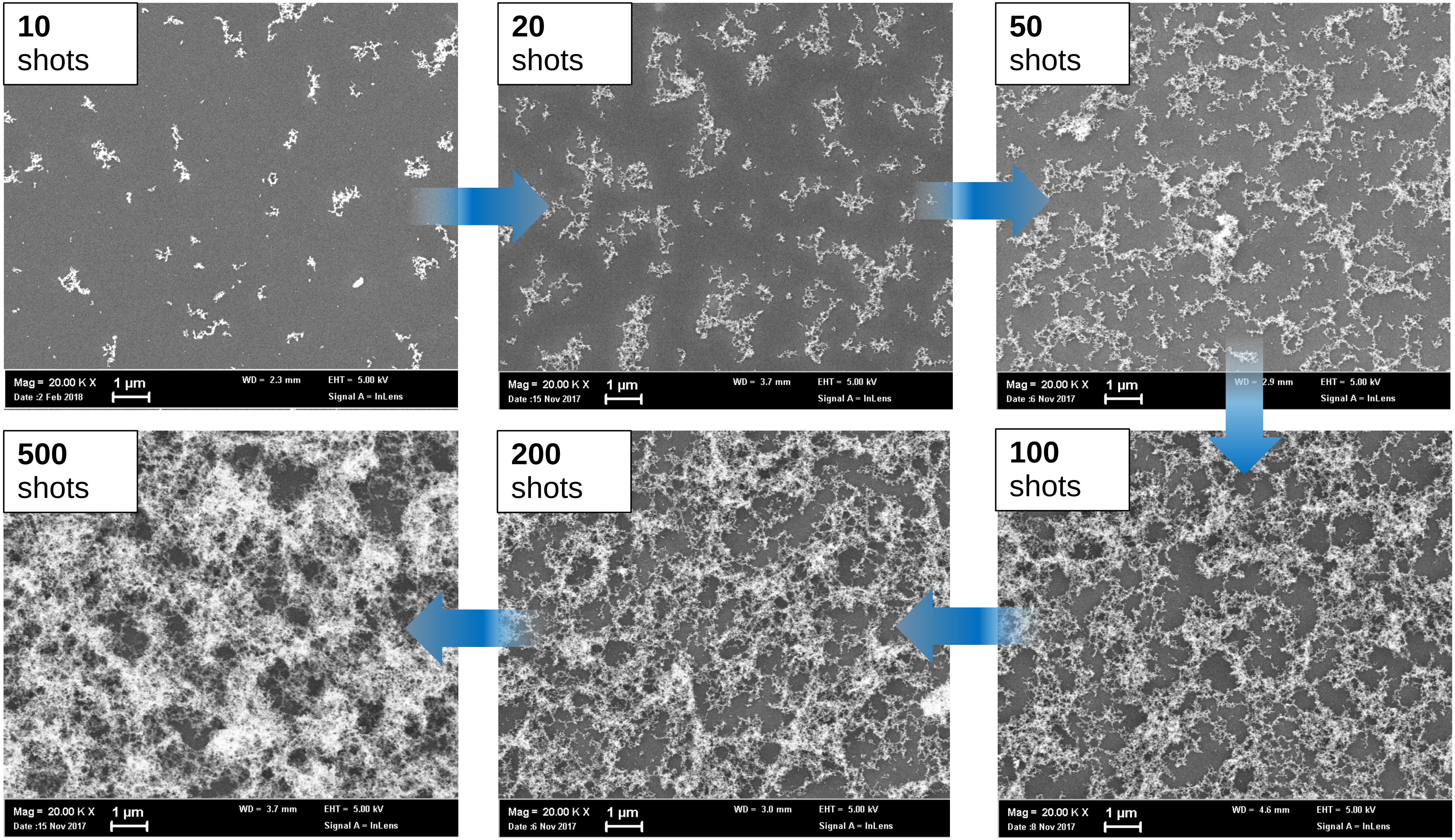

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2D-DLA fails!

Numerical simulation of foam growth

Few-shot experiments:

We have ruled out 2D diffusion limited aggregation

We have collected a valuable set of experimental data…

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Models:

- ➢ **The simplest: 3D DLA**
  - ☺ Computationally light, well know
  - ☹ One NP at the time → not ok for in-flight aggregation
- ➢ **Full-Physics Diffusion Limited Cluster Cluster Aggregation**
  - ☺ Keeps track each aggregate and reproduce the real dynamics
  - ☹ Computational cost explodes with N and box dimension
- ➢ **Simplified Diffusion Limited Cluster Cluster Aggregation**
  - ☹ Unable to describe the real dynamics
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Simplified Diffusion Limited Cluster-Cluster Aggregation

To reduce computational cost, aggregates are synthetized in a sub-box

1) **Nanoparticles**
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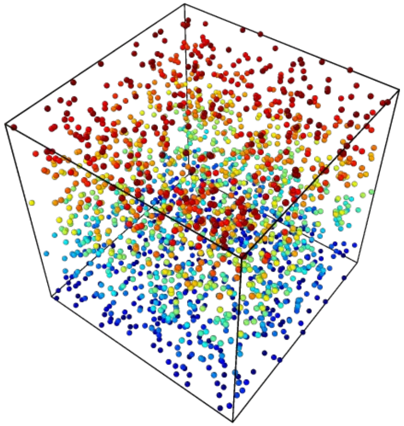

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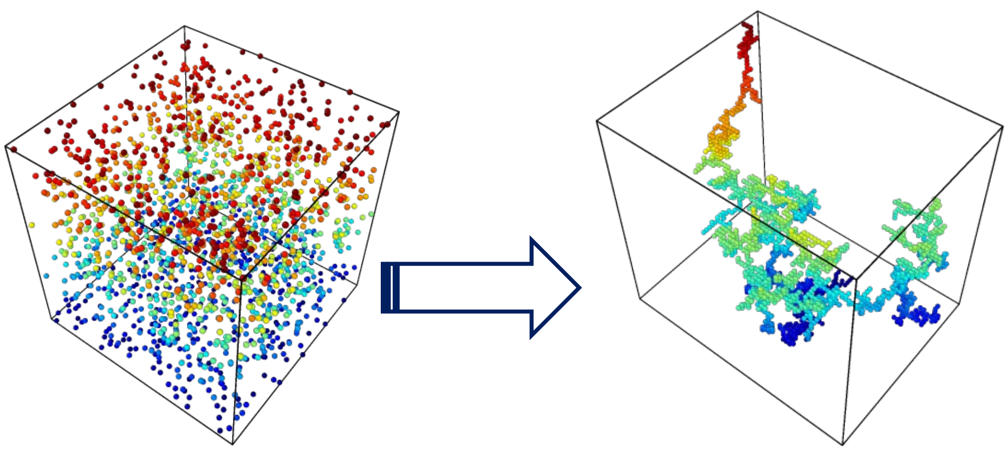
Aggregate size distribution is given as an input

→in this sense, it is **not predictive**

1) **Nanoparticles** in Brownian motion

3) **Formation of aggregates** (10-1000 NPs)



2) Irreversible sticking of NPs

Simplified Diffusion Limited Cluster-Cluster Aggregation

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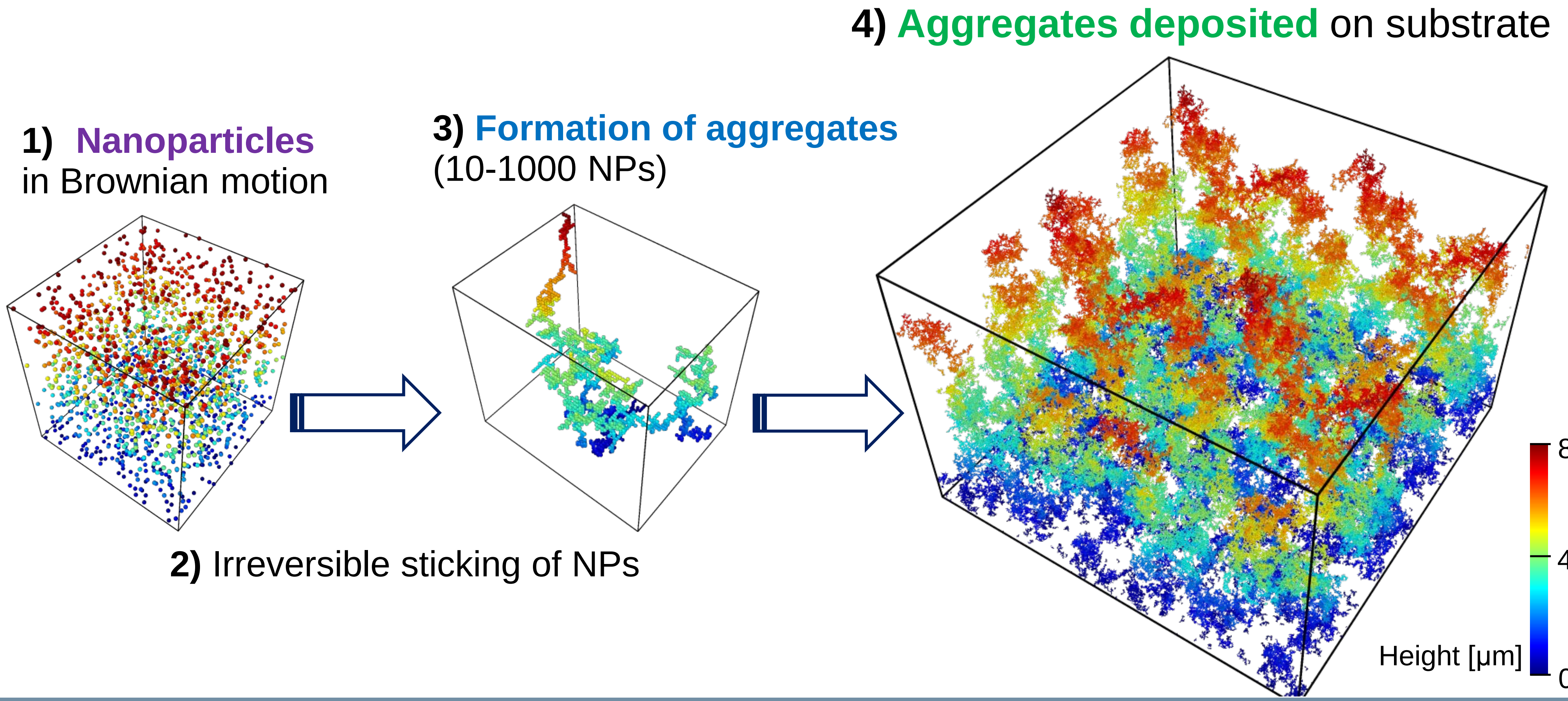
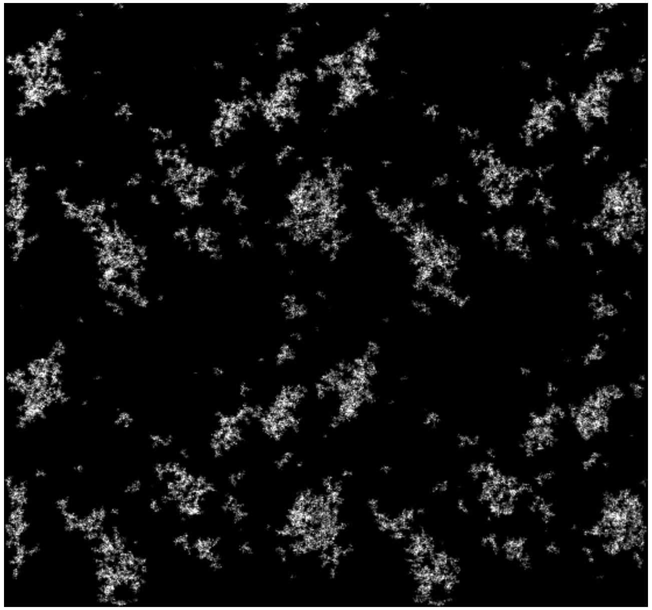
Aggregates are deposited one by one

→ **no information** about the aggregation **time scale**



Simulated growth by simplified-DLCCA



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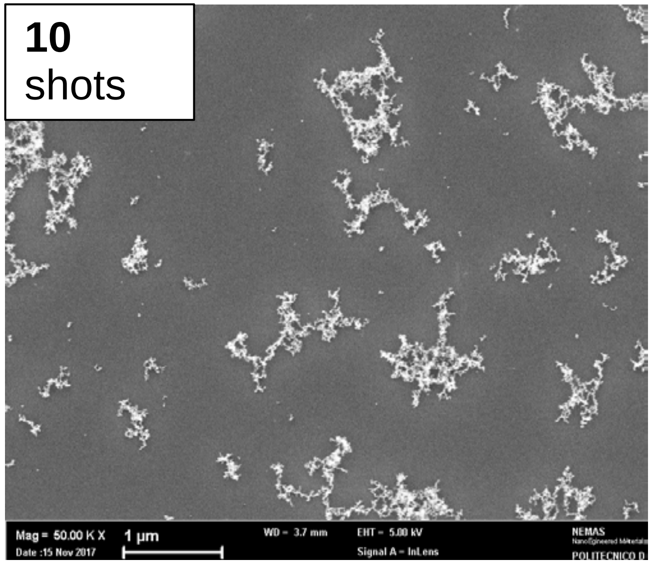

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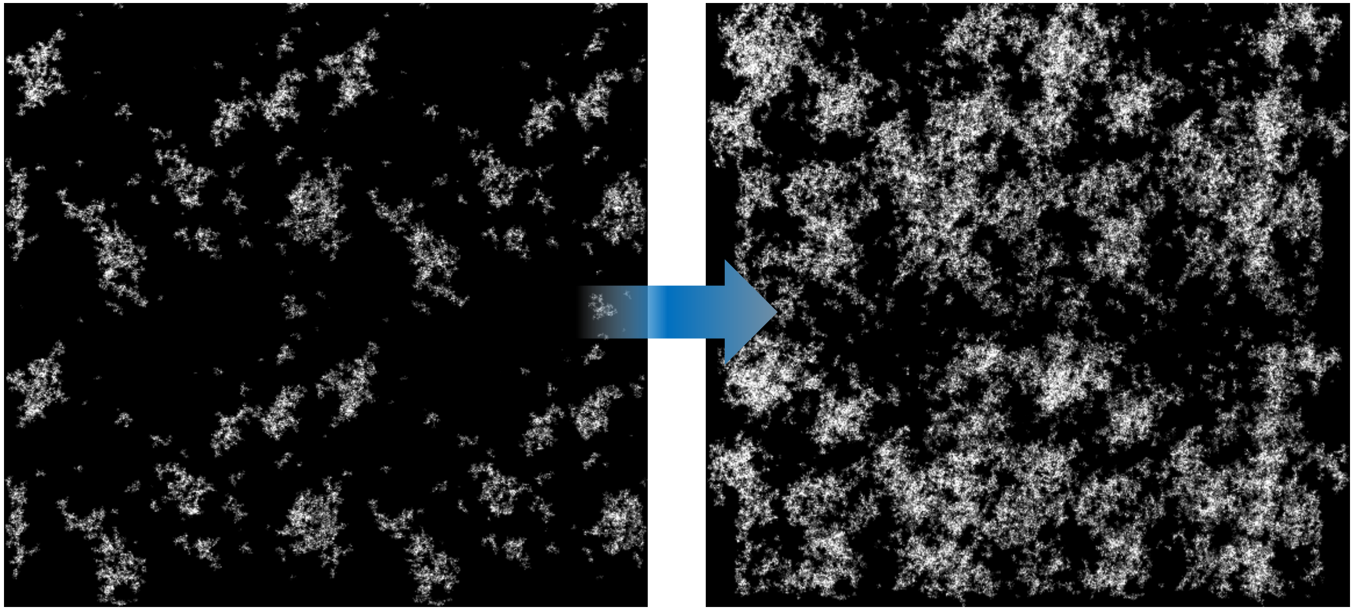

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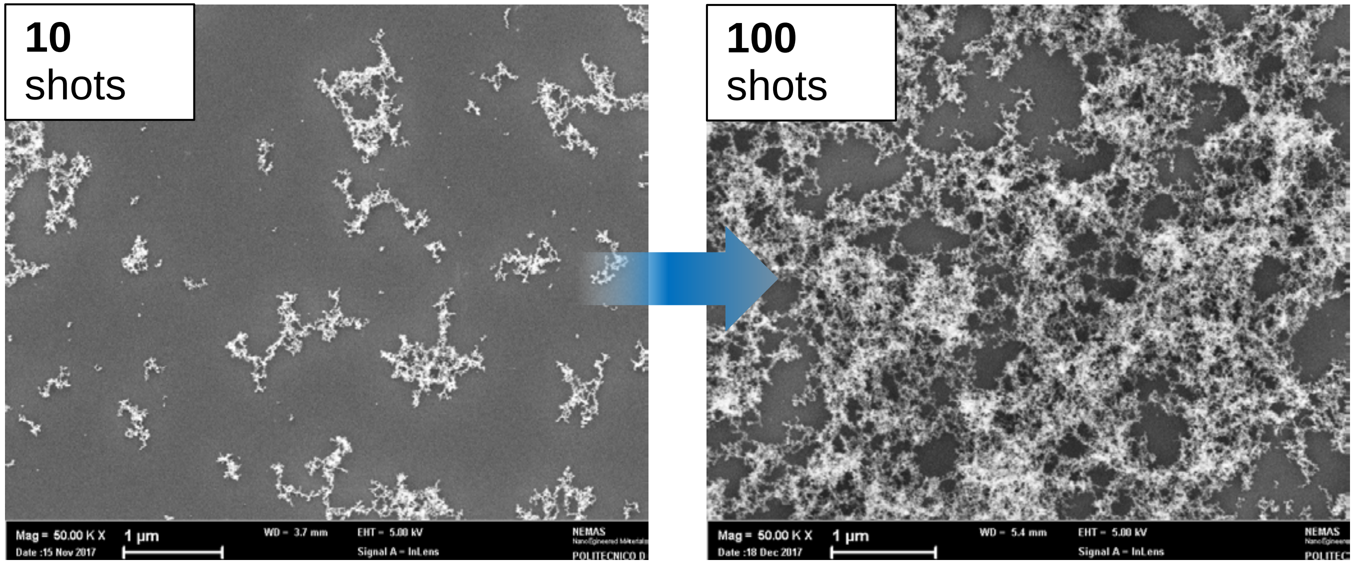

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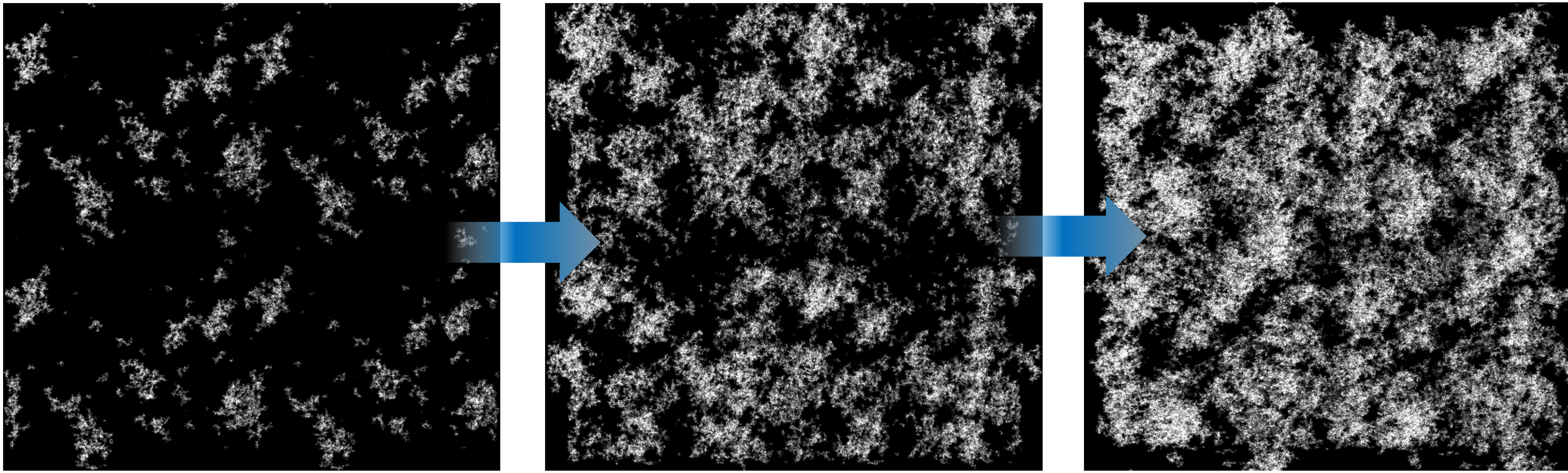

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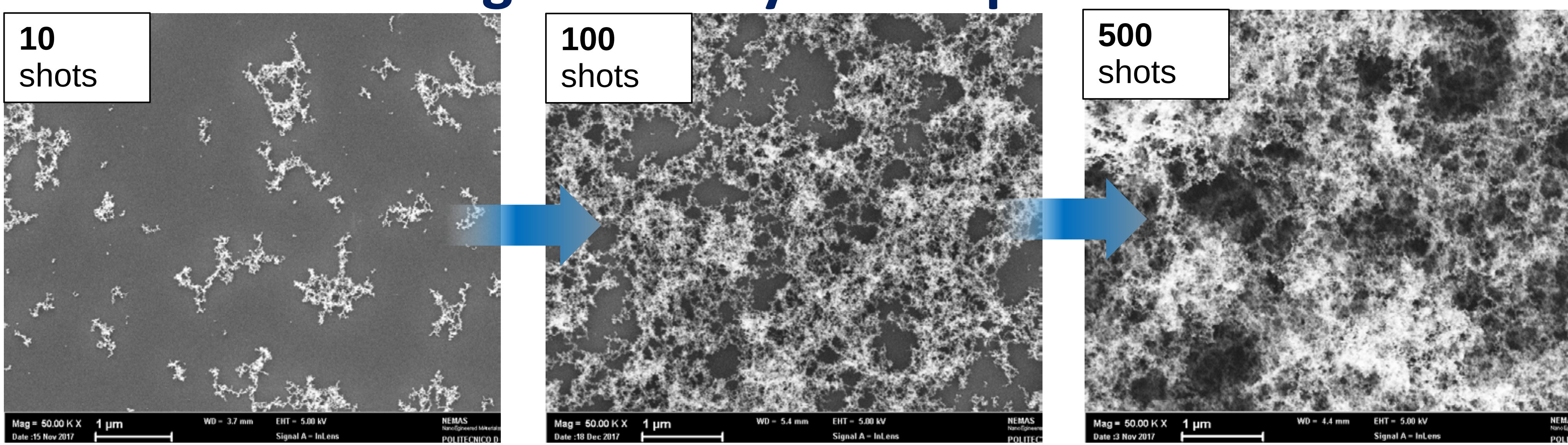
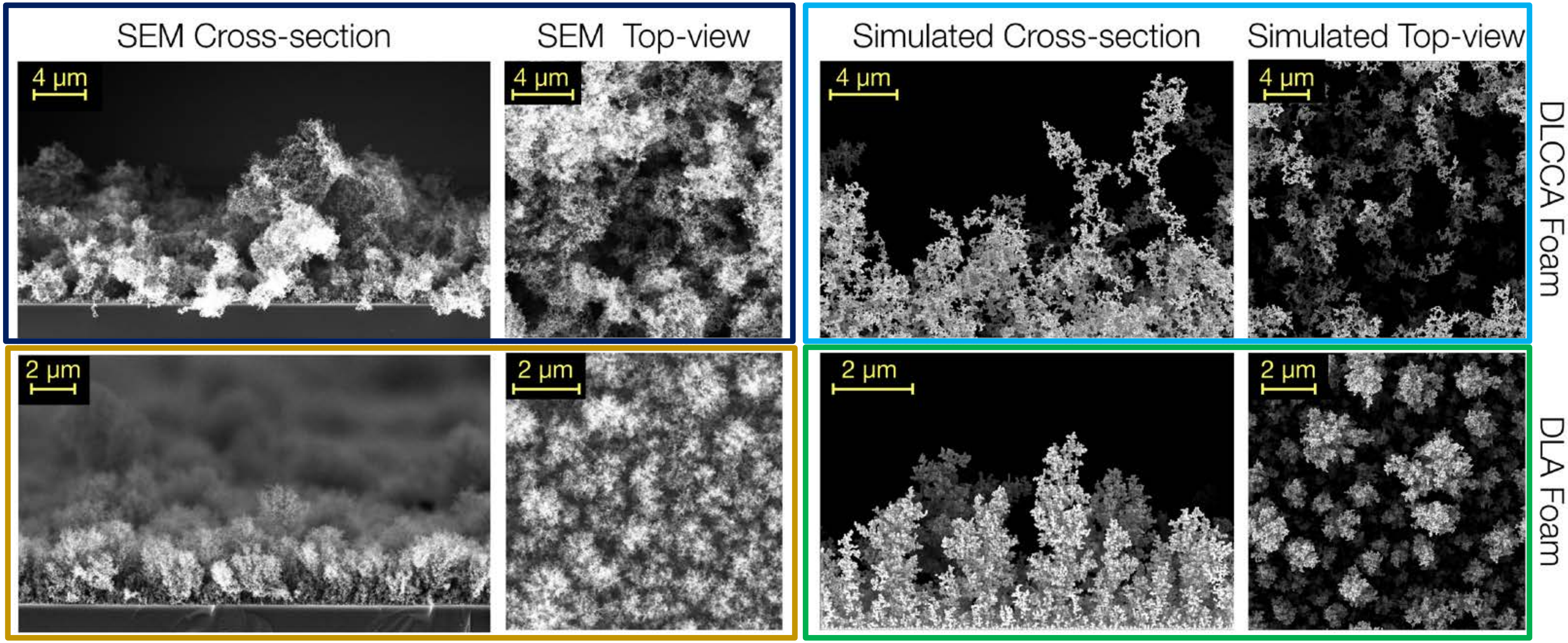


DLA vs DLCCA vs Reality

C foams

DLCCA simulation

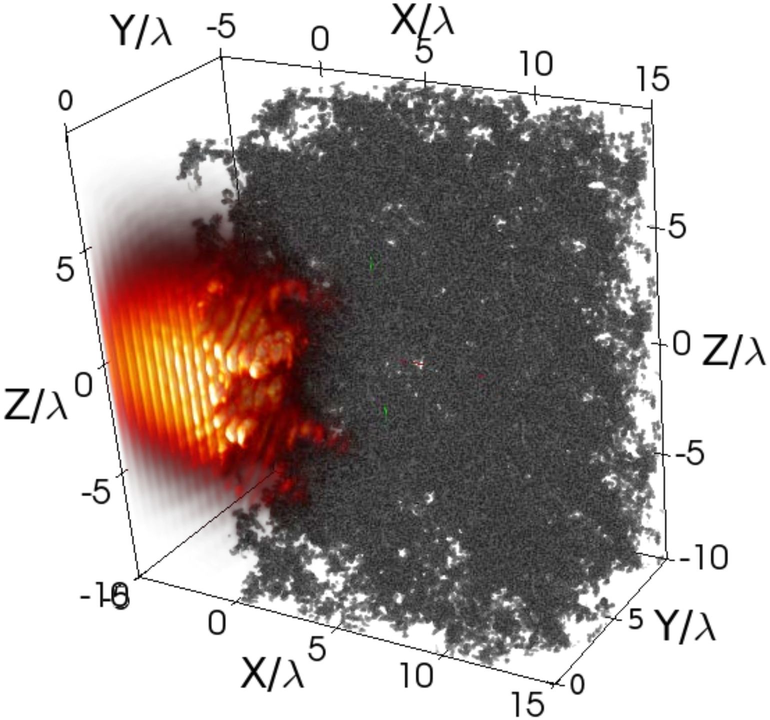


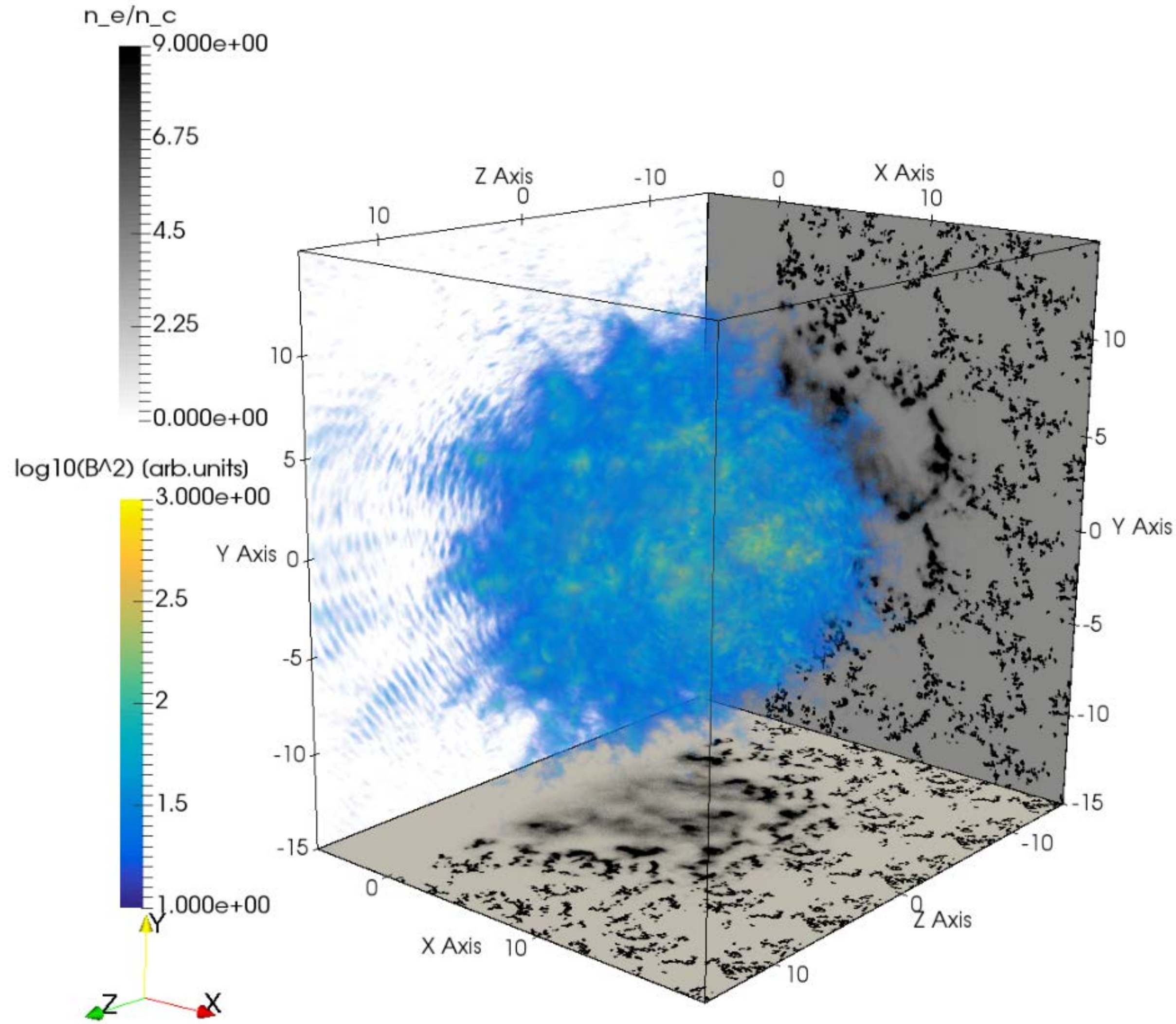
C nanotrees

3D-DLA simulation

L. Fedeli et al., *Sci. Rep*. (2018) 8:3834

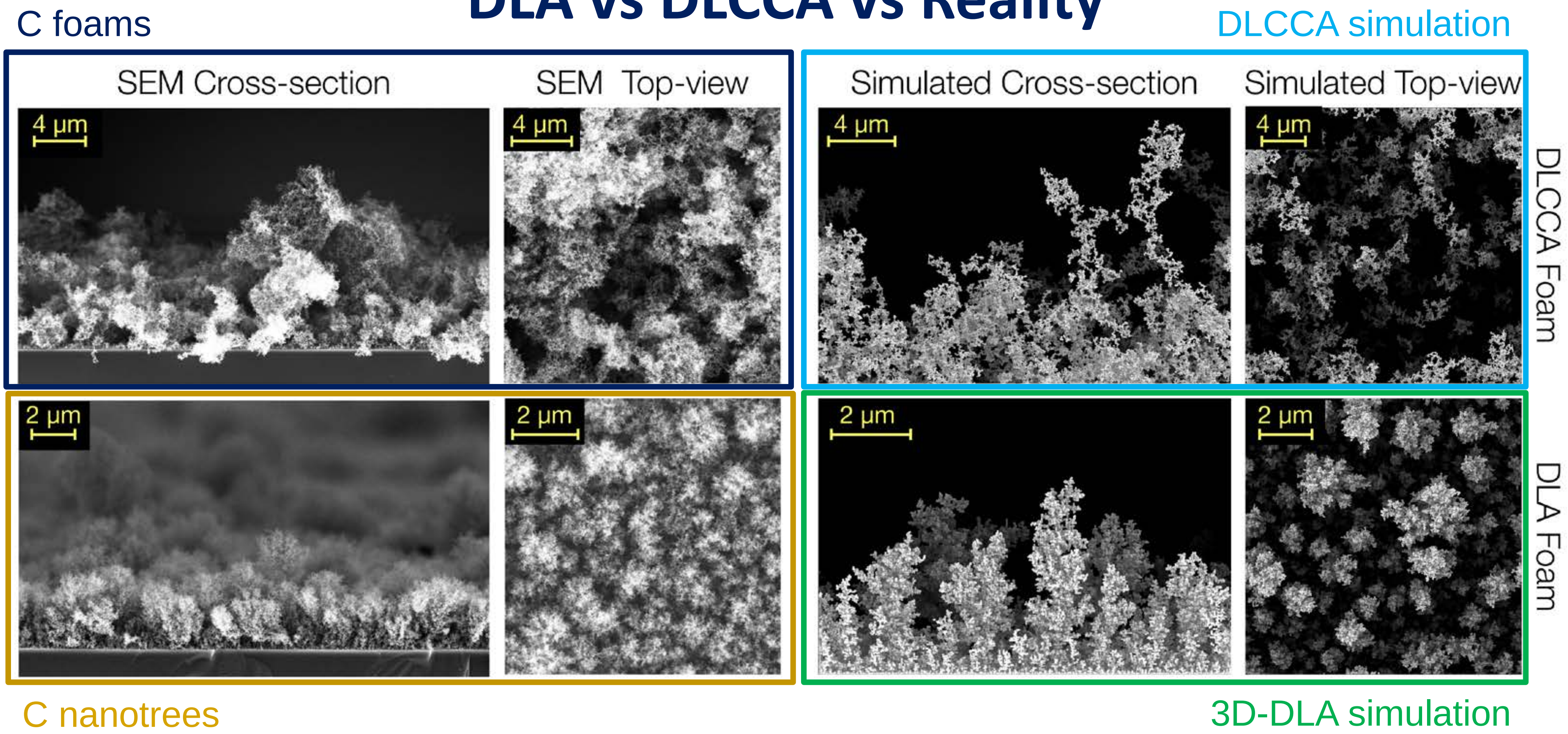
“numerical” foams are used in Laser-Plasma simulation





L. Fedeli et al., *Sci. Rep.* (2018) 8:3834
Arianna Formenti’s NanolabTak (23/04/18)

DLA vs DLCCA vs Reality



✓ Simplified-DLCCA reproduces foam morphology → the physics behind foam aggregation
But:
1) It is not predictive
2) Doesn't describe the dynamics (e.g. the aggregation timescale)

L. Fedeli et al., *Sci. Rep.* (2018) 8:3834

Let’s recap….

What we have learned so far:

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1st step: find a relation between 2R and t_{aggr}
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3rd step: test the hypothesis in PLD experiments:
can we predict 2R (and t_{aggr}) as a function of PLD parameters?

$$2R = f(PLD)$$?

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A scaling law for the aggregate size

Smoluchowski coagulation equation (1916)

$$\frac{\partial N_i(t)}{\partial t} = \frac{1}{2}\sum_{j=1}^{i-1} K_{i-j,j}\; N_{i-j}(t) N_j(t) - \sum_{j=1}^{\infty} K_{ij} N_i(t) N_j(t)$$

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$$K_{ij} = \sqrt{\frac{\pi k_B T}{2m(i+j)}}\,(2R_i + 2R_j)^2$$

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Fractal scaling:

$$R_i = R_{NP} \times (i)^{\frac{1}{d}}$$

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$$2R(t_{aggr}) = a\,(t_{aggr})^b$$

2R depends on t_{aggr} with **a power law b<1**; typically b ≈ 0.5 for DLCCA

Let's recap….

What we have learned so far:

1) Few-shots experiments: "in-flight" aggregation
2) Simplified-physics simulation: it's a DLCCA process

What is still missing:

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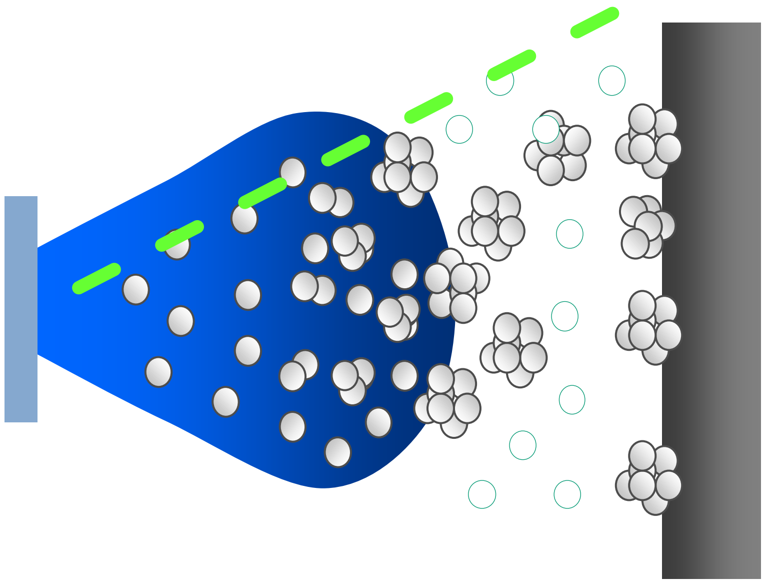
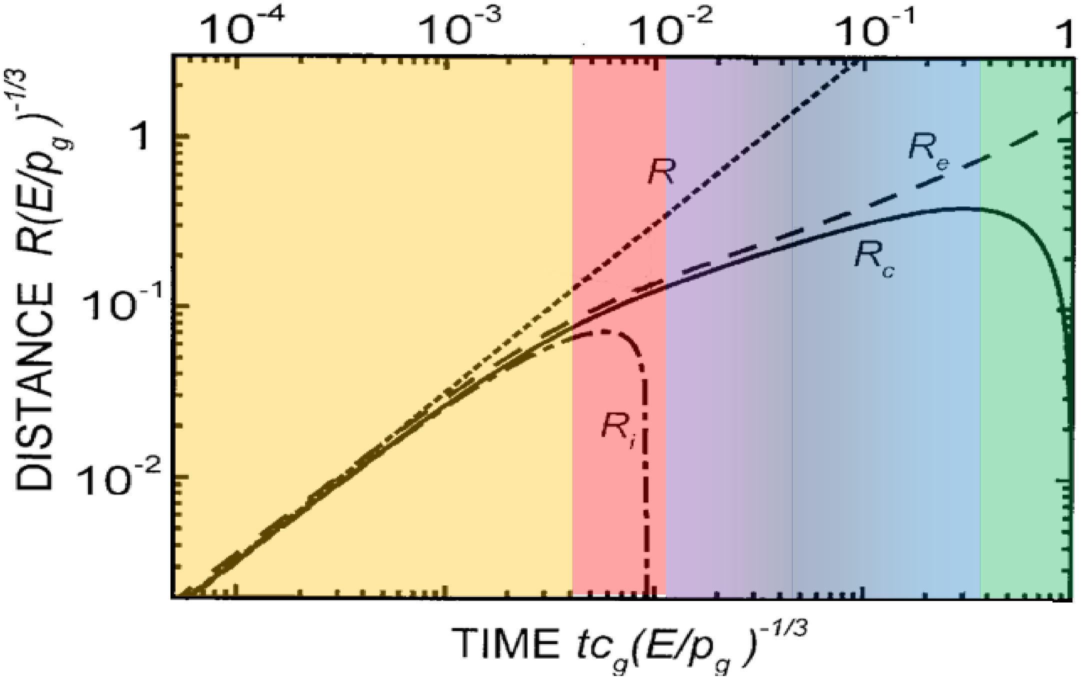
POLITECNICO MILANO 1863

Let's come back to plume dynamics…..



PLD plume dynamics in background gas is still an open research topic!



Adapted from: Arnolds et al., *Appl. Phys. A* 69 S87–S93 (1999)

A sketch of plume dynamics:

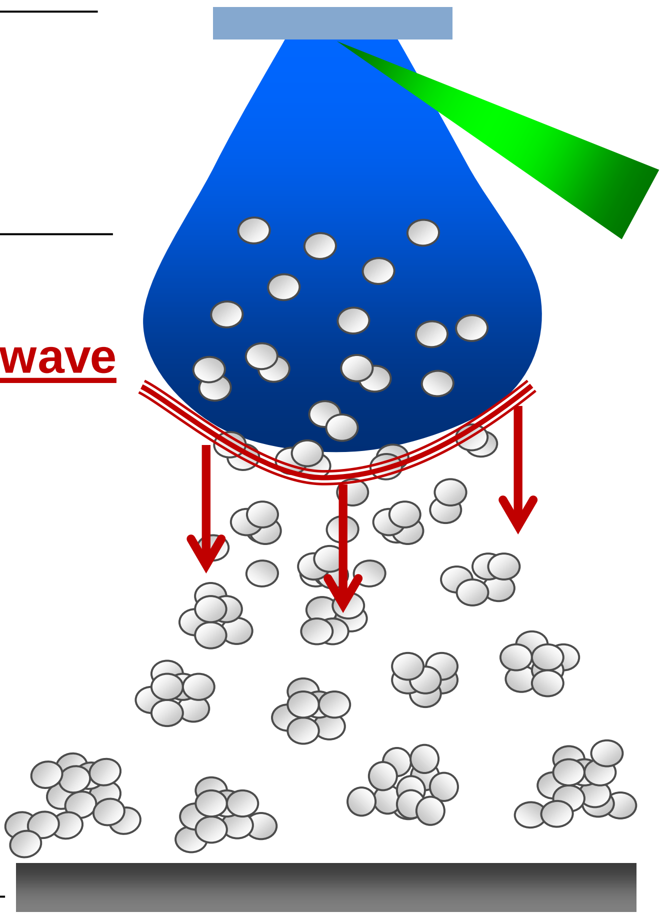
1) Adiabatic Expansion
2) Shock wave formation
3) Nanoparticle synthesis
4) Nanoparticle aggregation
5) Landing on substrate

A model (I) to find the aggregation time

t=0 — n^{th} **laser shot** on target

Adiabatic expansion

t ≈0 — **NPs generation**



t= time of flight — **Aggregate landing**

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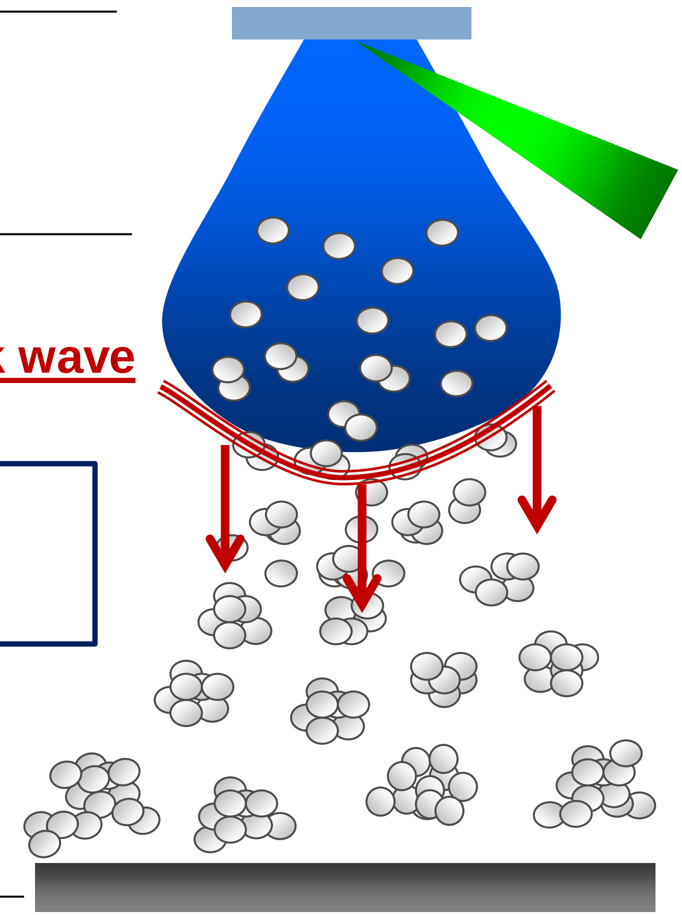

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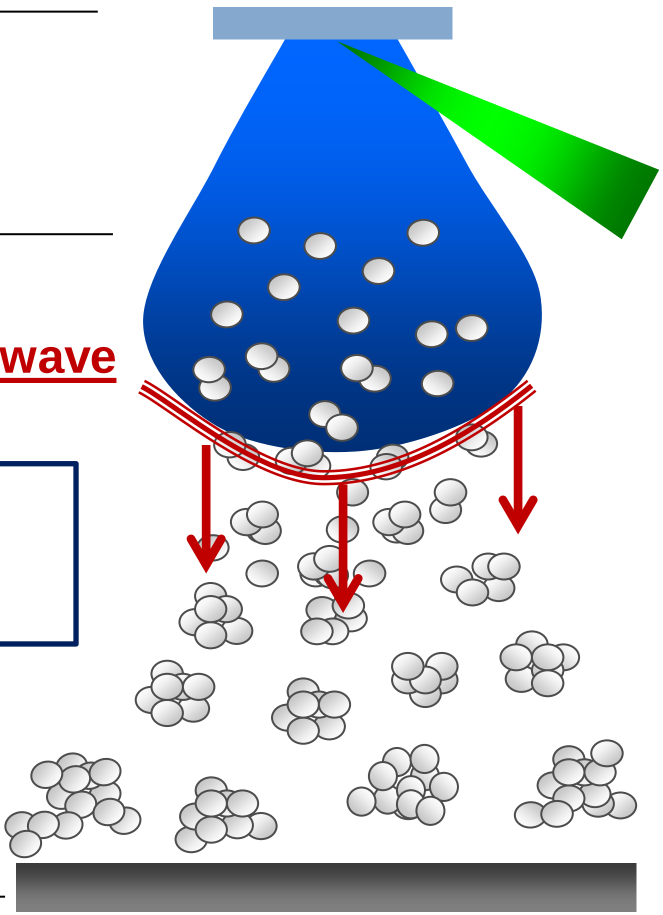

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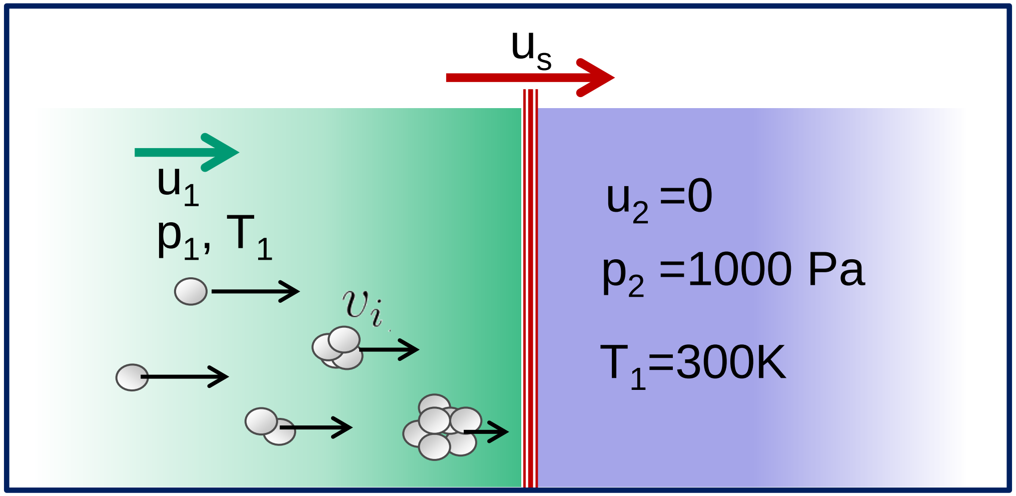
To calculate *t.o.f.* :

Stokes-Einstein equation

$$m_i \frac{\mathrm{d}v_i}{\mathrm{d}t} = 6\pi\eta R_i(u_1 - v_i)$$

+

Rankine –Hugoniot equations



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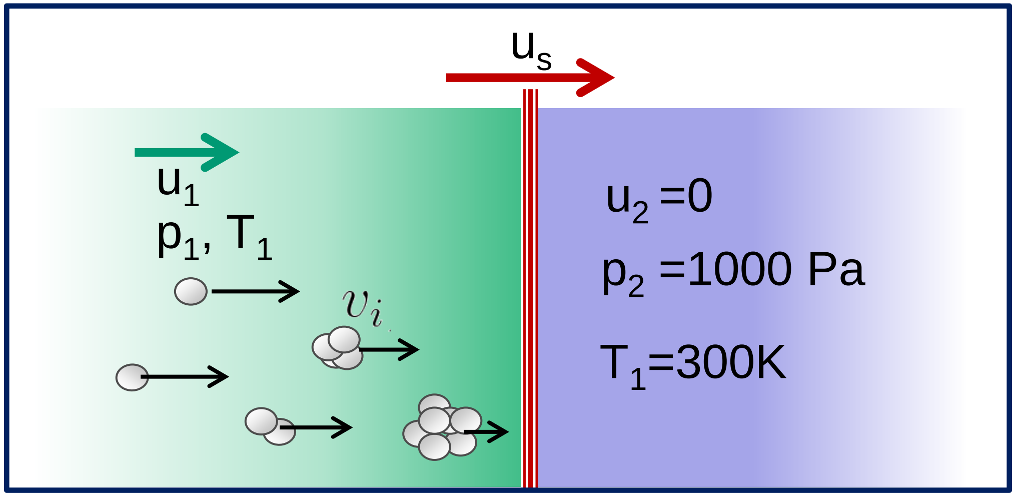

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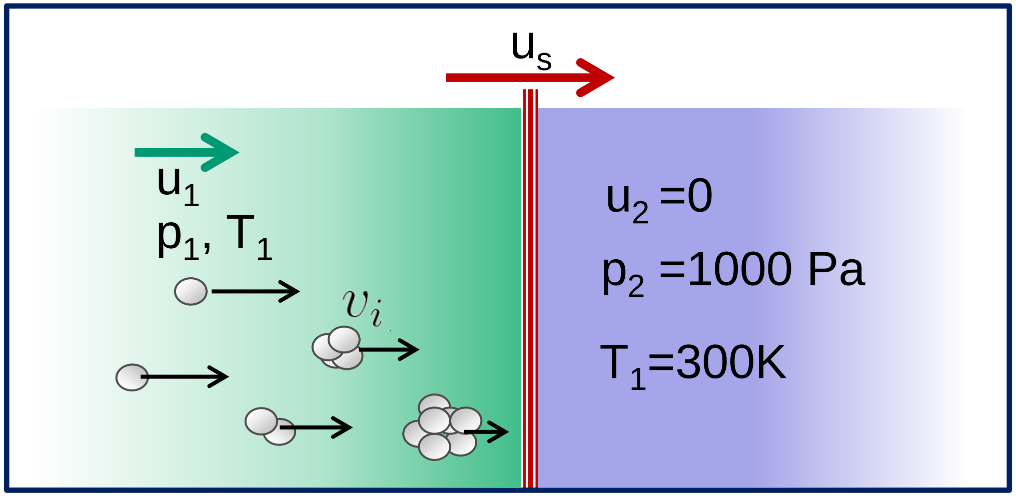

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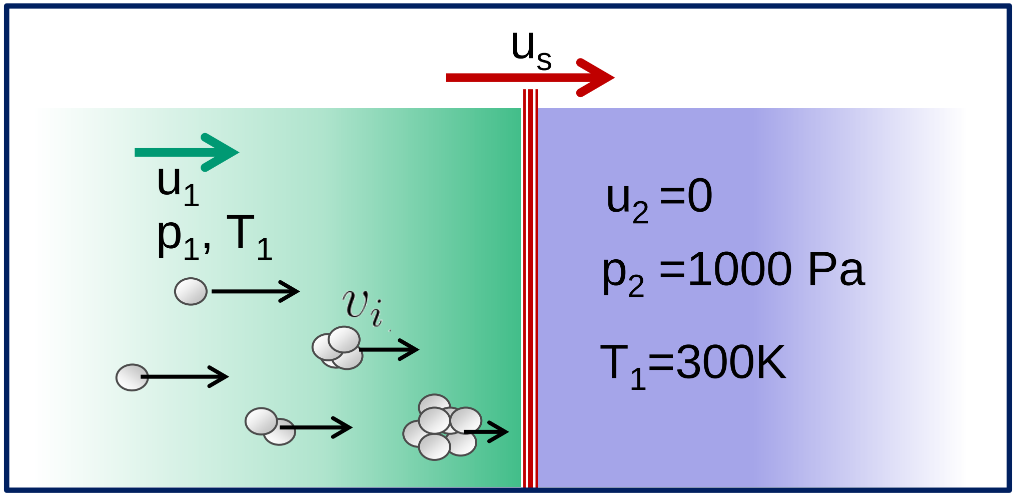

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Can be measured! **Can be controlled!**

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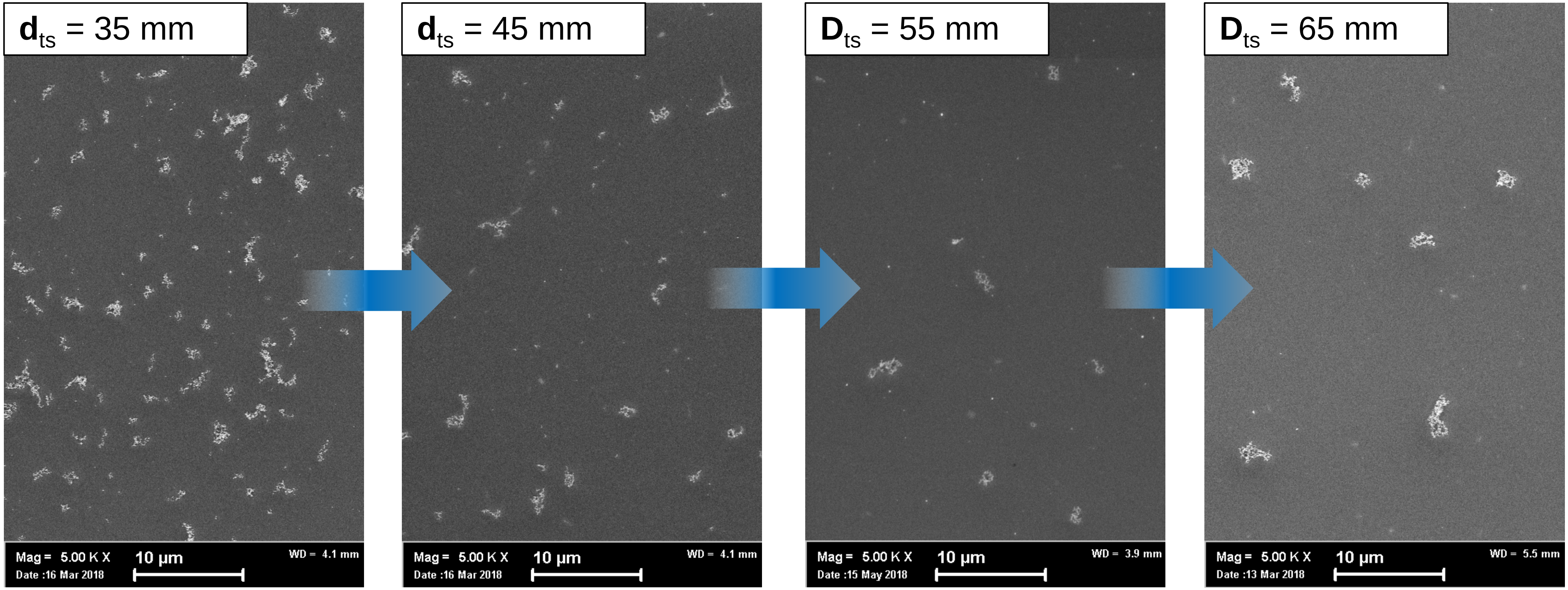

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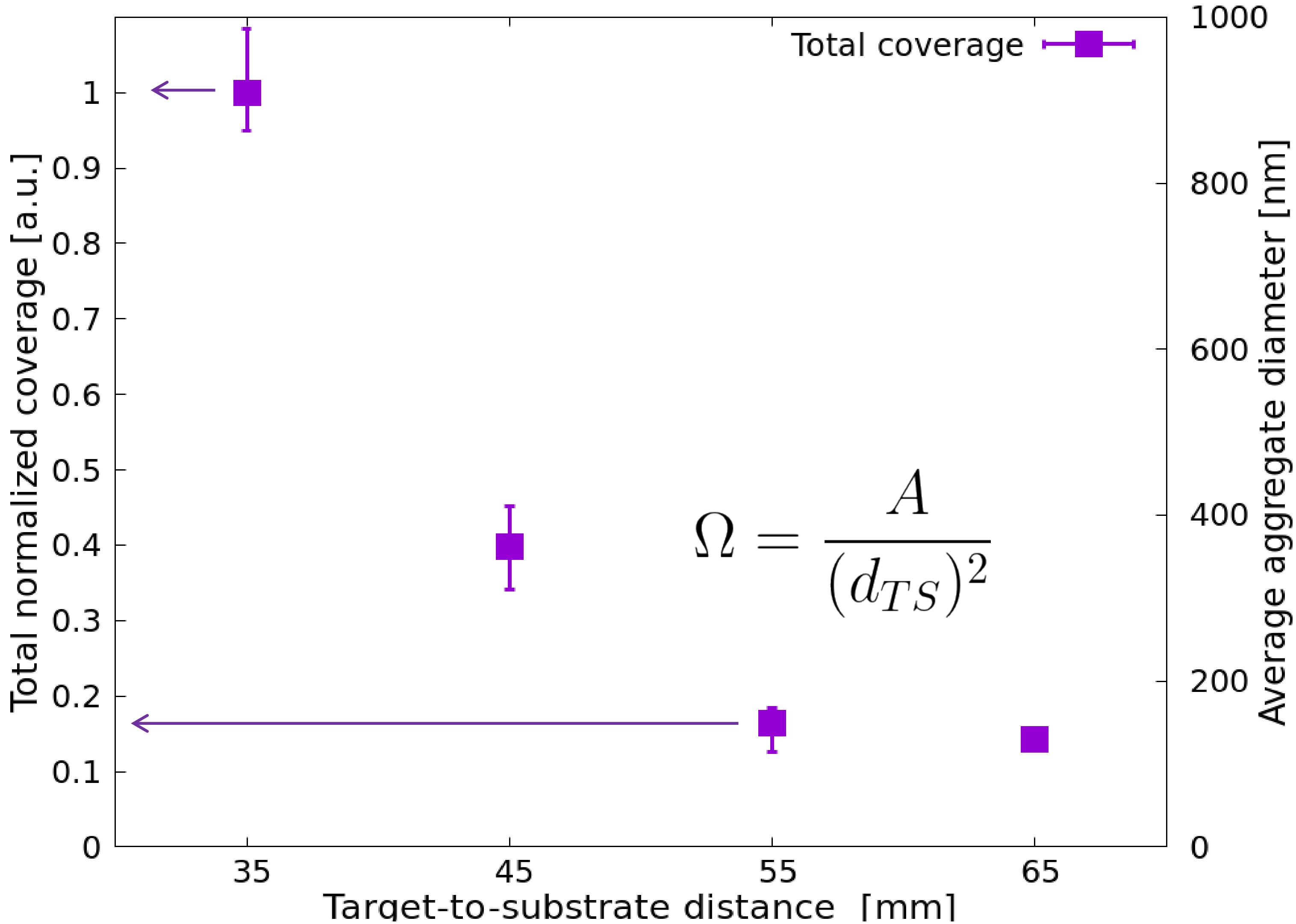

- Less coverage because of solid angle reduction

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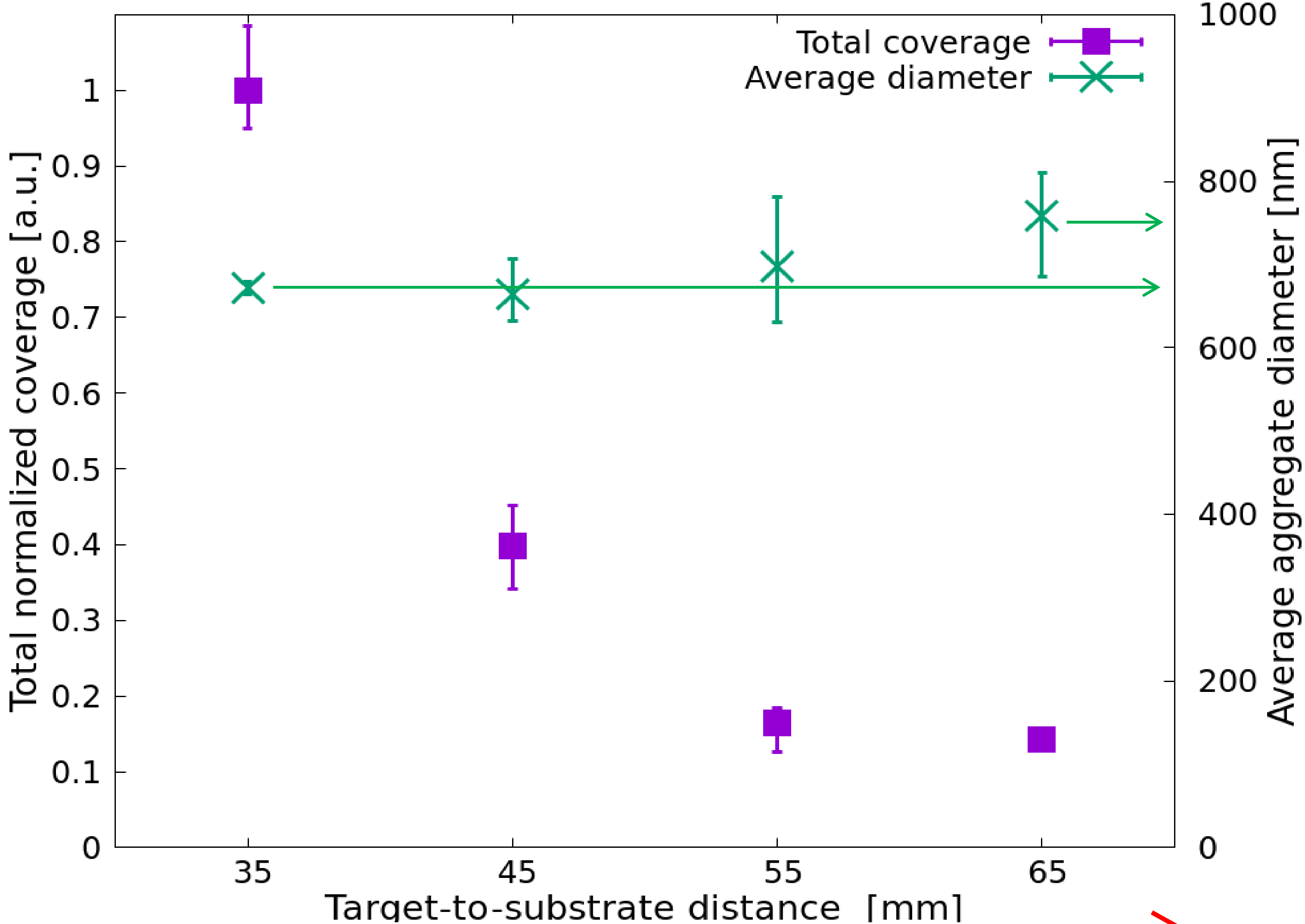

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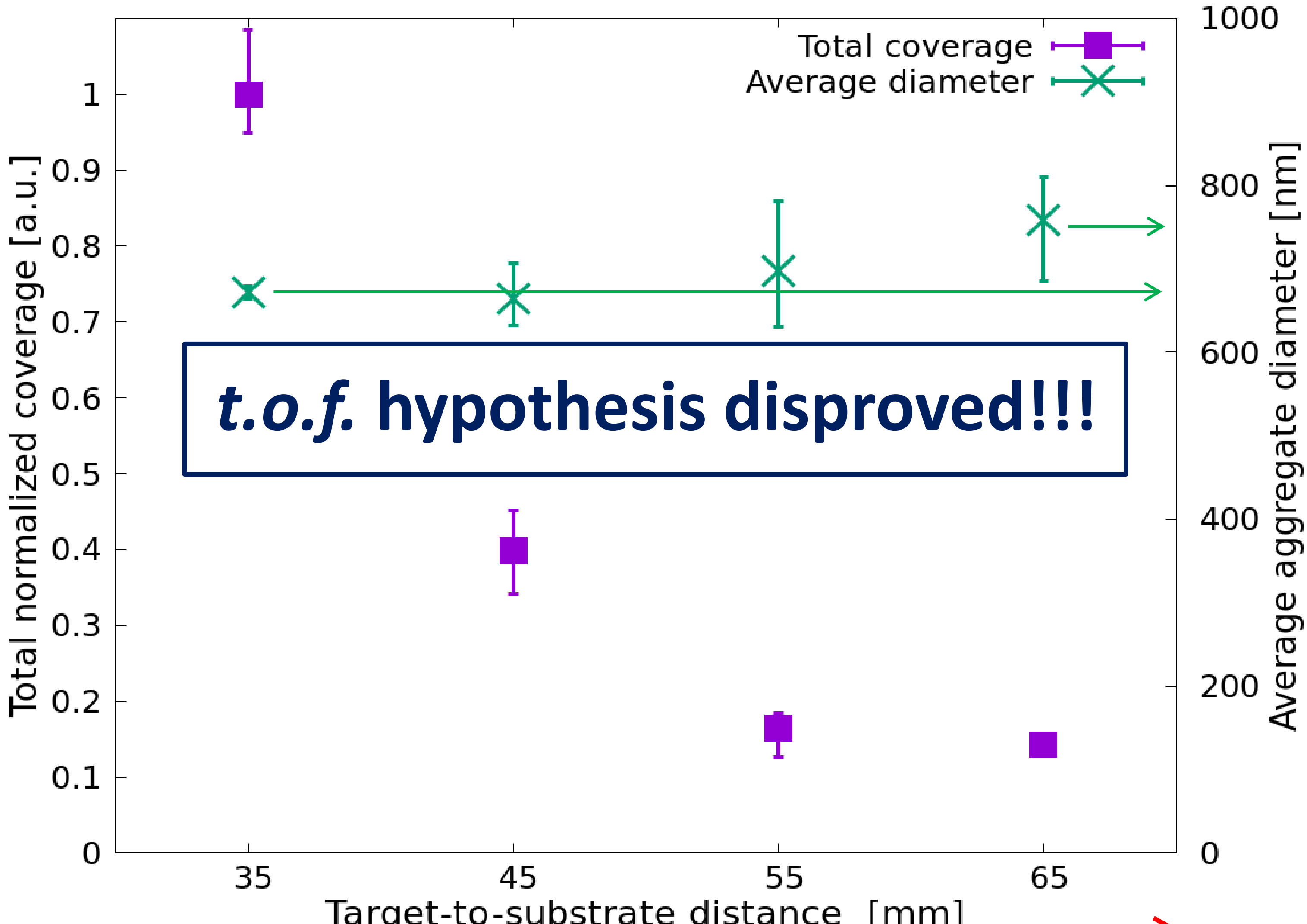

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A model (II) to find the aggregation time

t=0 — **n^th laser shot** on target

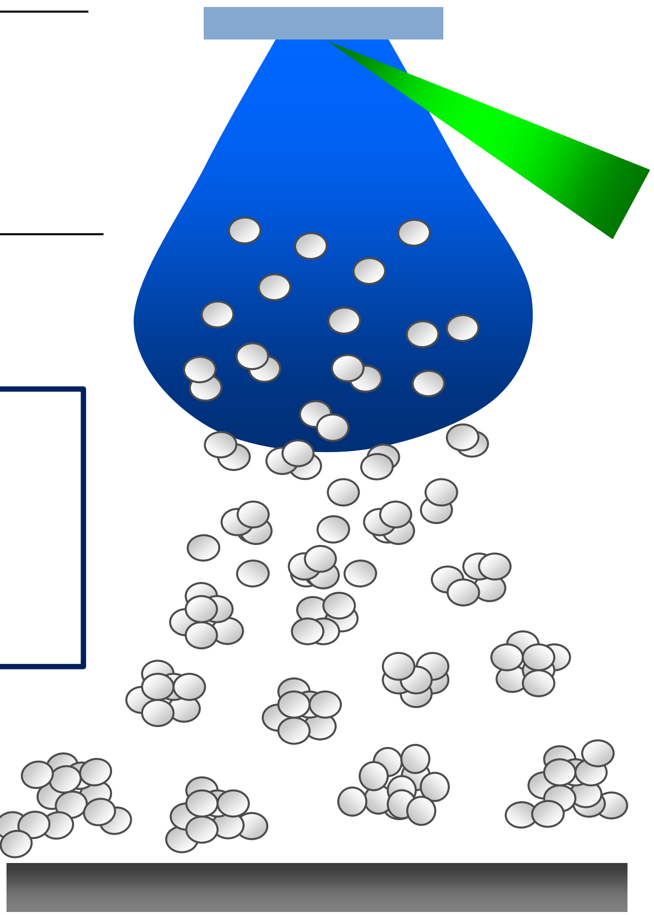
Adiabatic expansion

t ≈0 — NPs generation

Hypotheses (II):

1) n^th SW too quick to drag aggregates

t= $\frac{1}{R.R.}$ — **(n+1)^th laser shot** on target



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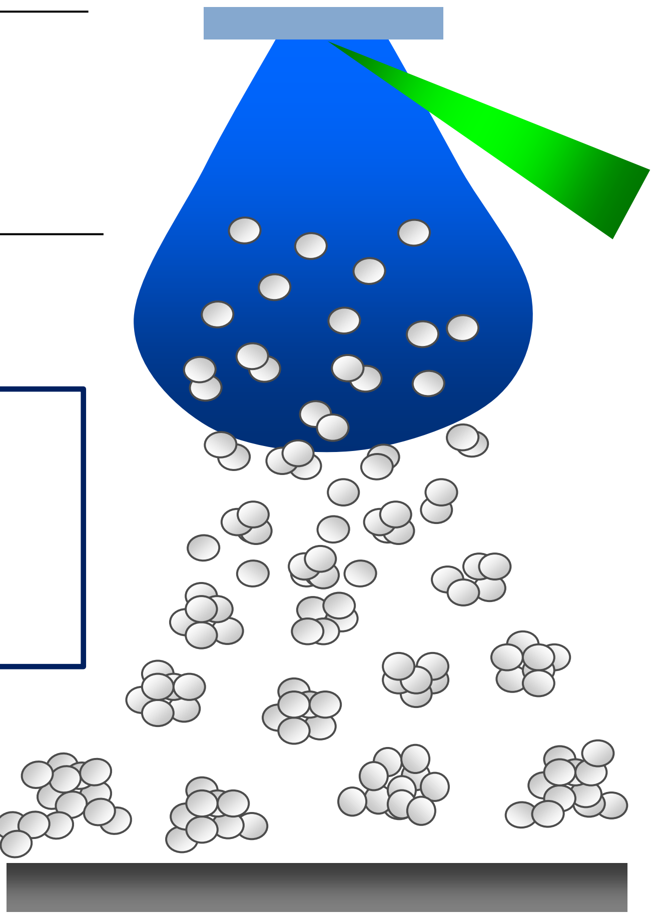
(Adiabatic expansion + NPs generation)

↓↓ **$(n+1)^{th}$ <u>Shock wave</u>** ↓↓



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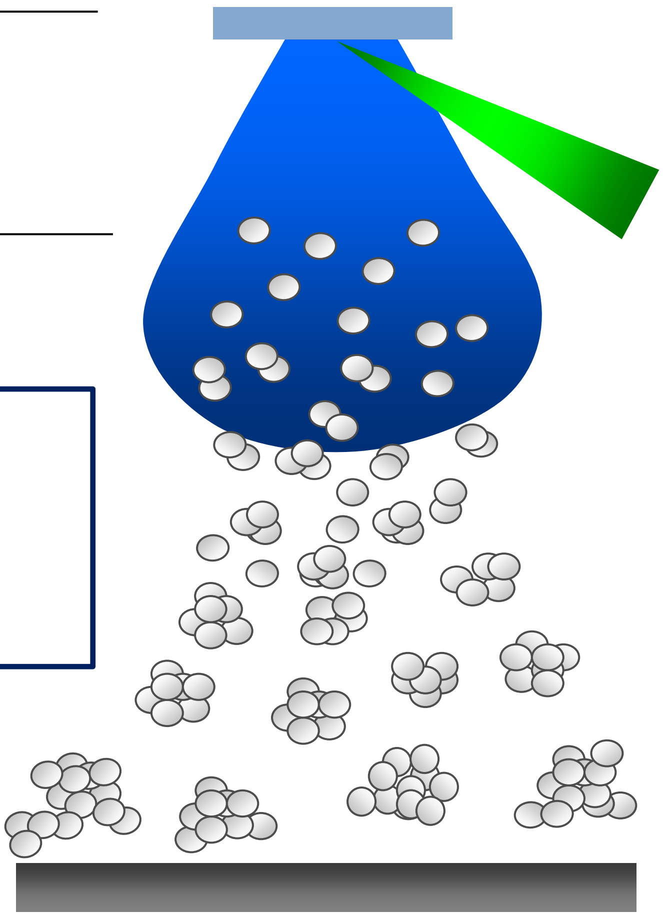
$(n+1)^{th}$ Shock wave

t= $\frac{1}{R.R.} + tof \approx \frac{1}{R.R.}$ — **Aggregate landing**



$$t_{aggr} \approx \frac{1}{R.R.}$$

Rep. Rate hypotheses:

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⇒

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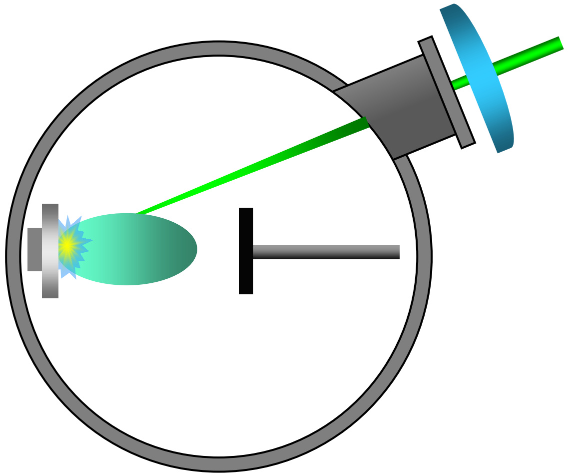
Can be measured! ($2R$)

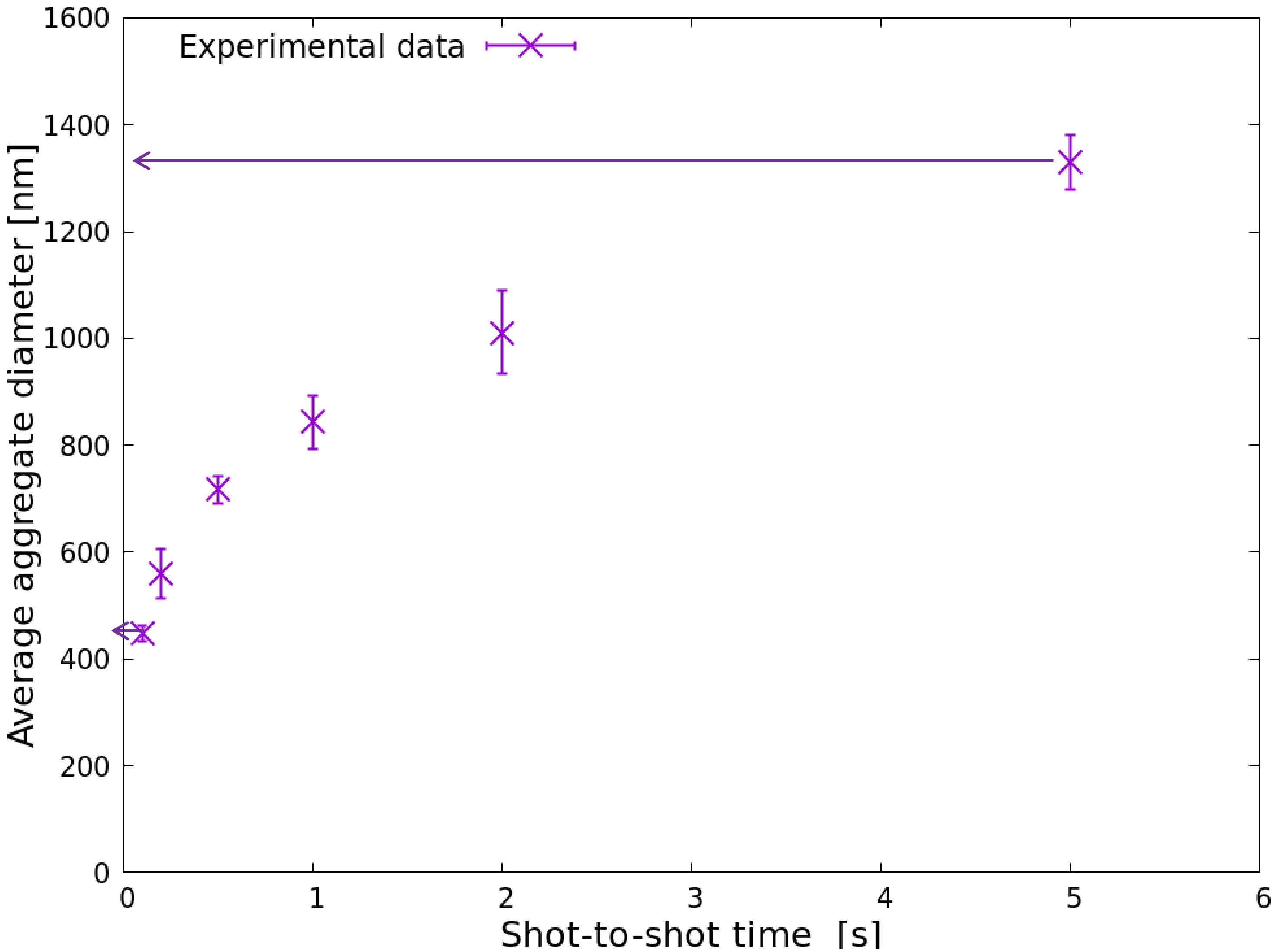
Can be controlled! (t_{sts})



PLD parameters:

10 shots
d_{ts} = 45 mm
Rep. Rate = 10 Hz, 5 Hz, 2 Hz, 1 Hz, 0.5 Hz, 0.2 Hz
Shot-to-shot time= 0.1 s, 0.2 s, 0.5 s, 1 s, 2 s, 5 s

Let’s test the “repetition rate” hypotesis…



- ➢ Average size 2R significantly affected by shot-to-shot time

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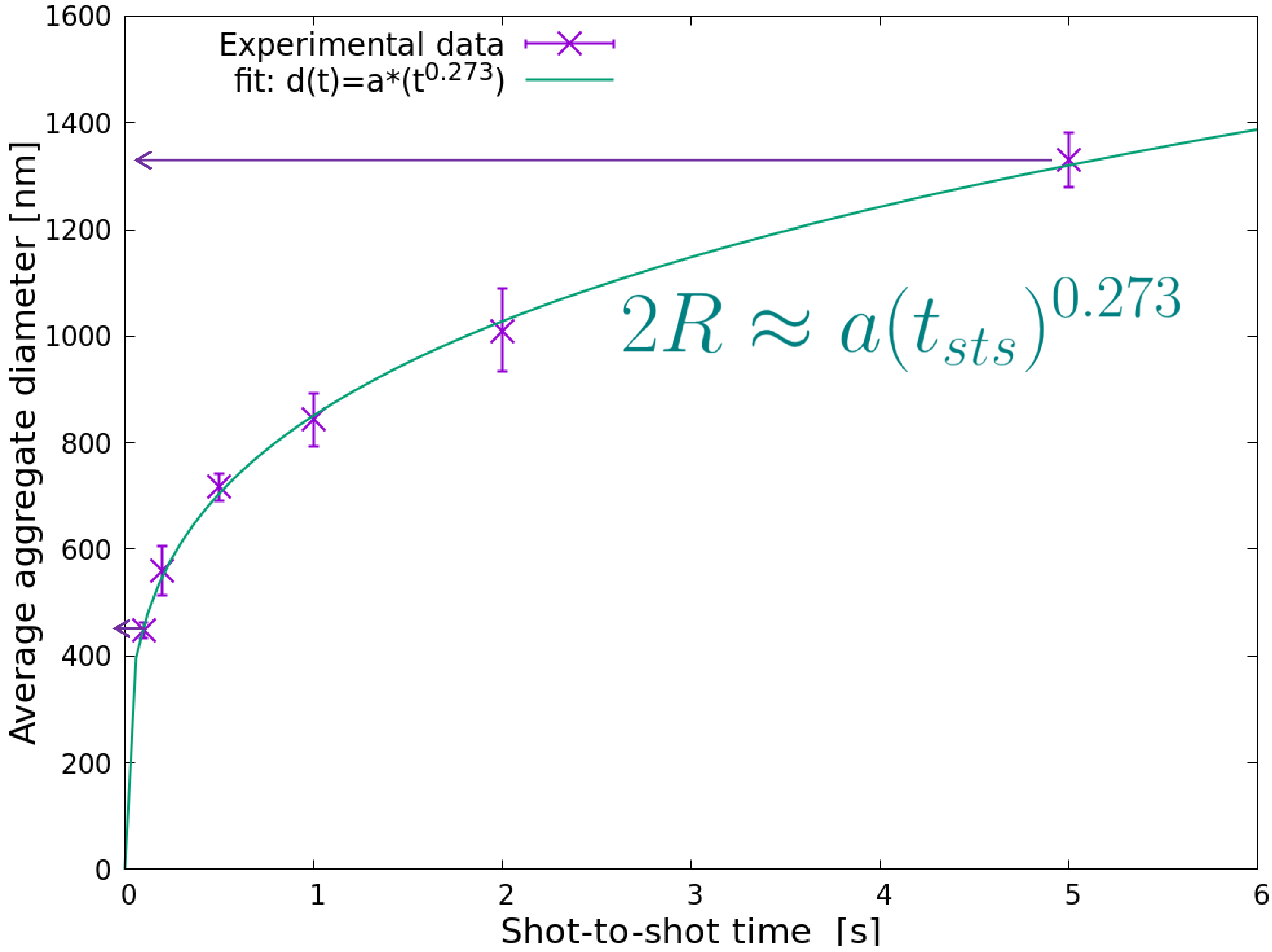

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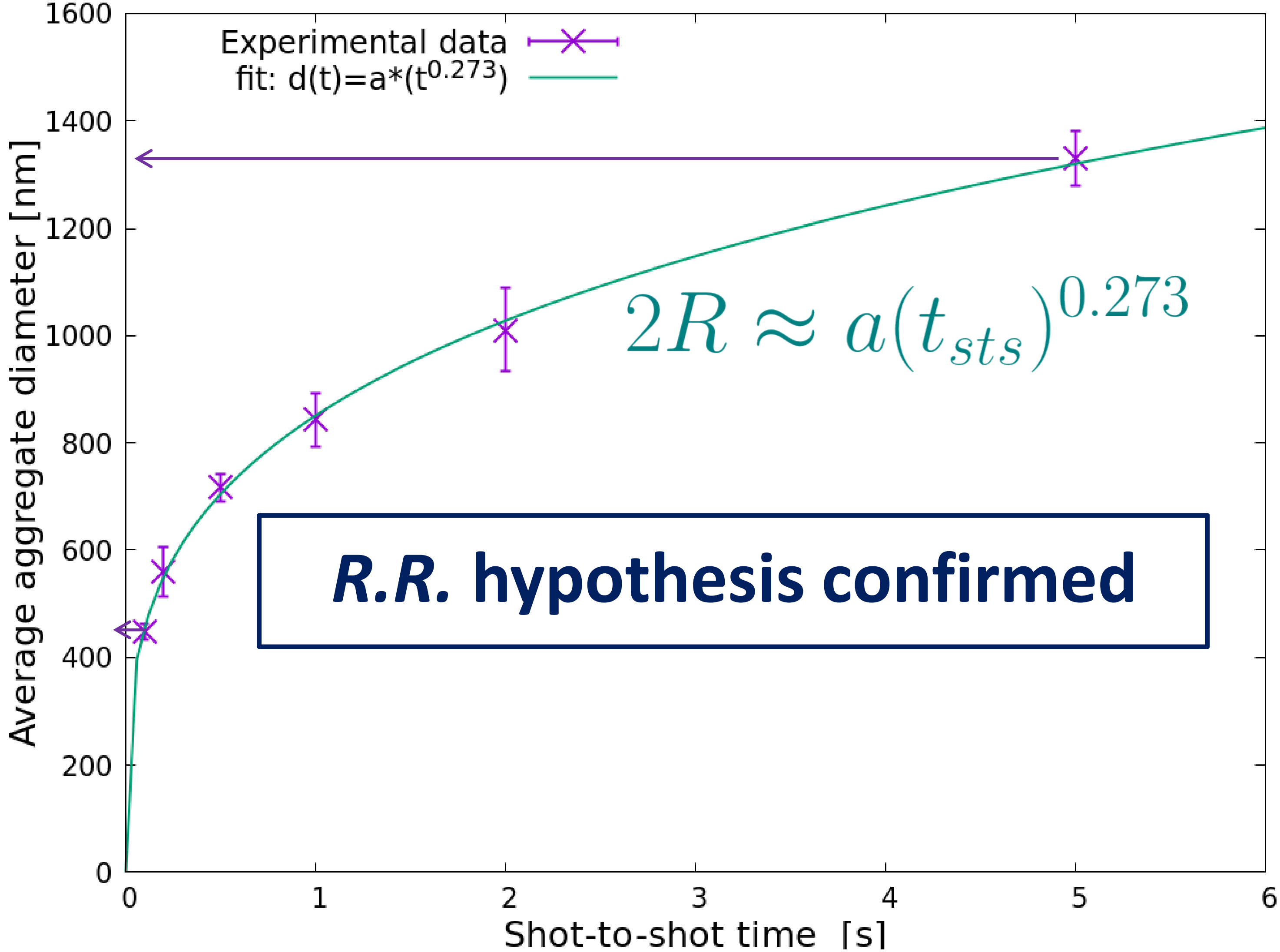

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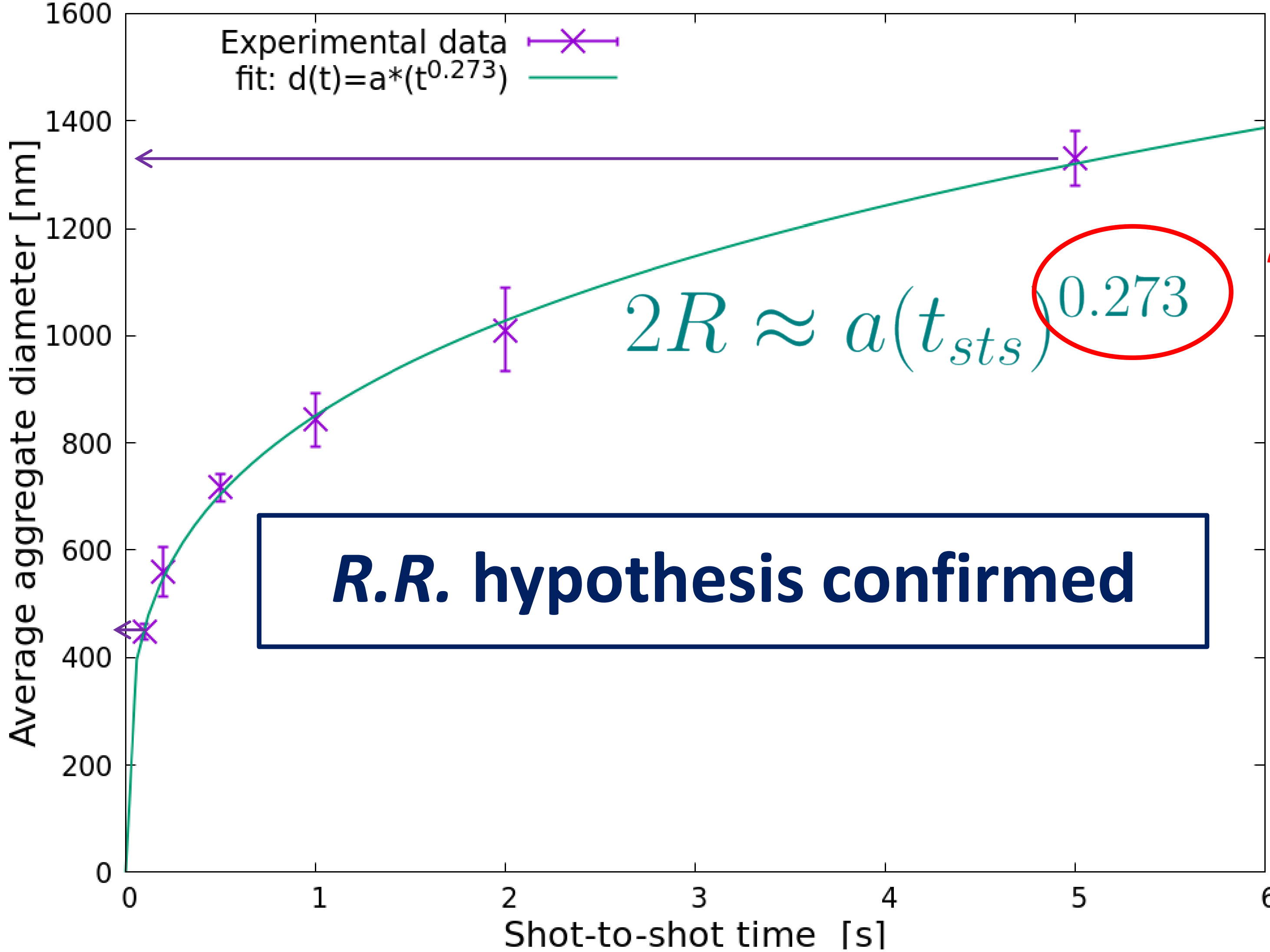

?

expected b ≈ 0.5

R.R. **hypothesis confirmed**

- Average size 2R significantly affected by shot-to-shot time
- **Experimental points nicely fitted by a power law!**

A summary:

We tried to answer to these questions:

How NPs aggregates and produce a foam?

How aggregation dynamics controls foam properties?

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There's still work to do

Why the exponent in 2R scaling law is roughly half than expected?

Does the model work for other materials and deposition conditions?

… even in different PLD regimes?

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ENSURE

... a brand-new fs-PLD is waiting for us!

Acknowledgment

The "ENSURE" team


M. Passoni


V. Russo

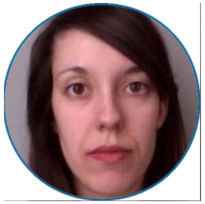

M. Zavelani-Rossi


D. Dellasega


A. Maffini


L. Fedeli


A. Pola


A. Formenti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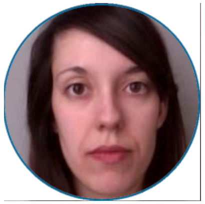
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....Thank you for your attention!

More info on our website

www.ensure.polimi.it