The direct characterization of the precursor colloidal phases in applications for sol-gel and mineral systems

Tomasz M. Stawski
stawski@gfz-potsdam.de

Rogier Besselink

GFZ Helmholtz Centre Potsdam

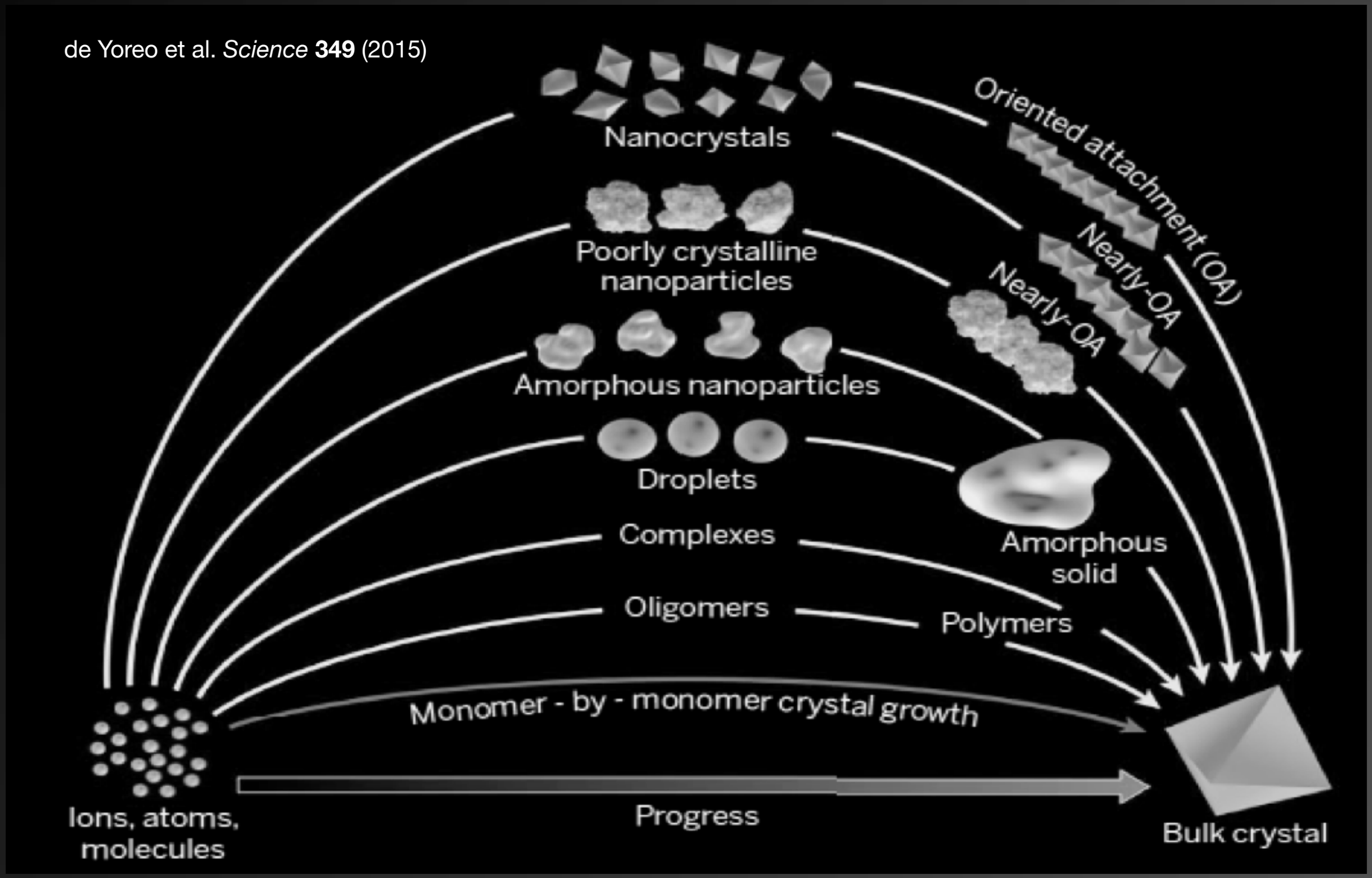
HELMHOLTZ ASSOCIATION

LinkedIn

ResearchGate

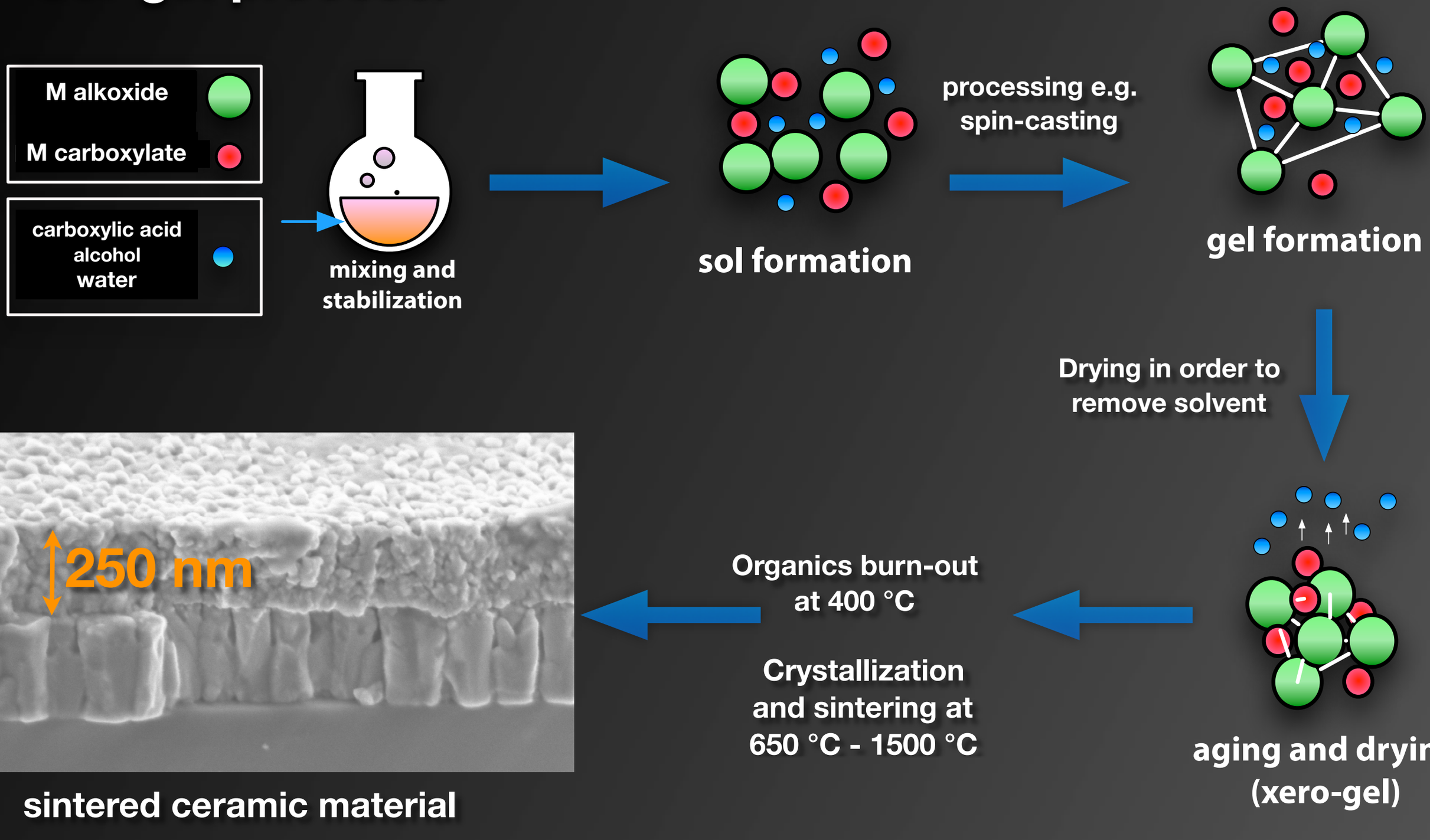
How do solids form from solution?

- **Crystallisation from supersaturated solutions:**

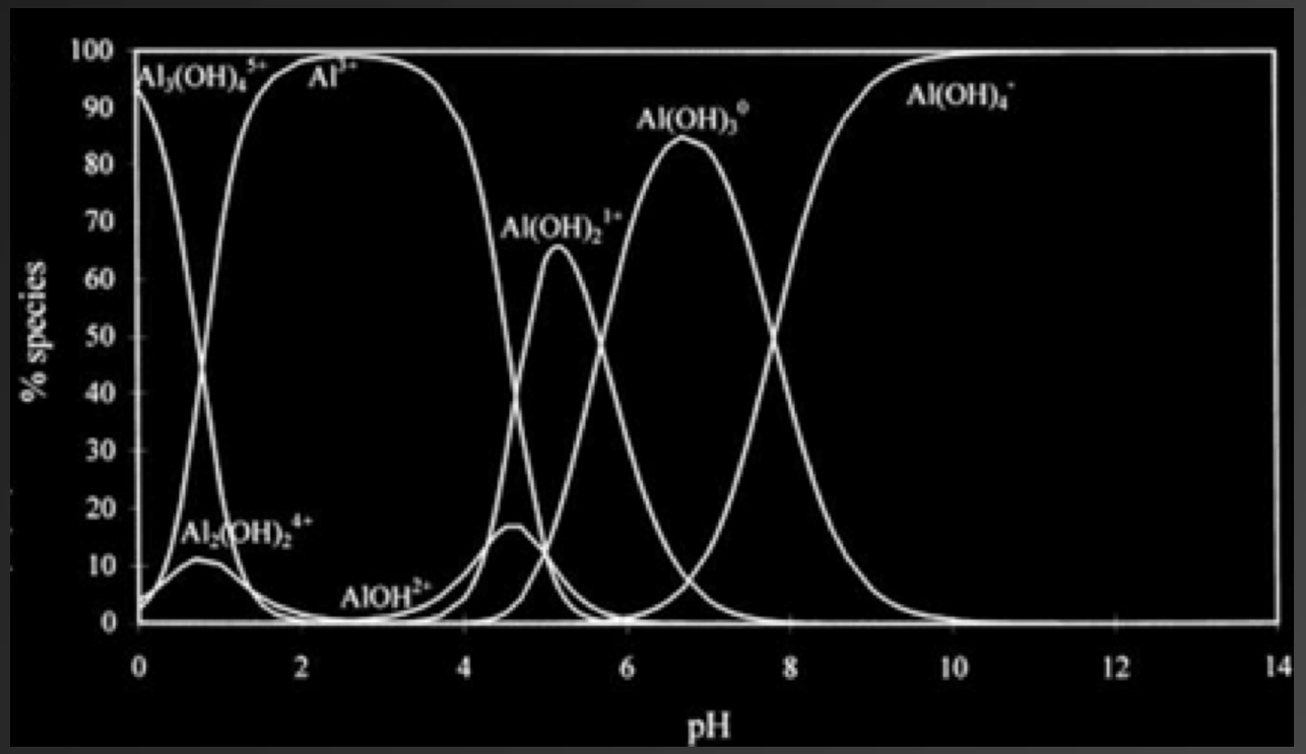


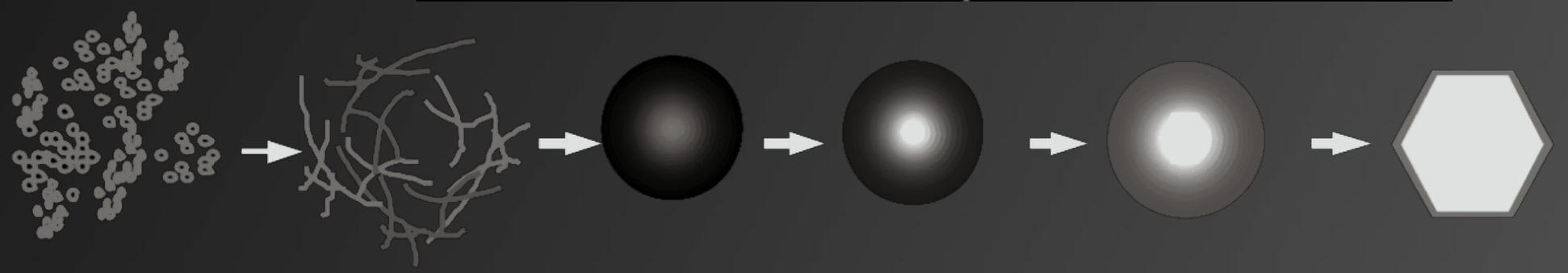
How do solids form from solution?

- Sol-gel process:

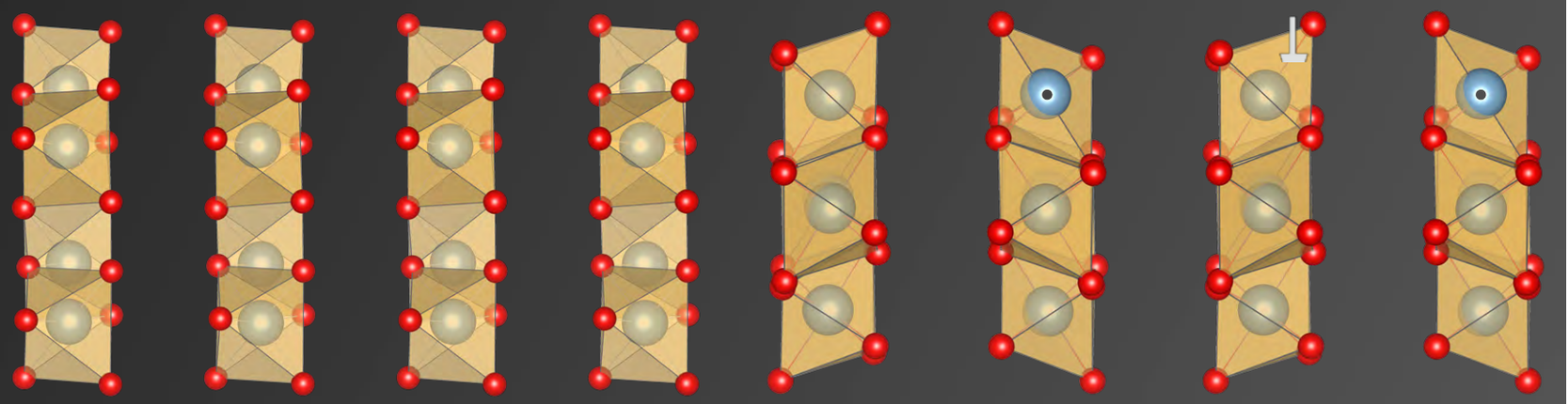


How do solids form from solution?





(1) < 1 nm (2) < 10 nm (3) < 30 nm (4) < 50 nm (5) < 100 nm (6) > 150 nm



Casey, *Chem. Rev.* 2006, 106, 1-16.

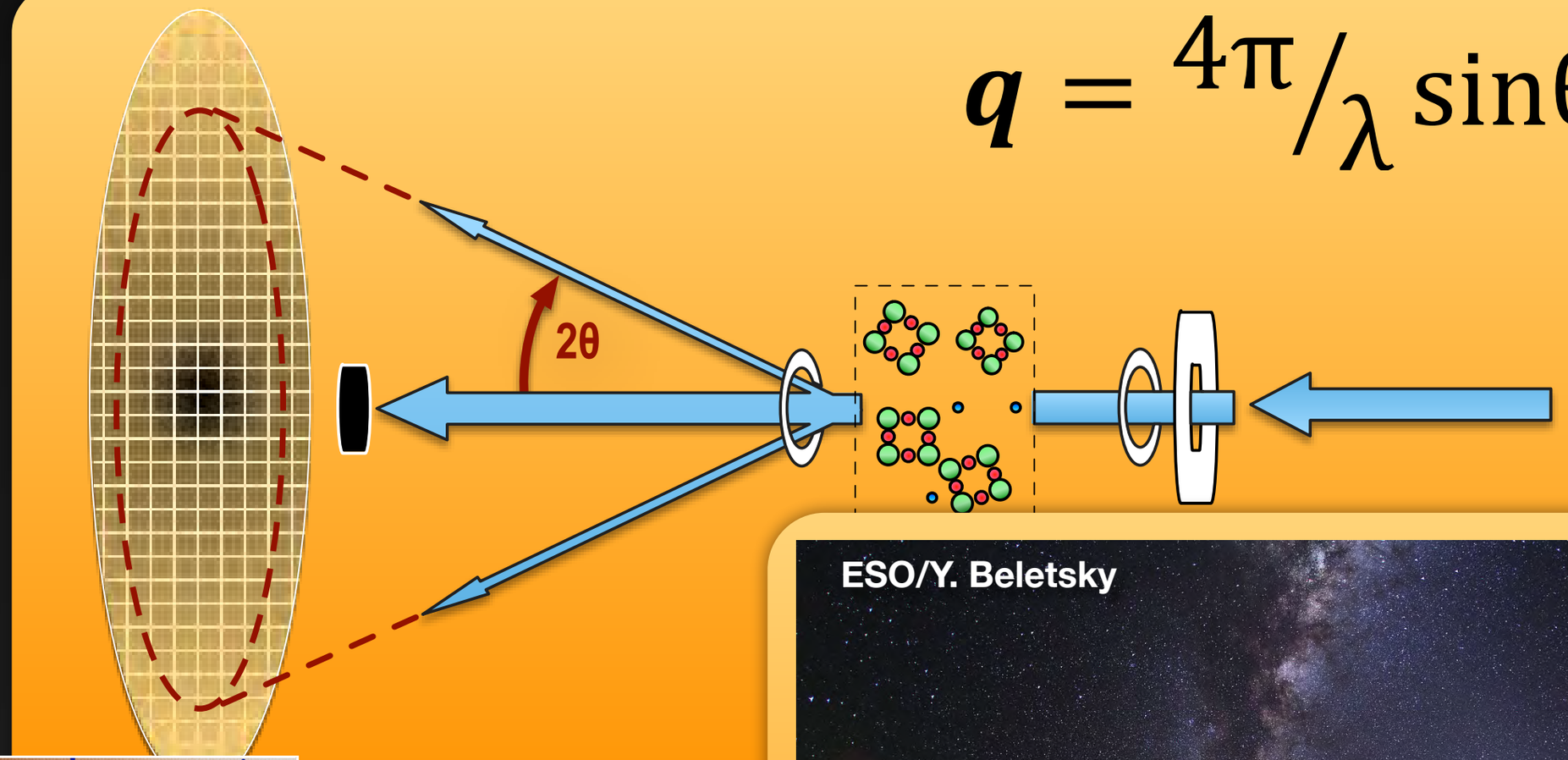
Li et al., *J. Cryst. Growth* 2005, 279, 508-520.

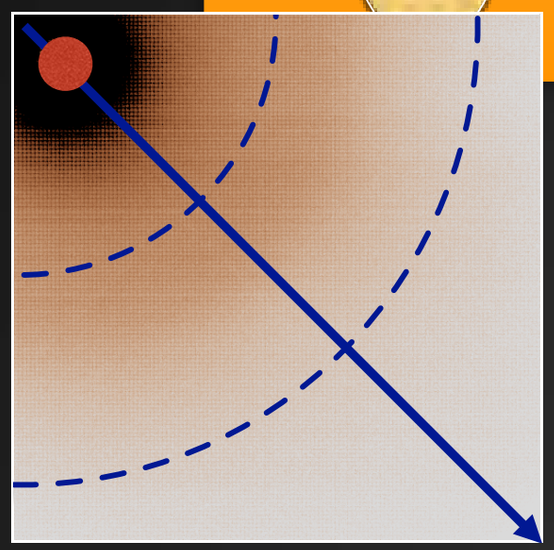
Panias et al. *Hydrometallurgy* 2001, 59, 15-29.

In situ and time-resolved characterization

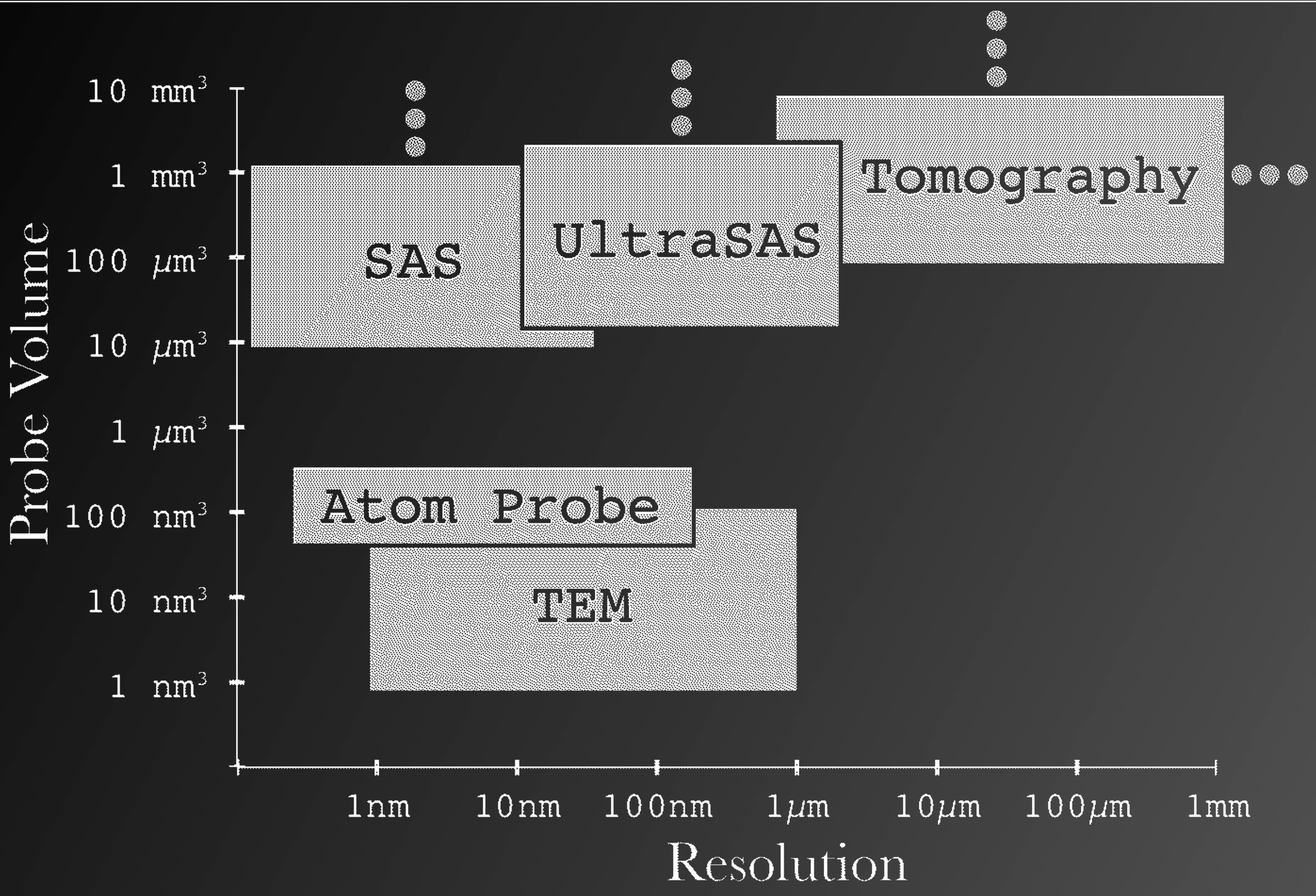
- In situ scattering methods (SAXS, WAXS, total scattering)

$$q = {}^{4\pi}/_{\lambda} \sin\theta$$



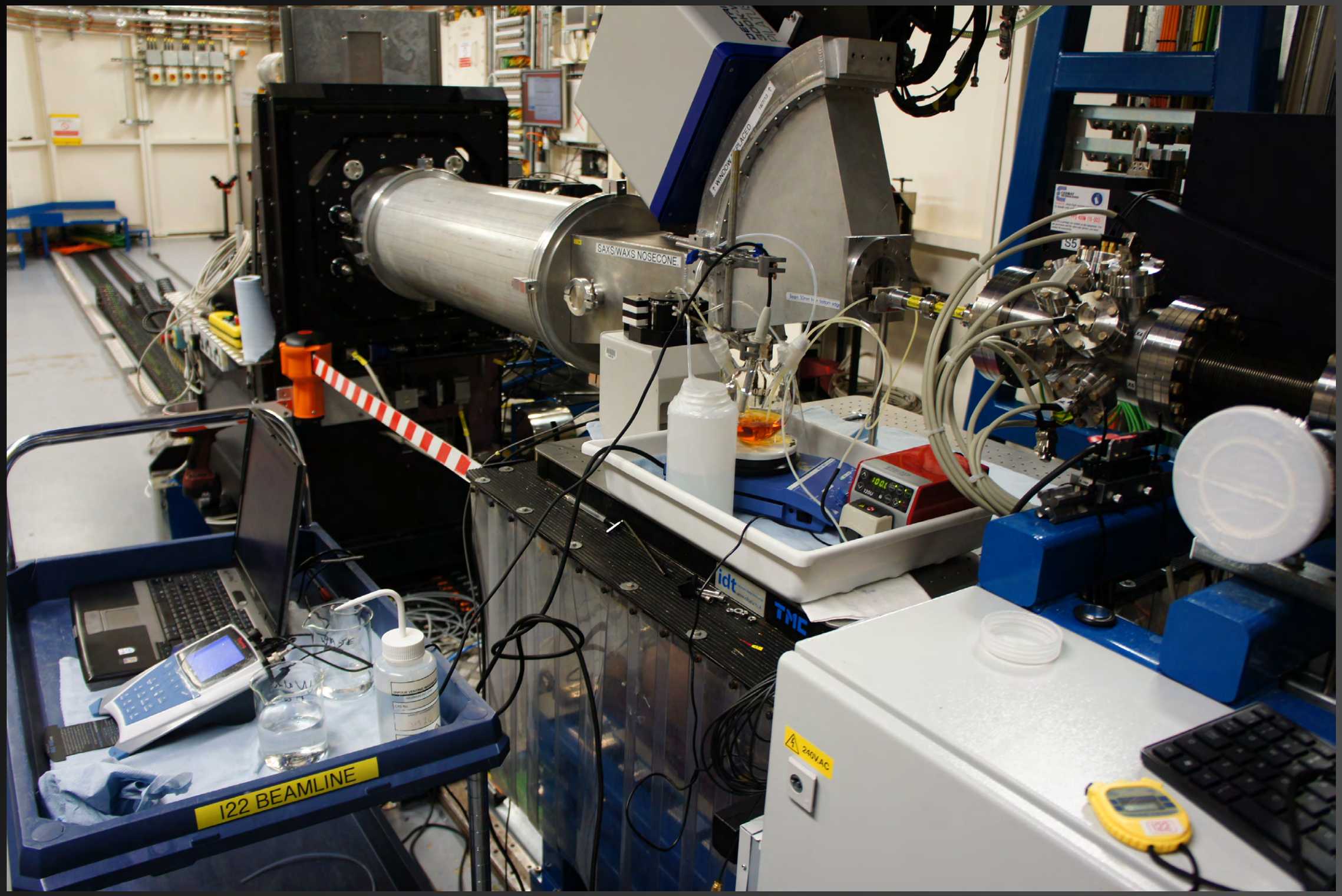


Scattering methods



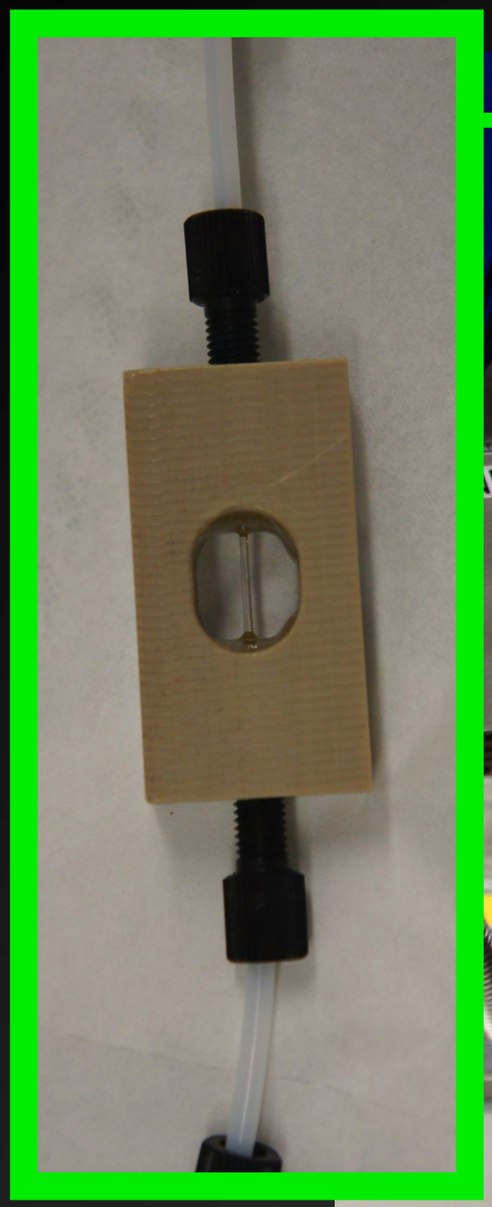
Pauw, *J. Phys.: Condens. Matter* 2013, 25, 383201

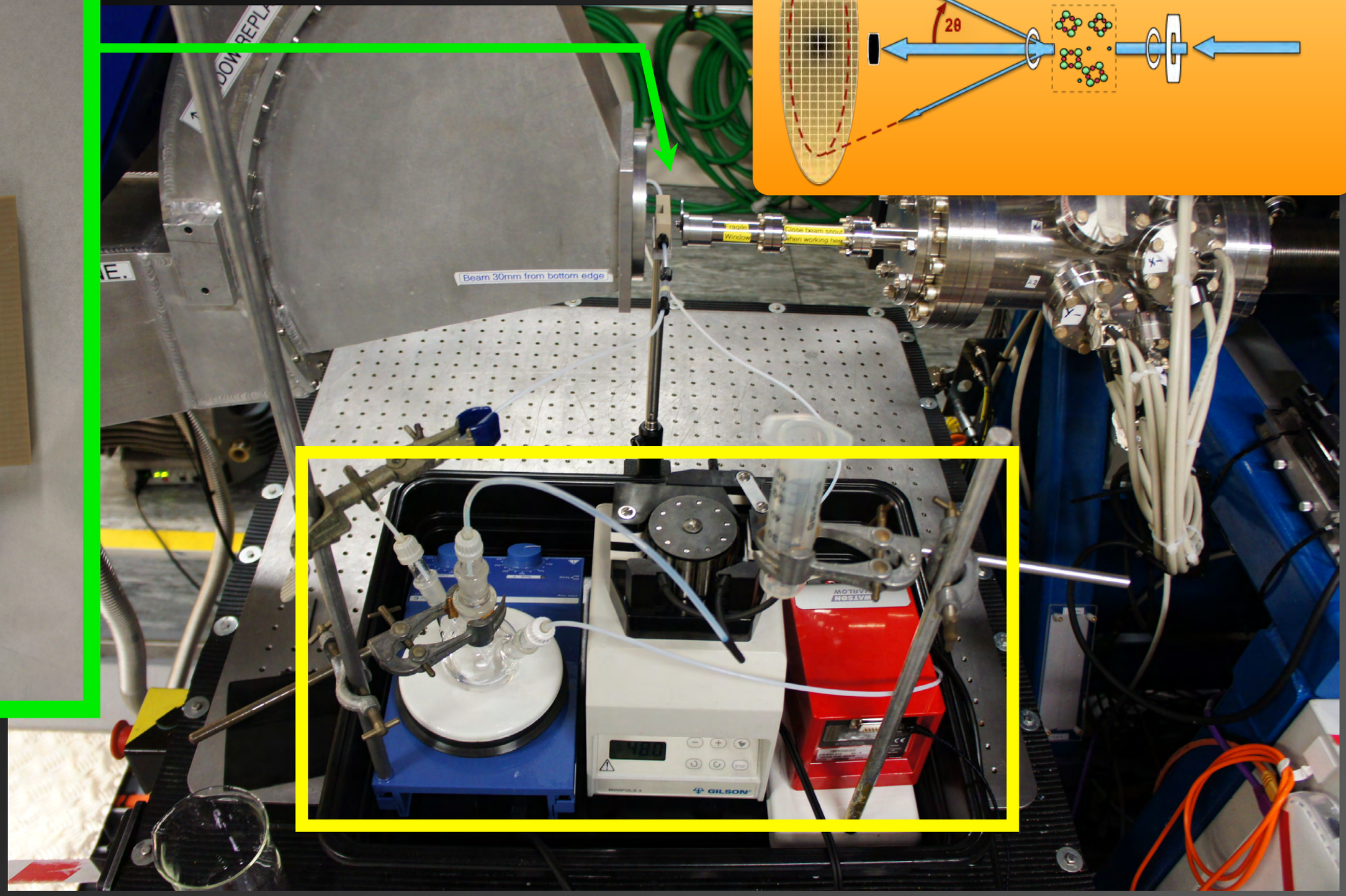
Scattering methods



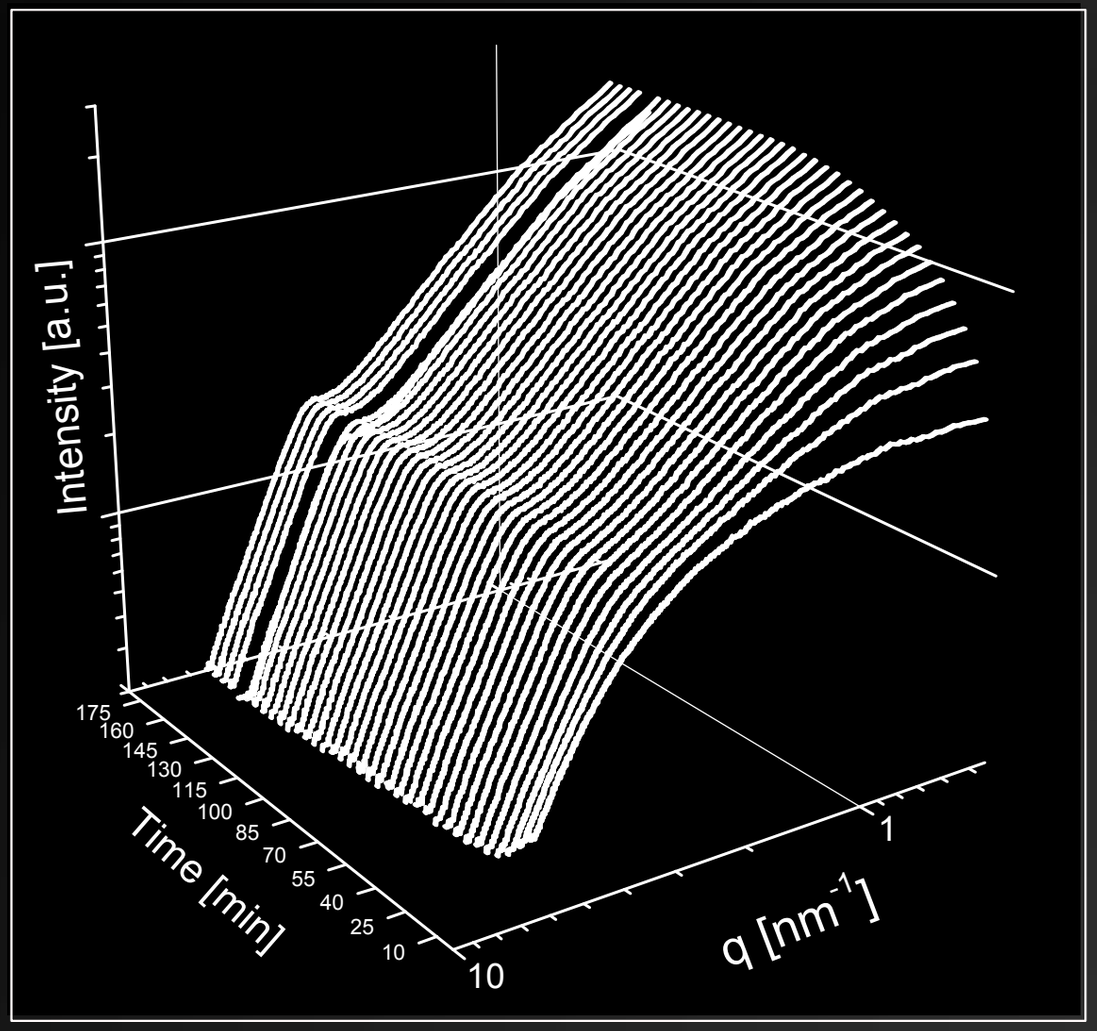
Scattering methods

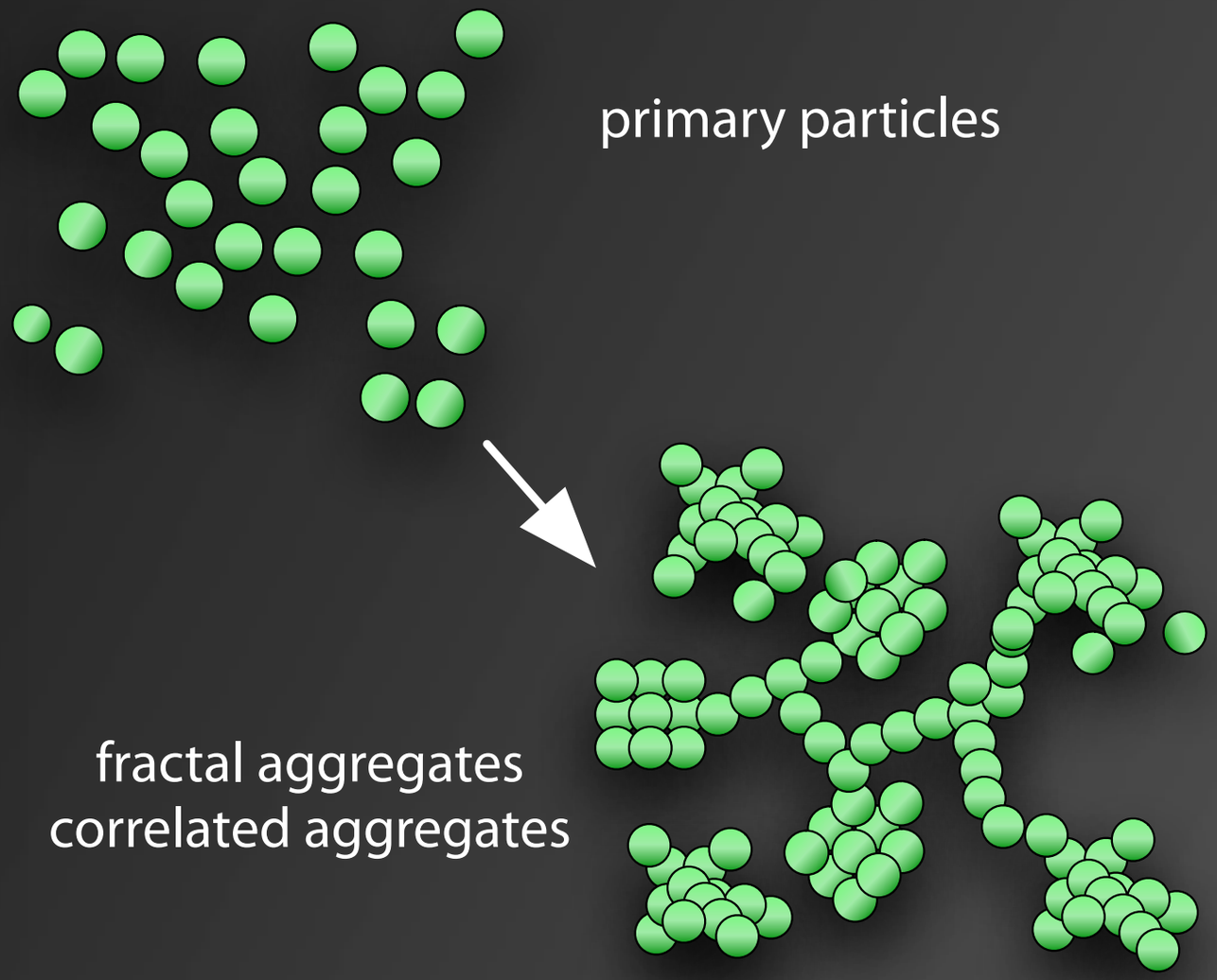
➡ In situ scattering methods

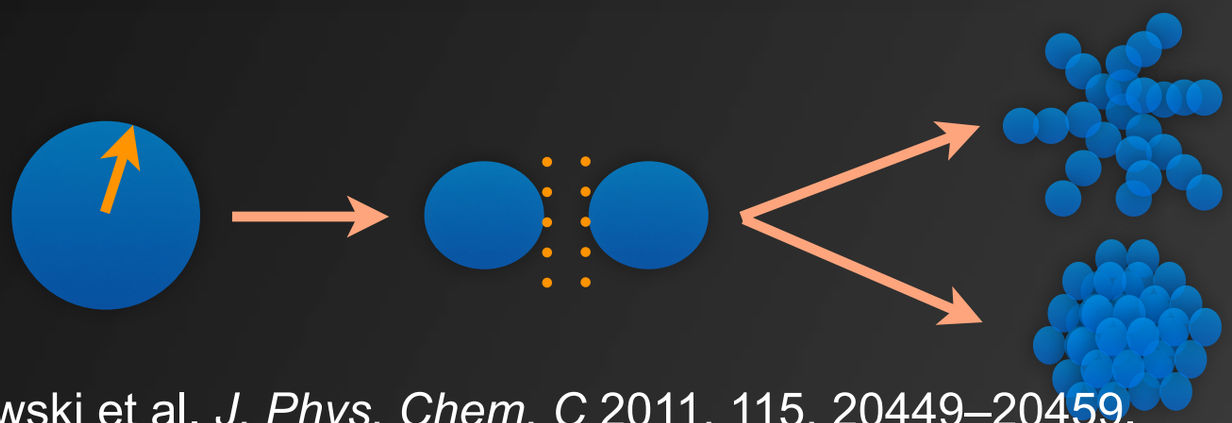




Scattering methods: gel formation







➡ **interaction type I**

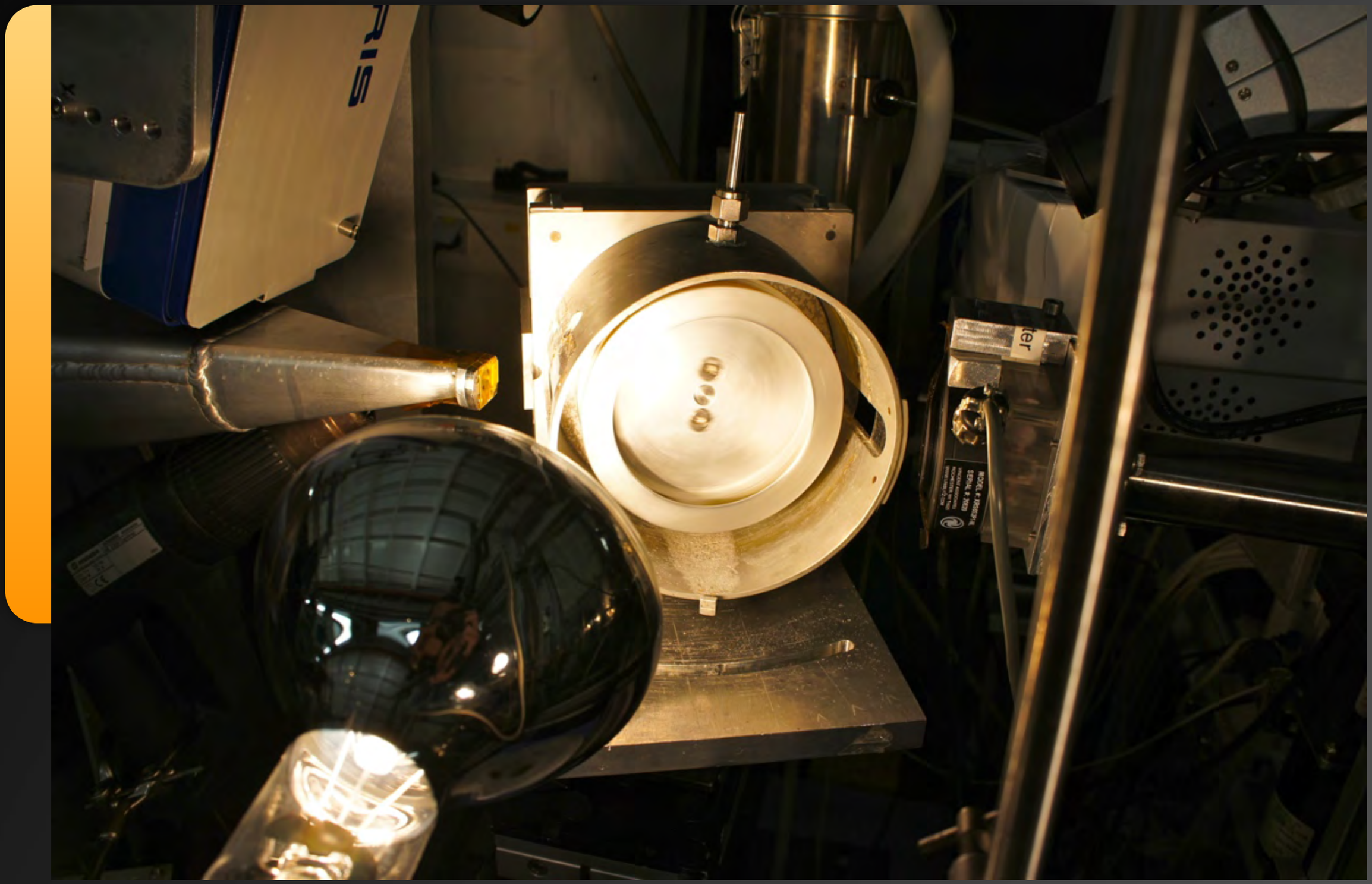
➡ **interaction type II**

Stawski et al. *J. Phys. Chem. C* 2011, 115, 20449–20459.

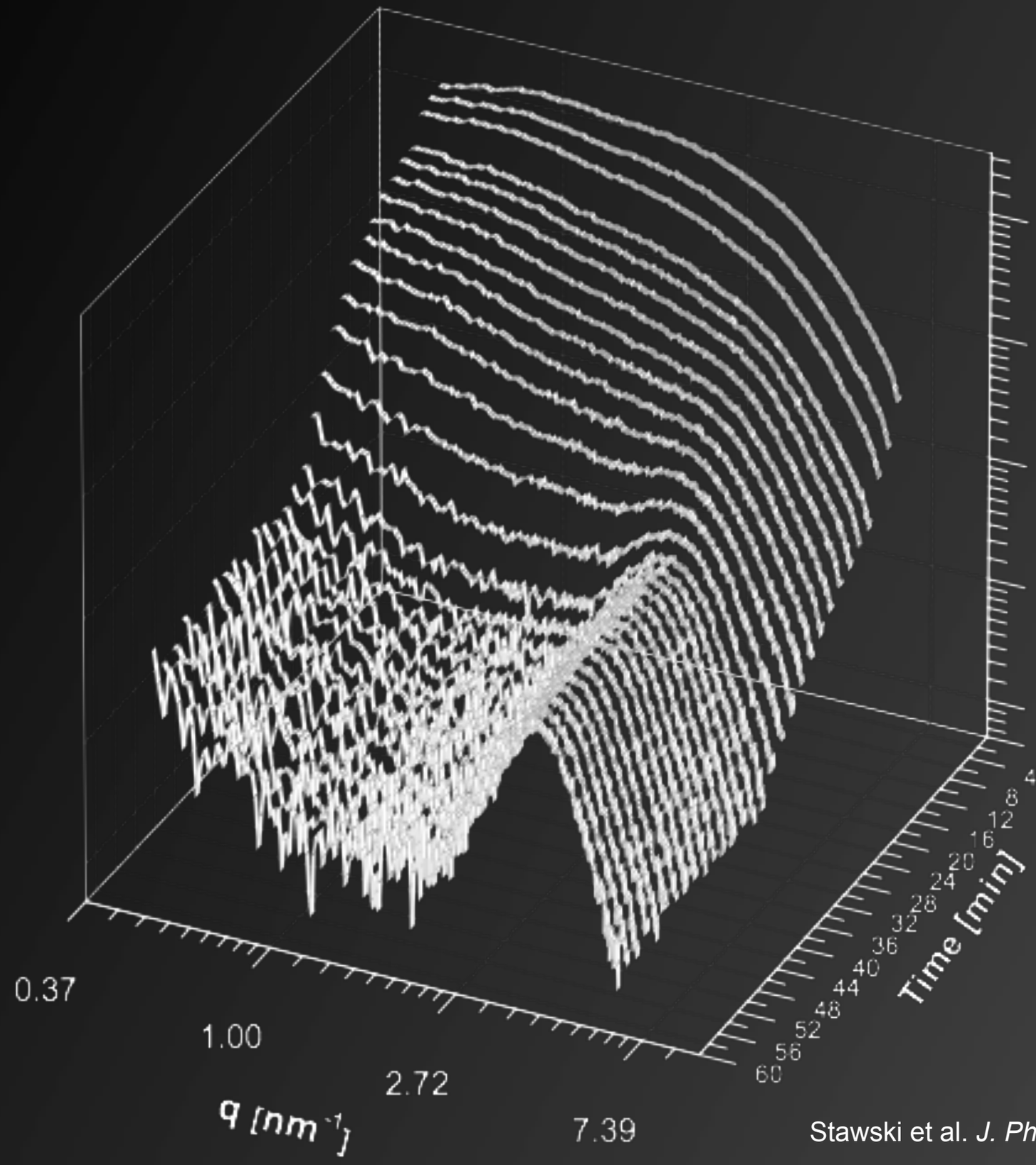
Besselink et al. *J. Colloid Inter.* 2013, 404, 24-35.

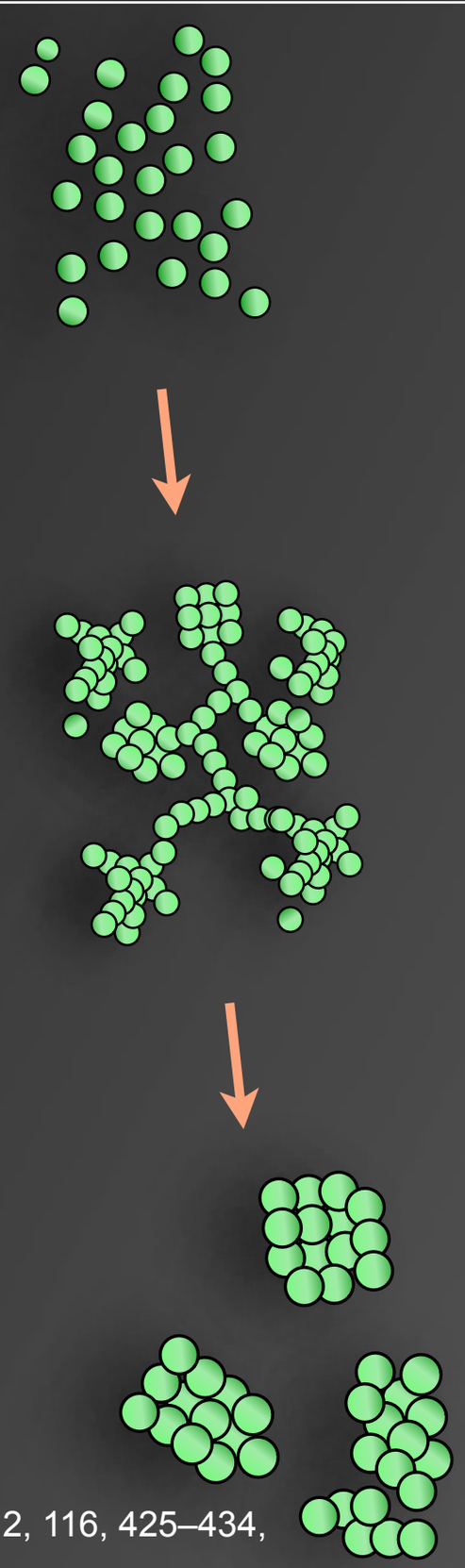
Scattering methods: drying

➡ In situ scattering methods



Scattering methods: drying



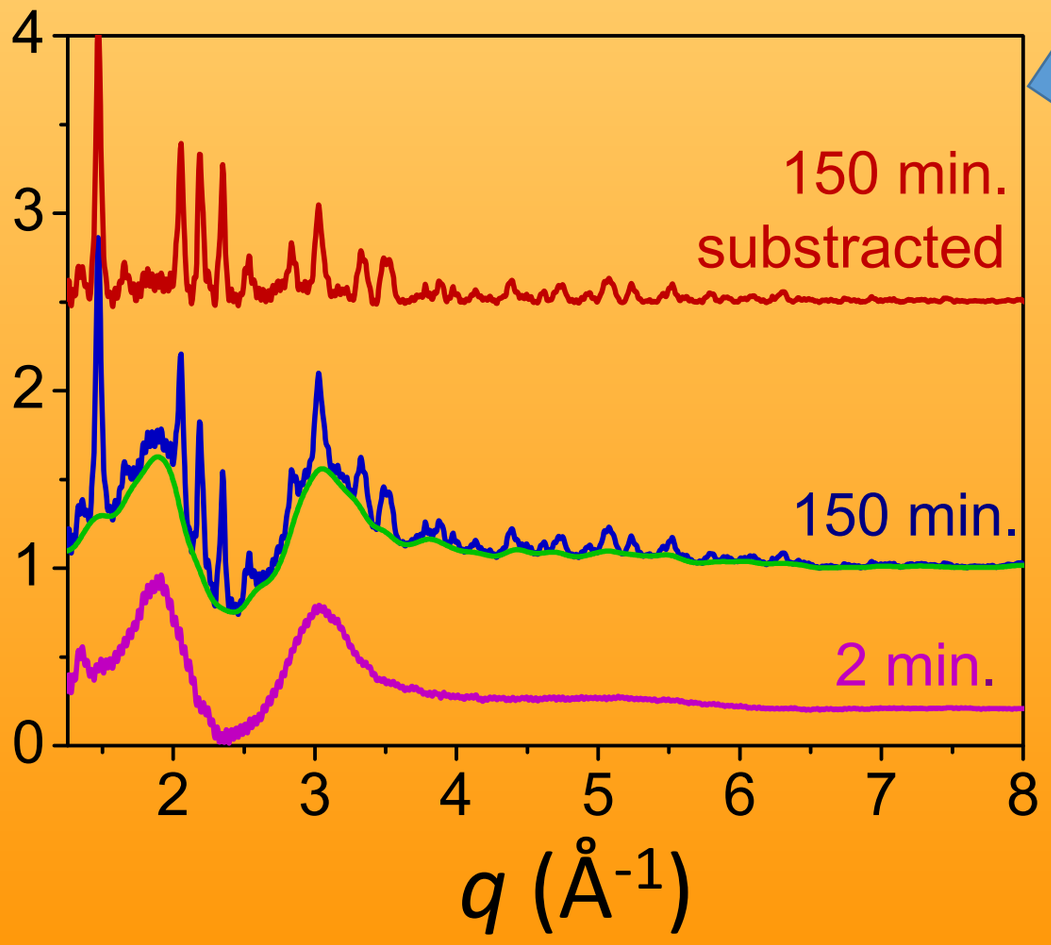


Stawski et al. *J. Phys. Chem. C* 2012, 116, 425–434,

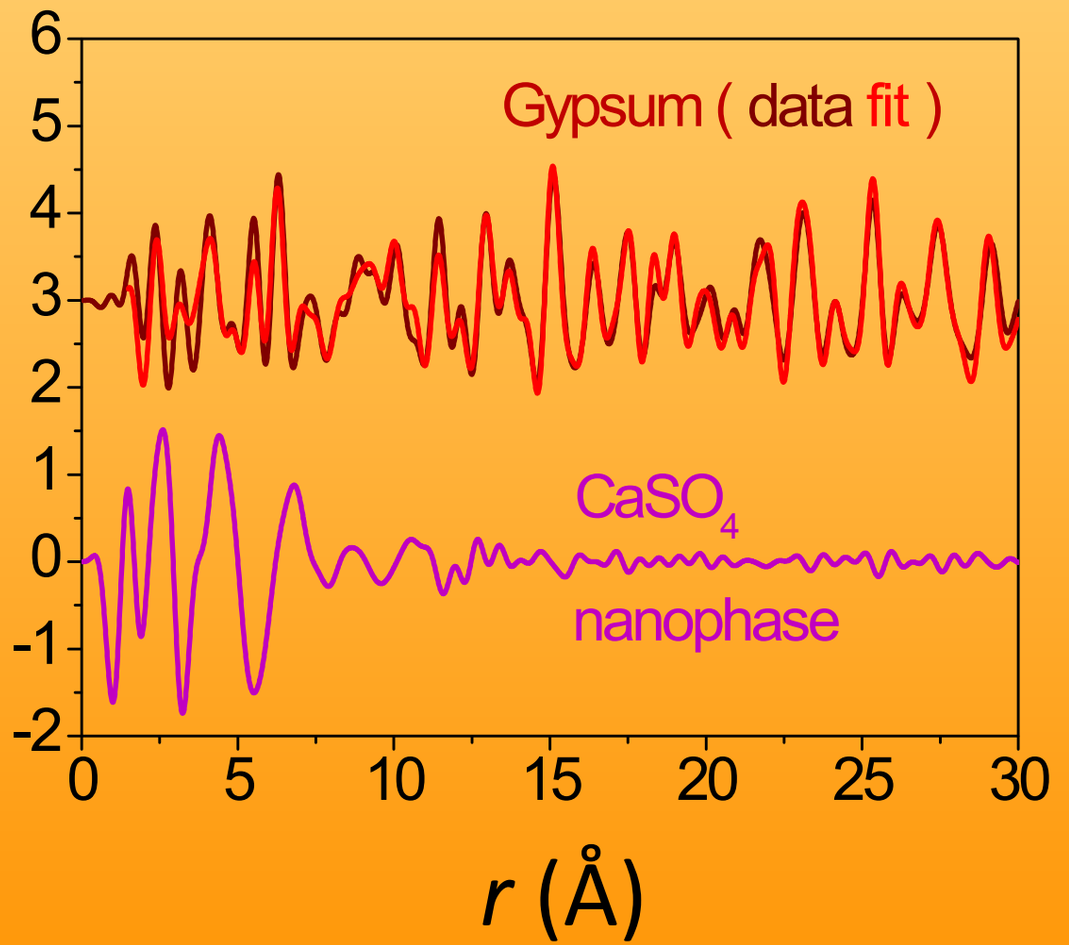
Stawski et al. *J. Phys. Chem. C* 2011, 115, 20449–20459.

Scattering methods: internal disorder

Inverse Fourier Transform



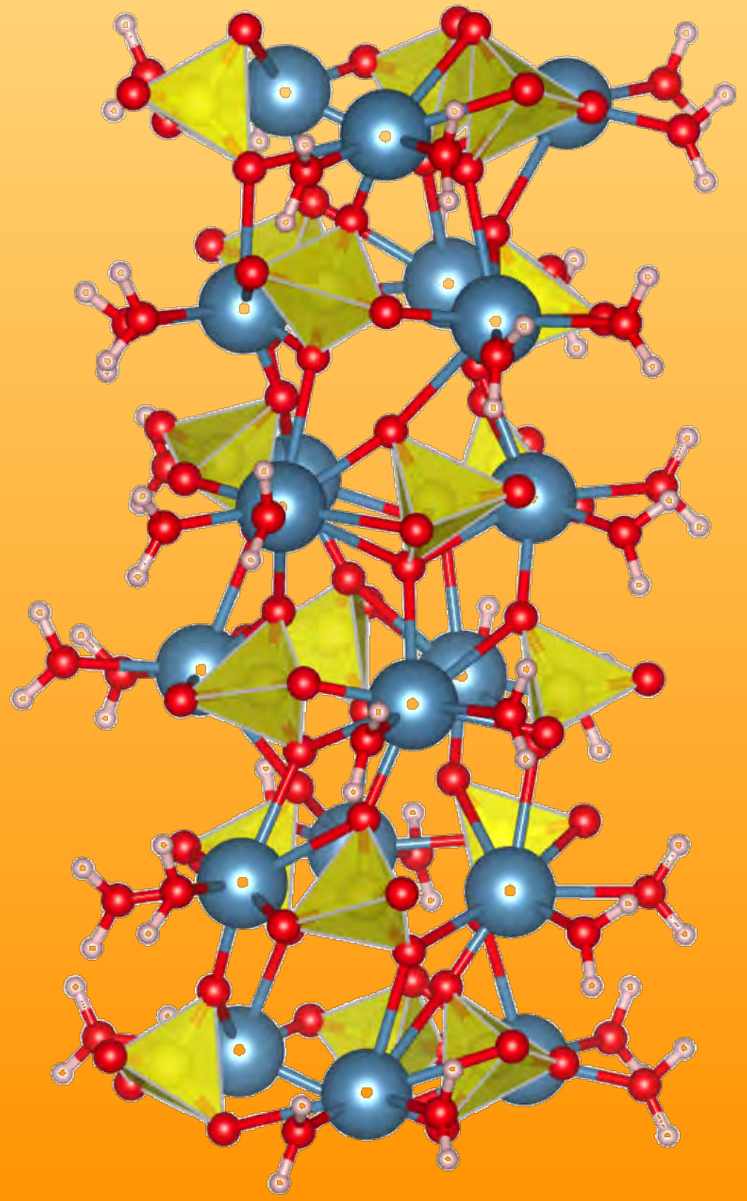
G (*r*)



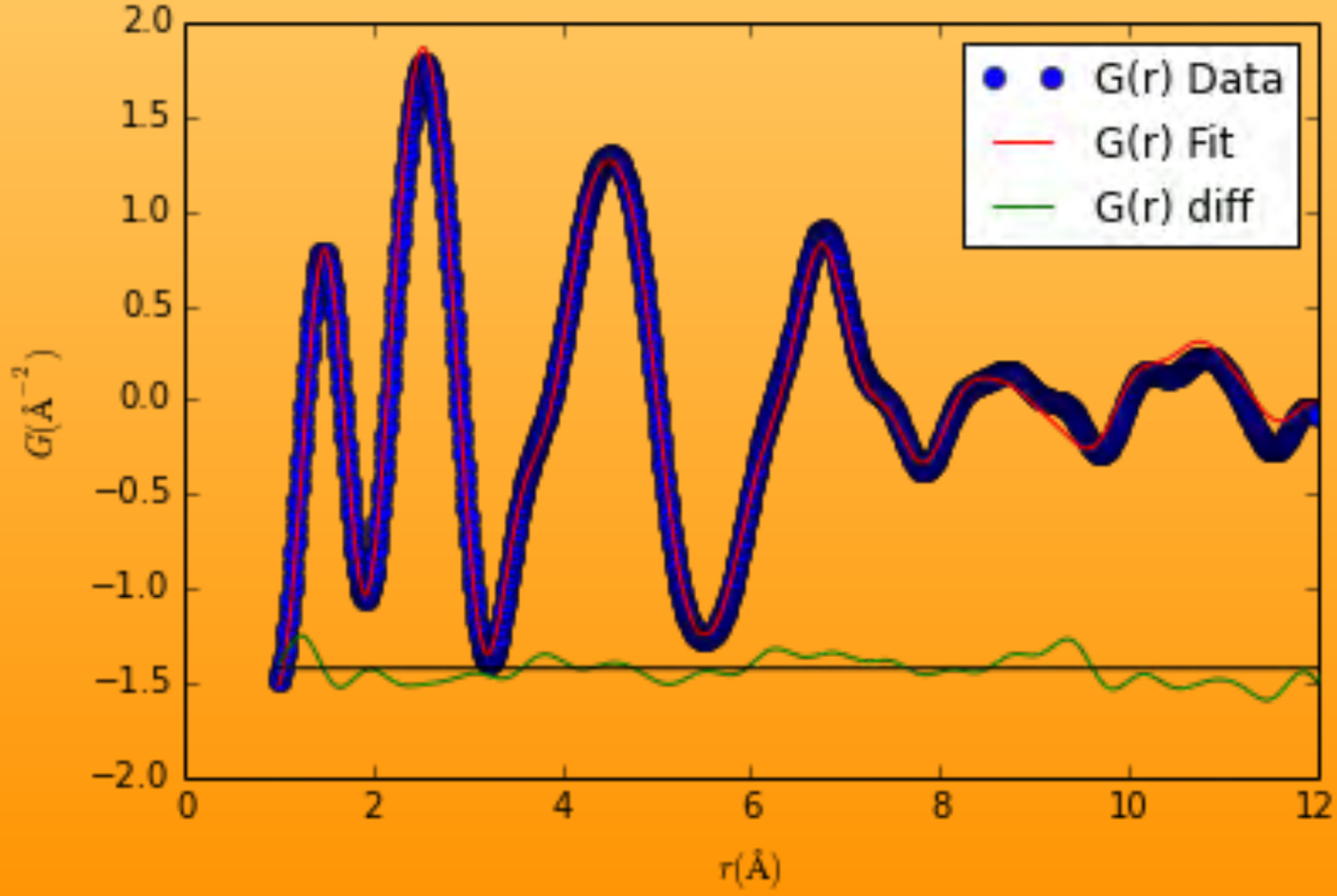
Stawski et al. *Nature Commun.* 2016, 7, 11177.

Stawski & Besselink *in prep.* 2016

Scattering methods: internal disorder

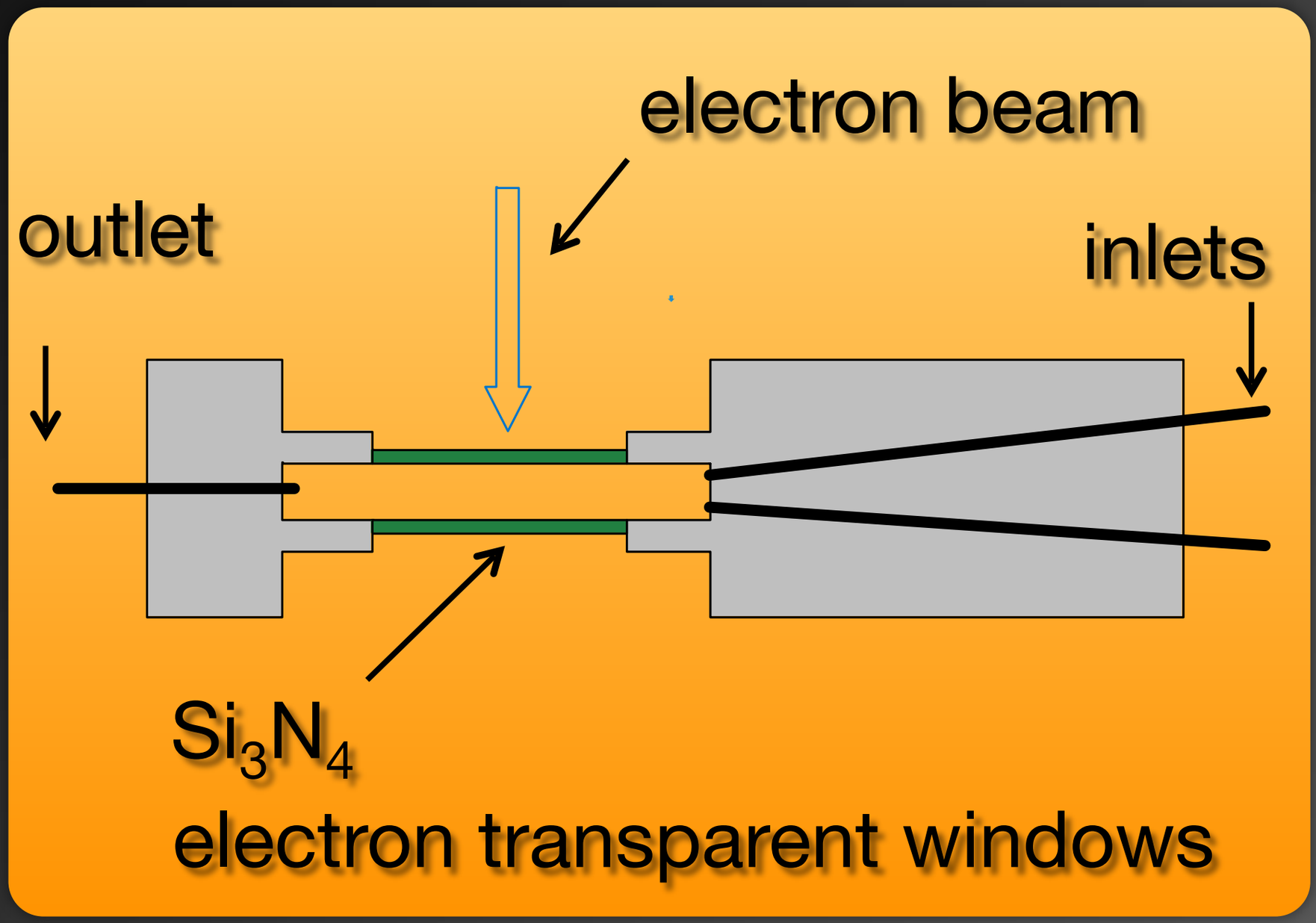


$CaSO_4$ nanophase:



Liquid-cell *in situ* TEM

JEOL 2200 FS
STEM/TEM



Conclusions

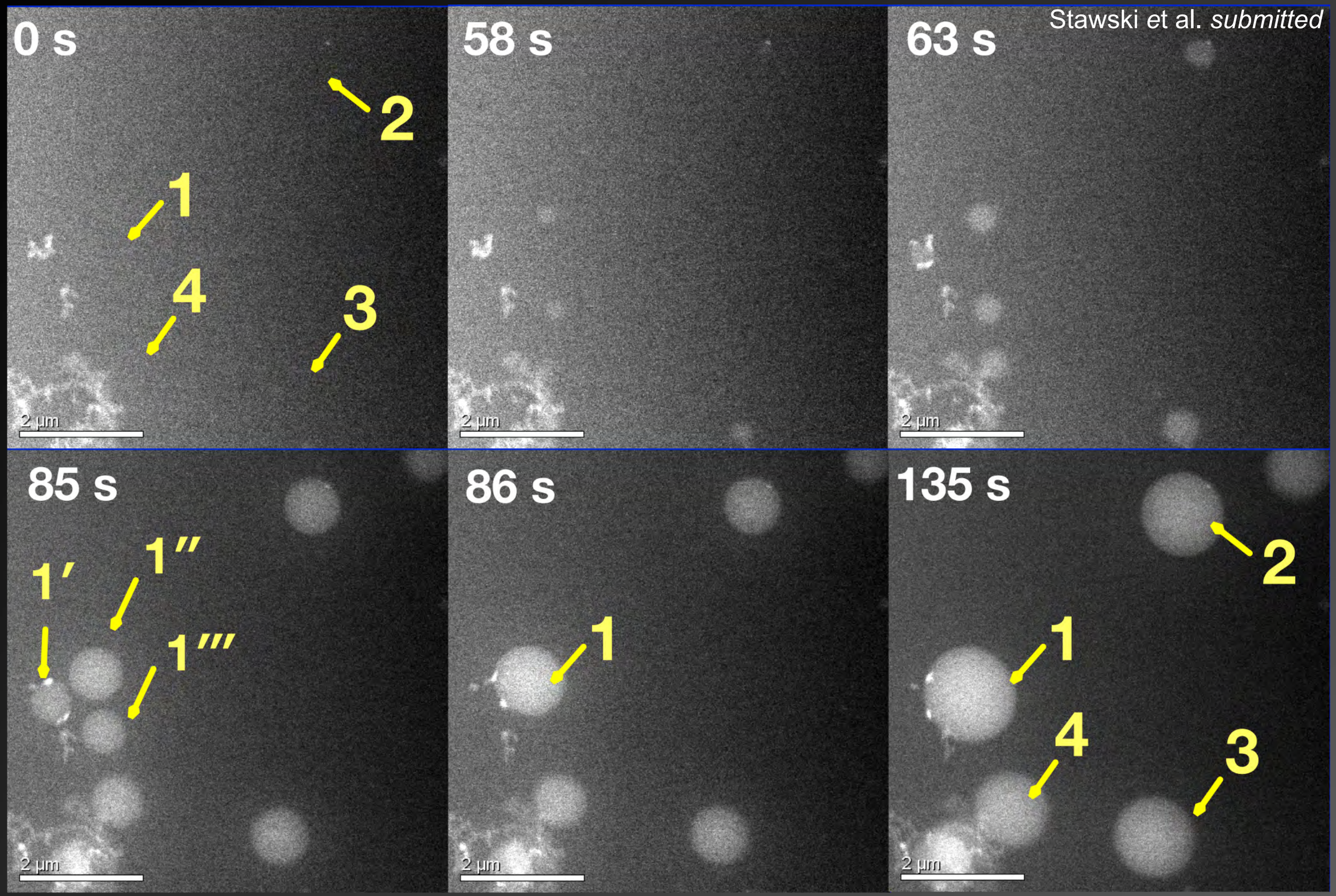
➡ **Colloid length-scales properties are crucial for understanding of the mechanisms of materials formation.**

➡ **Since colloids interact with the surrounding liquid medium, they should be characterised as *in situ* as possible.**

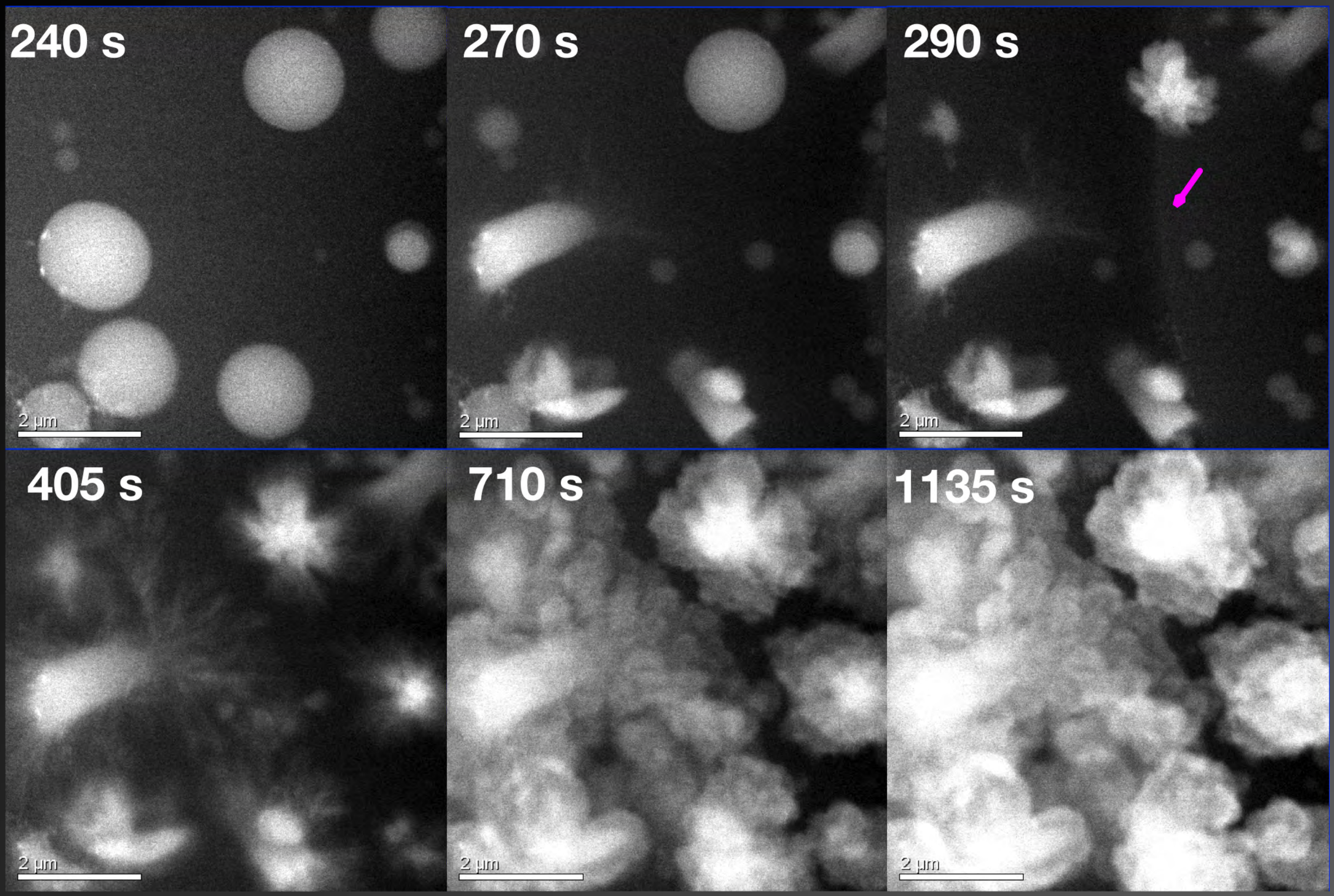
Acknowledgments

Thank you for your attention!

Micellar aggregates



Micellar aggregates



How do solids form from solution?

atomic structure

nanostructure

microstructure

Precursors:

Dissolved species

Liquid-liquid, liquid-solid interface:

Formation of clusters

Spectroscopy

Modelling

Colloids

Self-assembly

coalescence

network formation

Others

Solid products:

Amorphous solids

Crystalline solids

Solid-solid interface

Network formation

EM

Spectroscopy

Diffraction