

Synchrotron radiation and material science

Cinzia Giannini, IC-CNR

OUTLINE

International SILS School
Muggia, 01 / 10 / 2026

- SAXS – WAXS – Basic notions
- Scanning SAXS- WAXS Microscopy
- Nanocrystals and Nanocrystal-based Superlattices
- Scaffolds; Fibers

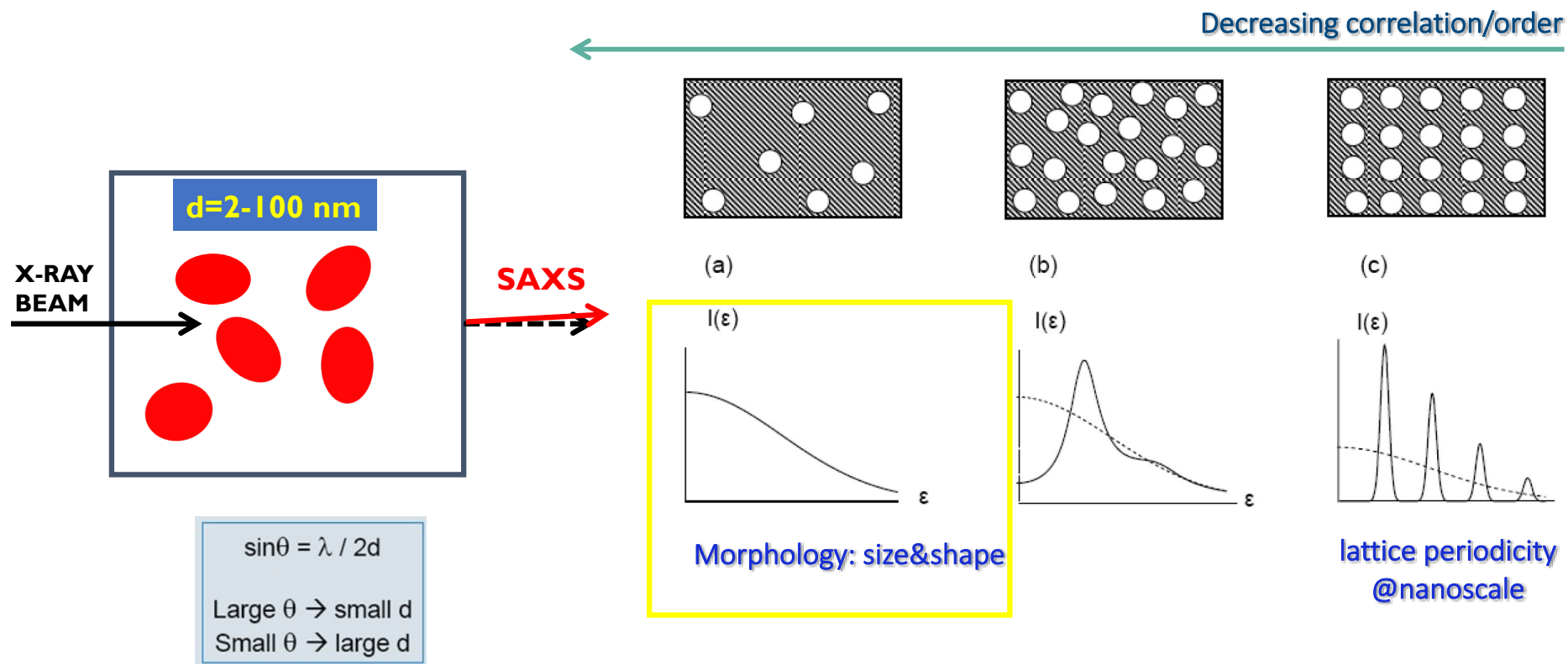
OUTLINE

International SILS School
Muggia, 01 / 10 / 2026

- SAXS – WAXS – Basic notions
- Scanning SAXS- WAXS Microscopy
- Nanocrystals and Nanocrystal-based Superlattices
- Scaffolds; Fibers

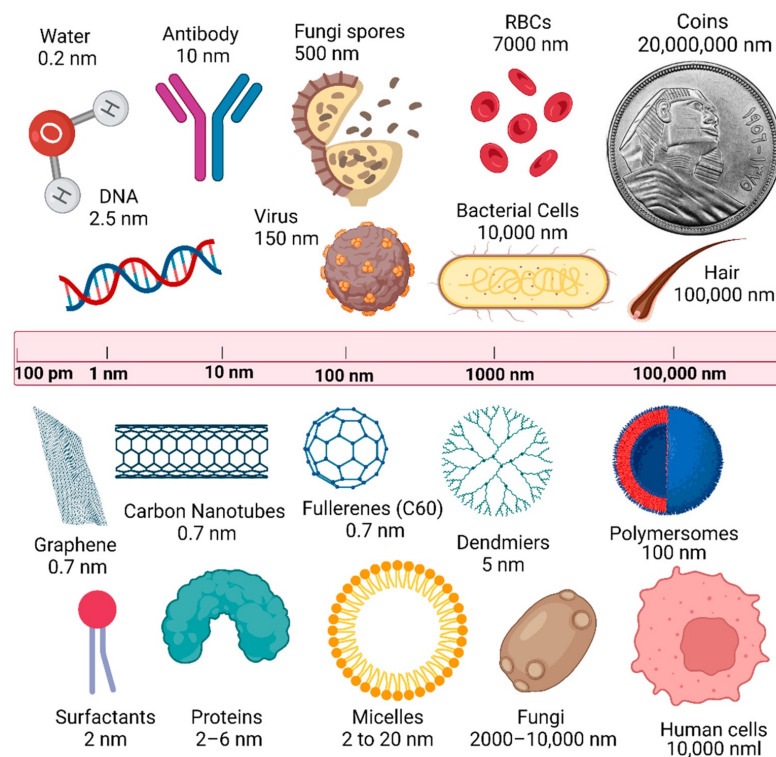
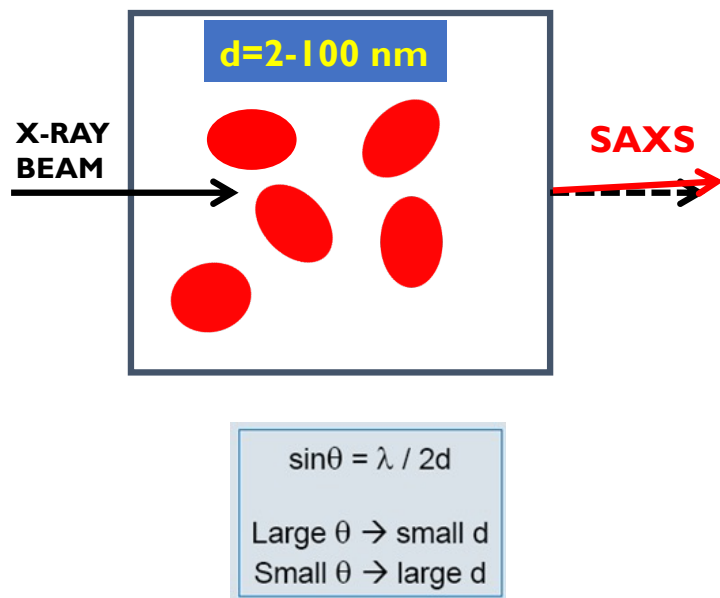
SMALL ANGLE X-RAY SCATTERING - SAXS

International SILS School
Muggia, 01 / 10 / 2026



SMALL ANGLE X-RAY SCATTERING - SAXS

International SILS School
Muggia, 01 / 10 / 2026

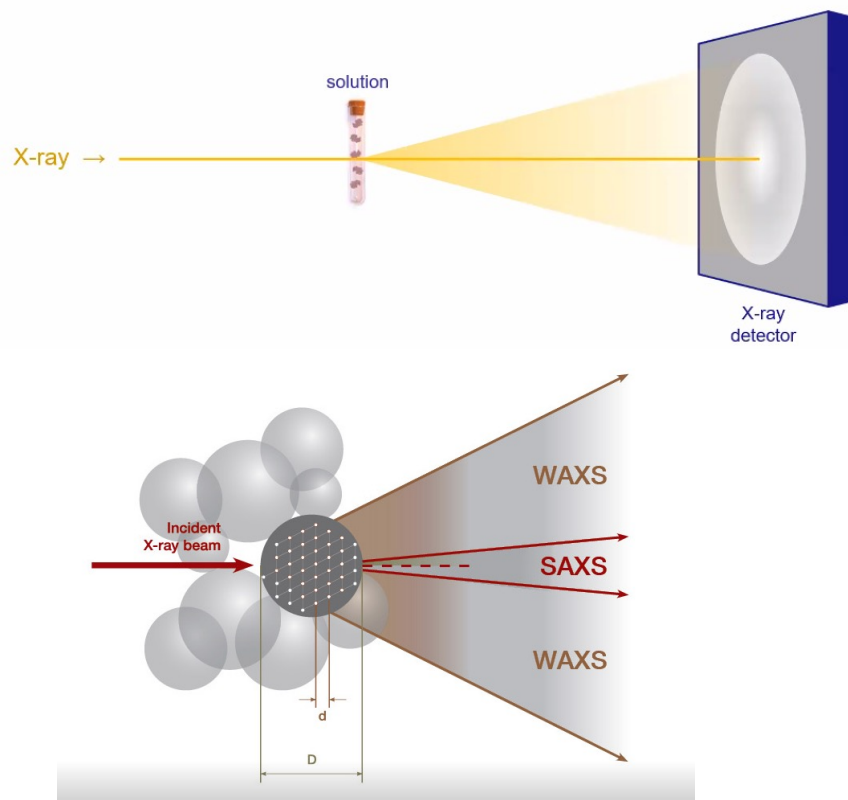


Nanomaterials **2022**, 12(2), 177;

<https://doi.org/10.3390/nano12020177>

SMALL ANGLE X-RAY SCATTERING - **SAXS**

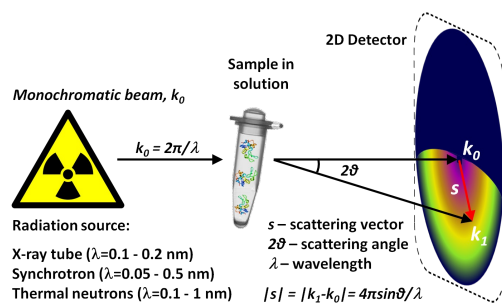
International SILS School
Muggia, 01 / 10 / 2026



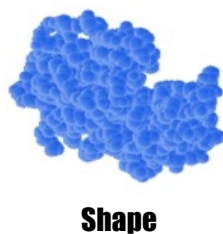
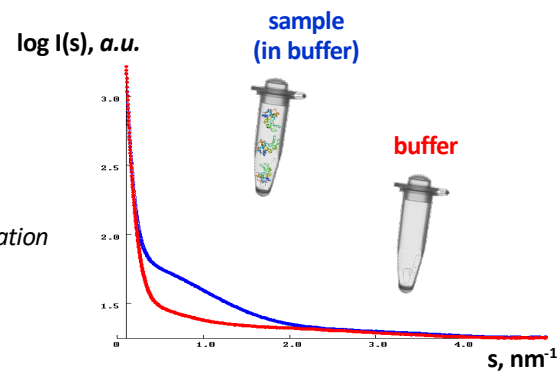
- transmission geometry
- X-ray optics that create a very narrow, yet highly intense incident X-ray beam are essential.
- This is because the comparatively weak scattering signal from the sample has to be measured in the immediate vicinity of the direct beam.
- It is also essential to use a 2D detector that has a high linearity, a high dynamic range and negligible intrinsic noise.

SMALL ANGLE X-RAY SCATTERING - SAXS

International SILS School
Muggia, 01 / 10 / 2026

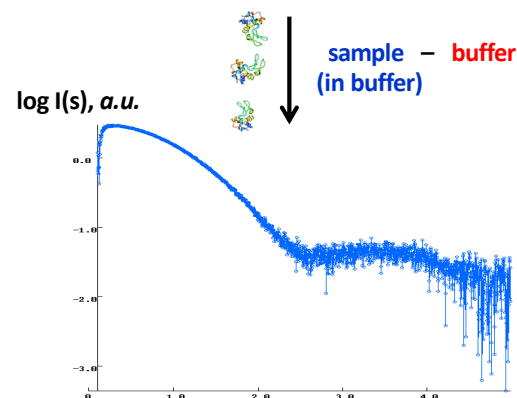


radially averaged
normalized:
sample concentration
incoming beam
exposure time



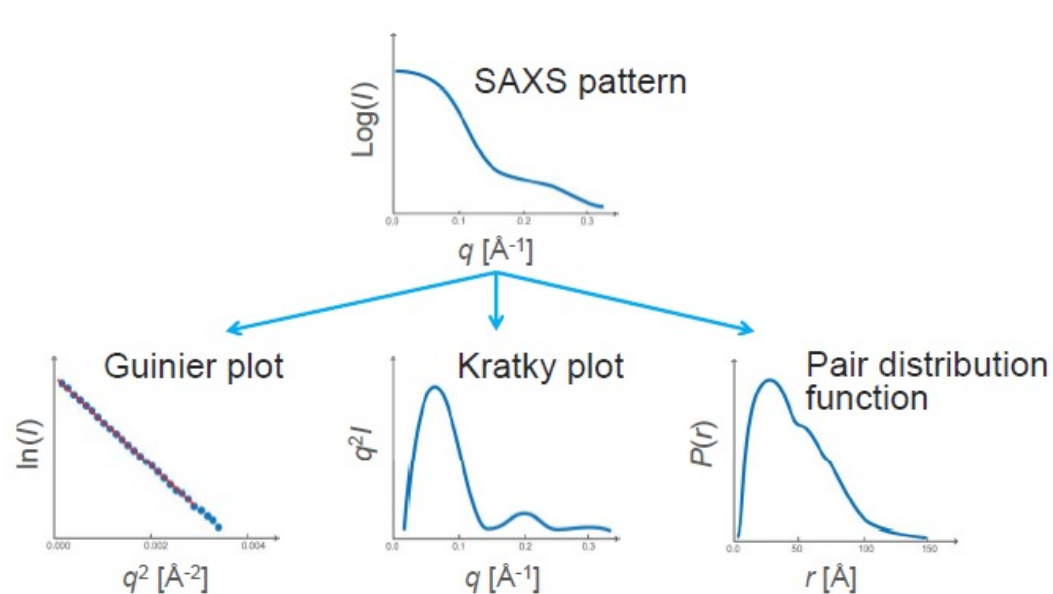
$$I(s) = 4\pi \int_0^D p(r) \frac{\sin sr}{sr} dr$$

for monodisperse systems the scattering is proportional to that of a single particle averaged over all orientations

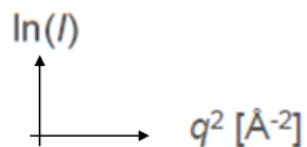


SMALL ANGLE X-RAY SCATTERING - SAXS

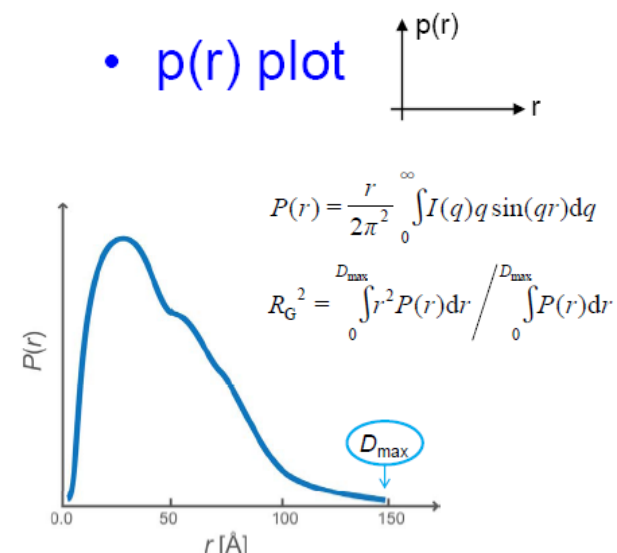
International SILS School
Muggia, 01 / 10 / 2026



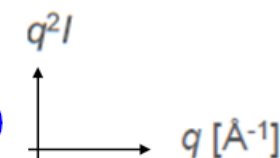
- Guinier plot (R_g , MM)



- $p(r)$ plot

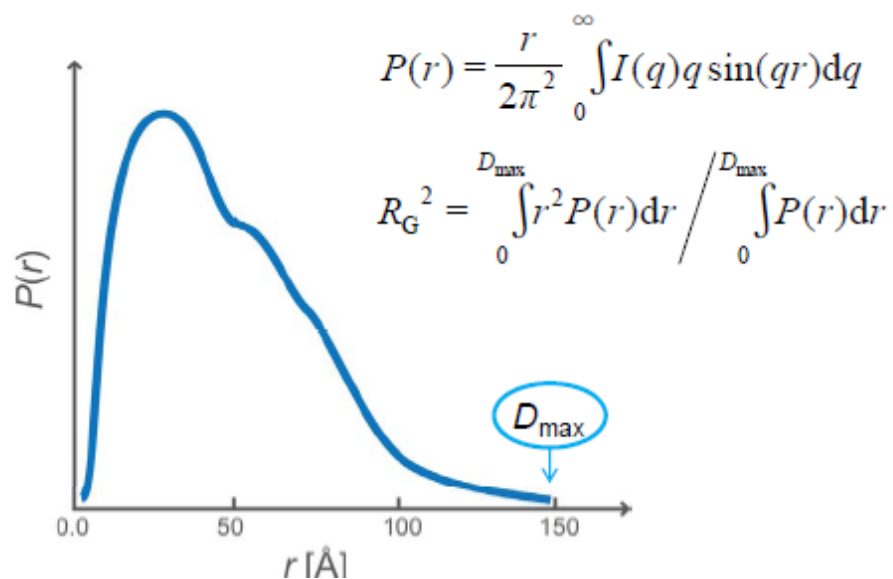


- Kratky plot (flexibility)

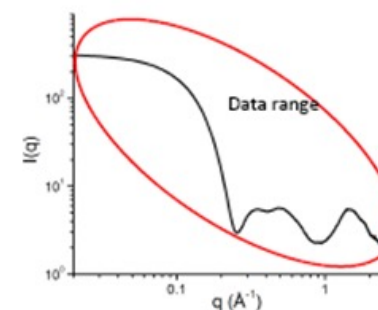


SMALL ANGLE X-RAY SCATTERING – SAXS/PDF

International SILS School
Muggia, 01 / 10 / 2026

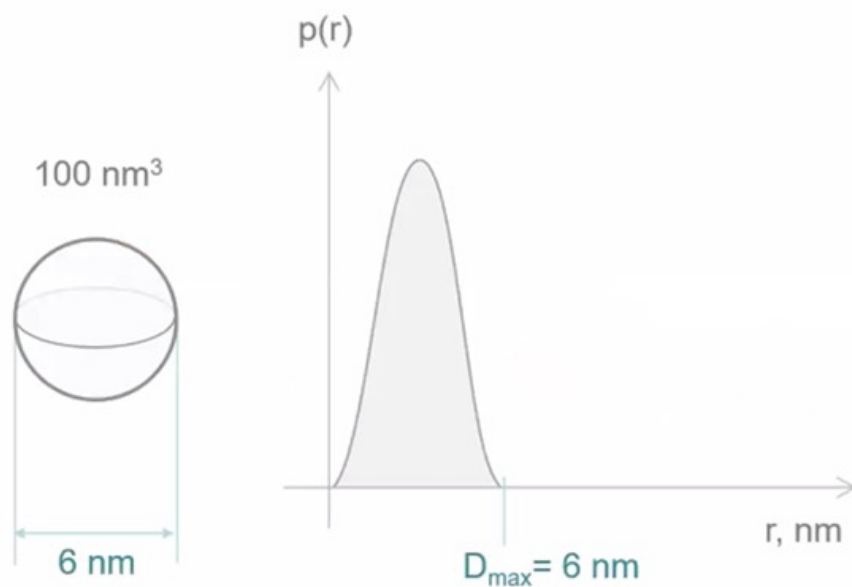


• $p(r)$ plot



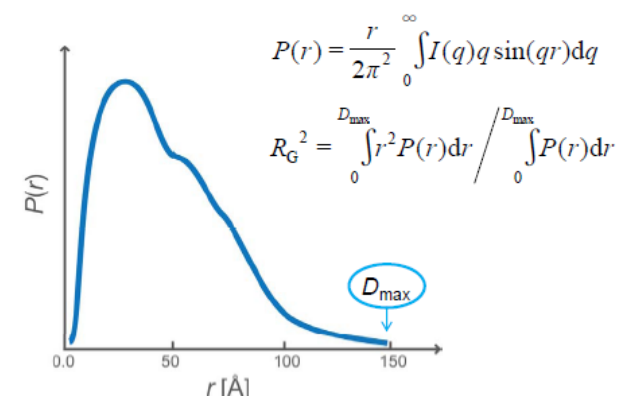
SMALL ANGLE X-RAY SCATTERING – SAXS/PDF

International SILS School
Muggia, 01 / 10 / 2026



• $p(r)$ plot

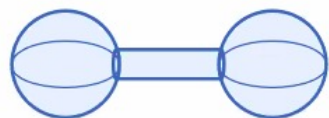
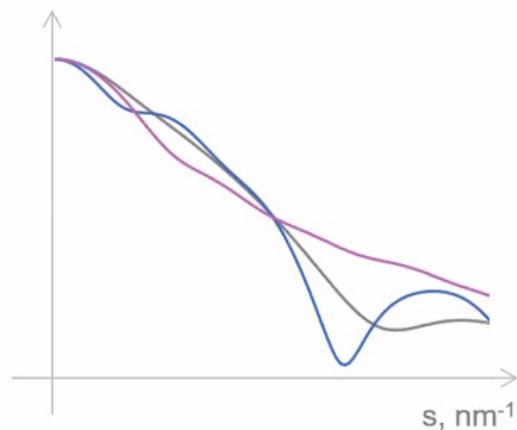
A small coordinate system with a vertical axis labeled $p(r)$ and a horizontal axis labeled r .



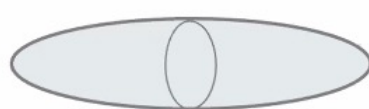
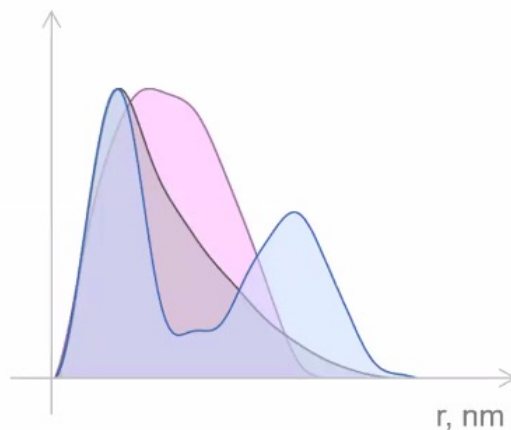
SMALL ANGLE X-RAY SCATTERING – SAXS/PDF

International SILS School
Muggia, 01 / 10 / 2026

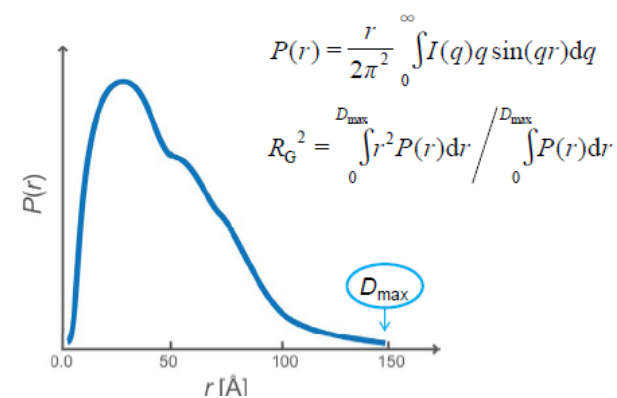
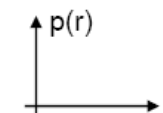
Log I(s)



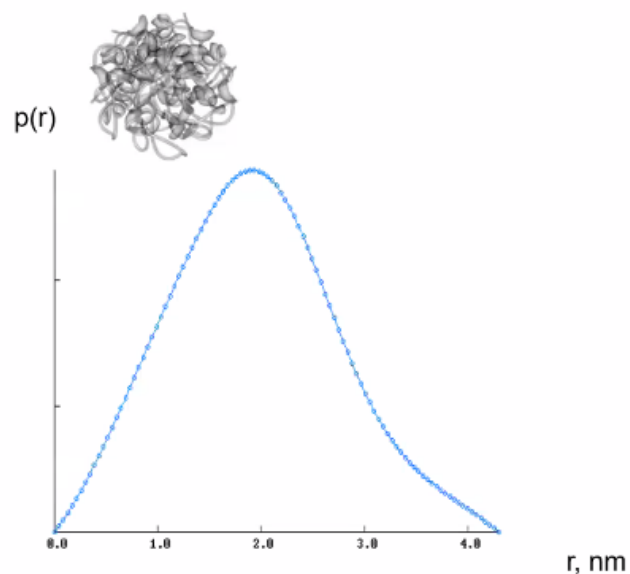
p(r)



• p(r) plot



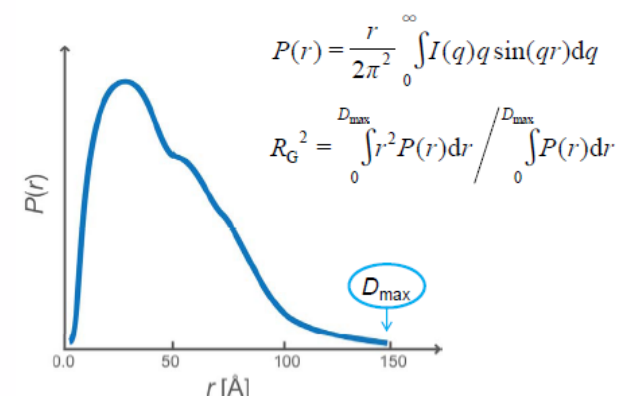
R_g and $I(0)$ from $p(r)$



$$I(0) = 4\pi \int_0^{D_{\max}} p(r) dr$$

$$R_g^2 = \frac{\int_0^{D_{\max}} r^2 p(r) dr}{2 \int_0^{D_{\max}} p(r) dr}$$

• $p(r)$ plot



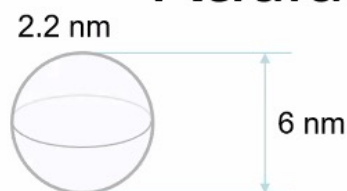
$$P(r) = \frac{r}{2\pi^2} \int_0^{\infty} I(q) q \sin(qr) dq$$

$$R_g^2 = \frac{\int_0^{D_{\max}} r^2 P(r) dr}{\int_0^{D_{\max}} P(r) dr}$$

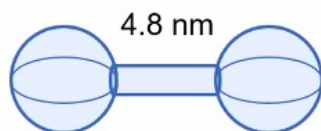
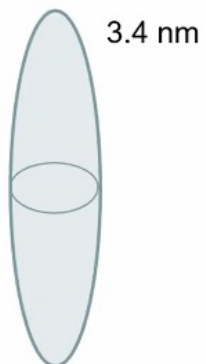
SMALL ANGLE X-RAY SCATTERING - SAXS

International SILS School
Muggia, 01 / 10 / 2026

Radius of gyration (R_g)



100 nm^3



Radius of gyration (R_g)

R_g^2 definition:

Average of square center-of-mass
distances in the molecule
weighted by the scattering length density

R_g is the quadratic mean of distances
to the center of mass weighted by
the contrast of electron density.

R_g is an *index of non sphericity*



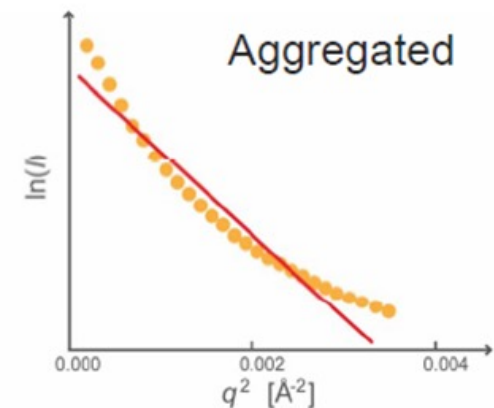
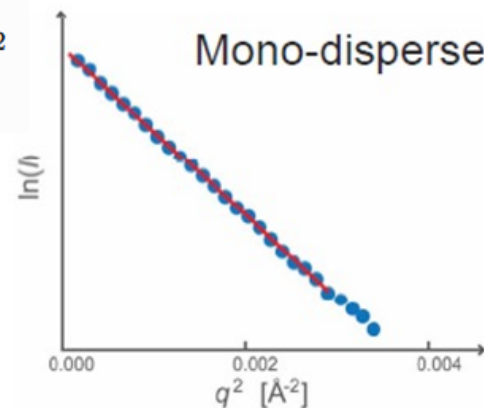
SMALL ANGLE X-RAY SCATTERING - SAXS

International SILS School
Muggia, 01 / 10 / 2026

- Guinier plot (R_g , MM)

$$I(q) = I(0) \exp\left(-\frac{q^2 R_g^2}{3}\right) \quad \ln I(q) = \ln I(0) - \frac{R_g^2}{3} q^2$$

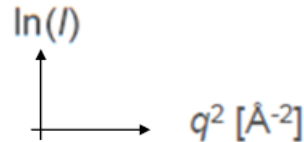
- Plot $\ln I$ vs. q^2
- Inner part will be a straight line
- Slope proportional to R_g^2 slope = $-R_g^2/3$
- IMPORTANT! Only valid near $q = 0$
- Usual limit: $R_g q_{\max} < 1.3$



SMALL ANGLE X-RAY SCATTERING - SAXS

International SILS School
Muggia, 01 / 10 / 2026

- Guinier plot (R_g , MM)



$$I(q) = I(0) \exp\left(-\frac{q^2 R_g^2}{3}\right) \quad \ln I(q) = \ln I(0) - \frac{R_g^2}{3} q^2$$

$$\text{slope} = -R_g^2/3$$

$$\text{intercept} = \ln I(0)$$

$$I(0) \propto M^2$$

$$\frac{I(0)_{\text{sample}}}{I(0)_{\text{standard}}} \approx \left(\frac{c_{\text{sample}} M_{\text{sample}}}{c_{\text{standard}} M_{\text{standard}}} \right)^2 \quad \text{where } c \text{ is concentration}$$

$$M_{\text{sample}} = M_{\text{standard}} \frac{c_{\text{standard}}}{c_{\text{sample}}} \sqrt{\frac{I(0)_{\text{sample}}}{I(0)_{\text{standard}}}}$$

Table 1

Macromolecular properties extracted from SAXS and SEC-SAXS data.

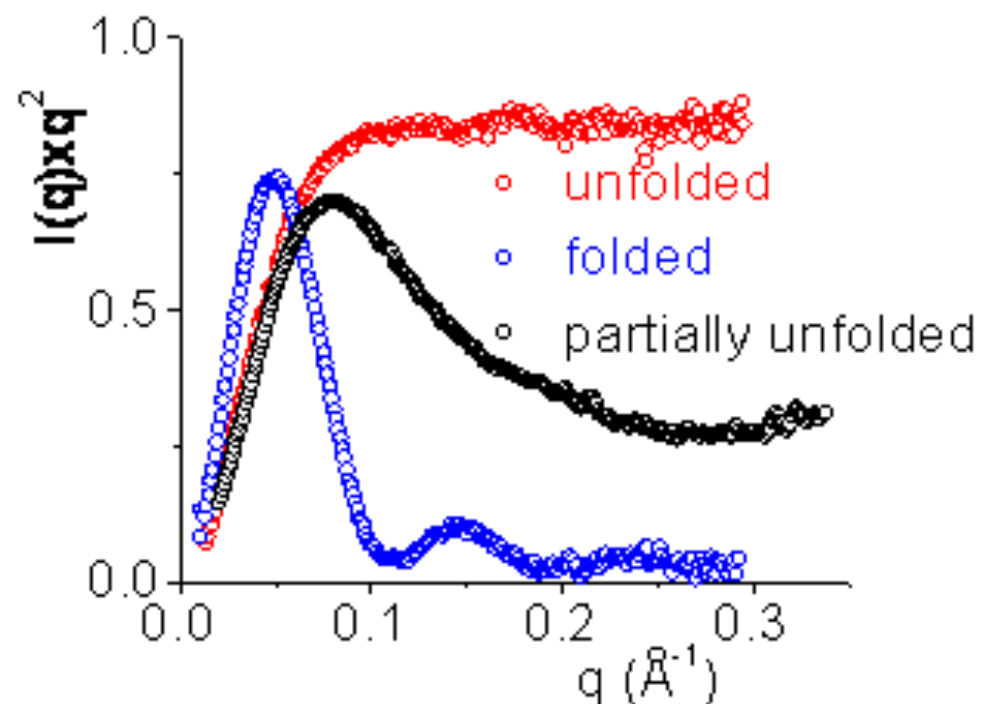
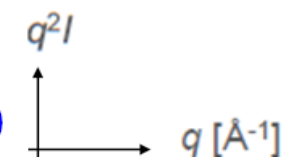
The experimental technique used for each dataset (batch SAXS or SEC-SAXS) is explicitly indicated in the second column, together with main experimental parameters: SDI, exposure time per frame and beam collimation. Comparative references to the SASBDB codes are provided, together with the expected molecular weight (MW_{exp}) and radius of gyration (Rg_{exp}) to validate the in-house measurements against structural data. SAXS profiles were analyzed by *ATSAS 4.1*, including *DAMMIF* *ab initio* modeling. The *ATSAS* package allows determination of the Rg value (here Rg_{data}) and different methods for the MW evaluation. Here, we report the evaluated MW_{data} along with its probability. In the 'Raw Data' column, black circles represent the experimental scattering data, while the red solid lines correspond to the fitted scattering curves.

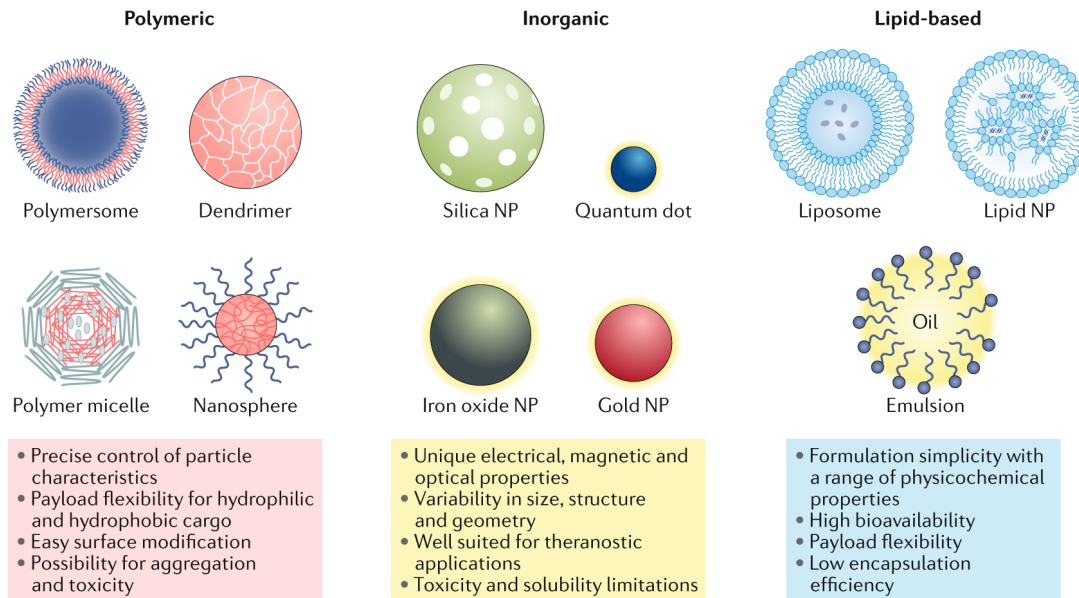
Protein (concentration)	Technique and Experimental Parameters	SASBDB code MW_{exp} (KDa) Rg_{exp} (nm)	MW_{data} (KDa) (Probability) Rg_{data} (nm)	BioSAXS Raw data and Fits	Pair Distance Distribution P(R)	DAMMIF
Lysozyme (6 mg/ml)	Batch SAXS 350 mm 300 s 0.9 x 0.9 mm ²	SASDUE4 MW_{exp} = 14 Rg_{exp} = 1.5	MW_{data} = 12 (55%) Rg_{data} = 1.5			
Carbonic Anhydrase II (7 mg/ml)	Batch SAXS 600 mm 60 s 1.3 x 1.3 mm ²	SASDFP8 MW_{exp} = 29 Rg_{exp} = 1.8	MW_{data} = 24 (31%) Rg_{data} = 1.9			
Human Insulin, Hexamer (2.5 mg/ml)	Batch SAXS 900 mm 60 s 0.9 x 0.9 mm ²	SASDE25 MW_{exp} = 35 Rg_{exp} = 2.0	MW_{data} = 29 (30%) Rg_{data} = 2.1			
Apo ferritin (10 mg/ml)	Batch SAXS 900 mm 60 s 0.9 x 0.9 mm ²	SASDE55 MW_{exp} = 476 Rg_{exp} = 5.1	MW_{data} = 318 (55%) Rg_{data} = 5.2			
Bovine Serum Albumin (injected 10 mg/ml), Monomer	SEC-SAXS 600 mm 30 s 1.3 x 1.3 mm ²	SASDBT4 MW_{exp} = 66 Rg_{exp} = 2.7	MW_{data} = 76 (32%) Rg_{data} = 2.8			
Bovine Serum Albumin (injected 10 mg/ml), Dimer	SEC-SAXS 600 mm 30 s 1.3 x 1.3 mm ²	SASDFR8 MW_{exp} = 133 Rg_{exp} = 4.0	MW_{data} = 208 (90%) Rg_{data} = 4.2			

SMALL ANGLE X-RAY SCATTERING - SAXS

International SILS School
Muggia, 01 / 10 / 2026

- Kratky plot (flexibility)





Drug delivery nanoparticles must be characterized in:
sizes, shapes, and morphologies

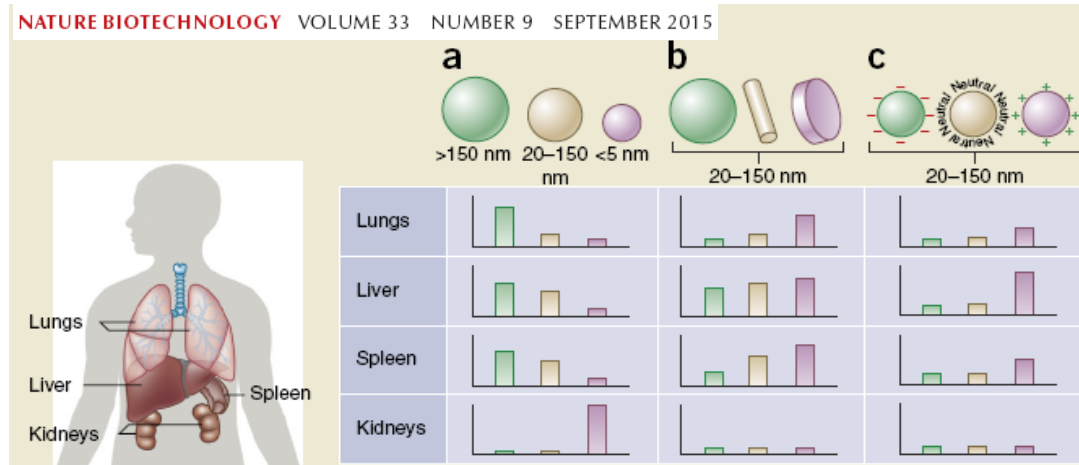
Review Article | Published: 04 December 2020

Engineering precision nanoparticles for drug delivery

[Michael J. Mitchell](#) , [Margaret M. Billingsley](#), [Rebecca M. Haley](#), [Marissa E. Wechsler](#), [Nicholas A. Peppas](#)

& [Robert Langer](#) 

Nature Reviews Drug Discovery **20**, 101–124 (2021) | [Cite this article](#)

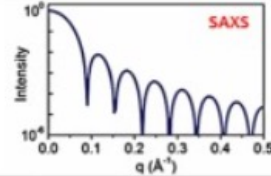
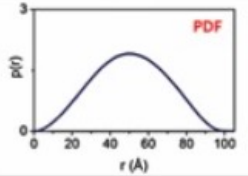
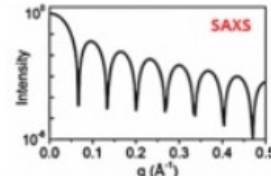
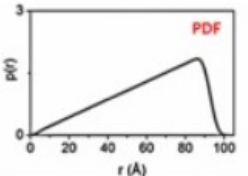
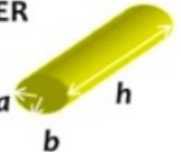
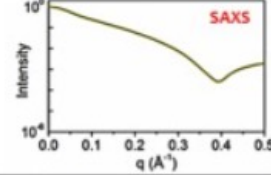
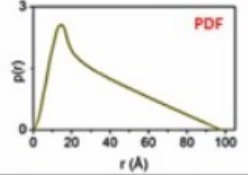
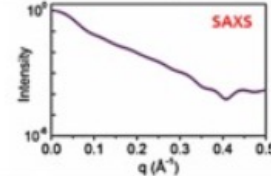
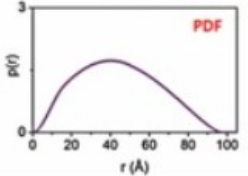
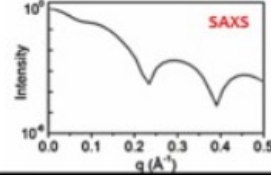
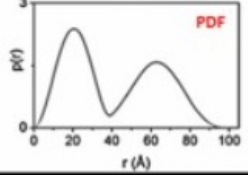


Size, structure and morphology play a crucial role in their functionality.

Characterizing their loaded and unloaded size and shape as a function of relevant human body parameters (e.g. pH or temperature), is therefore critical, especially if the characterization can be performed simulating the same condition of drug delivery, i.e. **dispersed in a buffer which is often water.**

SMALL ANGLE X-RAY SCATTERING - SAXS

International SILS School
Muggia, 01 / 10 / 2026

SPHERE 			$R_g^2 = \frac{3}{5} R^2$
HOLLOW SPHERE 			$R_g^2 = \frac{3 R_2^5 - R_1^5}{5 R_2^3 - R_1^3}$
CYLINDER 			$R_g^2 = \frac{a^2 + b^2}{4} + \frac{h^2}{12}$
FLAT DISK 			$R_g^2 = \frac{R^2}{2} + \frac{h^2}{12}$
DUMB BELLS 			$R_g^2 = \frac{\int_0^{d_{max}} P(r) r^2 dr}{\int_0^{d_{max}} P(r) dr}$



Progress in Materials Science
Volume 112, July 2020, 100667



Watching nanomaterials with X-ray eyes: Probing different length scales by combining scattering with spectroscopy

Cinzia Giannini^{a,*,1}, Vaclav Holy^{b,c,1}, Liberato De Caro^a, Lorenzo Mino^d, Carlo Lamberti^{b,1}

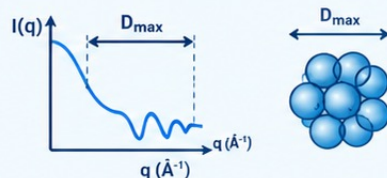
What can we extract from SAXS?

From the scattering pattern to the structural information

D_{max}

Maximum particle size

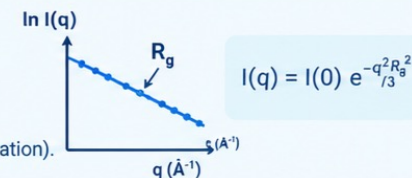
Provides the largest dimension of the scattering objects (e.g., particles, aggregates, pores).



R_g / I(0)

Radius of gyration / forward scattering intensity

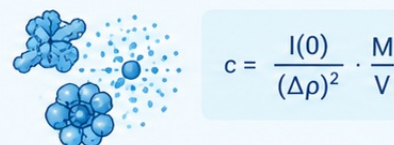
Provides the global size and shape of the particles and the scattering intensity at zero angle (related to concentration).



M/V

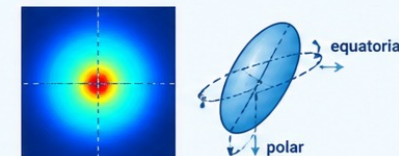
Mass / volume

Determines the amount of scattering material and allows the estimation of protein and nano-object characterization (e.g., concentration, molecular weight).



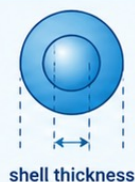
Ellipticity

Reveals the particle shape through the ratio of scattering in different directions (polar/equatorial asymmetry).



Shell thickness

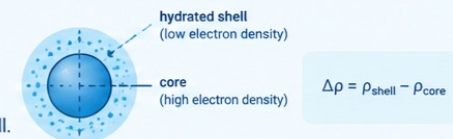
Gives information on the thickness of core-shell structures (e.g., micelles, vesicles, nanoparticles).



Δρ_{core} / Δρ_{shell}

Hydration fraction

Provides the fraction of hydration of the particle, related to the contrast difference between the core and the shell.



Aggregation number

Gives the number of monomers in self-assembled structures (e.g., micelles, nanoparticles).



JOURNAL OF
APPLIED
CRYSTALLOGRAPHY

ISSN 1600-5767

Shannon sampling based approach for the structural solution of nano-objects by laboratory and synchrotron SAXS data

Liberato De Caro,^{a*} Domenico Alberga,^a Francesco Scattarella,^a Teresa Sibillano,^a Vincenzo Mangini,^a Boyang Zhou,^b Thibaud Stoll^b and Cinzia Giannini^{a*}

“EUROPEAN SYNCHROTRON SAXS BEAMLINE LANDSCAPE (UPDATED)”



ESRF (European Synchrotron Radiation Facility)
Grenoble, France



ID02 – SAXS
(Structural Biology, BioSAXS)
BM26 – SAXS/WAXS
(High-throughput)
BM29 – BioSAXS
(Biological macromolecules)



SOLEIL Synchrotron
Saint-Aubin, France



SWING
(SAXS)
Solution scattering
& in situ experiments



ALBA, Barcelona, Spain



BL11 – NCD-SWEET
(SAXS/WAXS/GISAXS/GIWAXS)
Non-Crystalline Diffraction



Diamond Diamond Light Source
Didcot, UK



B21 – SAXS
(Biological and soft matter)
I22 – SAXS/WAXS
(materials, polymers)



MAX IV Laboratory,
Lund, Sweden



CoSAXS
(Coherent SAXS/WAXS)
BioSAXS, time-resolved SAXS,
XPCS



DESY / PETRA III
Hamburg, Germany



P03 – MINaXS
(U)SAXS, (GI)WAXS, GISAXS
P12 – BioSAXS
(biological samples)
P62 – SAXSMAT
(SAXS/WAXS, in situ/in operando,
high-throughput)



Elettra Sincrotrone Trieste, Trieste, Italy



5.2L – SAXS
(SAXS / GISAXS)
Materials & biological systems



Synchrotron Ring

X-ray Beam



Sample Stage



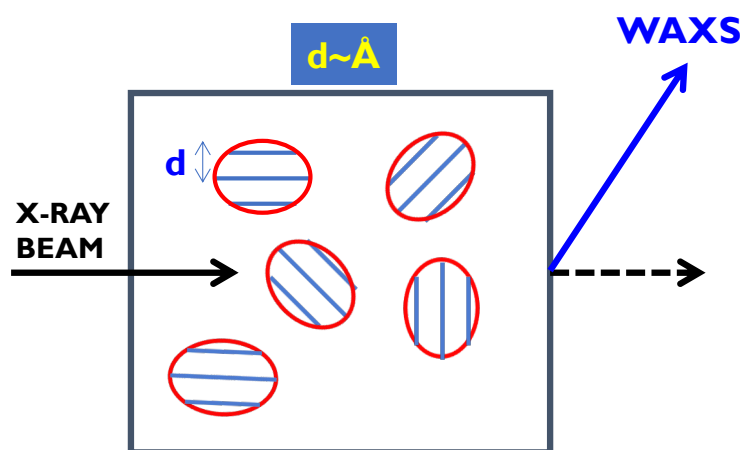
Detector

Beamline Labels



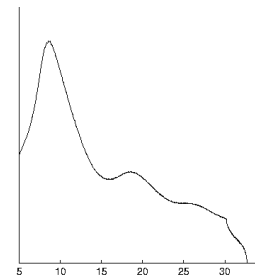
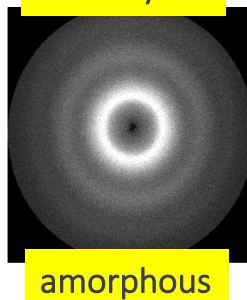
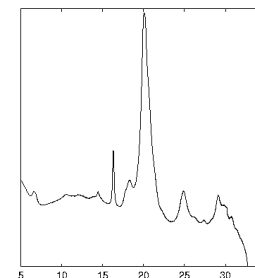
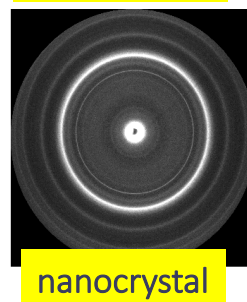
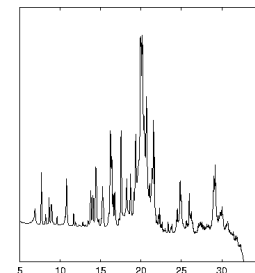
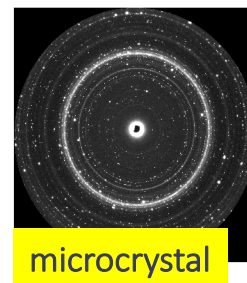
WIDE ANGLE X-RAY SCATTERING - WAXS

International SILS School
Muggia, 01 / 10 / 2026



$$\sin\theta = \lambda / 2d$$

Large $\theta \rightarrow$ small d
Small $\theta \rightarrow$ large d



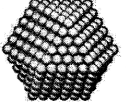
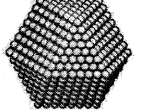
OUTLINE

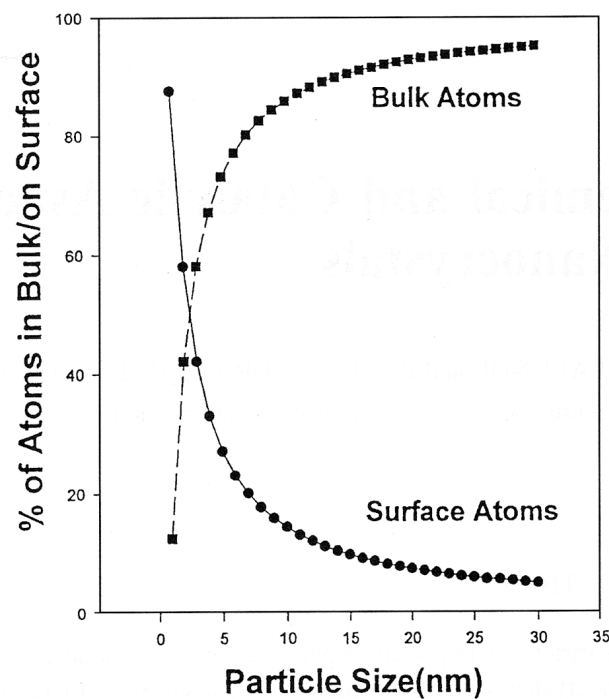
International SILS School
Muggia, 01 / 10 / 2026

- SAXS – WAXS – Basic notions
- Scanning SAXS- WAXS Microscopy
- **Nanocrystals** and Nanocrystal-based Superlattices
- Scaffolds; Fibers

WIDE ANGLE X-RAY SCATTERING - WAXS

International SILS School
Muggia, 01 / 10 / 2026

Full-shell Clusters		Total Number of Atoms	Surface Atoms (%)
1 Shell		13	92
2 Shells		55	76
3 Shells		147	63
4 Shells		309	52
5 Shells		561	45
7 Shells		1415	35



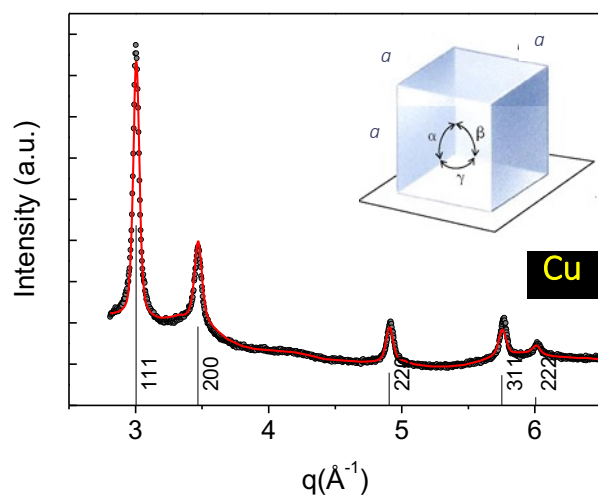
Nanoscale Materials in Chemistry,
Ed. K.J. Klabunde, Wiley, 2001

Kenneth J. Klabunde et al,
Nanocrystals as Stoichiometric Reagents
with Unique Surface Chemistry
J. Phys. Chem. 1996, 100, 12142-12153

WIDE ANGLE X-RAY SCATTERING - WAXS

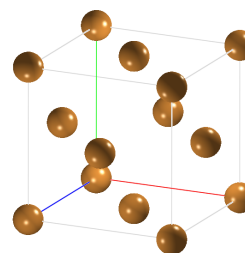
International SILS School
Muggia, 01 / 10 / 2026

What is the structure?



15.9 ± 0.5 along [1 1 1]

9.5 ± 0.5 along [2 0 0]

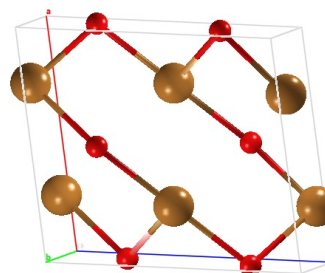
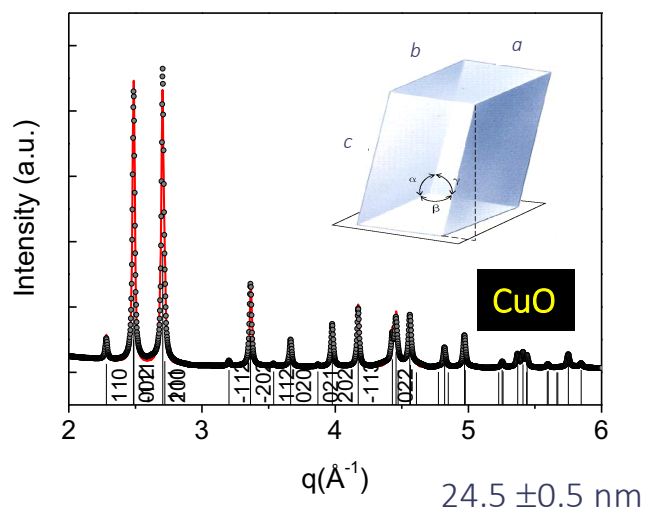


Identifier	9012043
Literature Reference	Otte, H. M., <i>Journal of Applied Physics</i> (1961), 32 , 1536
Formula	Cu
Compound Name	Copper
Synonym	
Space Group	F m -3 m
Cell Lengths	a 3.6150 b 3.6150 c 3.6150
Cell Angles	α 90 β 90 γ 90
Cell Volume	47.2416

WIDE ANGLE X-RAY SCATTERING - WAXS

International SILS School
Muggia, 01 / 10 / 2026

What is the structure?



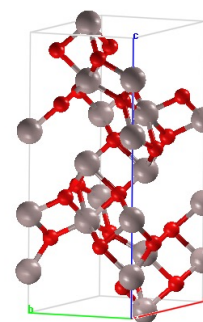
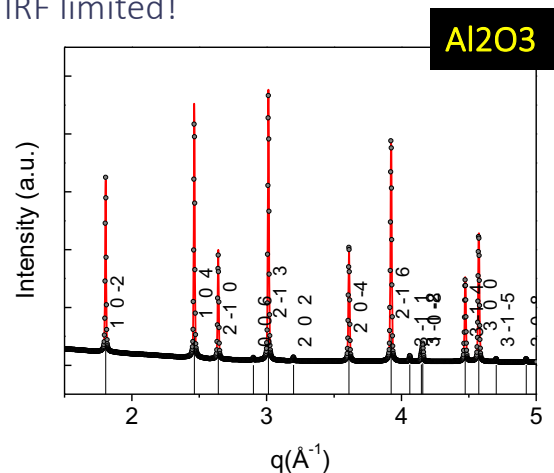
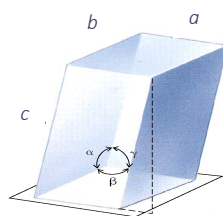
Identifier	9015924
Literature Reference	Calos, N. J., Forrester, J. S., Schaffer, G. B., <i>Journal of Solid State Chemistry</i> (1996), 122 , 273
Formula	Cu O
Compound Name	
Synonym	
Space Group	C 2/c
Cell Lengths	a 4.6832 b 3.4288 c 5.1297
Cell Angles	α 90 β 99.3086 γ 90
Cell Volume	81.2868

WIDE ANGLE X-RAY SCATTERING - WAXS

International SILS School
Muggia, 01 / 10 / 2026

What is the structure?

>100nm
IRF limited!

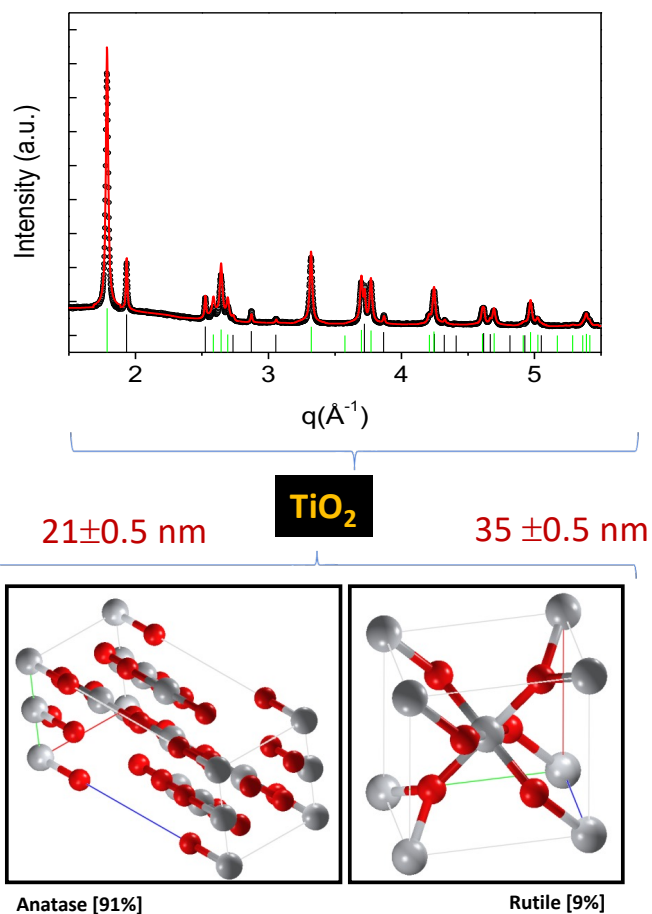


Identifier	9008081
Literature Reference	Newnham, R. E., de Haan, Y. M., <i>Zeitschrift für Kristallographie</i> (1962), 117 , 235
Formula	Al ₂ O ₃
Compound Name	
Synonym	
Space Group	R -3 c
Cell Lengths	a 4.7589 b 4.7589 c 12.991
Cell Angles	α 90 β 90 γ 120
Cell Volume	254.792
Z, Z'	Z: 0 Z': 0

WIDE ANGLE X-RAY SCATTERING - WAXS

International SILS School
Muggia, 01 / 10 / 2026

What is the structure?



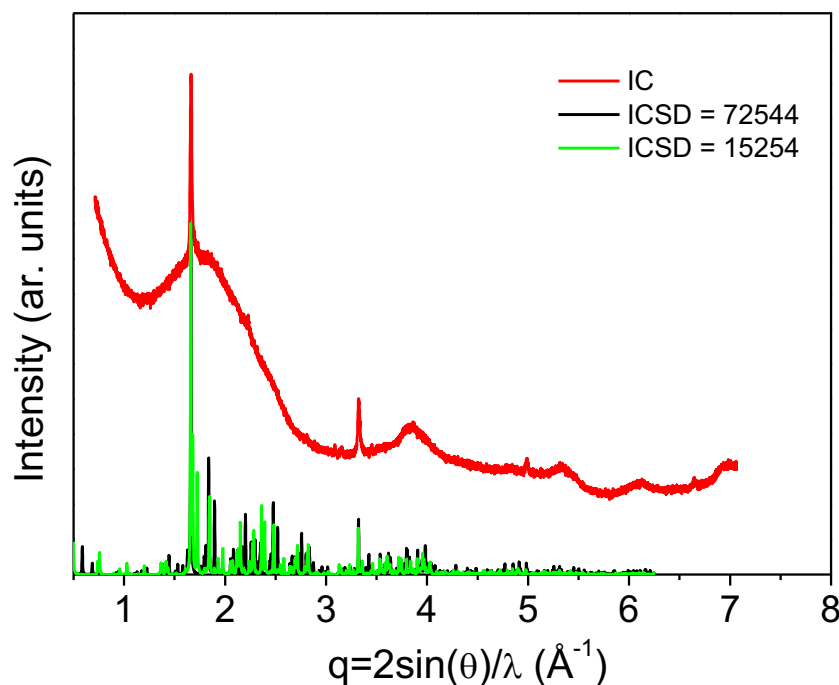
Identifier	7206075
Literature Reference	Rezaee, Masih, Mousavi Khoie, Seyyed Mohammad, Liu, Kun Hua, <i>CrystEngComm</i> (2011), 13 , 5055
Formula	$\text{O}_2 \text{ Ti}$
Compound Name	Titanium oxide - anatase
Synonym	
Space Group	$I 4_1/a m d$
Cell Lengths	a 3.7850 b 3.7850 c 9.5196
Cell Angles	α 90 β 90 γ 90
Cell Volume	136.38

Identifier	9007432
Literature Reference	Baur, W. H., <i>Acta Crystallographica</i> (1956), 9 , 515
Formula	$\text{O}_2 \text{ Ti}$
Compound Name	rutile
Synonym	
Space Group	$P 4_2/m n m$
Cell Lengths	a 4.594 b 4.594 c 2.959
Cell Angles	α 90 β 90 γ 90
Cell Volume	62.4492

WIDE ANGLE X-RAY SCATTERING - WAXS

International SILS School
Muggia, 01 / 10 / 2026

What is the structure?



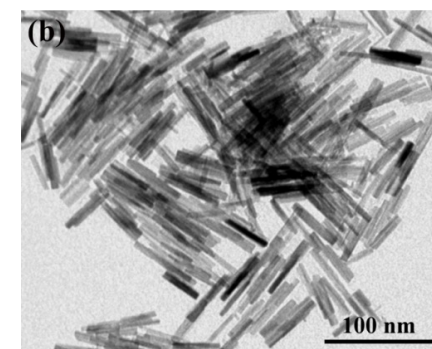
Data can be explained
either as:

$\text{W}_{18}\text{O}_{49}$ (ICSD=15254)

or

$\text{WO}_{2.626}$ (ICSD=72544)

phases.



WIDE ANGLE X-RAY SCATTERING - WAXS

International SILS School
Muggia, 01 / 10 / 2026

What is the structure?

72544-ICSD	
Current structure: 72544-ICSD	
Customise...	
Structure	
Diagram	
Atoms	
Bonds	
Contacts	
Centroids	
Planes	
Symmetry	
Distances	
Angles	
Torsions	
All Angles	
All Torsions	
Close	

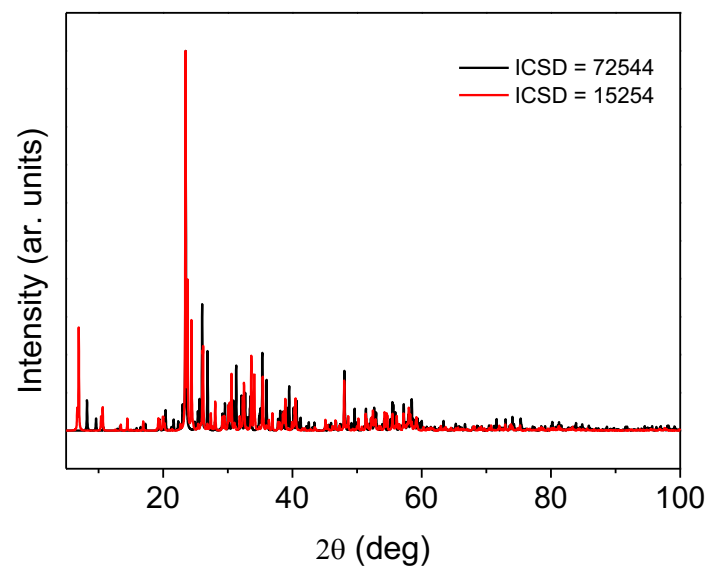
Identifier	72544-ICSD
Literature Reference	Barabanenkov, Yu.A.;Zakharov, N.D.;Zibrov, I.P.;Filonenko, V.P.;Werner, P.;Popov, A.I.;Val'kovskij, M.D., <i>Unknown</i> (0)
Formula	O _{2.625} W
Compound Name	Tungsten Oxide (1/2.6)
Synonym	
Space Group	P b a m
Cell Lengths	a 21.431(9) b 17.766(7) c 3.783(2)
Cell Angles	α 90 β 90 γ 90
Cell Volume	1440.35
Z, Z'	Z: 32 Z': 0
R-Factor (%)	7.5

WO_{2.626} (ICSD=72544)
orthorhombic

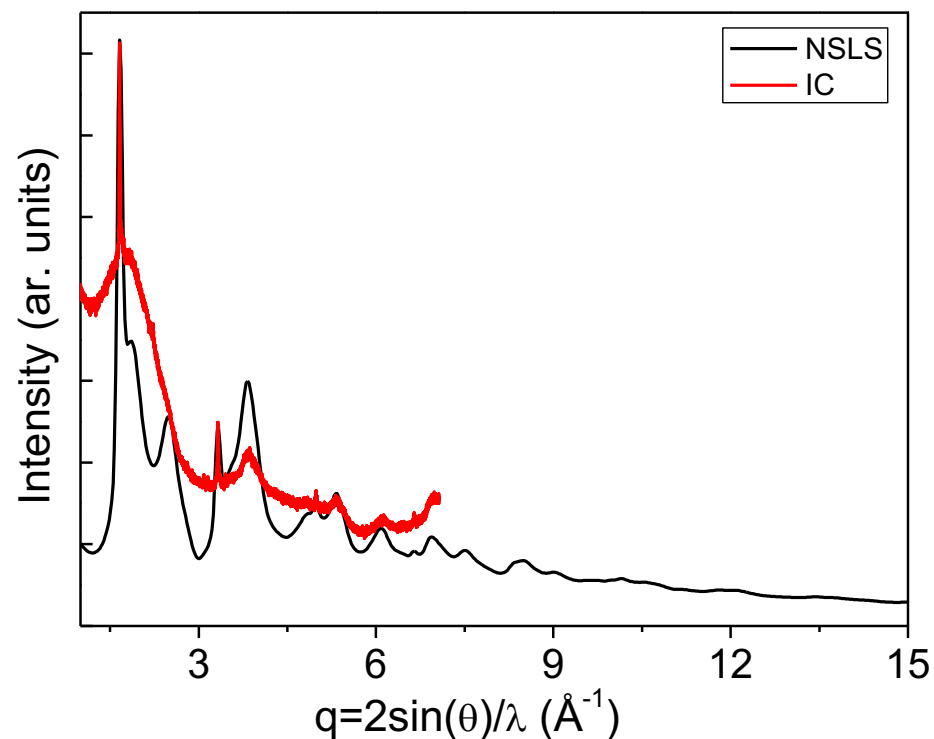
15254-ICSD	
Current structure: 15254-ICSD	
Customise...	
Structure	
Diagram	
Atoms	
Bonds	
Contacts	
Centroids	
Planes	
Symmetry	
Distances	
Angles	
Torsions	
All Angles	
All Torsions	
Close	

Identifier	15254-ICSD
Literature Reference	Viswanathan, K.;Brandt, K.;Salje, E., <i>Unknown</i> (0)
Formula	O ₄₉ W ₁₈
Compound Name	Tungsten Oxide (18/49)
Synonym	
Space Group	P 2/m
Cell Lengths	a 18.334 b 3.786 c 14.044
Cell Angles	α 90 β 115.2 γ 90
Cell Volume	882.052
Z, Z'	Z: 1 Z': 0
R-Factor (%)	6.5
Disorder	
Polymorph	

W₁₈O₄₉ (ICSD=15254)
monoclinic



What is the structure?

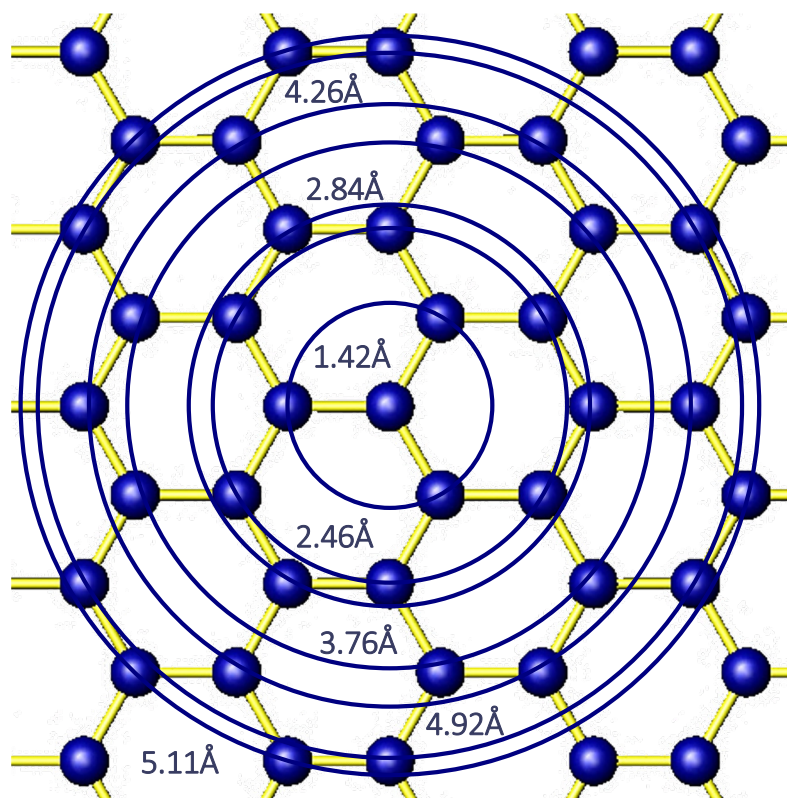


The XPD data collected at IC (IC-XPD) were acquired with a Bruker D8-Discover (3.3 kW) diffractometer equipped with a Cu source (8KeV, $\lambda=1.540562 \text{ \AA}$)

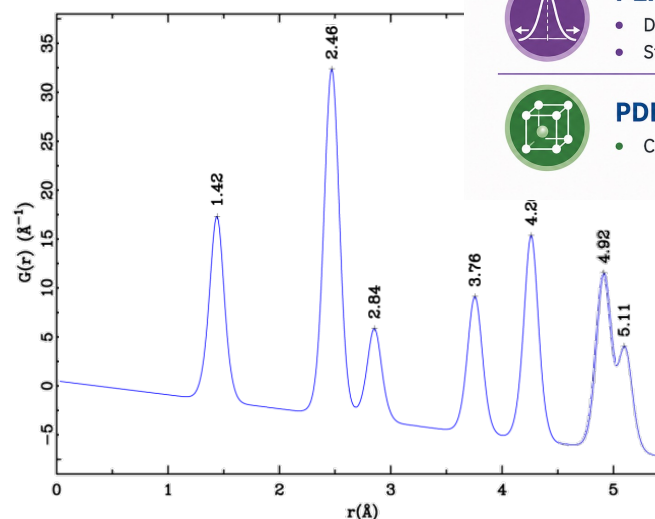
The NSLS data (NSLS-XPD) were measured using X-ray radiation with an energy of 66.7 keV ($\lambda=0.18597 \text{ \AA}$).

WIDE ANGLE X-RAY SCATTERING – WAXS/PDF

International SILS School
Muggia, 01 / 10 / 2026



Pair distribution function (PDF) gives the probability of finding an atom at a distance “ r ” from a given atom.



PEAK POSITIONS

- Bond lengths, interatomic distances



PEAK AREAS

- Coordination number



PEAK WIDTHS

- Dynamic disorder (ADPs)
- Static disorder



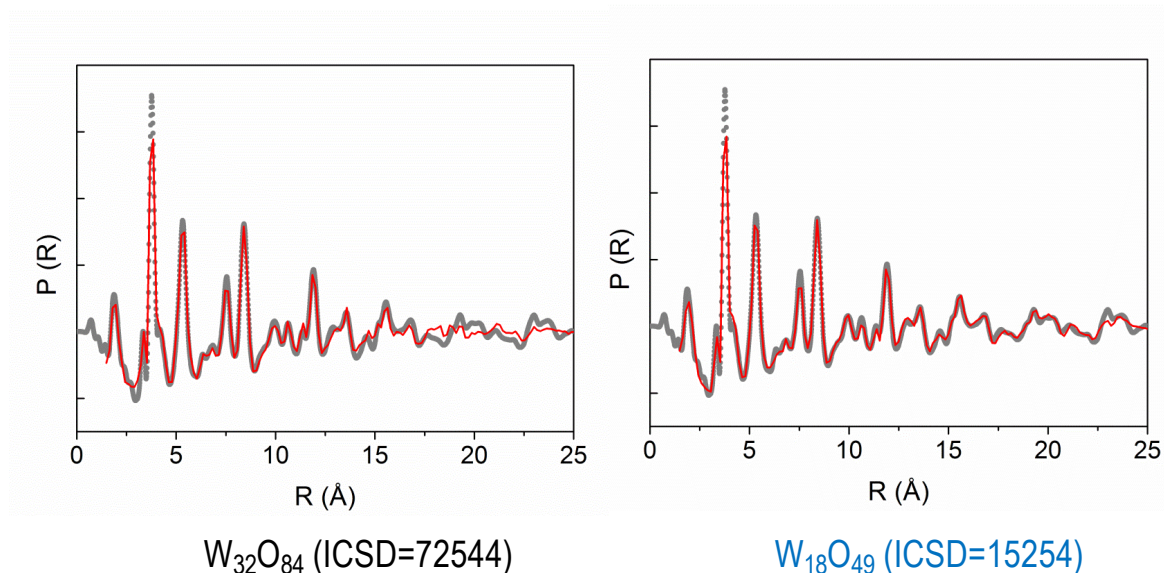
PDF PEAK DAMPING

- Crystallite size

WIDE ANGLE X-RAY SCATTERING – WAXS/PDF

International SILS School
Muggia, 01 / 10 / 2026

What is the structure?



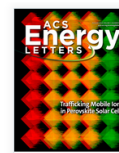
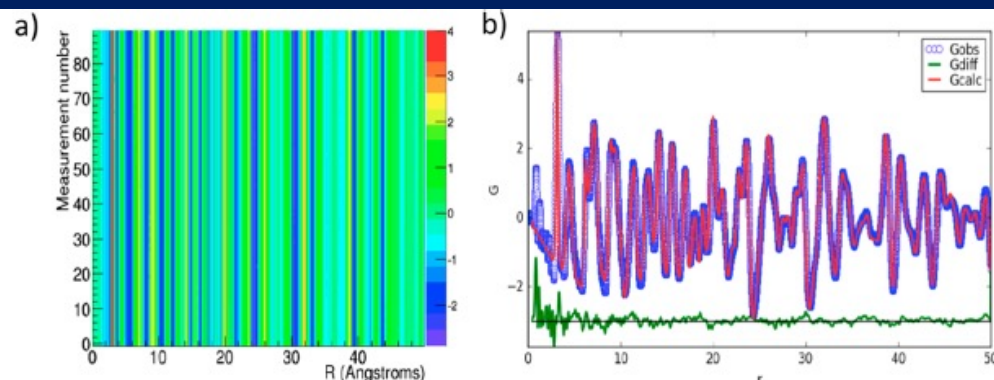
Results of the PDF data allowed to identify the **monoclinic $W_{18}O_{49}$ crystal phase** (ICSD # 15254); fitting proved that the actual stoichiometry was **$W_{16\pm0.4}O_{45\pm3}$**



NSLS data (NSLS-XPD) @ 66.7 keV

WIDE ANGLE X-RAY SCATTERING – WAXS/PDF

International SILS School
Muggia, 01 / 10 / 2026



Light-Induced Formation of Pb^{3+} Paramagnetic Species in Lead Halide Perovskites

Silvia Colella, Michela Todaro, Sofia Masi, Andrea Listorti, Davide Altamura, Rocco Caliendo*, Cinzia Giannini, Elisa Carignani, Marco Geppi, Daniele Meggiolaro, Gianpiero Buscarino*, Filippo De Angelis* and Aurora Rizzo

Cite This: *ACS Energy Lett.* 2018, 3, 8, 1840-1847

Publication Date: July 6, 2018

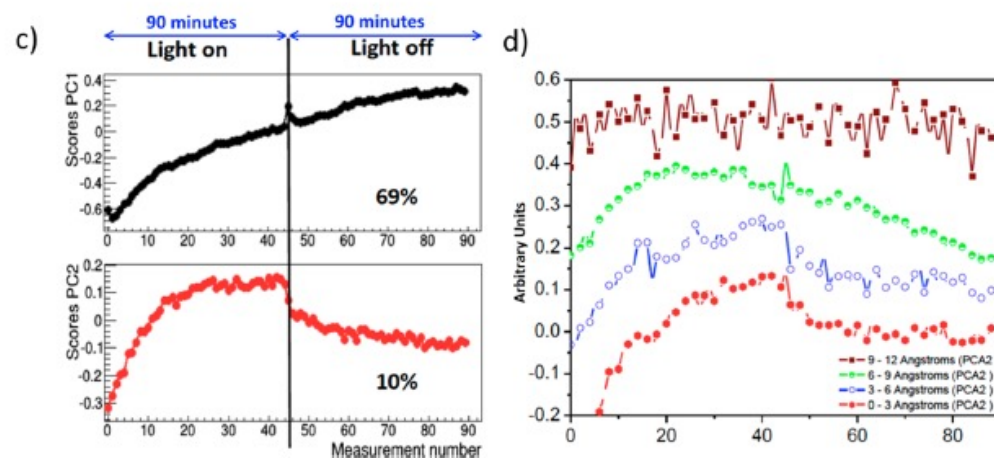
<https://doi.org/10.1021/acsenerylett.8b00944>

Copyright © 2018 American Chemical Society

ACS Energy Letters

Tetragonal MAPbI_3 (methyammonium (MA)).

Principal component analysis (PCA) on the PDF data matrix :

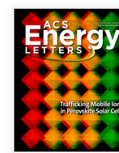


1. the first principal component (PC1) shows a monotonic trend not intrinsically related to illumination as it continues when the light is off. It captures structural variations induced by the X-ray illumination, as lattice distortion and thermal atomic fluctuations.
2. the second principal component (PC2) shows a light-related trend, with a steady increase when the light is on and a decrease (with a smaller time constant) when the light is off.

Figure 3. (a) PDF data matrix: the intensity is on a color scale. (b) PDF from the data matrix fitted by the MAPbI_3 tetragonal phase model. (c) Scores of first (PC1) and second (PC2) principal components resulting from PCA applied to the PDF data matrix. The percentage of explained variance by each component is reported. (d) PC2 scores obtained by PCA applied to the PDF data matrix in slices of 3 Å, for increasing interatomic distances. PC2 scores obtained for $R > 12$ Å show the same trend of those in the slice at 9–12 Å.

WIDE ANGLE X-RAY SCATTERING – WAXS/PDF

International SILS School
Muggia, 01 / 10 / 2026



Light-Induced Formation of Pb^{3+} Paramagnetic Species in Lead Halide Perovskites

Silvia Colella, Michela Todaro, Sofia Masi, Andrea Listorti, Davide Altamura, Rocco Caliendo*, Cinzia Giannini, Elisa Carignani, Marco Geppi, Daniele Meggiolaro, Gianpiero Buscarino*, Filippo De Angelis* and Aurora Rizzo

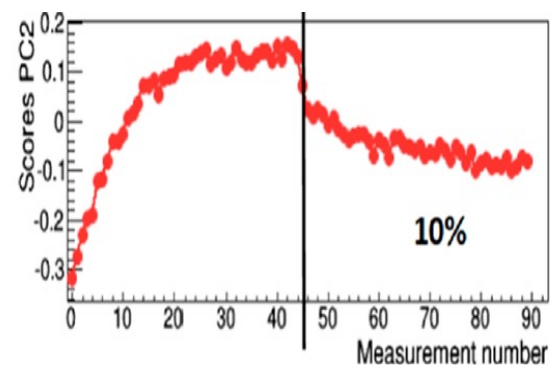
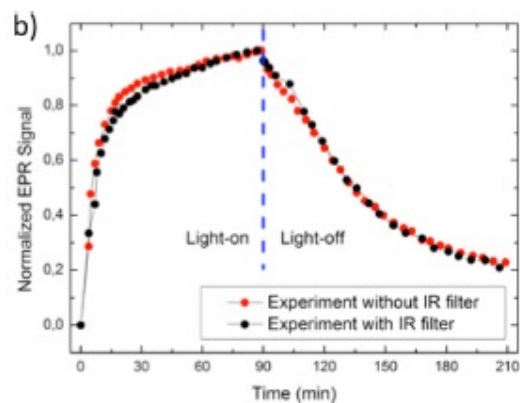
Cite This: *ACS Energy Lett.* 2018, 3, 8, 1840-1847

Publication Date: July 6, 2018

<https://doi.org/10.1021/acsenerylett.8b00944>

Copyright © 2018 American Chemical Society

ACS Energy Letters



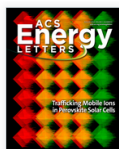
Tetragonal MAPbI_3 (methyammonium (MA)).

Principal component analysis (PCA) on the PDF data matrix :

3. the PC2 trend when the light is on and the recovery time when the light is off closely resembling the trend observed for the electron paramagnetic resonance (EPR) signal.

WIDE ANGLE X-RAY SCATTERING – WAXS/PDF

International SILS School
Muggia, 01 / 10 / 2026



Light-Induced Formation of Pb^{3+} Paramagnetic Species in Lead Halide Perovskites

Silvia Colella, Michela Todaro, Sofia Masi, Andrea Listorti, Davide Altamura, Rocco Calandro*, Cinzia Giannini, Elisa Carignani, Marco Geppi, Daniele Meggiolaro, Gianpiero Buscarino*, Filippo De Angelis* and Aurora Rizzo

Cite This: *ACS Energy Lett.* 2018, 3, 8, 1840-1847

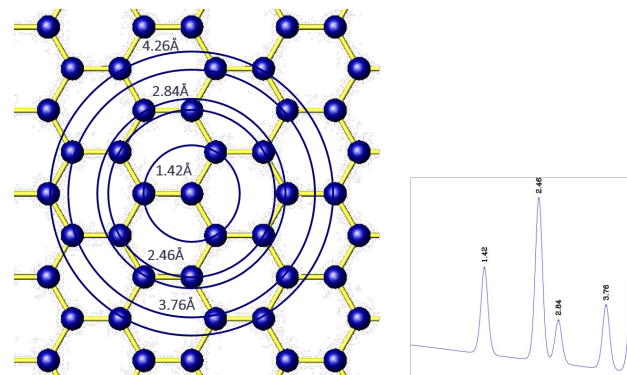
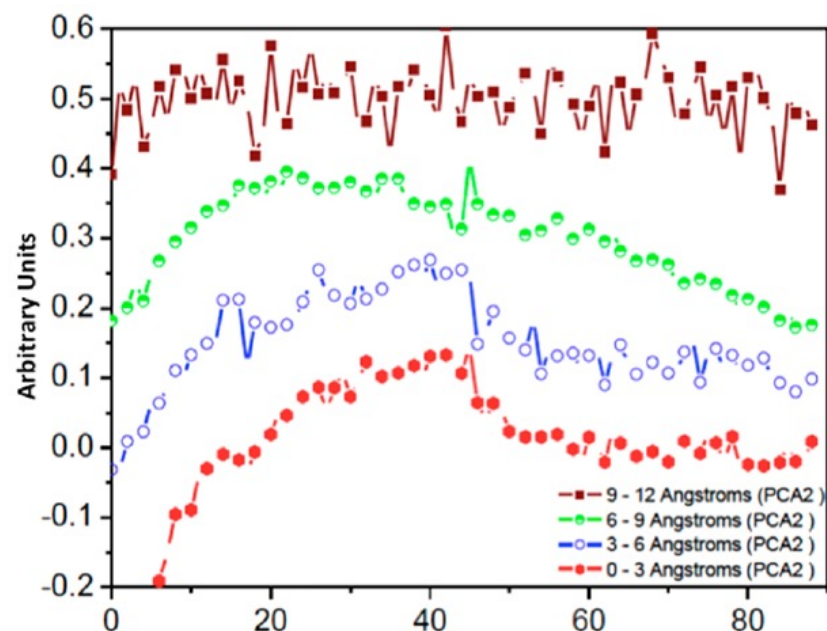
Publication Date: July 6, 2018

<https://doi.org/10.1021/acsenenerglett.8b00944>

Copyright © 2018 American Chemical Society

ACS Energy Letters

Tetragonal MAPbI_3 (methylammonium (MA)).



Principal component analysis (PCA) on the PDF data matrix :

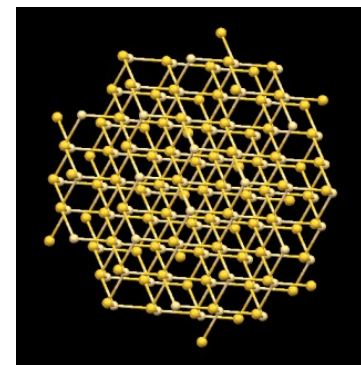
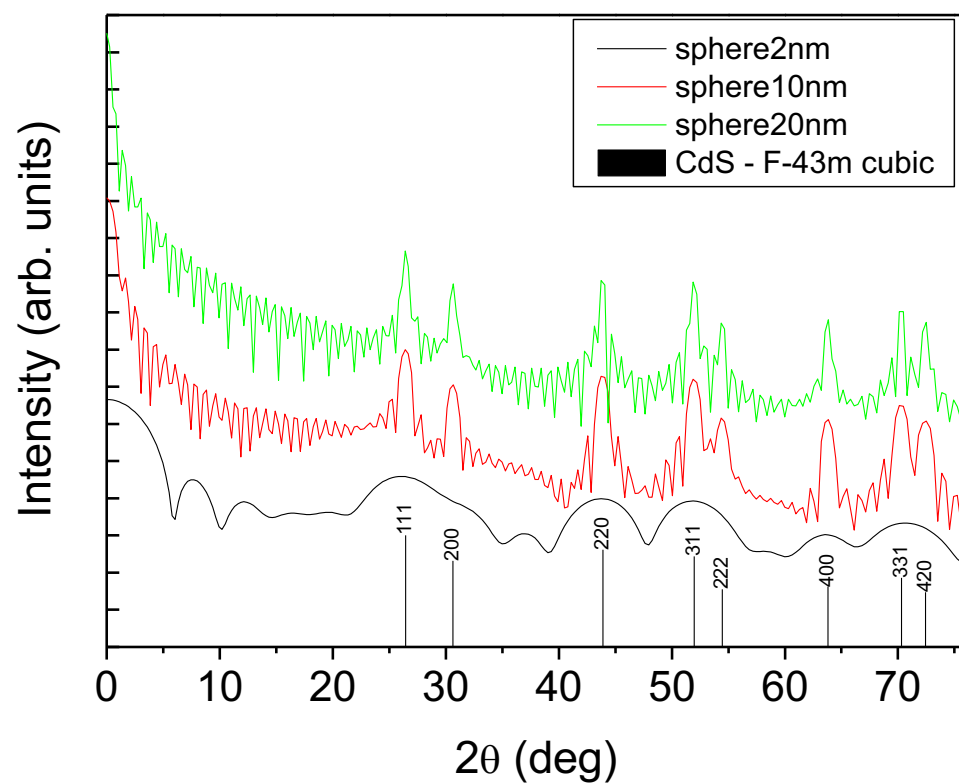
4. PCA was applied to the PDF data matrix in slices of 3 Å, for increasing interatomic distances. Notably, the resulting PC2 scores showed a **higher light-induced effect for the shorter interatomic distances ($R < 3$ Å)** and a progressive decrease for longer distances, until vanishing for $R > 9$ Å.

The considered range of distances (< 3 Å) is **consistent with the range of Pb–O distances (2.25 Å)**. Such distances are likely correlated with generation of paramagnetic species induced by illumination (**charge-killing defects in a range of distances compatible with Pb–O bonds**).

WIDE ANGLE X-RAY SCATTERING - WAXS

International SILS School
Muggia, 01 / 10 / 2026

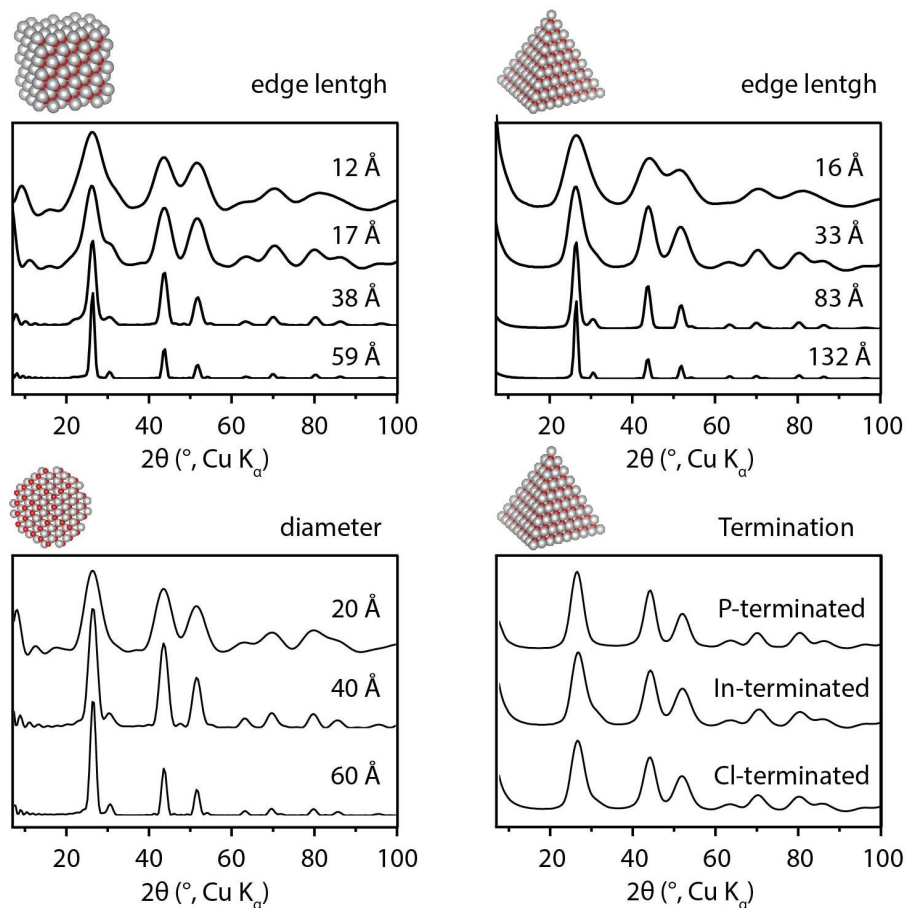
What is the domain size?



WIDE ANGLE X-RAY SCATTERING - WAXS

International SILS School
Muggia, 01 / 10 / 2026

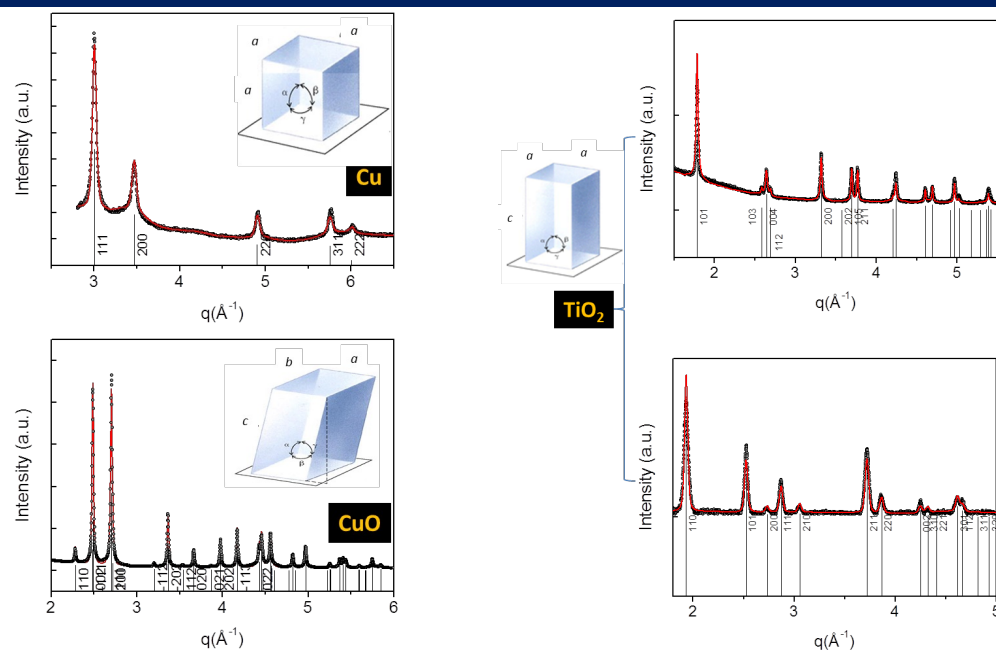
What is the domain size?



WIDE ANGLE X-RAY SCATTERING - WAXS

International SILS School
Muggia, 01 / 10 / 2026

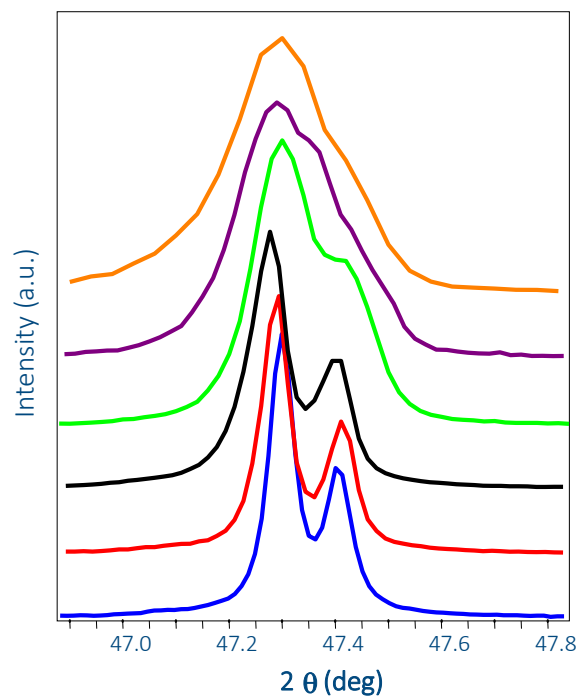
What is the domain size?



WIDE ANGLE X-RAY SCATTERING - **WAXS**

International SILS School
Muggia, 01 / 10 / 2026

What is the domain size?



Patterns collected from the same sample with different instruments and configurations



WIDE ANGLE X-RAY SCATTERING - WAXS

International SILS School
Muggia, 01 / 10 / 2026

Three-step procedure:

1. the instrumental resolution function (IRF)

was evaluated by fitting the XRD pattern of a LaB₆ NIST standard recorded under the same experimental conditions as those used for measuring the powders.

2. the crystal structure models of the crystalline phases previously identified, here tetragonal TiO₂ anatase (space group I4₁/amd;

cell parameters: a=b=3.7835430 Å and c=9.614647 Å; α=β=γ=90°) and tetragonal TiO₂ rutile (space group p 4₂/m n m; cell parameters: a=b = 4.59365 Å; c=2.95874 Å; α= β =γ=90°) were provided to the program.

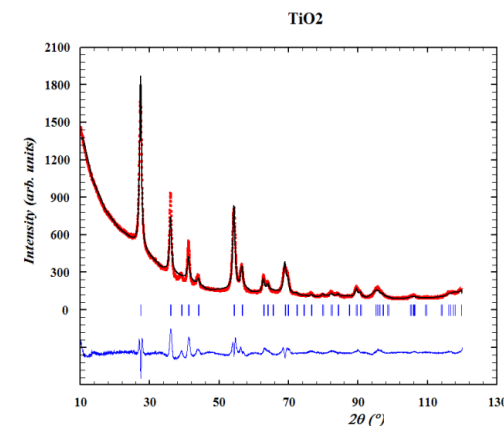
3. the inhomogeneous peak broadening of the XRD pattern reflections was described by a phenomenological model based on a modified Scherrer formula:

$$\beta_{h,k,l} = \frac{\lambda}{D_{h,k,l} \cos \theta} = \frac{\lambda}{\cos \theta} \sum_{imn} a_{imp} y_{imp}(\theta_{h,k,l}, \Phi_{h,k,l})$$

size contribution to the integral width of the (h,k,l) reflection

real
spherical
harmonics

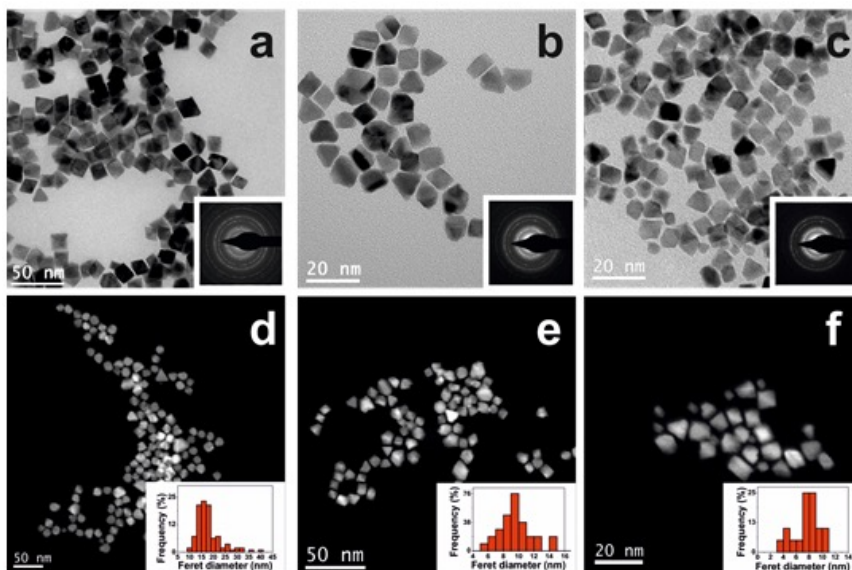
What is the domain size?



WIDE ANGLE X-RAY SCATTERING - WAXS

International SILS School
Muggia, 01 / 10 / 2026

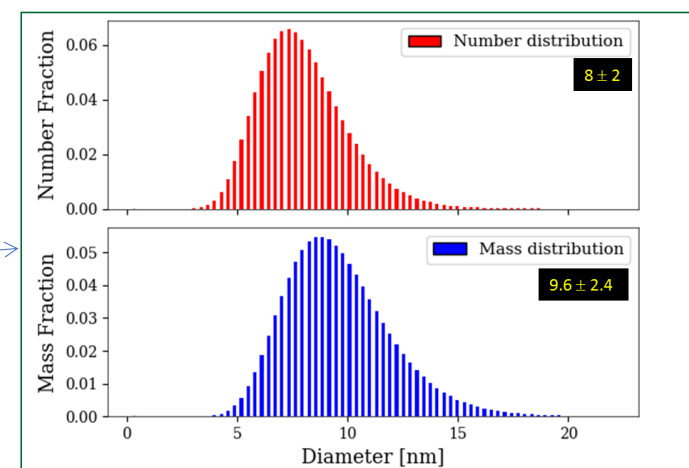
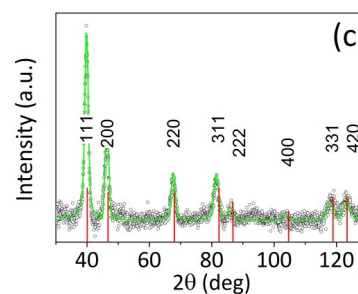
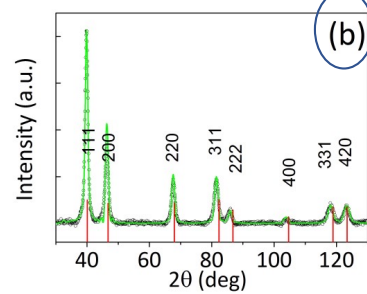
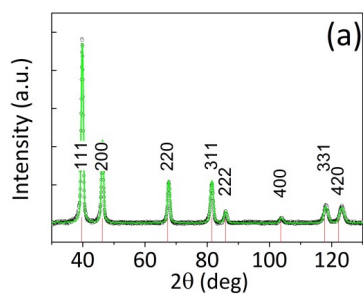
What is the domain size?



Pt NC18: 18 ± 2 nm

Pt NC10: 10 ± 2 nm

Pt NC7: 7 ± 1 nm

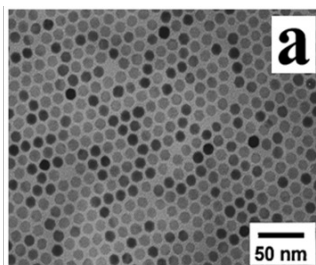


WIDE ANGLE X-RAY SCATTERING - **WAXS**

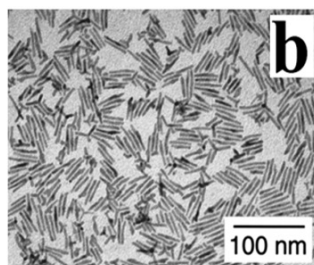
International SILS School
Muggia, 01 / 10 / 2026

What is the size/shape?

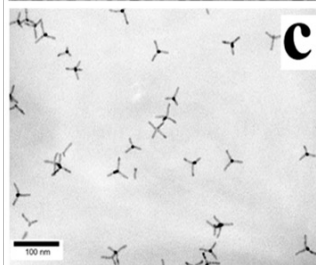
γ -Fe₂O₃ spheres



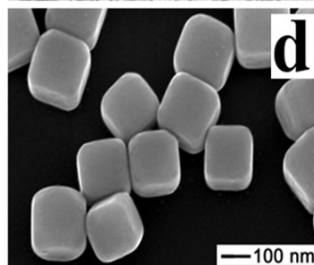
CdSe nanorods



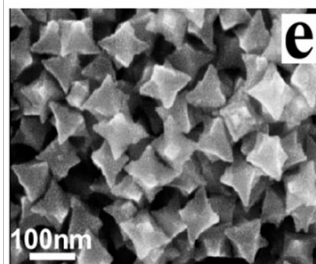
CdTe tetrapods



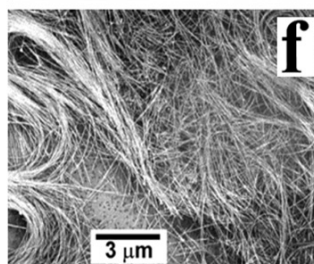
Ag nanocubes



PbSe stars



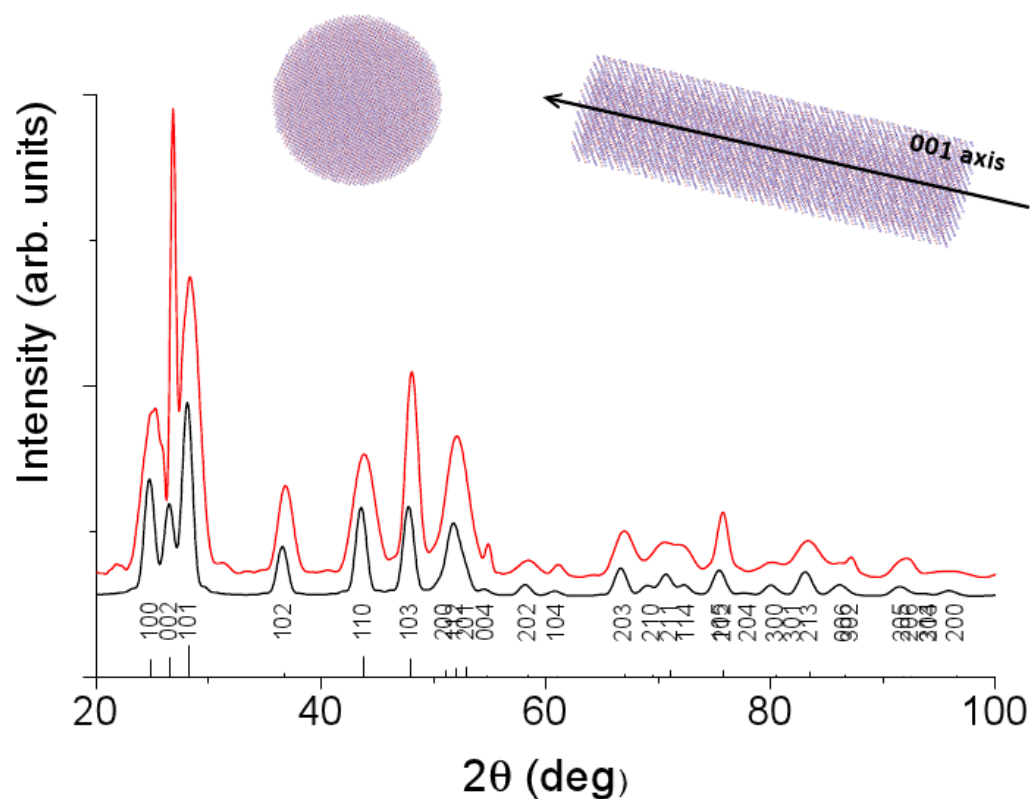
PbSe nanowires



WIDE ANGLE X-RAY SCATTERING - WAXS

International SILS School
Muggia, 01 / 10 / 2026

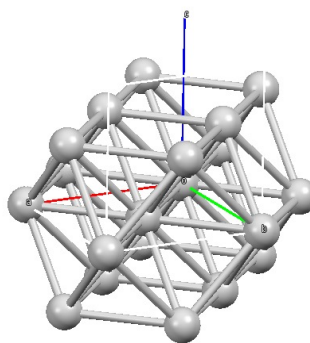
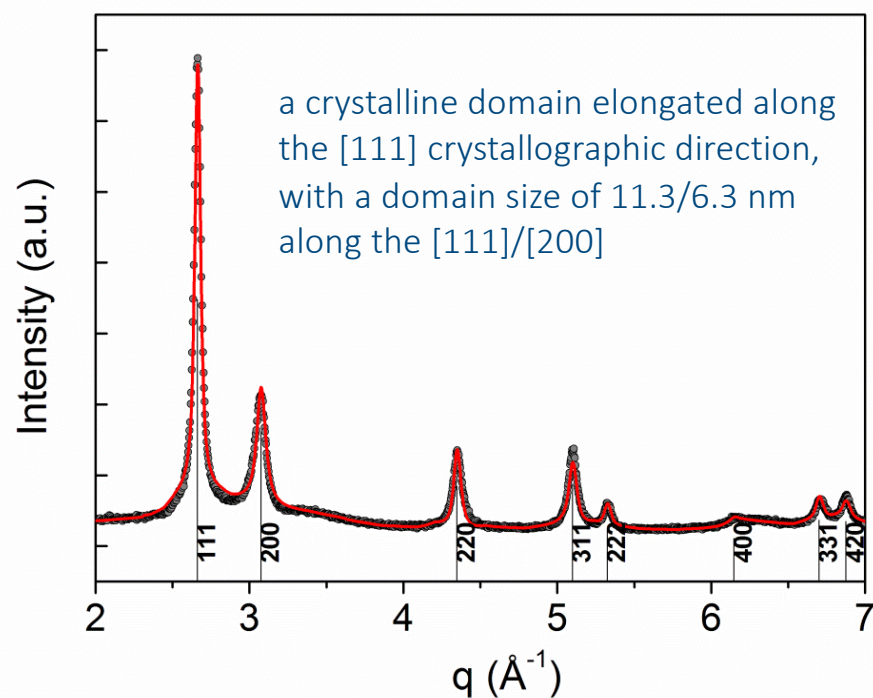
What is the size/shape?



WIDE ANGLE X-RAY SCATTERING - WAXS

International SILS School
Muggia, 01 / 10 / 2026

What is the size/shape?



Identifier	1100136
Literature Reference	Spreadborough, J, Christian, J W, <i>Journal of Scientific Instruments</i> (1959), 36 , 116
Formula	Ag
Compound Name	Silver - 3C
Synonym	
Space Group	F m $\bar{3}$ m
Cell Lengths	a 4.0855(1) b 4.0855(1) c 4.0855(1)
Cell Angles	α 90 β 90 γ 90
Cell Volume	68.1923
Z, Z'	Z : 4 Z' : 0

WIDE ANGLE X-RAY SCATTERING - WAXS

International SILS School
Muggia, 01 / 10 / 2026

What is the size/shape?

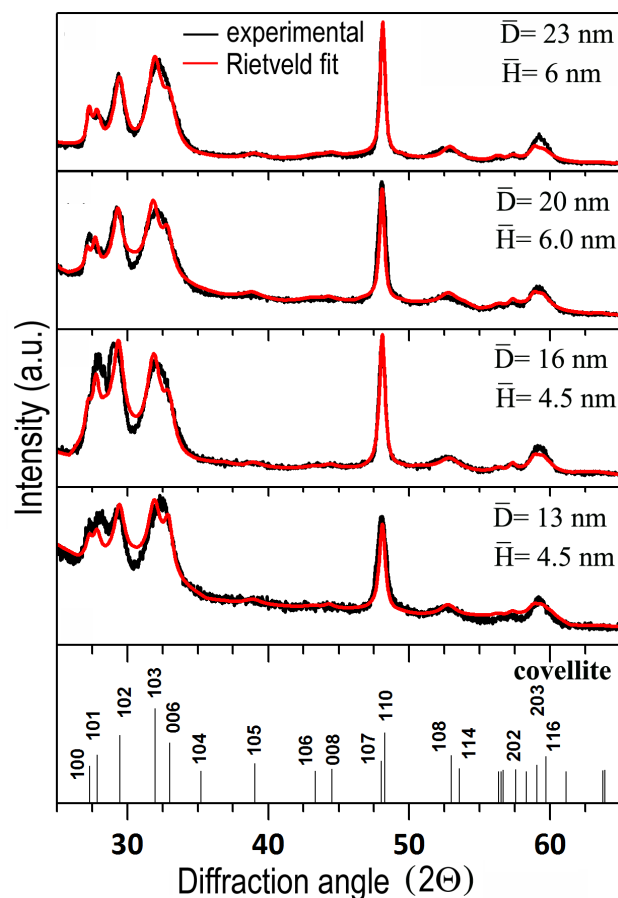


TABLE 1. Mean Coherent Domain Size, D_{hkl} , Estimated upon Rietveld-Fitting the XRD Patterns Shown in Figure 2

$\bar{D}$ (nm) ^a	$\bar{H}$ (nm) ^a	D_{110} (nm) ^c	D_{100} (nm) ^c	D_{006} (nm) ^c
13 ± 2	4.5 ± 0.5	15 ± 1	15 ± 2	8 ± 2
16 ± 2	5.5 ± 0.5	16 ± 1	16 ± 1	6 ± 1
20 ± 2	6.0 ± 0.5	20 ± 1	20 ± 1	6 ± 1
23 ± 2	6.0 ± 0.5	21 ± 1	22 ± 1	6 ± 1

^a Mean sizes estimated by TEM by statistical analysis. ^c Minor discrepancies between the XRD-derived and TEM-measured sizes can be attributed to the presence of self-organized ND superstructures within the ND powders deposited on the silicon substrates for analysis. Micrometer-size domains of columnar face-to-face stacked NDs, in which the degree of intracolumnar ND ordering along the *c*-axis direction prevailed over the degree of intercolumnar alignment in the perpendicular orientations (Figure 1g–i), can be expected to hold higher structural coherence along the [001] and, to a lower extent, along the [010] and [110], relative to their fully disordered ND ensemble counterparts.

ACS NANO

Metallic-like Stoichiometric Copper Sulfide Nanocrystals: Phase- and Shape-Selective Synthesis, Near-Infrared Surface Plasmon Resonance Properties, and Their Modeling

Yi Xie †, Luigi Carbone §*, Concetta Nobile §, Vincenzo Grillo †, Stefania D'Agostino †#, Fabio Della Sala †§, Cinzia Giannini Δ, Davide Altamura Δ, Christian Oelsner Ø, Carola Kryschki Ø, and P. Davide Cozzoli §††

ACS Nano, 2013, 7 (8), pp 7352–7369
DOI: 10.1021/nn403035e
Publication Date (Web): July 16, 2013

WIDE ANGLE X-RAY SCATTERING - WAXS

International SILS School
Muggia, 01 / 10 / 2026

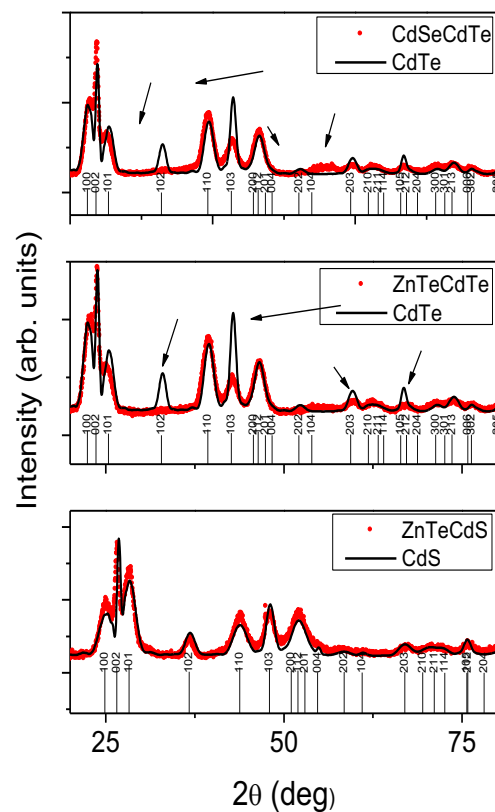
What is the effect of defects?

JACS
JOURNAL OF THE AMERICAN CHEMICAL SOCIETY

Article

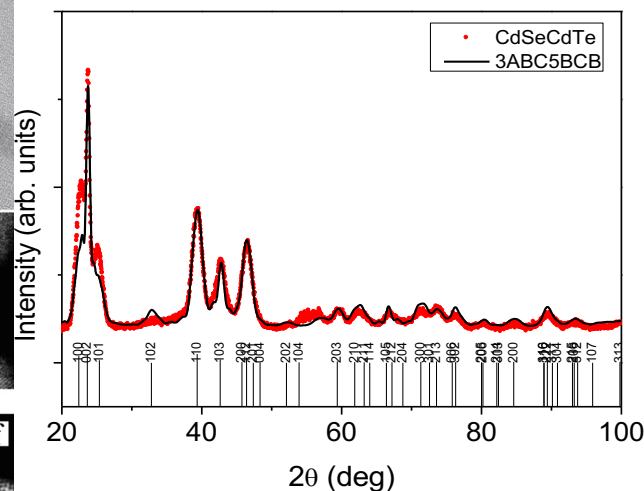
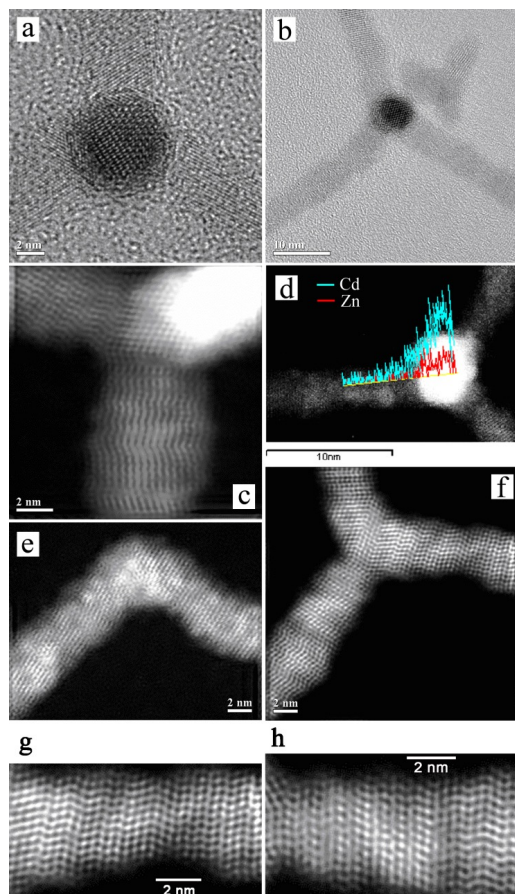
Tetrapod-Shaped Colloidal Nanocrystals of II#VI Semiconductors Prepared by Seeded Growth

Angela Fiore, Rosanna Mastria, Maria Grazia Lupo, Guglielmo Lanzani, Cinzia Giannini, Elvio Carlino, Giovanni Morello, Milena De Giorgi, Yanqin Li, Roberto Cingolani, and Liberato Manna
J. Am. Chem. Soc., 2009, 131 (6), 2274-2282 • DOI: 10.1021/ja807874e • Publication Date (Web): 26 January 2009



WIDE ANGLE X-RAY SCATTERING - WAXS

International SILS School
Muggia, 01 / 10 / 2026



What is the effect of defects?

JACS
JOURNAL OF THE AMERICAN CHEMICAL SOCIETY

Article

Tetrapod-Shaped Colloidal Nanocrystals of II#VI Semiconductors Prepared by Seeded Growth

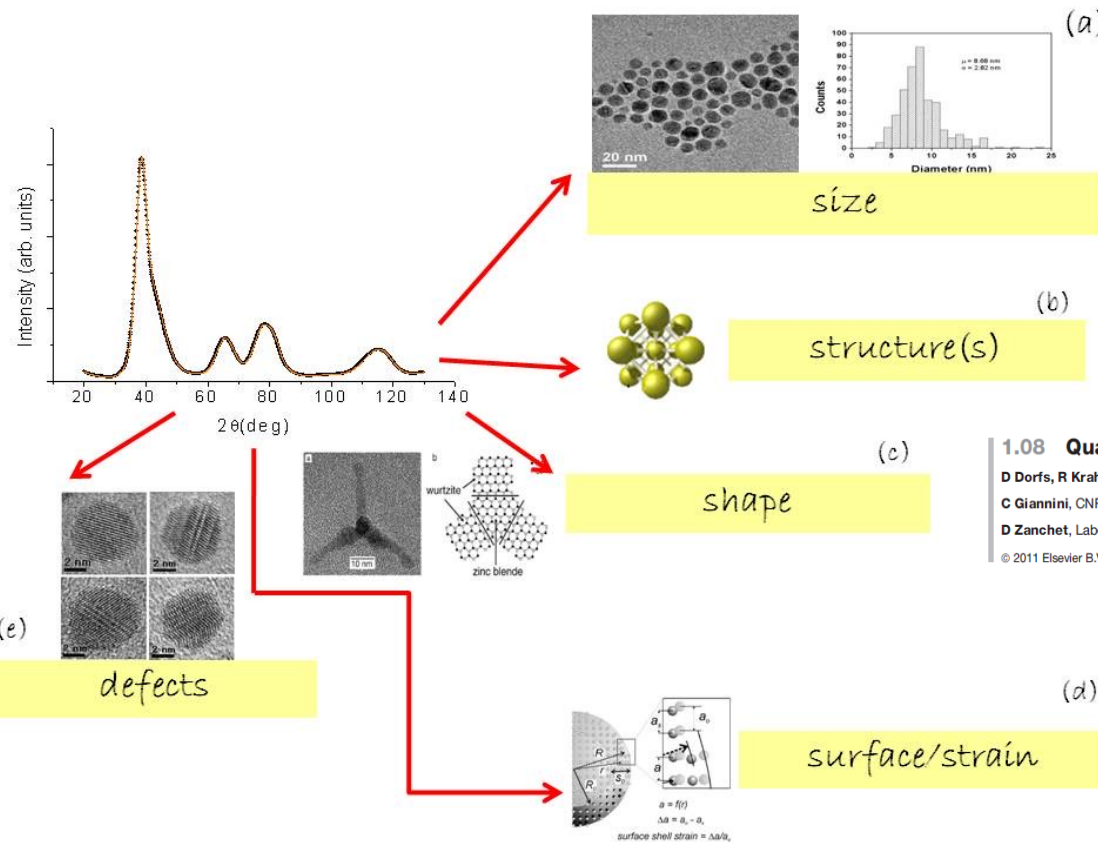
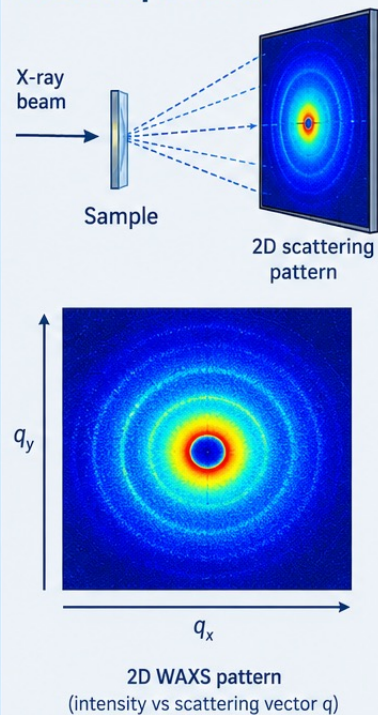
Angela Fiore, Rosanna Mastria, Maria Grazia Lupo, Guglielmo Lanzani, Cinzia Giannini, Elvio Carlino, Giovanni Morello, Milena De Giorgi, Yanqin Li, Roberto Cingolani, and Liberato Manna
J. Am. Chem. Soc., 2009, 131 (6), 2274-2282 • DOI: 10.1021/ja807874e • Publication Date (Web): 26 January 2009

HRTEM image of a ZnTe/CdTe tetrapod. It is remarkable to note the changes of the lattice fringes contrast along the arms of the tetrapod, which indicates the presence of regions with either different orientations, or structure, or composition

What can we extract from WAXS?

From the scattering pattern to the structural information

WAXS experiment



1.08 Quantum Dots: Synthesis and Characterization

D Dorfs, R Krahne, A Falqui, and L Manna, Istituto Italiano di Tecnologia, Genoa, Italy

C Giannini, CNR-Istituto di Cristallografia (IC), Bari, Italy

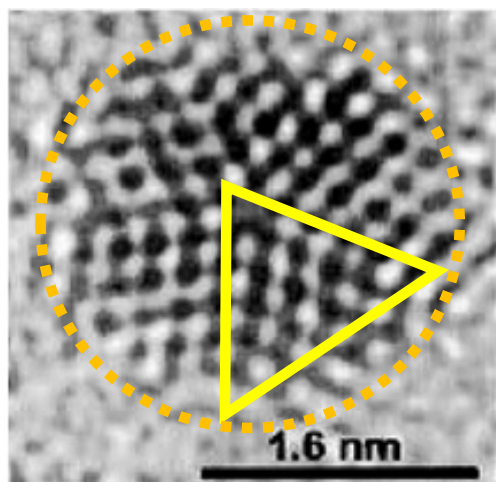
D Zanchet, Laboratório Nacional de Luz Síncrotron, Campinas-SP, Brazil

© 2011 Elsevier B.V. All rights reserved.

WIDE ANGLE X-RAY SCATTERING - **WAXS**

International SILS School
Muggia, 01 / 10 / 2026

WAXS:
nanocrystal structure
domain size
anisotropy of the crystalline domain (crystal domain shape)



crystalline domain -
WAXS

NP shape / size
SAXS

"EUROPEAN SYNCHROTRON WAXS / PDF BEAMLINE LANDSCAPE"



ESRF (European Synchrotron Radiation Facility)
Grenoble, France



ID15A – High Energy/PDF
(Materials chemistry)

ID22 – High Resolution
(Powder Diffraction)

ID31 – Materials Science/in situ
(Hard X-rays)



SOLEIL Synchrotron
Saint-Aubin, France



CRISTAL
(High Resolution
Powder & PDF)



ALBA, Barcelona, Spain



BL04-MSPD
(Materials Science
Powder Diffraction/PDF)

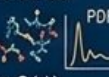


Diamond Light Source
Didcot, UK



I11 – High Resolution Powder
(Diffraction & WAXS)

I15-1 – PDF
(Local Structure/Atomic G(r))



MAX IV Laboratory,
Lund, Sweden



DanMAX

(Danish Materials Science /
WAXS / PDF)



DESY / PETRA III
Hamburg, Germany



PETRA III

P02.1 – Powder
Diffraction/PDF
(in situ materials)

P21.1 – High Energy
Diffraction/PDF



Elettra Sincrotrone Trieste, Trieste, Italy



MCX

(Material Characterization
by X-rays/WAXS)



Synchrotron Ring

X-ray Beam



Sample Stage



Detector

Beamline Labels



OUTLINE

International SILS School
Muggia, 01 / 10 / 2026

- SAXS – WAXS – Basic notions
- Scanning SAXS- WAXS Microscopy
- **Nanocrystals and Nanocrystal-based Superlattices**
- Scaffolds; Fibers

Nanocrystals Assembly: hierarchical structure

International SILS School
Muggia, 01 / 10 / 2026

ACS Nano > Vol 18/Issue 23 > Article

Subscribed

Cite Share Jump to Expand

REVIEW | May 30, 2024

Nanocrystal Assemblies: Current Advances and Open Problems

Carlos L. Bassani, Greg van Anders, Uri Banin, Dmitry Baranov, Qian Chen, Marjolein Dijkstra, Michael S. Dimitriyev, Efi Efrati, Jordi Faraudo, Oleg Gang, Nicola Gaston, Ramin Golestanian, G. Ivan Guerrero-Garcia, Michael Gruenwald, Amir Haji-Akbari, Maria Ibáñez, Matthias Karg, Tobias Kraus, Byeongdu Lee, Reid C. Van Lehn, Robert J. Macfarlane, Bortolo M. Moggetti, Arash Nikoubashman, Saeed Osat, Oleg V. Prezhdov, Grant M. Rotskoff, Leonor Saiz, An-Chang Shi, Sara Skrabalak, Ivan I. Smalyukh, Mario Tagliazucchi, Dmitri V. Talapin, Alexei V. Tkachenko, Sergei Tretiak, David Vaknin, Asaph Widmer-Cooper, Gerard C. L. Wong, Xingchen Ye, Shan Zhou, Eran Rabani, Michael Engel, and Alex Travesset*

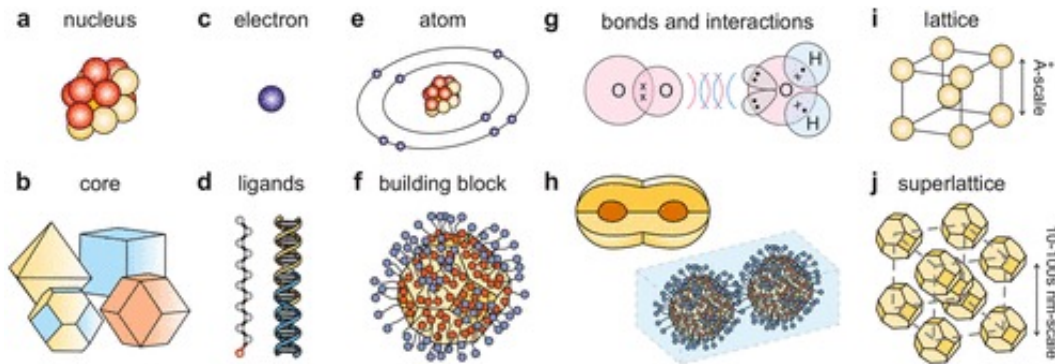


Figure 1. Analogy between NCs and atoms.

The crystal structure can be described as an assembly of «building blocks»

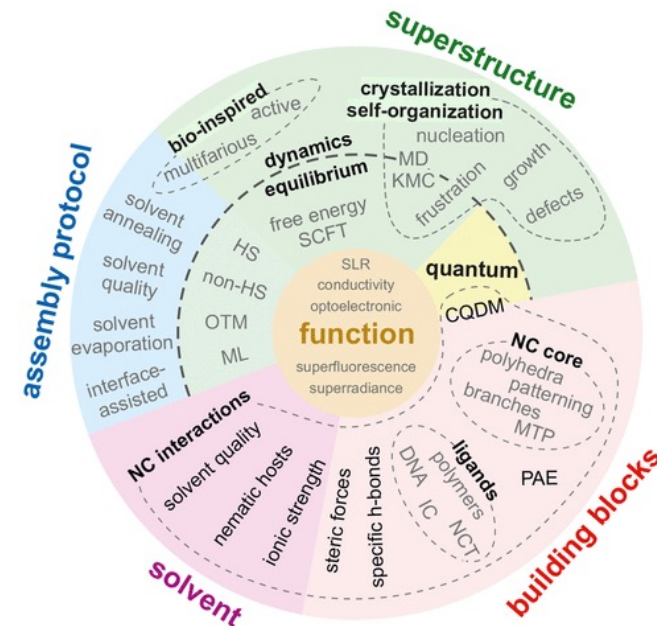
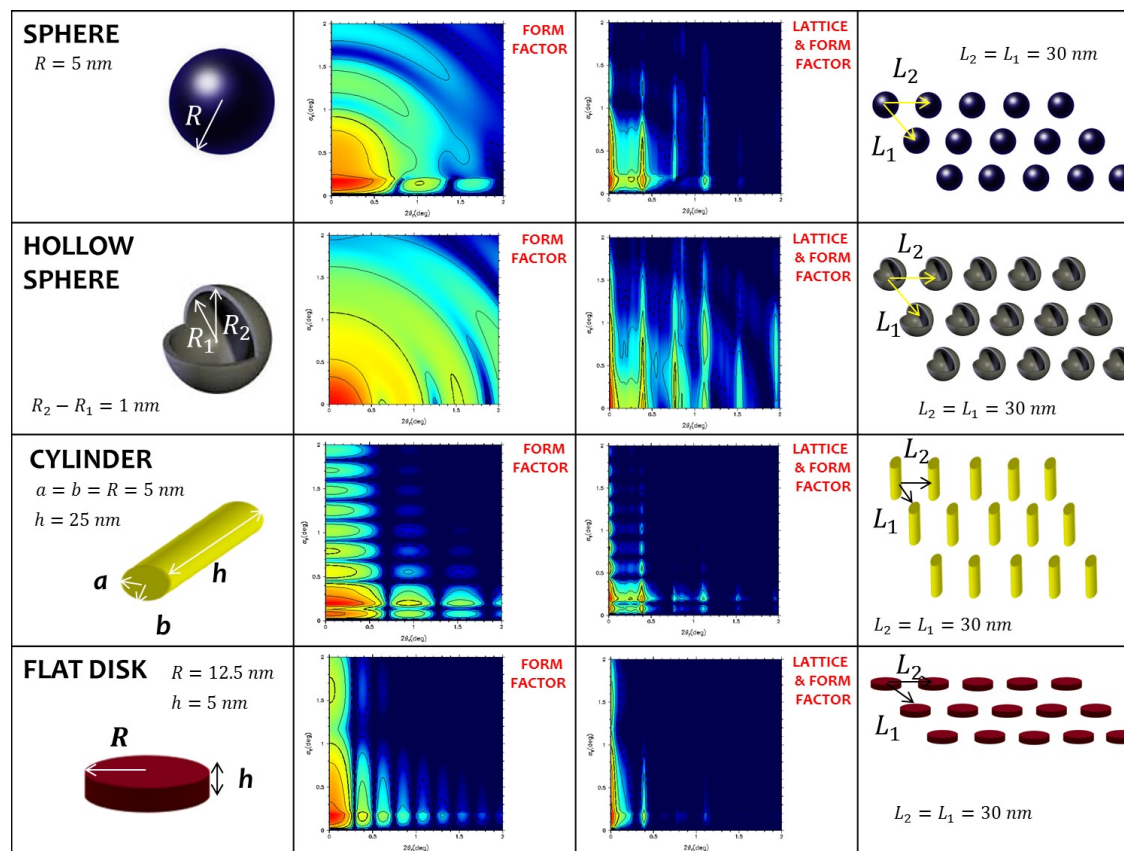


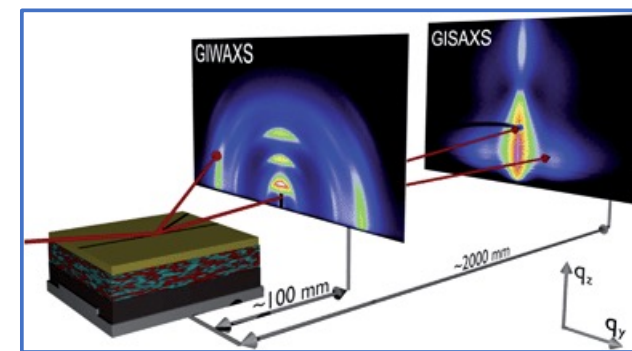
Figure 2. ...to assemble NCs into superstructures to a given desired function.

SUB-MOLECULAR (GIWAXS) SUPRA-MOLECULAR (GISAXS)

International SILS School
Muggia, 01 / 10 / 2026



Reflection geometry



INTECH
open source | open access

Nanomaterials and Nanotechnology

OPEN ACCESS
ARTICLE

Assembled Nanostructured Architectures Studied By Grazing Incidence X-Ray Scattering

Invited Feature Article

Davide Altamura¹, Teresa Sibillano¹, Dritan Siliqi¹, Liberato De Caro¹ and Cinzia Giannini^{1,*}

¹ Istituto di Cristallografia, Sede di Bari, Bari, Italy
* Corresponding author: cinzia.giannini@ic.cnr.it

 **Consiglio Nazionale
delle Ricerche**

SUB-MOLECULAR (GIWAXS) SUPRA-MOLECULAR (GISAXS)

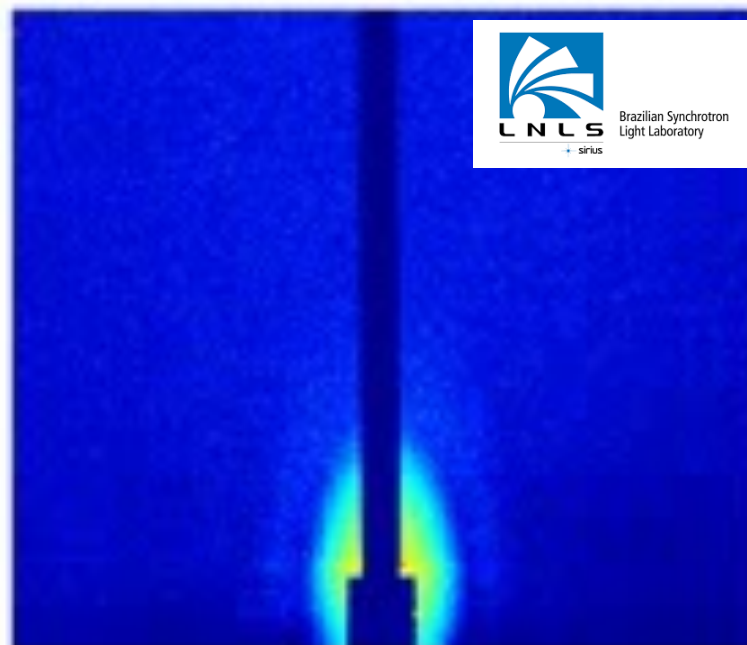
International SILS School
Muggia, 01 / 10 / 2026

Hydrogen 1 H 1.01																		Helium 2 He 4.00														
Lithium 3 Li 6.94	Beryllium 4 Be 9.01											Boron 5 B 10.81	Carbon 6 C 12.01	Nitrogen 7 N 14.01	Oxygen 8 O 16.00	Fluorine 9 F 19.00	Neon 10 Ne 20.18															
Sodium 11 Na 22.99	Magnesium 12 Mg 24.31											Aluminum 13 Al 26.98	Silicon 14 Si 28.09	Phosphorus 15 P 30.97	Sulfur 16 S 32.07	Chlorine 17 Cl 35.45	Argon 18 Ar 39.95															
Potassium 19 K 39.10	Calcium 20 Ca 40.08	Scandium 21 Sc 44.96	Titanium 22 Ti 47.88	Vanadium 23 V 50.94	Chromium 24 Cr 52.00	Manganese 25 Mn 54.94	Iron 26 Fe 55.85	Cobalt 27 Co 58.93	Nickel 28 Ni 58.69	Copper 29 Cu 63.55	Zinc 30 Zn 65.39	Gallium 31 Ga 69.72	Germanium 32 Ge 72.61	Arsenic 33 As 74.92	Selenium 34 Se 78.96	Bromine 35 Br 79.90	Krypton 36 Kr 83.80															
Rubidium 37 Rb 85.47	Strontium 38 Sr 87.62	Yttrium 39 Y 88.91	Zirconium 40 Zr 91.22	Niobium 41 Nb 92.91	Molybdenum 42 Mo 95.94	Technetium 43 Tc (98)	Ruthenium 44 Ru 101.07	Rhodium 45 Rh 102.91	Palladium 46 Pd 106.42	Silver 47 Ag 107.87	Cadmium 48 Cd 112.41	Indium 49 In 114.82	Tin 50 Sn 118.71	Antimony 51 Sb 121.76	Tellurium 52 Te 127.60	Iodine 53 I 126.90	Xenon 54 Xe 131.29															
Cesium 55 Cs 132.91	Barium 56 Ba 137.33	57-70 *	Lutetium 71 Lu 174.97	Hafnium 72 Hf 178.49	Tantalum 73 Ta 180.95	Tungsten 74 W 183.84	Rhenium 75 Re 186.21	Osmium 76 Os 190.23	Iridium 77 Ir 192.22	Platinum 78 Pt 195.08	Gold 79 Au 196.97	Mercury 80 Hg 200.59	Thallium 81 Tl 204.38	Lead 82 Pb 207.20	Bismuth 83 Bi 208.98	Polonium 84 Po (209)	Astatine 85 At (210)	Radon 86 Rn (222)														
Francium 87 Fr (223)	Radium 88 Ra (226)	89-102 **	Lawrencium 103 Lr (262)	Rutherfordium 104 Rf (267)	Dubnium 105 Db (268)	Seaborgium 106 Sg (271)	Bohrium 107 Bh (272)	Hassium 108 Hs (270)	Meitnerium 109 Mt (276)	Darmstadtium 110 Ds (281)	Roentgenium 111 Rg (280)	Copernicium 112 Cn (285)	Ununtrium 113 Uut (284)	Ununquadium 114 Uuq (289)	Ununpentium 115 Uup (288)	Ununhexium 116 Uuh (293)	Ununseptium 117 Uus (294?)	Ununoctium 118 Uuo (294)														
																			Lanthanum 57 La 138.91	Cerium 58 Ce 140.12	Praseodymium 59 Pr 140.91	Neodymium 60 Nd 144.24	Promethium 61 Pm (145)	Samarium 62 Sm 150.36	Europium 63 Eu 151.97	Gadolinium 64 Gd 157.25	Terbium 65 Tb 158.93	Dysprosium 66 Dy 162.50	Holmium 67 Ho 164.93	Erbium 68 Er 167.26	Thulium 69 Tm 168.93	Ytterbium 70 Yb 173.04
																			Actinium 89 Ac (227)	Thorium 90 Th 232.04	Protactinium 91 Pa 231.04	Uranium 92 U 238.03	Neptunium 93 Np (237)	Plutonium 94 Pu (244)	Americium 95 Am (243)	Curium 96 Cm (247)	Berkelium 97 Bk (247)	Californium 98 Cf (251)	Einsteinium 99 Es (252)	Fermium 100 Fm (257)	Mendelevium 101 Md (258)	Nobelium 102 No (259)

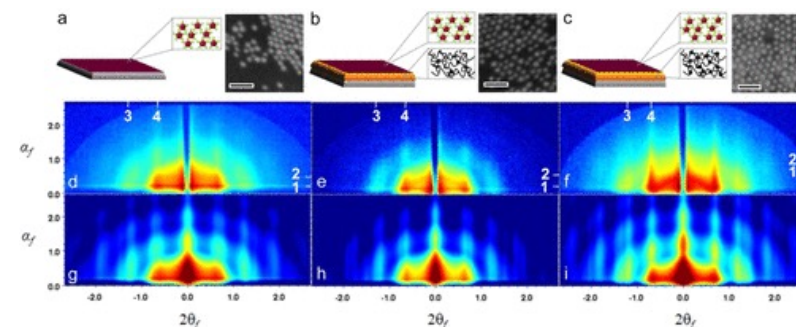
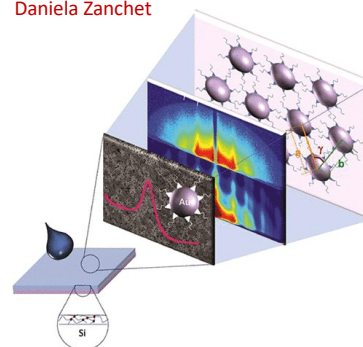
Gold Nanocrystals, sphere~12 nm

SUB-MOLECULAR (GIWAXS) SUPRA-MOLECULAR (GISAXS)

International SILS School
Muggia, 01 / 10 / 2026



Daniela Zanchet



CrystEngComm

PAPER



Cite this: CrystEngComm, 2014, 16, 9482

GISAXS and GIWAXS study on self-assembling processes of nanoparticle based superlattices†

M. Corricelli,^{†,ab} D. Altamura,^{†,c} M. L. Curri,^b T. Sibillano,^c D. Siliqi,^c A. Mazzone,^c N. Depalo,^b E. Fanizza,^{ab} D. Zanchet,^a C. Giannini^{*c} and M. Striccoli^{*b}

GISAXS in situ Experiment performed at the XRD2 beamline in LNLS-Campinas, Brazil

Gold Nanocrystals, sphere ~12 nm



Lucia Curri



Marinella Striccoli

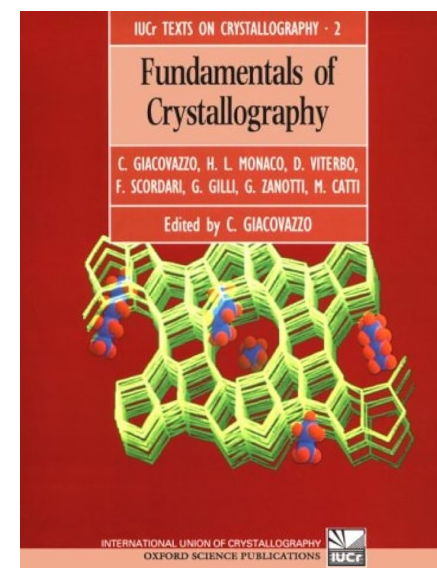
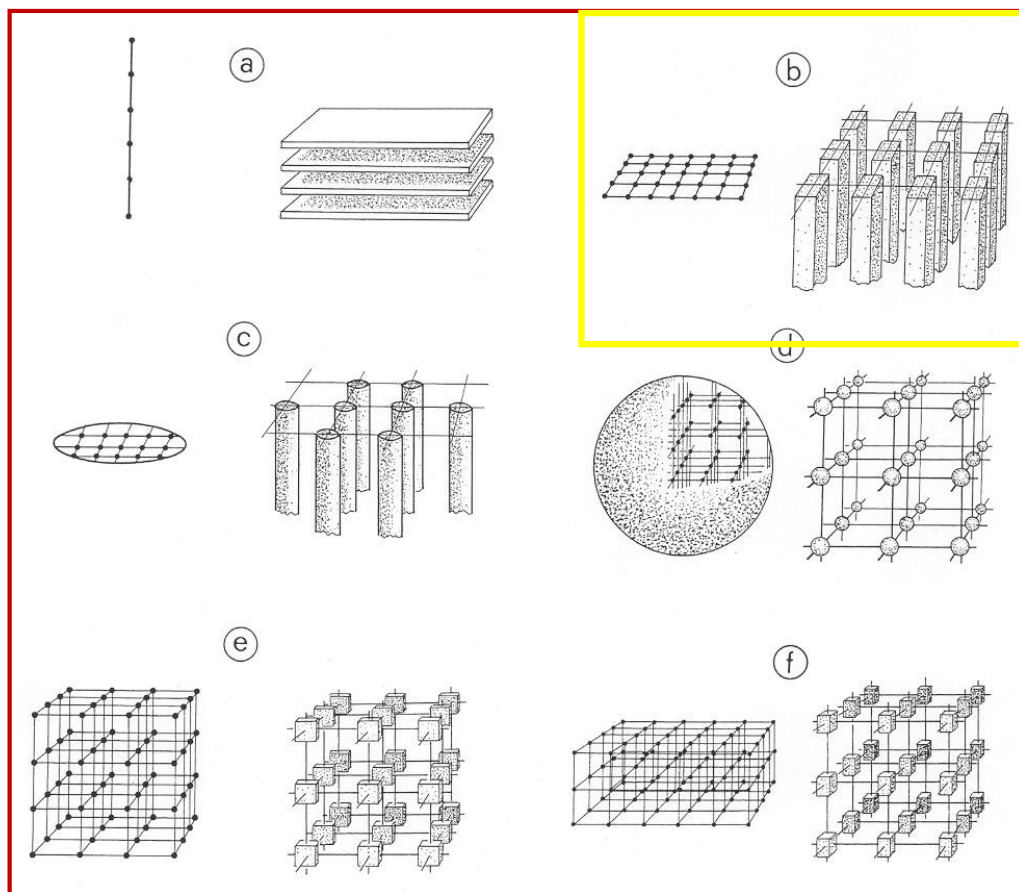


UNIVERSITÀ
DEGLI STUDI DI BARI
ALDO MORO

Consiglio Nazionale
delle Ricerche

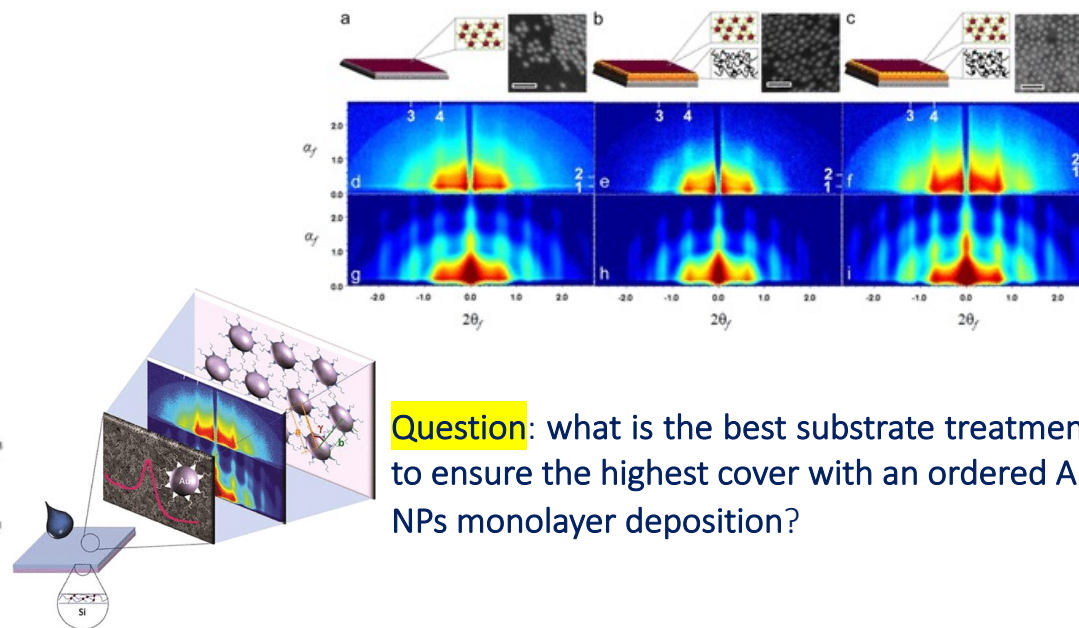
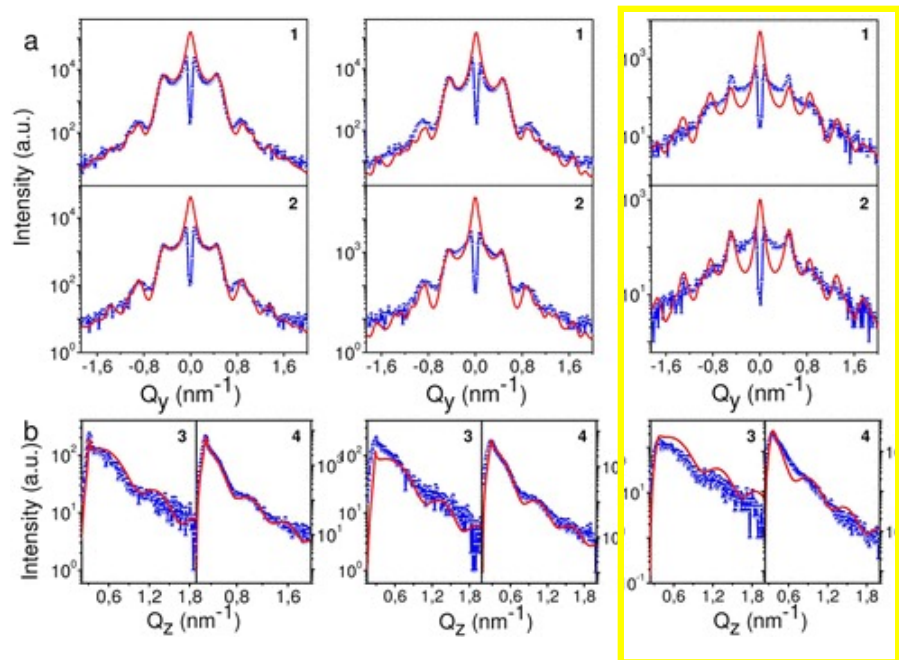
CRYSTALLOGRAPHY

International SILS School
Muggia, 01 / 10 / 2026



SUB-MOLECULAR (GIWAXS) SUPRA-MOLECULAR (GISAXS)

International SILS School
Muggia, 01 / 10 / 2026



Question: what is the best substrate treatment to ensure the highest cover with an ordered Au NPs monolayer deposition?

Conclusion:

GISAXS revealed for Au NPs deposited on Si/SiO₂ substrate, it was found that $a = 14.0 \pm 0.5$ nm and $b = 15.0 \pm 0.5$ nm, forming an angle of $\gamma = 70^\circ \pm 2^\circ$. In the case of Au NPs deposited on the **Si/TL substrate**, NPs form a distorted hexagonal lattice with $a = b = 15.0 \pm 0.5$ nm basis vectors, forming an angle $\gamma = 50^\circ$ on average, possibly taking on larger values, up to 65° .

Gold Nanocrystals, sphere~12 nm

SUB-MOLECULAR (GIWAXS) SUPRA-MOLECULAR (GISAXS)

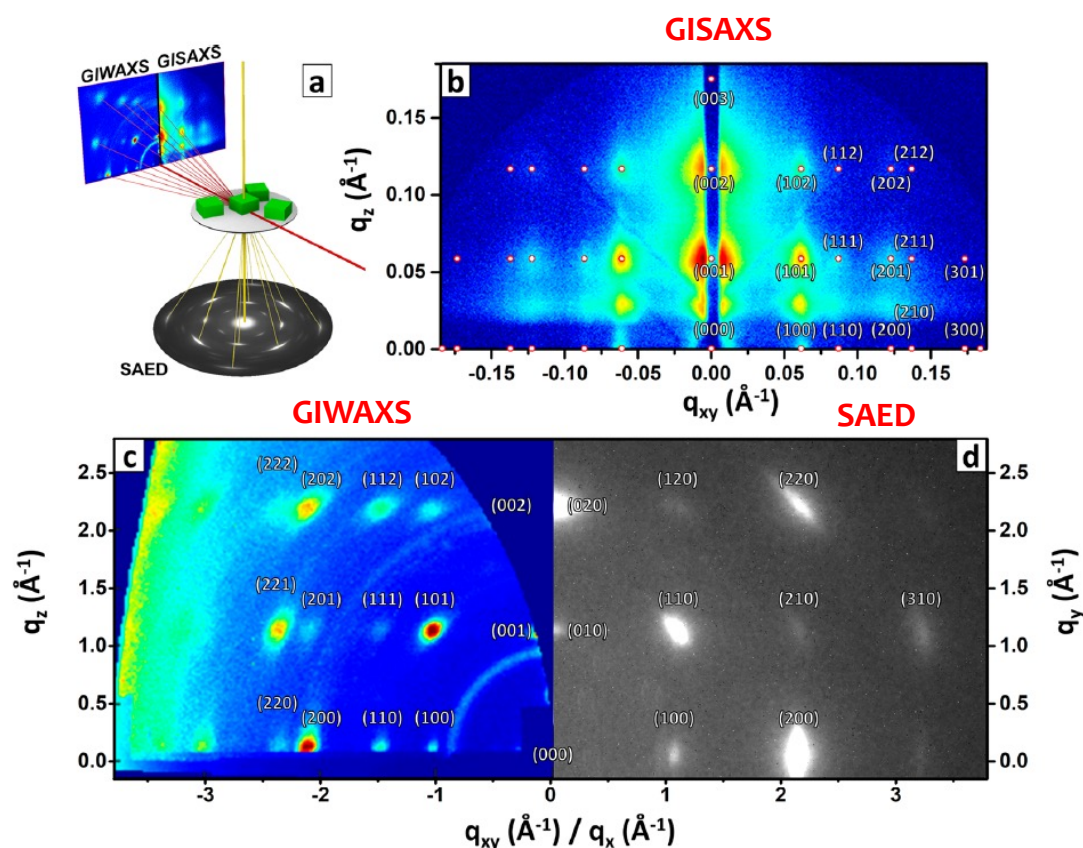
International SILS School
Muggia, 01 / 10 / 2026

Hydrogen 1 H 1.01																		Helium 2 He 4.00	
Lithium 3 Li 6.94	Beryllium 4 Be 9.01											Boron 5 B 10.81	Carbon 6 C 12.01	Nitrogen 7 N 14.01	Oxygen 8 O 16.00	Fluorine 9 F 19.00	Neon 10 Ne 20.18		
Sodium 11 Na 22.99	Magnesium 12 Mg 24.31											Aluminum 13 Al 26.98	Silicon 14 Si 28.09	Phosphorus 15 P 30.97	Sulfur 16 S 32.07	Chlorine 17 Cl 35.45	Argon 18 Ar 39.95		
Potassium 19 K 39.10	Calcium 20 Ca 40.08	Scandium 21 Sc 44.96	Titanium 22 Ti 47.88	Vanadium 23 V 50.94	Chromium 24 Cr 52.00	Manganese 25 Mn 54.94	Iron 26 Fe 55.85	Cobalt 27 Co 58.93	Nickel 28 Ni 58.69	Copper 29 Cu 63.55	Zinc 30 Zn 65.39	Gallium 31 Ga 69.72	Germanium 32 Ge 72.61	Arsenic 33 As 74.92	Selenium 34 Se 78.96	Bromine 35 Br 79.90	Krypton 36 Kr 83.80		
Rubidium 37 Rb 85.47	Strontium 38 Sr 87.62	Yttrium 39 Y 88.91	Zirconium 40 Zr 91.22	Niobium 41 Nb 92.91	Molybdenum 42 Mo 95.94	Technetium 43 Tc (98)	Ruthenium 44 Ru 101.07	Rhodium 45 Rh 102.91	Palladium 46 Pd 106.42	Silver 47 Ag 107.87	Cadmium 48 Cd 112.41	Indium 49 In 114.82	Tin 50 Sn 118.71	Antimony 51 Sb 121.76	Tellurium 52 Te 127.60	Iodine 53 I 126.90	Xenon 54 Xe 131.29		
Cesium 55 Cs 132.91	Barium 56 Ba 137.33	57-70 *	Lutetium 71 Lu 174.97	Hafnium 72 Hf 178.49	Tantalum 73 Ta 180.95	Tungsten 74 W 183.84	Rhenium 75 Re 186.21	Osmium 76 Os 190.23	Iridium 77 Ir 192.22	Platinum 78 Pt 195.08	Gold 79 Au 196.97	Mercury 80 Hg 200.59	Thallium 81 Tl 204.38	Lead 82 Pb 207.20	Bismuth 83 Bi 208.98	Polonium 84 Po (209)	Astatine 85 At (210)	Radon 86 Rn (222)	
Francium 87 Fr (223)	Radium 88 Ra (226)	89-102 **	Lawrencium 103 Lr (262)	Rutherfordium 104 Rf (267)	Dubnium 105 Db (268)	Seaborgium 106 Sg (271)	Bohrium 107 Bh (272)	Hassium 108 Hs (270)	Meitnerium 109 Mt (276)	Darmstadtium 110 Ds (281)	Roentgenium 111 Rg (280)	Copernicium 112 Cn (285)	Ununtrium 113 Uut (284)	Ununquadium 114 Uuq (289)	Ununpentium 115 Uup (288)	Ununhexium 116 Uuh (293)	Ununseptium 117 Uus (294?)	Ununoctium 118 Uuo (294)	
			Lanthanum 57 La 138.91	Cerium 58 Ce 140.12	Praseodymium 59 Pr 140.91	Neodymium 60 Nd 144.24	Promethium 61 Pm (145)	Samarium 62 Sm 150.36	Europium 63 Eu 151.97	Gadolinium 64 Gd 157.25	Terbium 65 Tb 158.93	Dysprosium 66 Dy 162.50	Holmium 67 Ho 164.93	Erbium 68 Er 167.26	Thulium 69 Tm 168.93	Ytterbium 70 Yb 173.04			
			Actinium 89 Ac (227)	Thorium 90 Th 232.04	Protactinium 91 Pa 231.04	Uranium 92 U 238.03	Neptunium 93 Np (237)	Plutonium 94 Pu (244)	Americium 95 Am (243)	Curium 96 Cm (247)	Berkelium 97 Bk (247)	Californium 98 Cf (251)	Einsteinium 99 Es (252)	Fermium 100 Fm (257)	Mendelevium 101 Md (258)	Nobelium 102 No (259)			

Cesium Lead Bromide Nanocrystals; cube ~13 nm

SUB-MOLECULAR (GIWAXS) SUPRA-MOLECULAR (GISAXS)

International SILS School
Muggia, 01 / 10 / 2026



Cesium Lead Bromide Nanocrystals ; cube ~ 13 nm

Stefano Toso



Dmitry Baranov

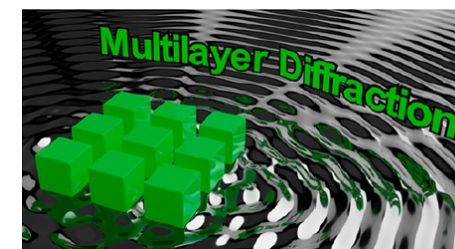


Liberato Manna



iit

ISTITUTO
ITALIANO DI
TECNOLOGIA



Conclusion:

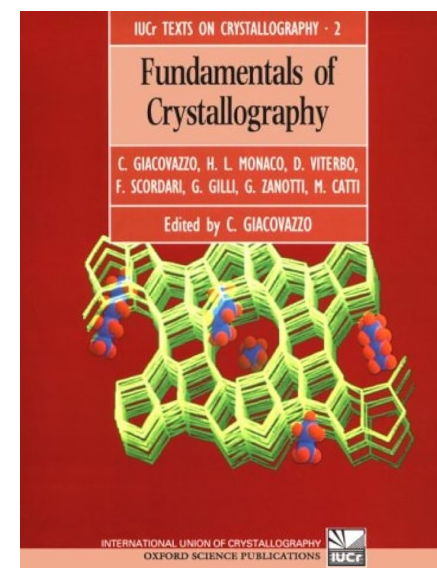
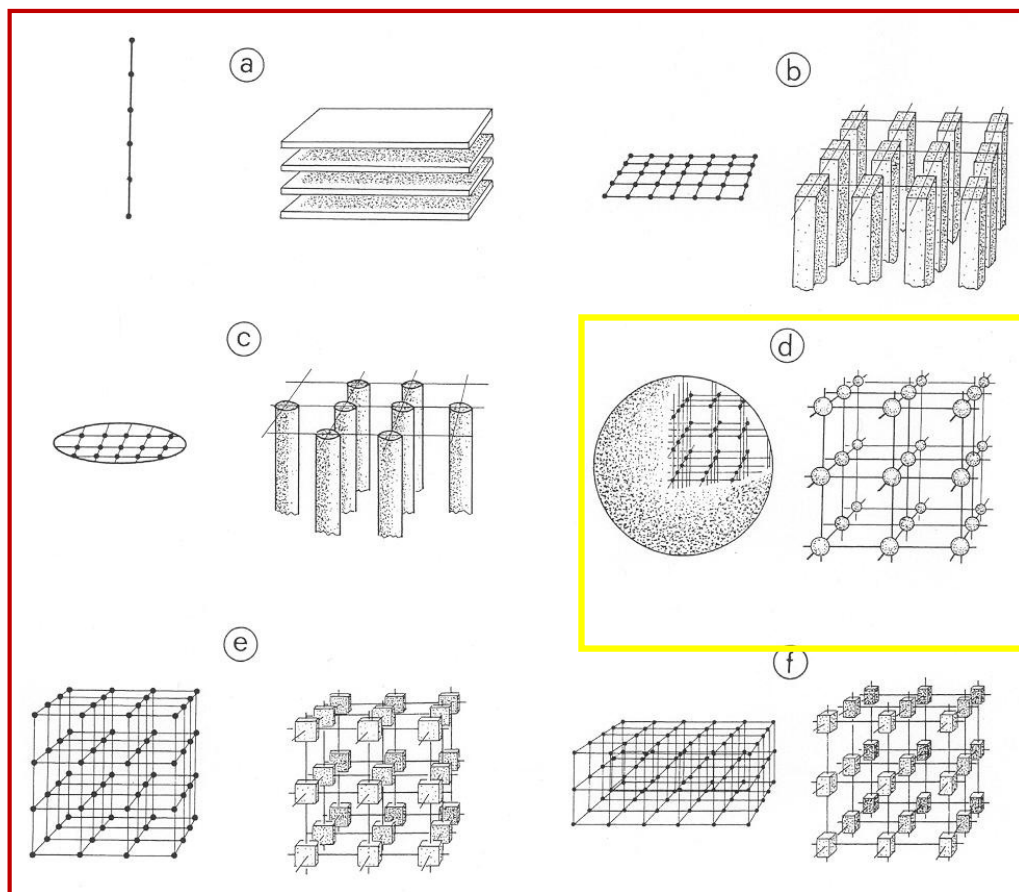
in-depth characterization by

- **GISAXS** revealed (001)-oriented 3D assemblies of CsPbBr₃ nanocrystals in simple-cubic symmetry
- **GIWAXS** revealed (001)-oriented CsPbBr₃ nanocrystals



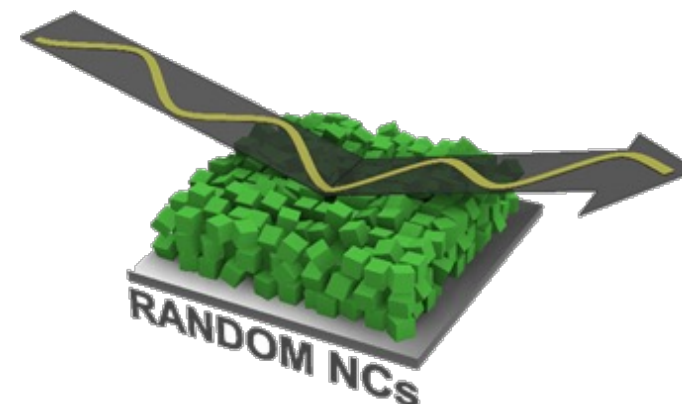
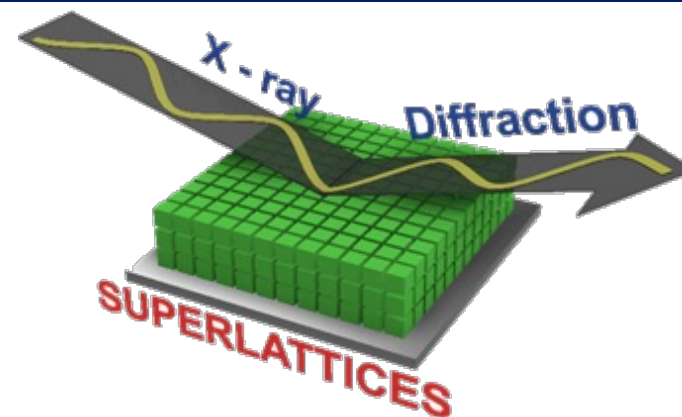
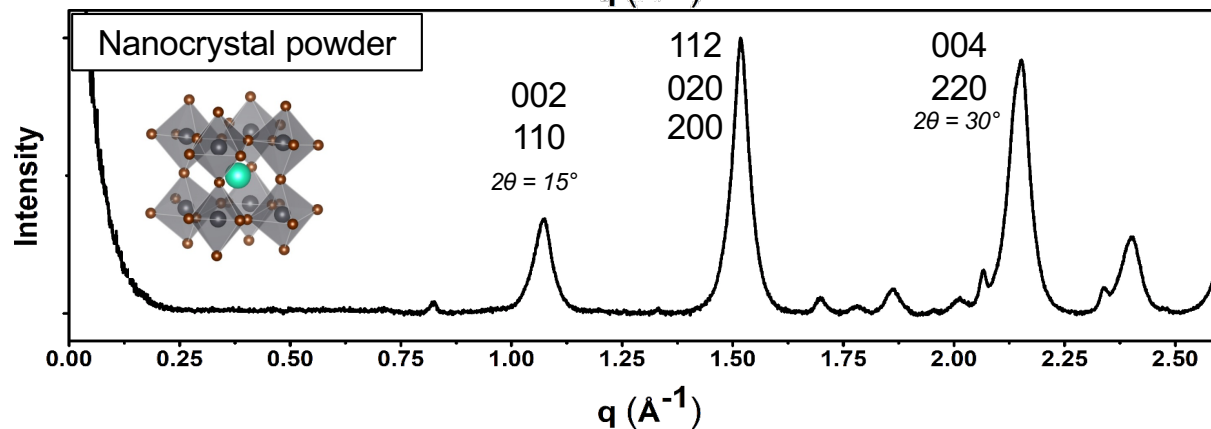
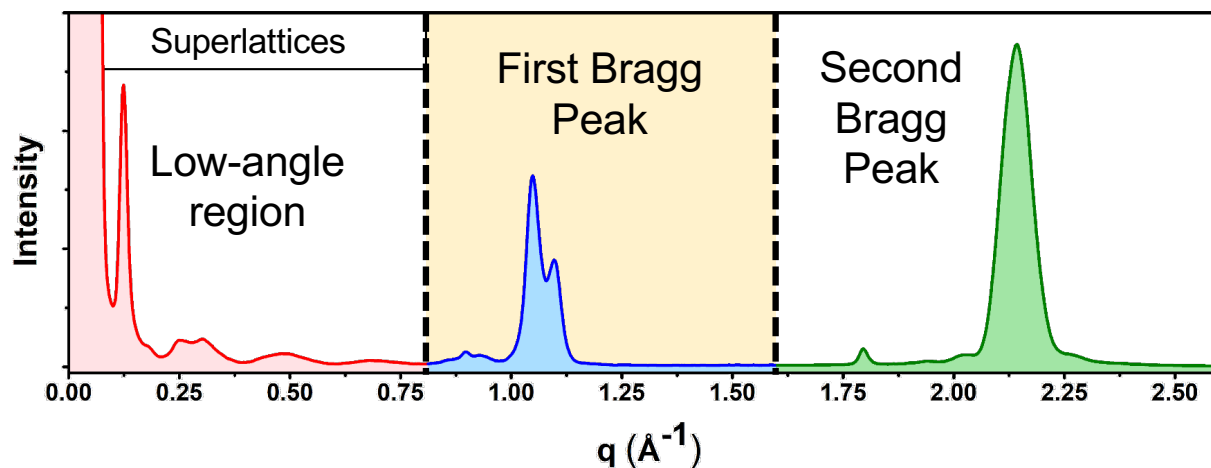
CRYSTALLOGRAPHY

International SILS School
Muggia, 01 / 10 / 2026



SUB-MOLECULAR (GIWAXS) SUPRA-MOLECULAR (GISAXS)

International SILS School
Muggia, 01 / 10 / 2026



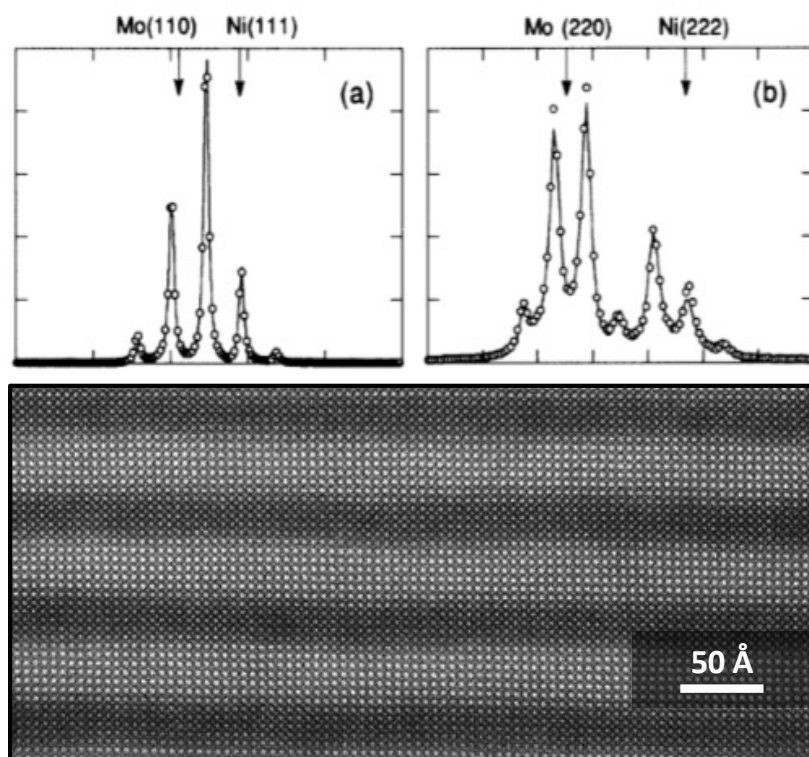
SUB-MOLECULAR (GIWAXS) SUPRA-MOLECULAR (GISAXS)

International SILS School
Muggia, 01 / 10 / 2026

VOLUME 45, NUMBER 16

Structural refinement of superlattices from x-ray diffraction

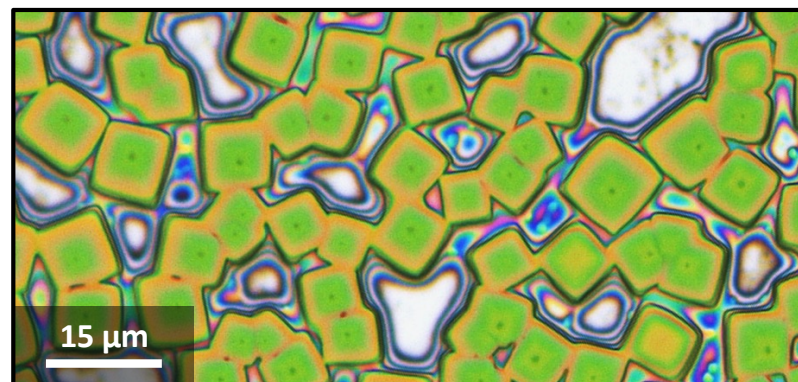
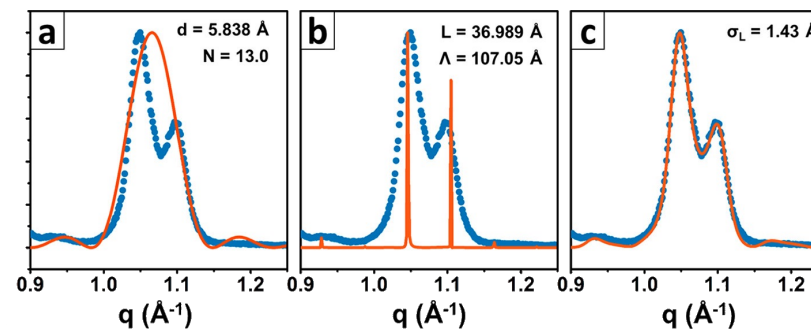
Eric E. Fullerton and Ivan K. Schuller



Multilayer Diffraction Reveals That Colloidal Superlattices Approach the Structural Perfection of Single Crystals

ACS NANO

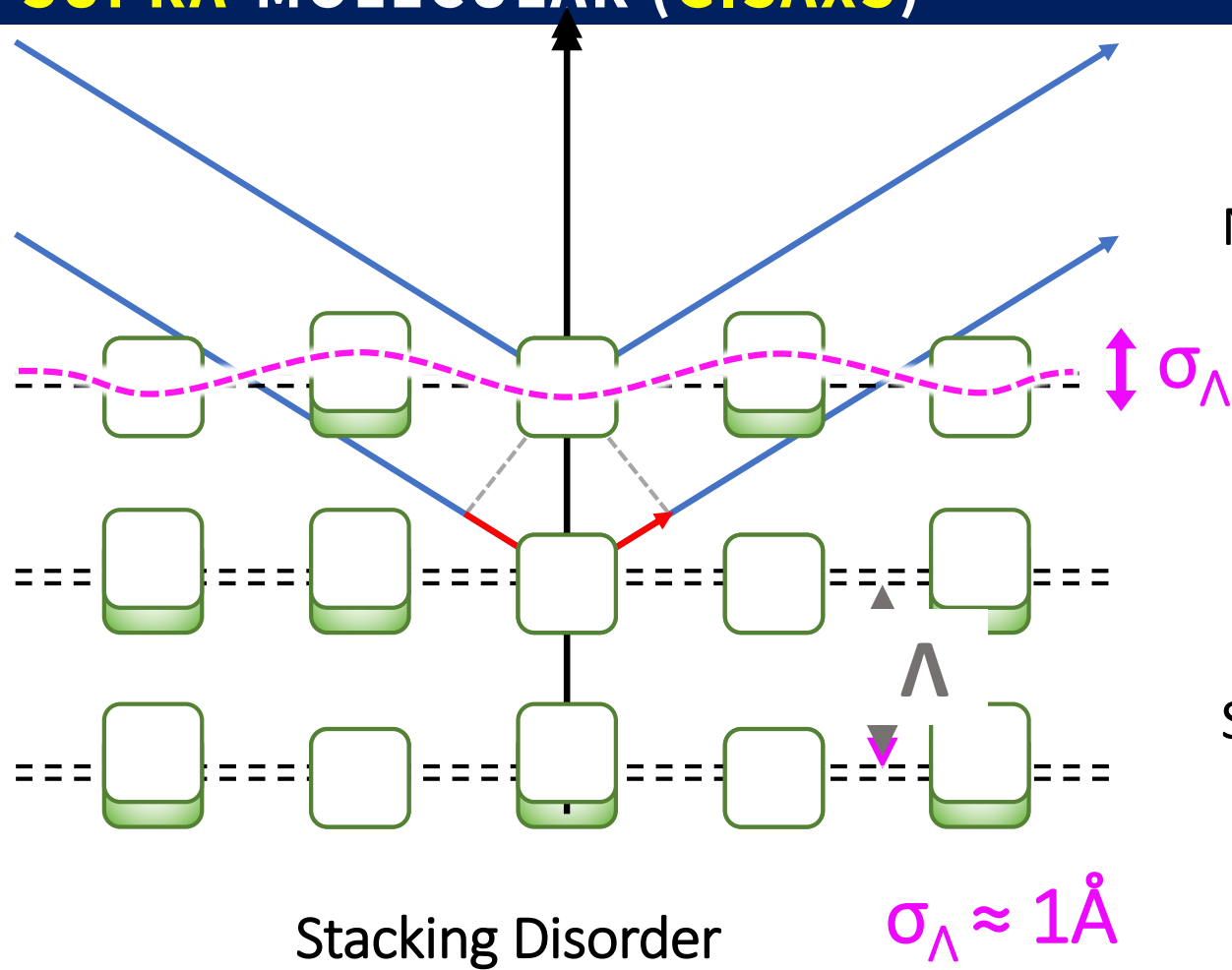
Stefano Toso,[†] Dmitry Baranov,^{*,‡} Davide Altamura, Francesco Scattarella, Jakob Dahl, Xingzhi Wang, Sergio Marras, A. Paul Alivisatos, Andrej Singer, Cinzia Giannini,^{*} and Liberato Manna^{*}



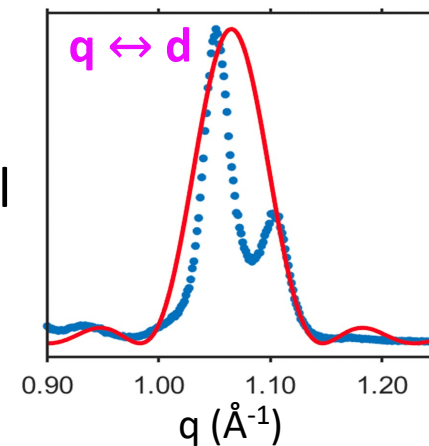
Nazionale
erche

SUB-MOLECULAR (GIWAXS) SUPRA-MOLECULAR (GISAXS)

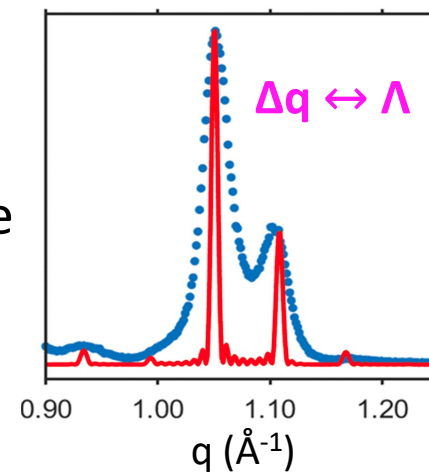
International SILS School
Muggia, 01 / 10 / 2026

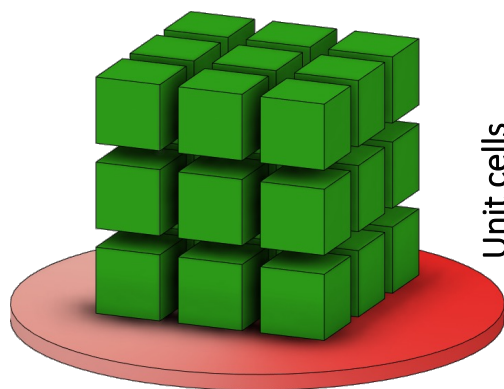
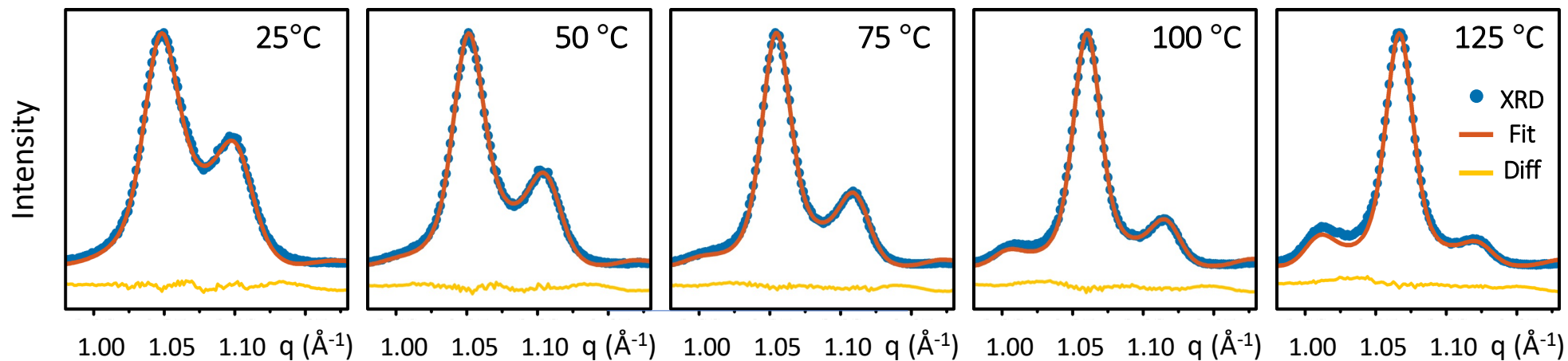


Individual
Nanocrystal

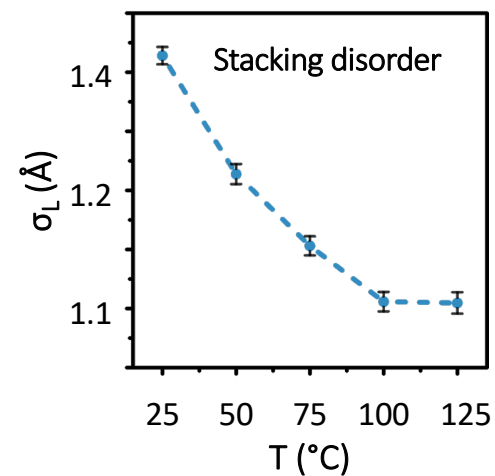
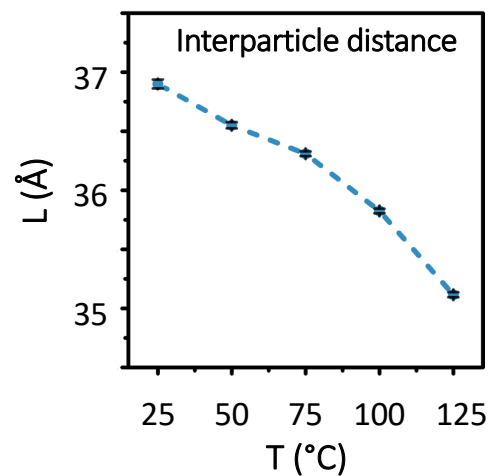
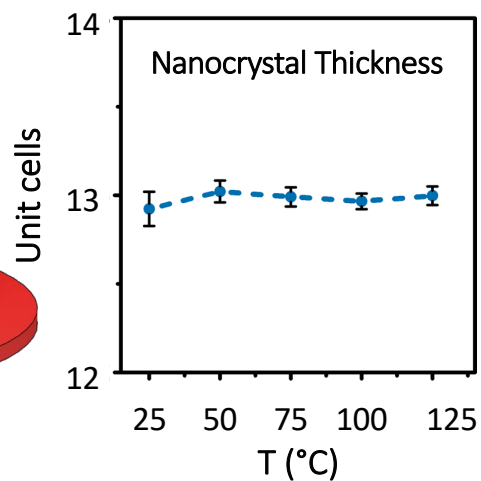


Ideal
Superlattice





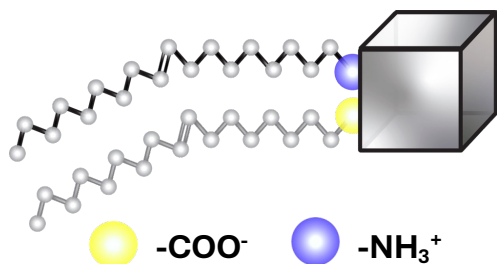
Thermal Annealing



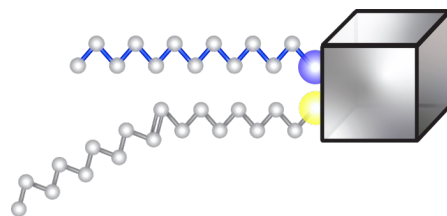
SUB-MOLECULAR (GIWAXS) SUPRA-MOLECULAR (GISAXS)

International SILS School
Muggia, 01 / 10 / 2026

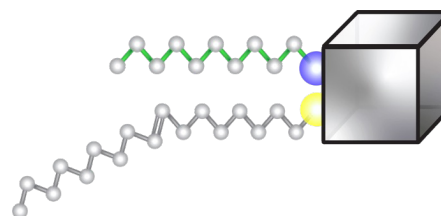
Oleylamine
C-18



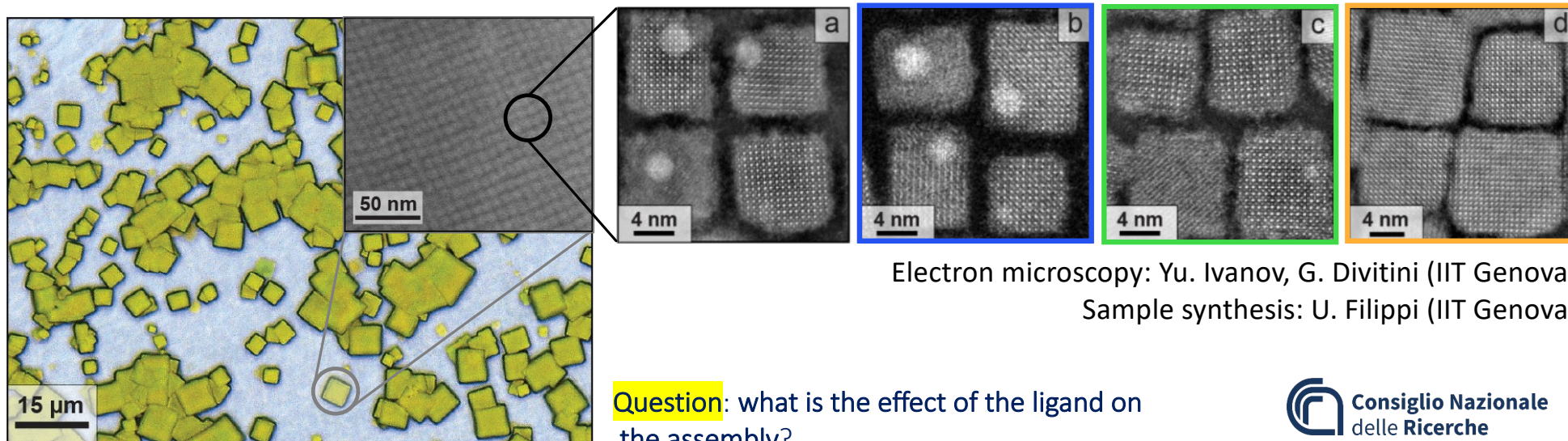
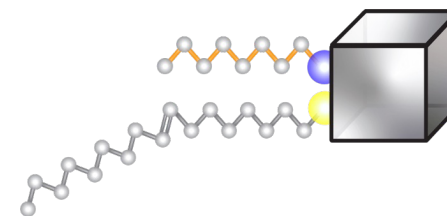
Dodecylamine
C-12



Decylamine
C-10



Octylamine
C-8

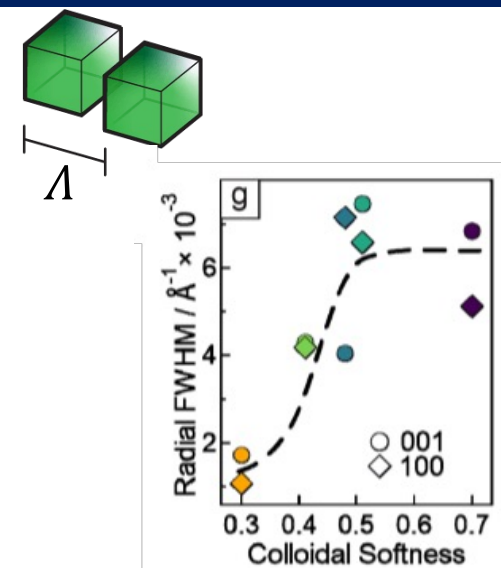
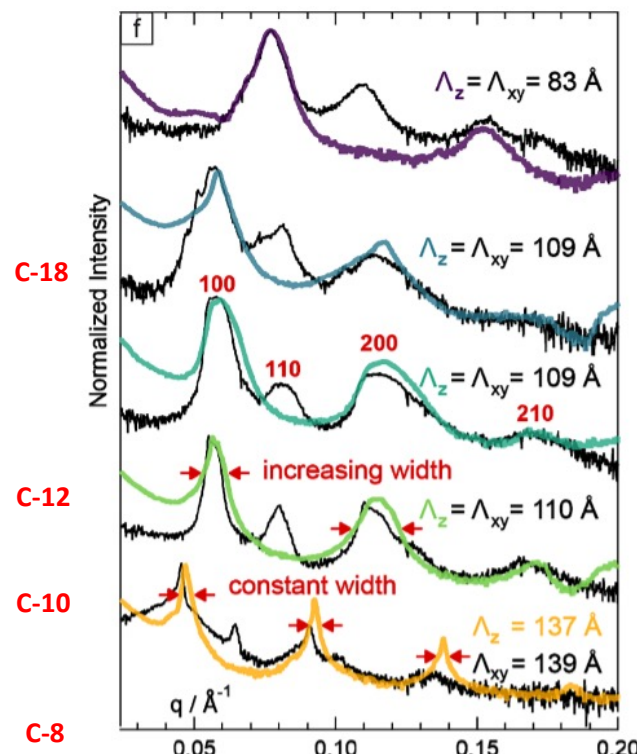
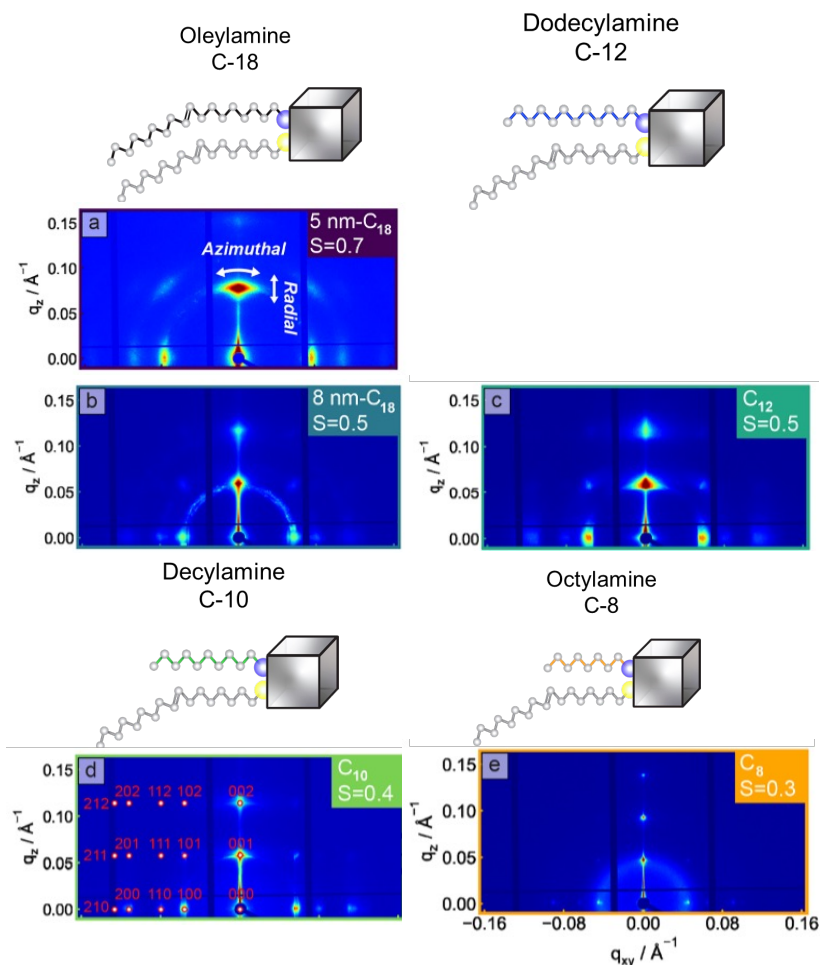


Electron microscopy: Yu. Ivanov, G. Divitini (IIT Genova)
Sample synthesis: U. Filippi (IIT Genova)

Question: what is the effect of the ligand on the assembly?

SUPRA-MOLECULAR (GISAXS)

International SILS School
Muggia, 01 / 10 / 2026

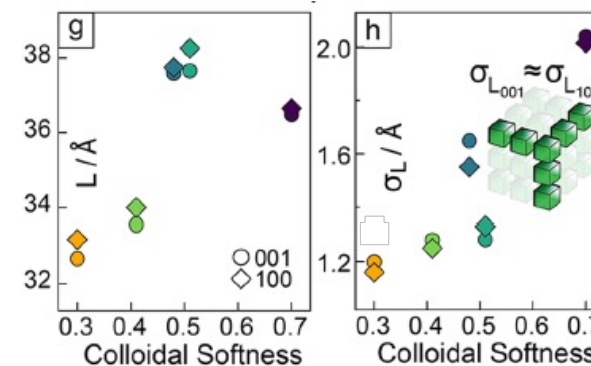
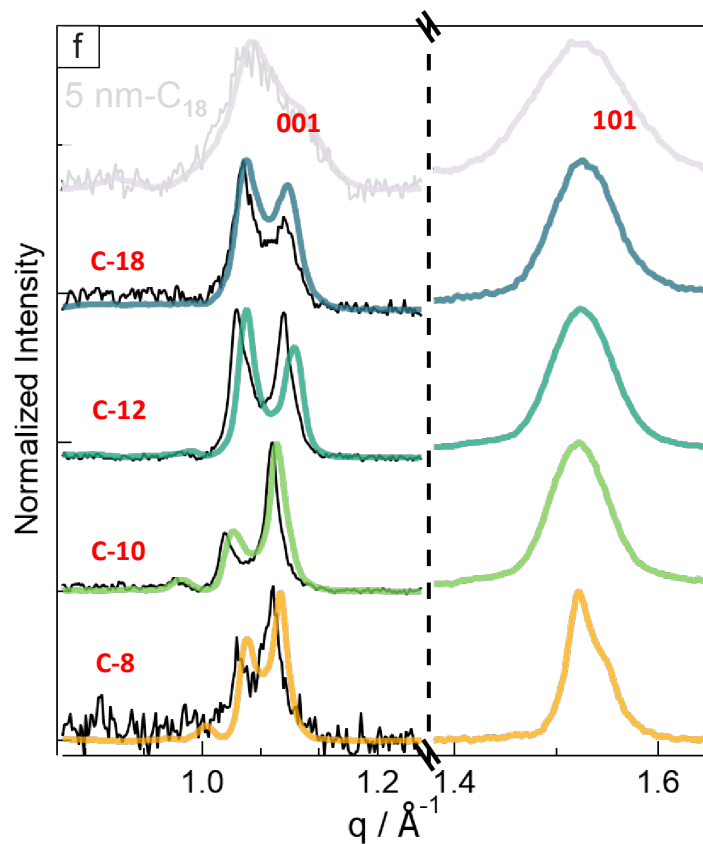
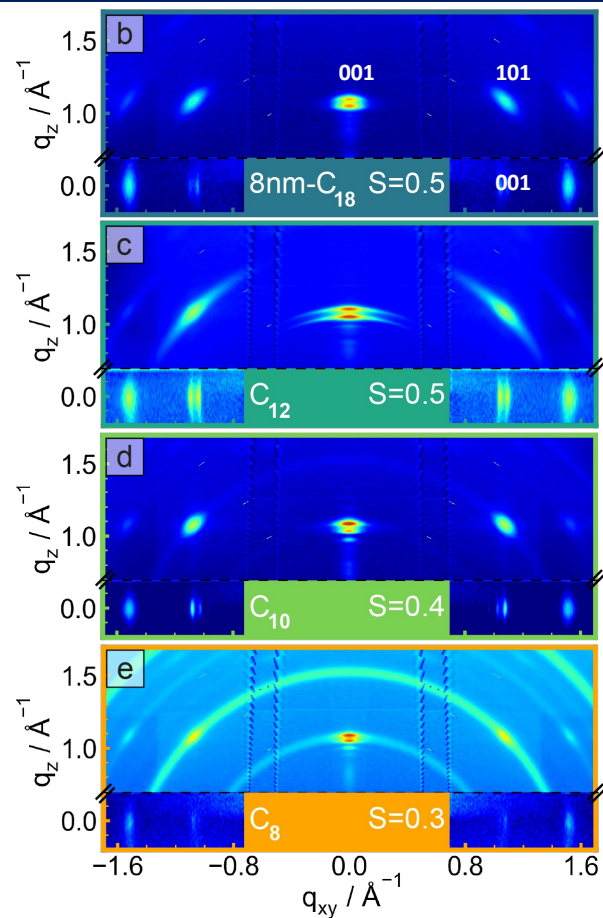


colloidal softness S of nanocrystals, defined as the ratio between the ligand shell thickness and the edge length of nanocrystals ($S = L/NC_{\text{edge}}$)

The GISAXS patterns were indexed as **cubic space group $Pm\bar{3}m$** using SUNBIM4.0 software

SUB-MOLECULAR (GIWAXS)

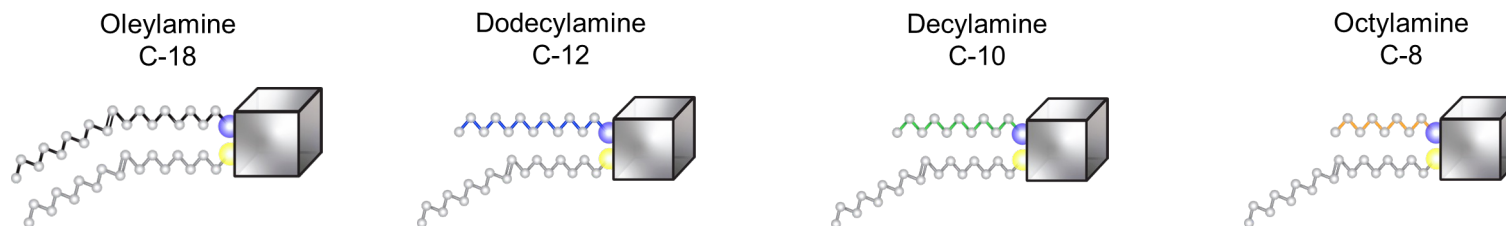
International SILS School
Muggia, 01 / 10 / 2026



GIWAXS patterns were indexed as
orthorhombic structure
(Pbnm, ICSD No. 98009-7851)

CONCLUSION

International SILS School
Muggia, 01 / 10 / 2026

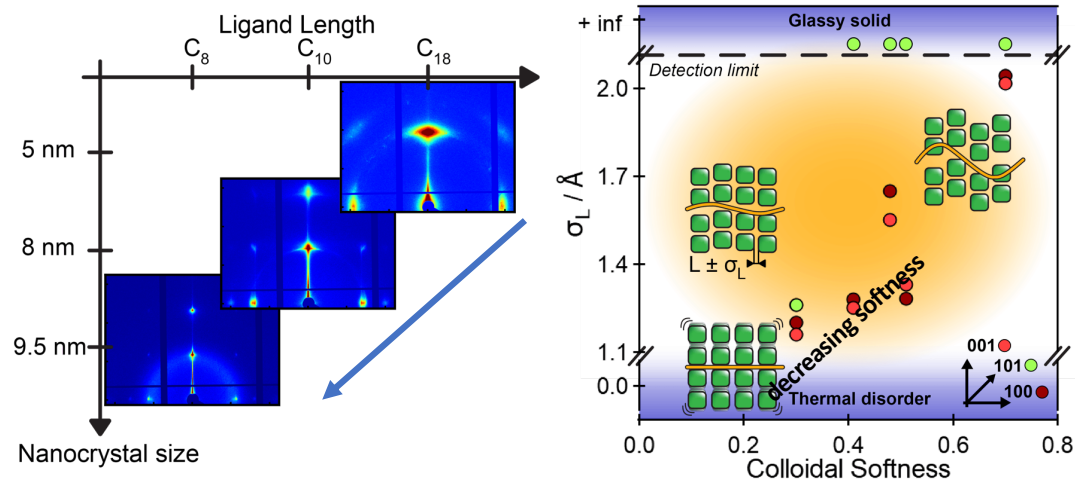


Ligand length

NC size

$$\text{NC softness} = L / \text{NC}_{\text{size}}$$

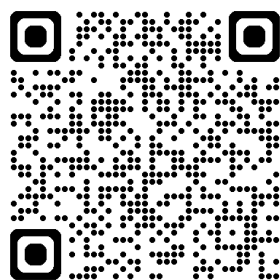
NC softness



colloidal softness S of nanocrystals, defined as the ratio between the ligand shell thickness and the edge length of nanocrystals ($S = L / \text{NC}_{\text{edge}}$)

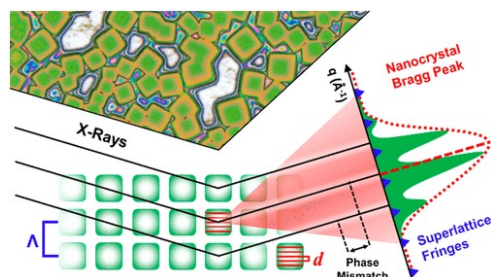
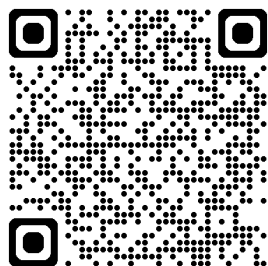
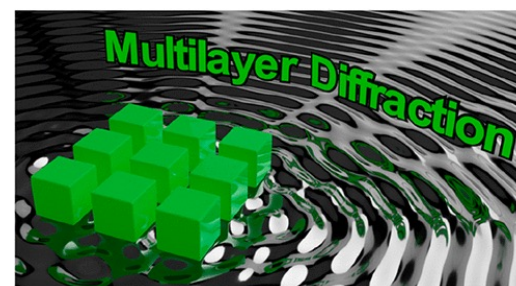
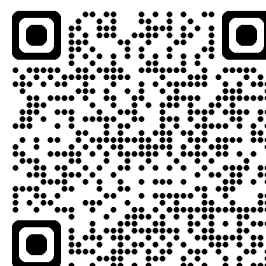
Conclusion: the interparticle spacing (L) and average nanocrystal displacement (σ_L) decrease with the shortening of the amine ligand

Key References



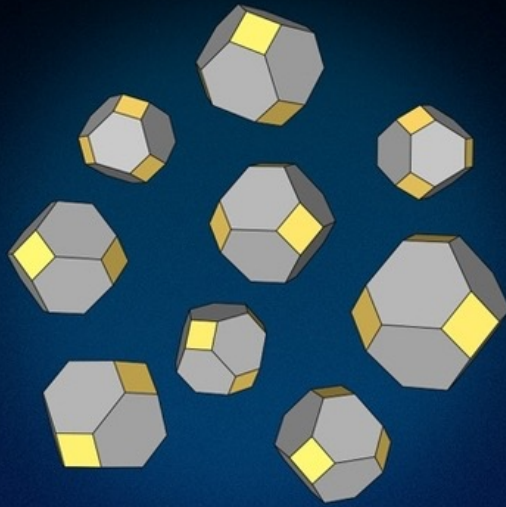
“Sinusoidal Displacement Describes Disorder in CsPbBr₃ Nanocrystal Superlattices” by Filippi U, Toso S, Ferreira MG, Tallarini L, Ivanov YP, Scattarella F, Haghighat V, Chen H, Landberg M, Divitini G, Wallentin J, Giannini C, Manna L, Baranov D,. *ACS Nano* **2026**, 20, 4, 3867–3877

“Collective Diffraction Effects in Perovskite Nanocrystal Superlattices” by Toso S, Baranov D, Filippi U, Giannini C, Manna L, Acc. *Chem. Res.* **2023**, 56, 66–76.

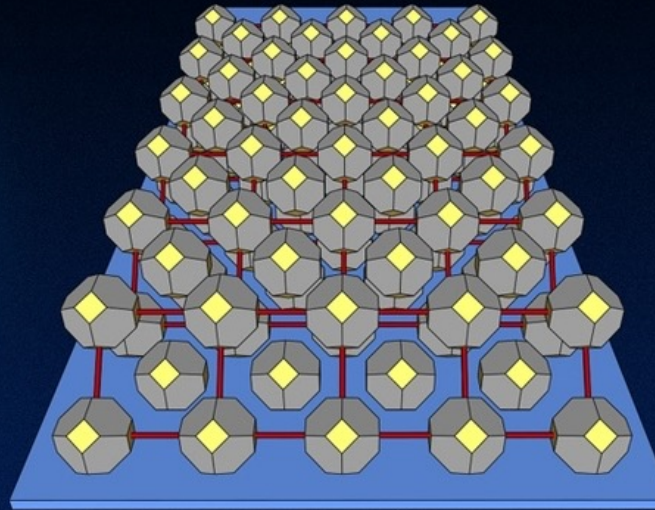


“Multilayer Diffraction Reveals That Colloidal Superlattices Approach the Structural Perfection of Single Crystals” by Toso S, Baranov D, Altamura D, Scattarella F, Dahl J, Wang XZ, Marras S, Alivisatos AP, Singer A, Giannini C, Manna L, *ACS Nano* **2021**, 15, 6243–6256.

initial state: colloidal suspension



*final state: atomically aligned
BCC superlattice*



Materials: X-ray diffraction/scattering and data modeling helps to gain insight into artificially ordered superlattices.

OUTLINE

International SILS School
Muggia, 01 / 10 / 2026

- SAXS – WAXS – Basic notions
- Scanning SAXS- WAXS Microscopy
- Nanocrystals and Nanocrystal-based Superlattices
- Scaffolds; Fibers

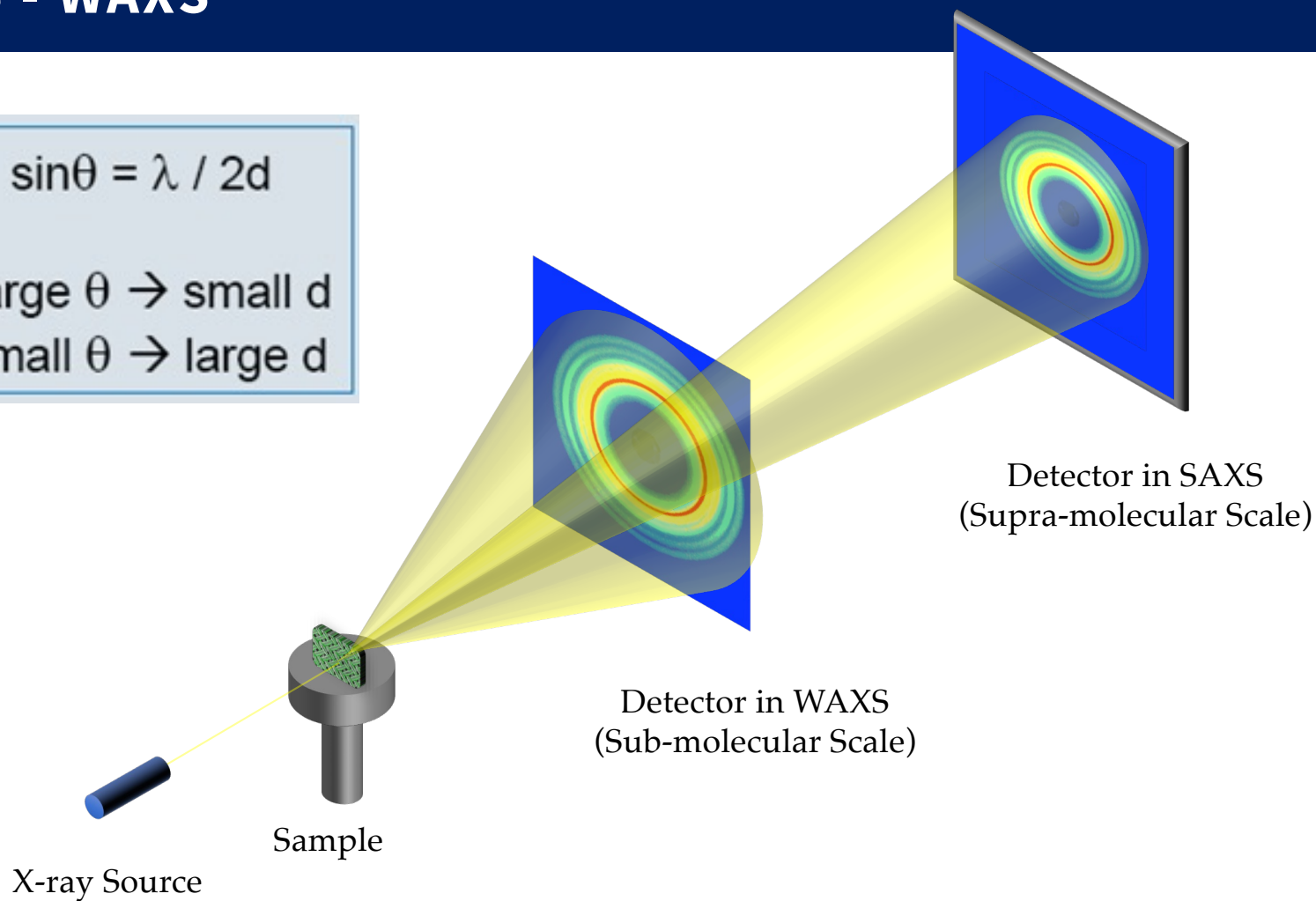
SAXS - WAXS

International SILS School
Muggia, 01 / 10 / 2026

$$\sin\theta = \lambda / 2d$$

Large $\theta \rightarrow$ small d

Small $\theta \rightarrow$ large d



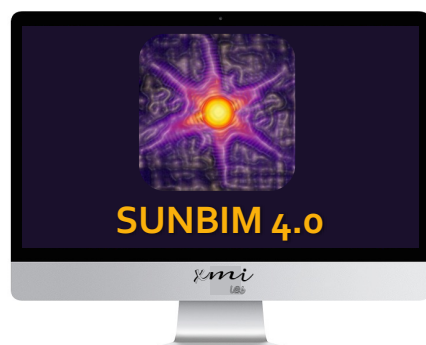
SCANNING MICROSCOPY

International SILS School
Muggia, 01 / 10 / 2026

SUNBIM: a package for X-ray imaging of nano- and biomaterials using SAXS, WAXS, GISAXS and GIWAXS techniques

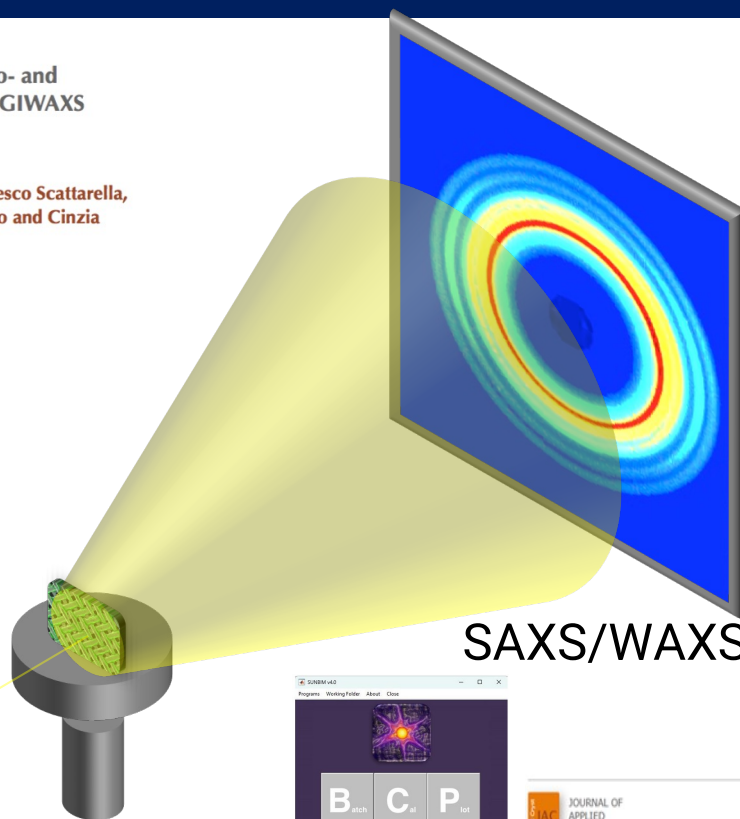
Dritan Siliqi, Liberato De Caro, Massimo Ladisa, Francesco Scattarella, Annamaria Mazzone, Davide Altamura, Teresa Sibillano and Cinzia Giannini

J. Appl. Cryst. (2016). **49**, 1107–1114



Scan Mode

X-ray source



SAXS/WAXS Detector



JOURNAL OF
APPLIED
CRYSTALLOGRAPHY
ISSN: 1600-5767

SUNBIM 4.0 software: new developments in small- and wide-angle X-ray scattering data analysis for scanning mode and grazing-incidence geometry

Francesco Scattarella,* Davide Altamura,* Teresa Sibillano,* Liberato De Caro,* Dritan Siliqi* and Cinzia Giannini*

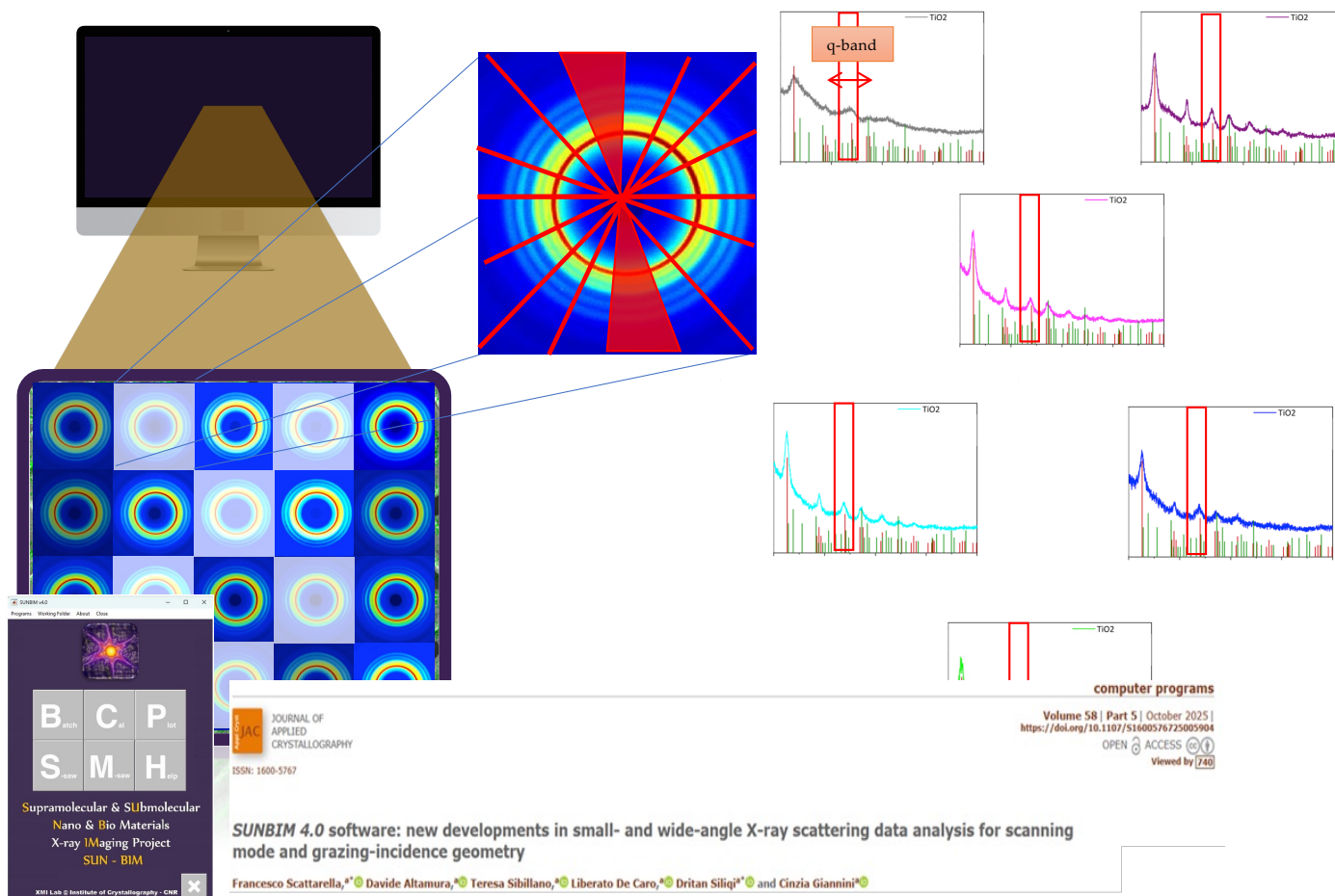
computer programs

Volume 58 | Part 5 | October 2025 |
<https://doi.org/10.1107/S1600576725005904>

OPEN ACCESS
Viewed by 740

BASIC SCANNING X-RAY MICROSCOPY

International SILS School
Muggia, 01 / 10 / 2026

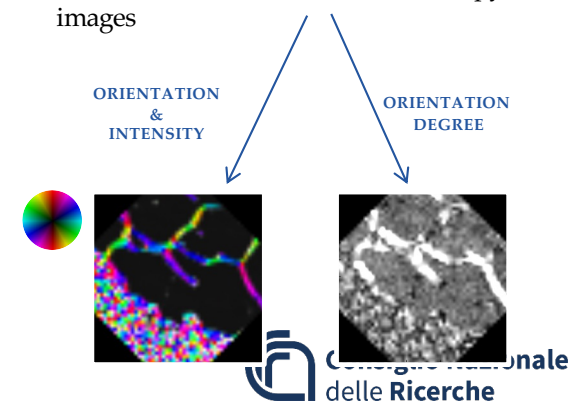


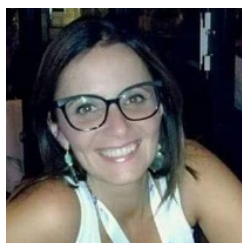
Multi-modal imaging approach: transform a mesh of two-dimensional SWAXS frames into microscopy images (*Bunk et al. New J. Phys. 11, 123016, 2009*)

- Each 2D frame of the mesh is folded into a one-dimensional profile
- From a specific 1D profile, properly selected from the composite map, the user has to choose one or more diffraction peaks (a q range)

Orientation & Intensity

Intensity distribution along the azimuth of the selected rings is analysed for each 2D frame and transformed into microscopy images





Maria Grazia Raucci



Luigi Ambrosio



3D Osteochondral Scaffold

- ✓ Mimic the gradient structure of healthy osteochondral tissue

Scanning Small- and Wide-Angle X-ray Scattering Microscopy Selectively Probes HA Content in Gelatin/Hydroxyapatite Scaffolds for Osteochondral Defect Repair

Davide Altamura,[†] Stella G. Pastore,[‡] Maria G. Raucci,[§] Dritan Siliqi,[†] Fabio De Pascalis,[§] Michele Nacucchi,[§] Luigi Ambrosio,^{‡,||} and Cinzia Giannini^{§,||}

[†]Institute of Crystallography (IC), National Research Council, Bari 70125, Italy

[‡]Institute of Polymers, Composites, and Biomaterials (IPCB), National Research Council, Naples, Italy

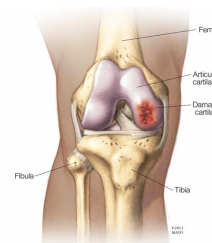
[§]Italian National Agency for New Technologies, Energy and Sustainable Economic Development (ENEA), Brindisi 72100, Italy

^{||}Department of Chemical Sciences and Materials Technology (DSCTM), National Research Council, Rome 000133, Italy

Polymer	Characteristic
Collagen	Major component of natural bone tissue. Excellent biocompatibility and cell -binding. Relatively weak mechanical stiffness and rapid biodegradation rate
Gelatin	Denaturalized form of collagen. Similar properties as collagen
Silk-fibroin (SF)	Structural protein of silk fibers. High mechanical strength and thermal stability. Flexible processability.
Chitosan	Polysaccharide. Good biocompatibility, antibacterial properties. Relatively weak mechanical properties
Alginate	Soluble in water, good biocompatibility
Hyaluronic acid	Soluble in water, good biocompatibility, Easy manipulation.
Poly(lactic acid) (PLA), poly(glycolic acid) (PGA) and copolymer PLGA	Tunable physical and chemical properties by adjusting the copolymer ratio.
Polycaprolactone (PCL)	Good mechanical strength, Slow degradation rate

Gelatin/HA scaffold (sol gel procedure)

- viscous gelatin solution, **type B (pH = 5)**, was prepared in distilled water at 40°C with constant adjustments of the pH (10-10.5).
- Simultaneously calcium nitrate $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ and diammonium hydrogen phosphate $(\text{NH}_4)_2\text{HPO}_4$ solutions were dissolved in distilled water and stirred for 2 hours.
- The **Ca^{2+} solution** was added to gelatin and mixed at 250 rpm for 2 hours at 40°C; after this time a **phosphate solution** was added to the system.
- The alkaline environment was reached by using NH_4OH solution.
- The samples were treated at **40°C for 24 hours (stirring at 85 rpm) and sonicated for 3 hours** to obtain an homogeneous system.
- After that, the gel system was poured into the Teflon moulds (5 x 10 mm) quenched to -80 °C and **freeze-dried for 48 hours**.
- For the preparation, **Ca/P ratio was fixed at 1.67** and the **HA contents with respect to gelatin-HA were considered at 30 wt%**. The gelatin amount with respect to the total water content is fixed at 10% w/v



Scanning Small- and Wide-Angle X-ray Scattering Microscopy Selectively Probes HA Content in Gelatin/Hydroxyapatite Scaffolds for Osteochondral Defect Repair

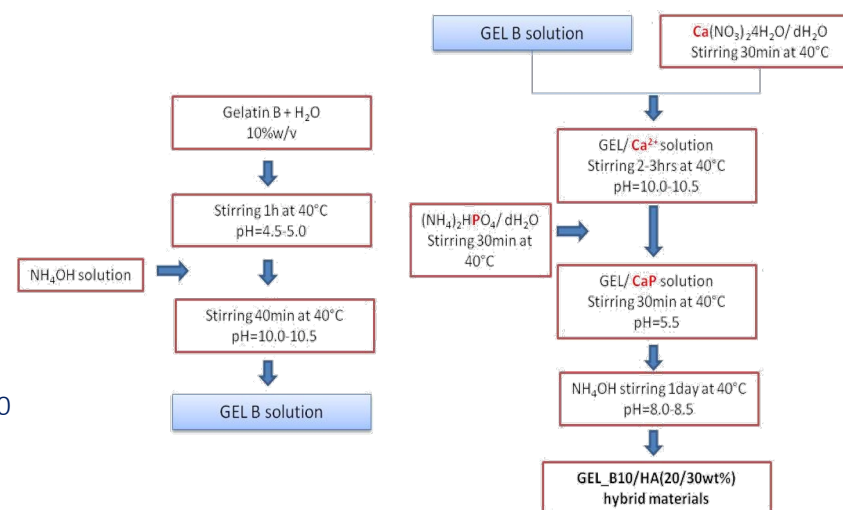
Davide Altamura,[†] Stella G. Pastore,[‡] Maria G. Raucci,[§] Dritan Siliqi,[†] Fabio De Pascalis,[§] Michele Nacucchi,[§] Luigi Ambrosio,^{‡,||} and Cinzia Giannini^{§,¶}

[†]Institute of Crystallography (IC), National Research Council, Bari 70125, Italy

[‡]Institute of Polymers, Composites, and Biomaterials (IPCB), National Research Council, Naples, Italy

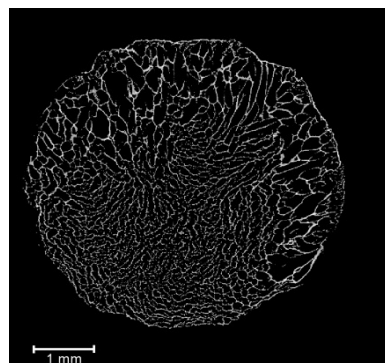
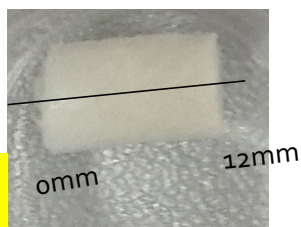
[§]Italian National Agency for New Technologies, Energy and Sustainable Economic Development (ENEA), Brindisi 72100, Italy

^{||}Department of Chemical Sciences and Materials Technology (DSCTM), National Research Council, Rome 000133, Italy



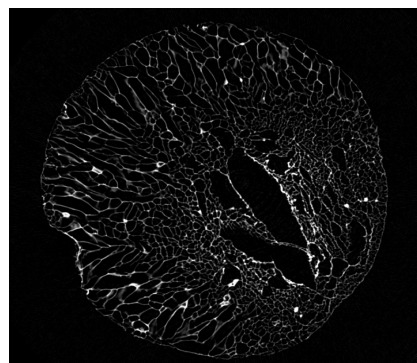
μ CT - μ tomography

Gel/HA



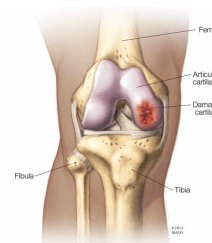
Gel

(d)



Gel/HA

(e)

ACS APPLIED MATERIALS
& INTERFACESResearch Article
www.acsami.org

Scanning Small- and Wide-Angle X-ray Scattering Microscopy Selectively Probes HA Content in Gelatin/Hydroxyapatite Scaffolds for Osteochondral Defect Repair

Davide Altamura,[†] Stella G. Pastore,[‡] Maria G. Raucci,[§] Dritan Siliqi,[†] Fabio De Pascalis,[§] Michele Nacucchi,[§] Luigi Ambrosio,^{‡,||} and Cinzia Giannini^{†,§,||}

[†]Institute of Crystallography (IC), National Research Council, Bari 70125, Italy

[‡]Institute of Polymers, Composites, and Biomaterials (IPCB), National Research Council, Naples, Italy

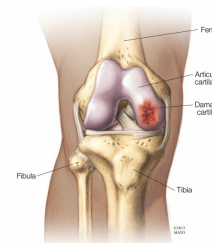
[§]Italian National Agency for New Technologies, Energy and Sustainable Economic Development (ENEA), Brindisi 72100, Italy

^{||}Department of Chemical Sciences and Materials Technology (DSCTM), National Research Council, Rome 000133, Italy

Micro-CT cross-sections clearly show the presence of **open and connected pores** in the structure of the gelatin/HA matrix.

Such a porous structure plays a critical role in the specific tissue formation, allowing migration and proliferation of osteoblasts and mesenchymal stem cells, besides improving mechanical interlocking between the implant biomaterial and the surrounding natural bone

X-ray Transmission



ACS APPLIED MATERIALS
INTERFACES

Research Article
www.acsami.org

Scanning Small- and Wide-Angle X-ray Scattering Microscopy Selectively Probes HA Content in Gelatin/Hydroxyapatite Scaffolds for Osteochondral Defect Repair

Davide Altamura,[†] Stella G. Pastore,[‡] Maria G. Raucci,[§] Dritan Siliqi,[†] Fabio De Pascalis,[§] Michele Nacucchi,[§] Luigi Ambrosio,^{‡,||} and Cinzia Giannini^{†,§,||}

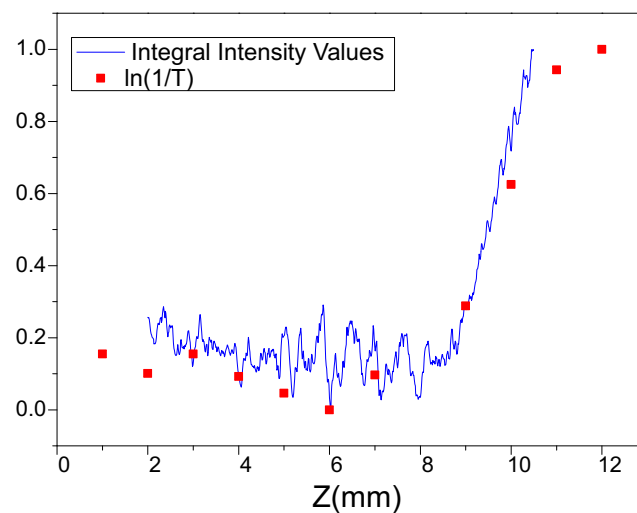
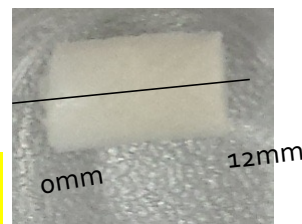
[†]Institute of Crystallography (IC), National Research Council, Bari 70125, Italy

[‡]Institute of Polymers, Composites, and Biomaterials (IPCB), National Research Council, Naples, Italy

[§]Italian National Agency for New Technologies, Energy and Sustainable Economic Development (ENEA), Brindisi 72100, Italy

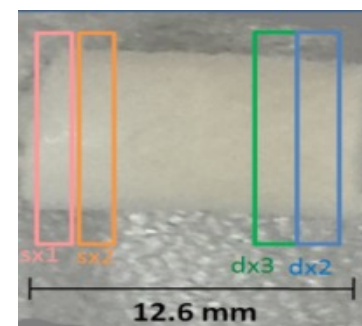
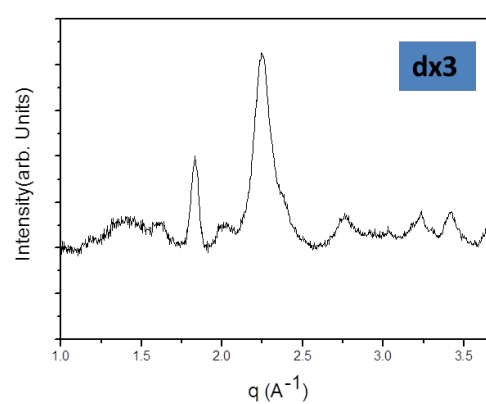
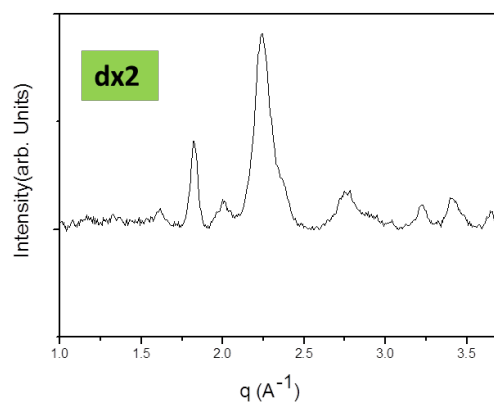
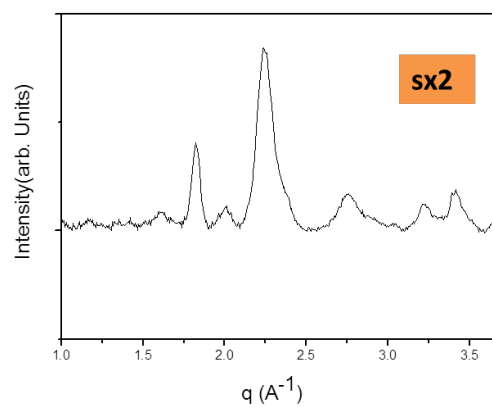
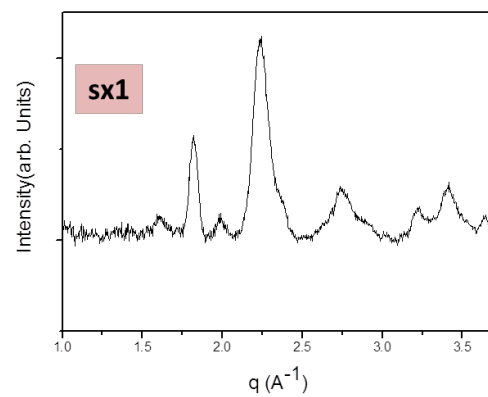
^{||}Department of Chemical Sciences and Materials Technology (DSCTM), National Research Council, Rome 000133, Italy

Gel/HA



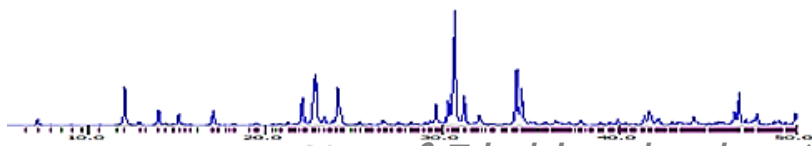
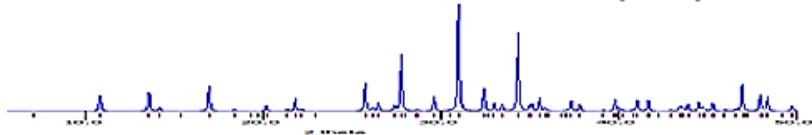
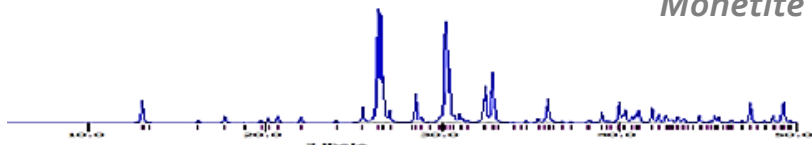
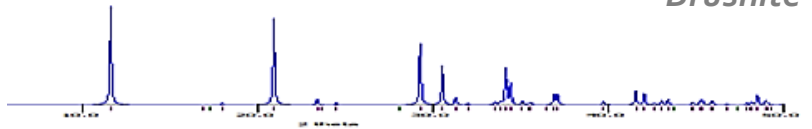
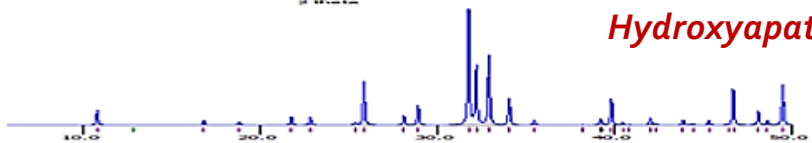
— μ CT

• WAXS

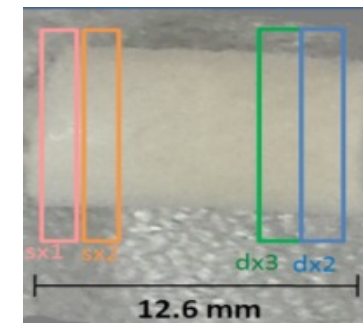
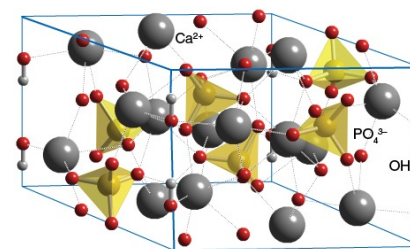


Gel/HA

WAXS

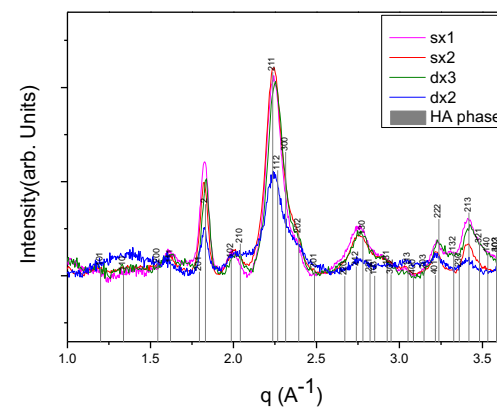
α -Tricalcium phosphate (TCP) *β -Tricalcium phosphate (TCP)**Monetite**Brushite**Hydroxyapatite*

Calcium Posphates crystals

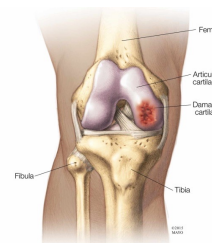


$a = 9.4154 \text{ \AA}$	$b = 9.4154 \text{ \AA}$	$c = 6.8792 \text{ \AA}$
$\alpha = 90^\circ$	$\beta = 90^\circ$	$\gamma = 120^\circ$
SG= P63/m		

Gel/HA



WAXS



ACS APPLIED MATERIALS
INTERFACES

Research Article
www.acsami.org

Scanning Small- and Wide-Angle X-ray Scattering Microscopy Selectively Probes HA Content in Gelatin/Hydroxyapatite Scaffolds for Osteochondral Defect Repair

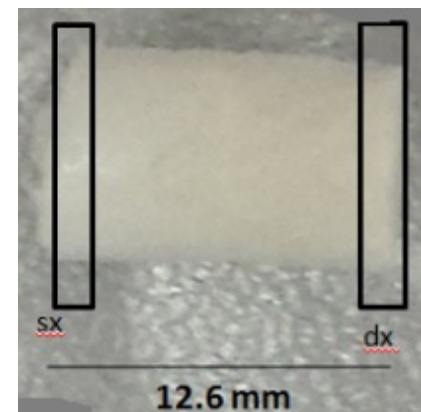
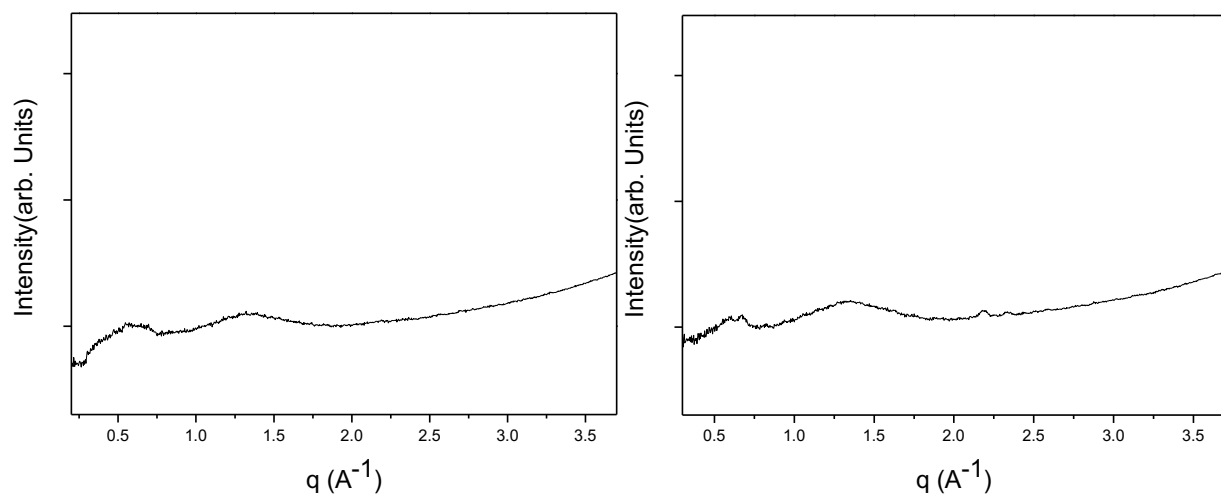
Davide Altamura,[†] Stella G. Pastore,[‡] Maria G. Raucci,[§] Dritan Siliqi,[†] Fabio De Pascalis,[§] Michele Nacucchi,[§] Luigi Ambrosio,^{‡,||} and Cinzia Giannini^{†,§,||}

[†]Institute of Crystallography (IC), National Research Council, Bari 70125, Italy

[‡]Institute of Polymers, Composites, and Biomaterials (IPCB), National Research Council, Naples, Italy

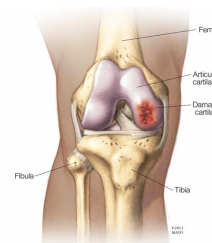
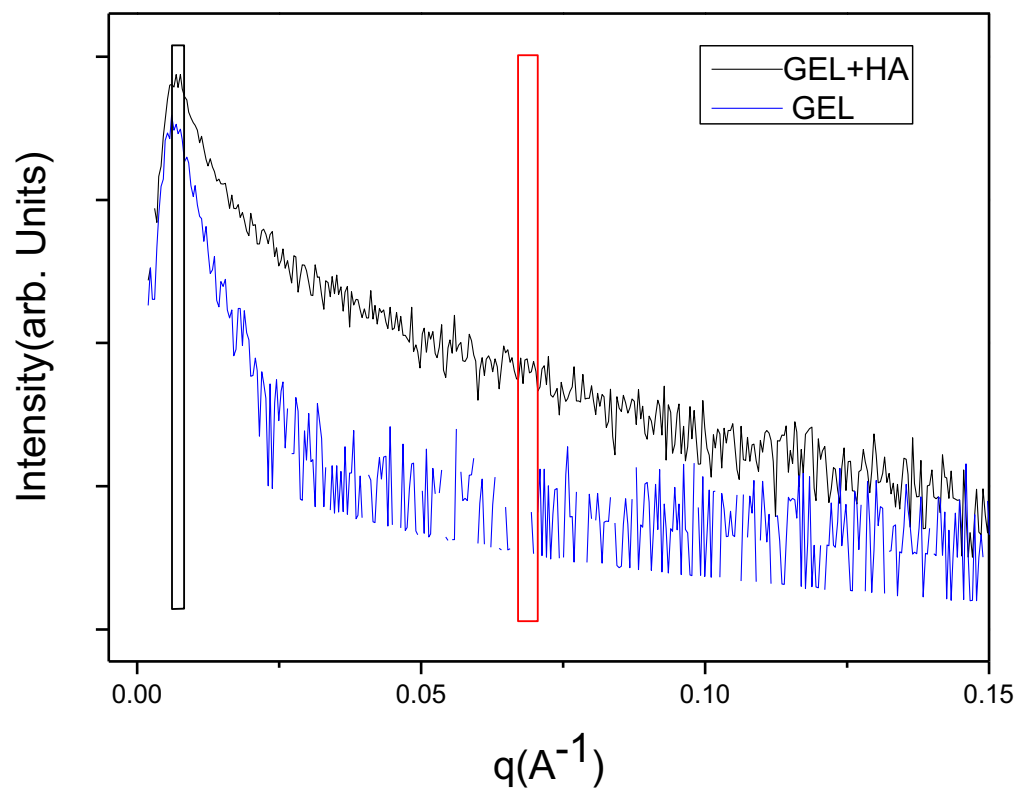
[§]Italian National Agency for New Technologies, Energy and Sustainable Economic Development (ENEA), Brindisi 72100, Italy

^{||}Department of Chemical Sciences and Materials Technology (DSCTM), National Research Council, Rome 000133, Italy



Gel

SAXS



Scanning Small- and Wide-Angle X-ray Scattering Microscopy Selectively Probes HA Content in Gelatin/Hydroxyapatite Scaffolds for Osteochondral Defect Repair

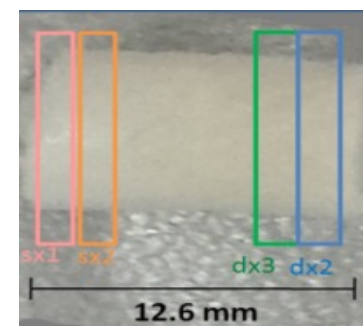
Davide Altamura,[†] Stella G. Pastore,[†] Maria G. Raucci,[‡] Dritan Siliqi,[†] Fabio De Pascalis,[§] Michele Nacucchi,[§] Luigi Ambrosio,^{‡,||} and Cinzia Giannini^{†,§,||}

[†]Institute of Crystallography (IC), National Research Council, Bari 70125, Italy

[‡]Institute of Polymers, Composites, and Biomaterials (IPCB), National Research Council, Naples, Italy

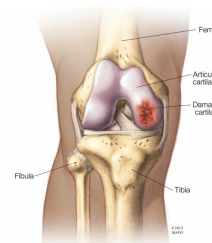
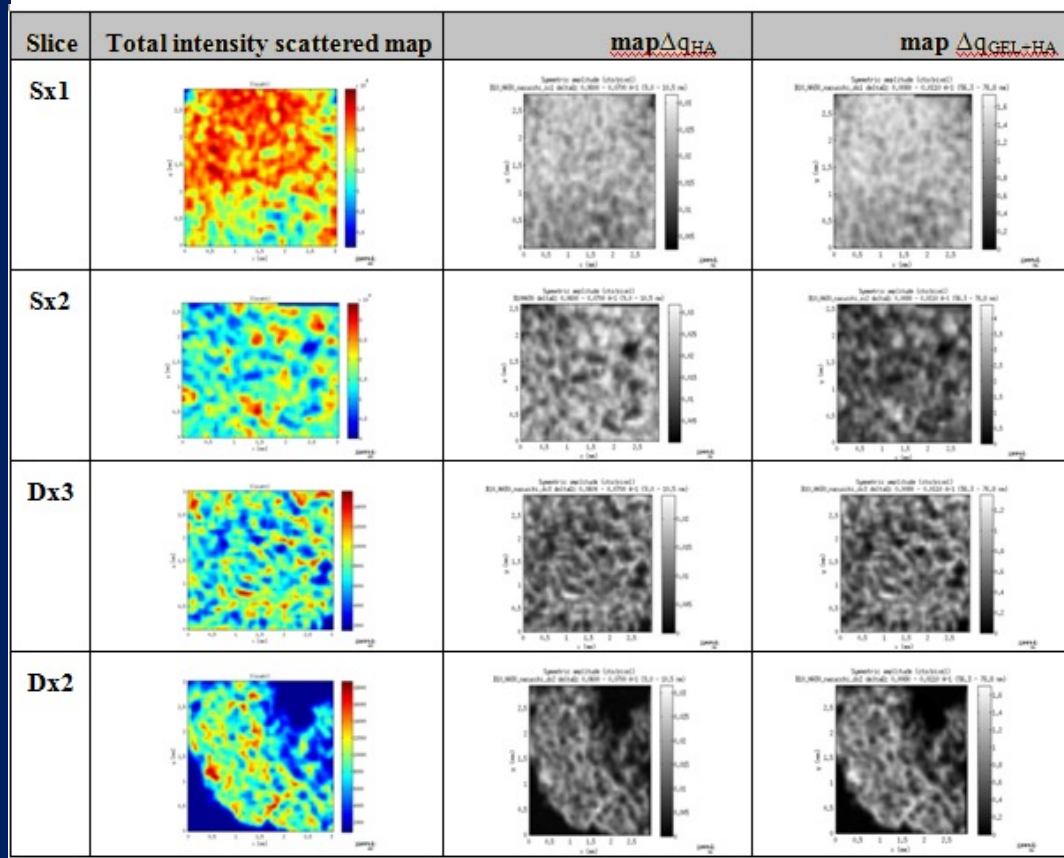
[§]Italian National Agency for New Technologies, Energy and Sustainable Economic Development (ENEA), Brindisi 72100, Italy

^{||}Department of Chemical Sciences and Materials Technology (DSCTM), National Research Council, Rome 000133, Italy



Gel/HA

Scanning SAXS microscopy



Scanning Small- and Wide-Angle X-ray Scattering Microscopy Selectively Probes HA Content in Gelatin/Hydroxyapatite Scaffolds for Osteochondral Defect Repair

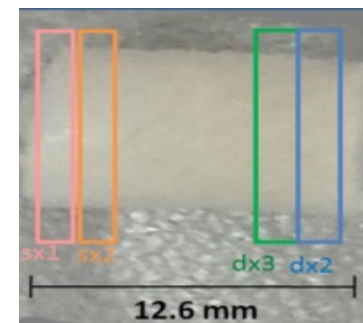
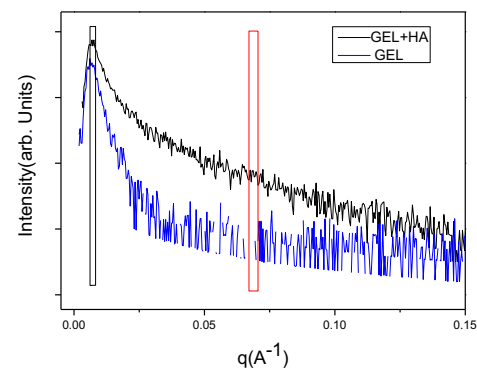
Davide Altamura,[†] Stella G. Pastore,[‡] Maria G. Raucci,[§] Dritan Siliqi,[†] Fabio De Pascalis,[§] Michele Nacucchi,[§] Luigi Ambrosio,^{‡,§} and Cinzia Giannini^{†,§}

[†]Institute of Crystallography (IC), National Research Council, Bari 70125, Italy

[‡]Institute of Polymers, Composites, and Biomaterials (IPCB), National Research Council, Naples, Italy

[§]Italian National Agency for New Technologies, Energy and Sustainable Economic Development (ENEA), Brindisi 72100, Italy

[§]Department of Chemical Sciences and Materials Technology (DSCMT), National Research Council, Rome 000133, Italy



Gel/HA

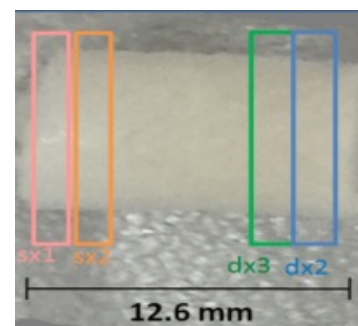
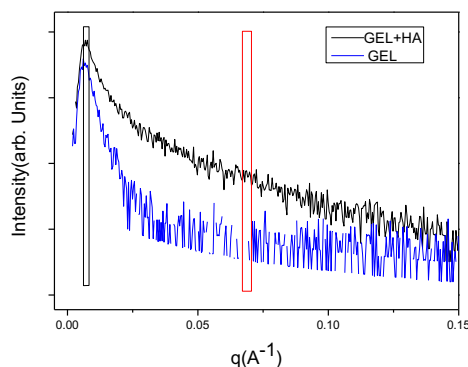
Scanning SAXS microscopy

Gel/HA	I(Sx1)	I(Sx2)	I(Dx3)	I(Dx2)
$\Delta q: 0.06-0.07 \text{ \AA}^{-1}$	10.38023709	10.24687648	15.00989277	12.92347856
$\Delta q: 0.008-0.011 \text{ \AA}^{-1}$	186.4373245	284.7826989	266.9744148	241.964543
Gel	I(Sx)		I(Dx)	
$\Delta q: 0.008-0.011 \text{ \AA}^{-1}$	116.36115		103.22153	

An intensity increases in the $\Delta q: 0.06-0.07 \text{ \AA}^{-1}$ range by **36%** resulted, from the left to the right side of the scaffold whereas only $\sim 8-10\%$ increase in the $\Delta q: 0.008-0.011 \text{ \AA}^{-1}$



HA GRADIENT



Gel/HA

Scanning Small- and Wide-Angle X-ray Scattering Microscopy Selectively Probes HA Content in Gelatin/Hydroxyapatite Scaffolds for Osteochondral Defect Repair

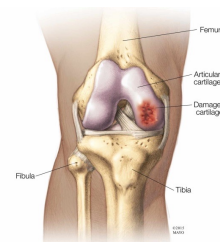
Davide Altamura,[†] Stella G. Pastore,[‡] Maria G. Raucci,[§] Dritan Siliqi,[†] Fabio De Pascalis,[§] Michele Nacucchi,[§] Luigi Ambrosio,^{‡,||} and Cinzia Giannini^{§,¶}

[†]Institute of Crystallography (IC), National Research Council, Bari 70125, Italy

[‡]Institute of Polymers, Composites, and Biomaterials (IPCB), National Research Council, Naples, Italy

[§]Italian National Agency for New Technologies, Energy and Sustainable Economic Development (ENEA), Brindisi 72100, Italy

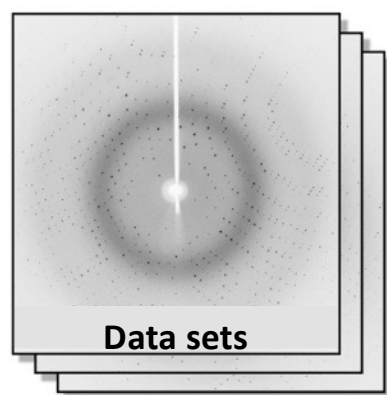
^{||}Department of Chemical Sciences and Materials Technology (DSCTM), National Research Council, Rome 000133, Italy



TYPE OF CRYSTALS

International SILS School
Muggia, 01 / 10 / 2026

Single-crystals



Single crystals

Single crystal



Twinned crystal



Crystal with mosaic spread



Textured sample



Polycrystalline crystals

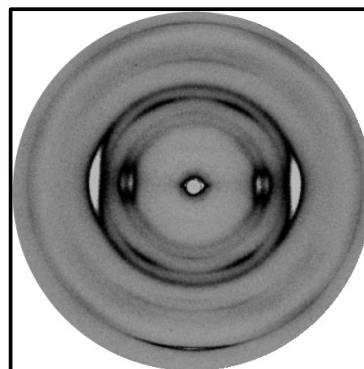
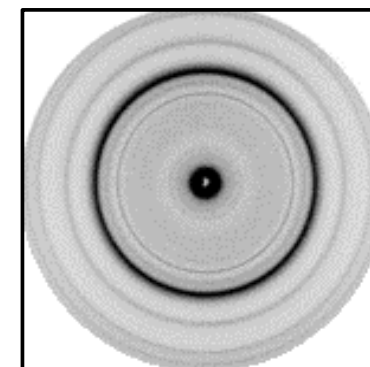
Powder sample



Nanocrystalline powder



Poly-crystals

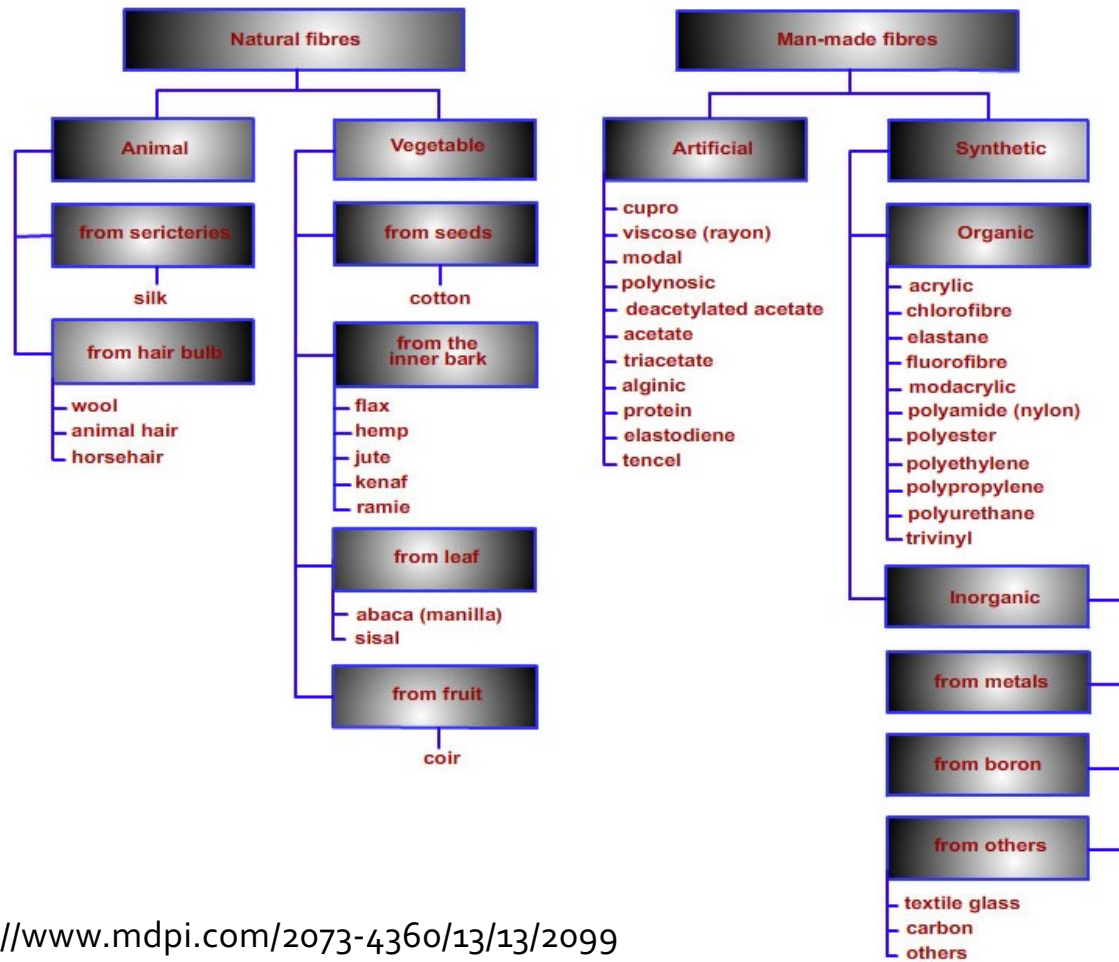


Fibers

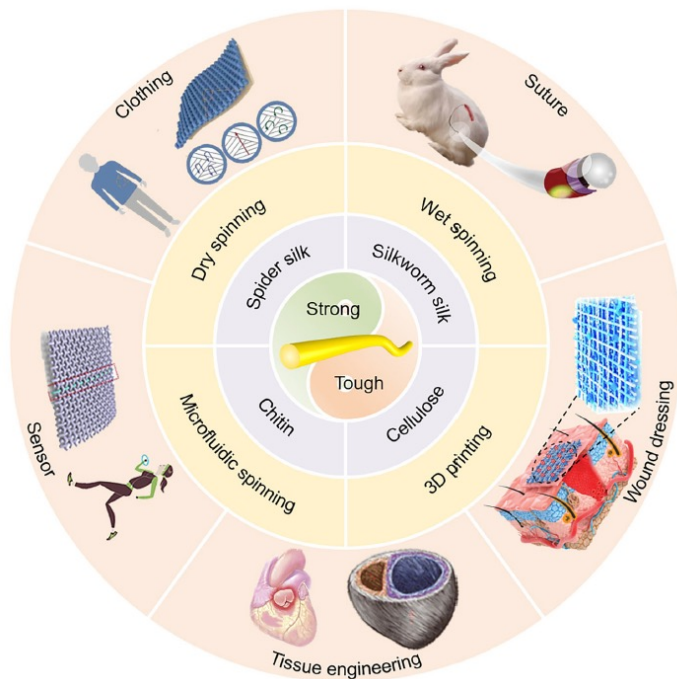


TYPE OF FIBERS

International SILS School
Muggia, 01 / 10 / 2026



<https://www.mdpi.com/2073-4360/13/13/2099>



BioFibers – whether natural or man-made – are semi-crystalline materials featuring **crystalline nanodomains** embedded within an amorphous (or highly disordered) matrix and **organized hierarchically**.

scientific commentaries



IUCrJ

ISSN 2052-2525

Exploring fibers with X-ray eyes

Cinzia Giannini*

Istituto di Cristallografia, Consiglio Nazionale delle Ricerche (CNR), Via Giovanni Amendola, 122/o, 70126 Bari, Italy.
*Correspondence e-mail: cinzia.giannini@cnr.it

DOI: 10.1002/mba2.70005

REVIEW ARTICLE

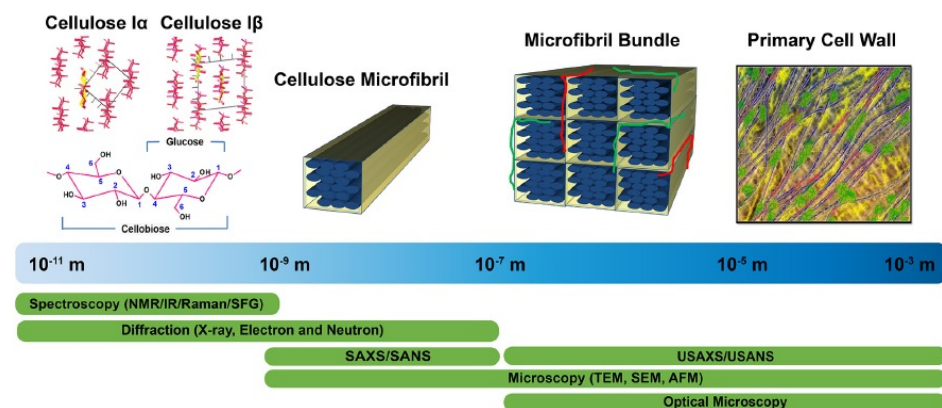
MedComm
BIOMATERIALS AND APPLICATIONS
WILEY

Design of strong and tough biofibers and their biomedical applications

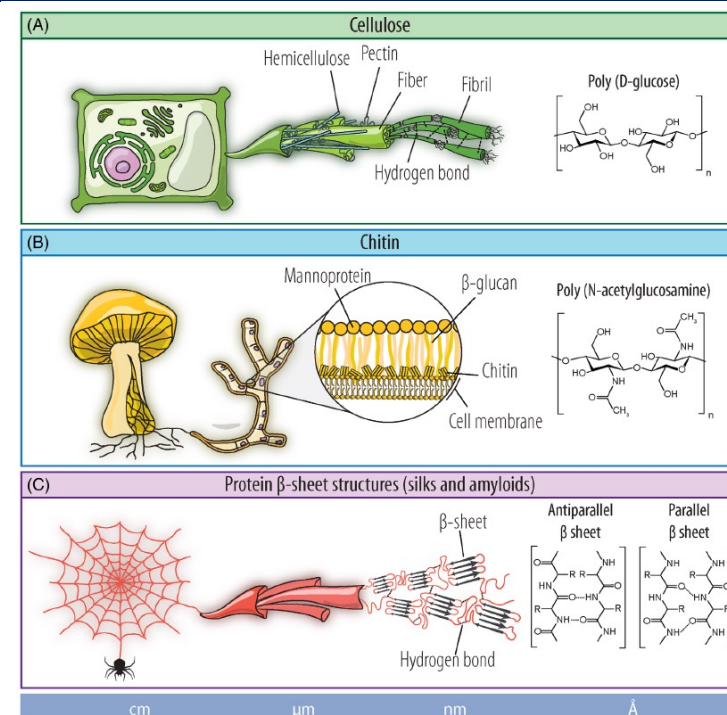
 **Consiglio Nazionale
delle Ricerche**

TOOLS : **hierarchical** structure characterization

International SILS School
Muggia, 01 / 10 / 2026



Technique	Length scale d (nm)
uSAXS	1000
SAXS	100
SAXS	10
WAXS	1
WAXS	0.1



DOI: 10.1002/pol.20230126

REVIEW

JOURNAL OF
POLYMER SCIENCE
WILEY

Hierarchical biopolymer-based materials and composites

**Consiglio Nazionale
delle Ricerche**



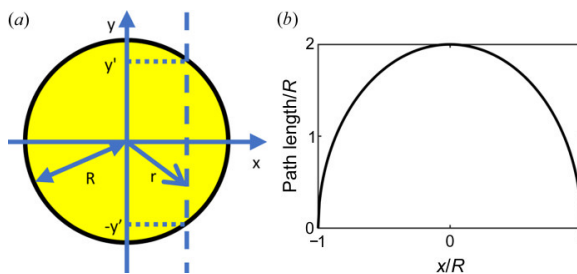
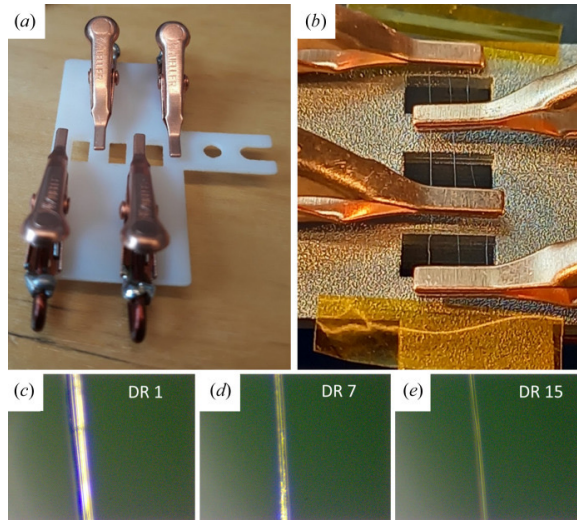
Progress and Opportunities in the
Characterization of Cellulose – An
Important Regulator of Cell Wall
Growth and Mechanics

Scanning **WAXS** microscopy on CELLULOSE FIBERS

International SILS School
Muggia, 01 / 10 / 2026

IUCrJ
ISSN: 2052-2525
NEUTRON | SYNCHROTRON

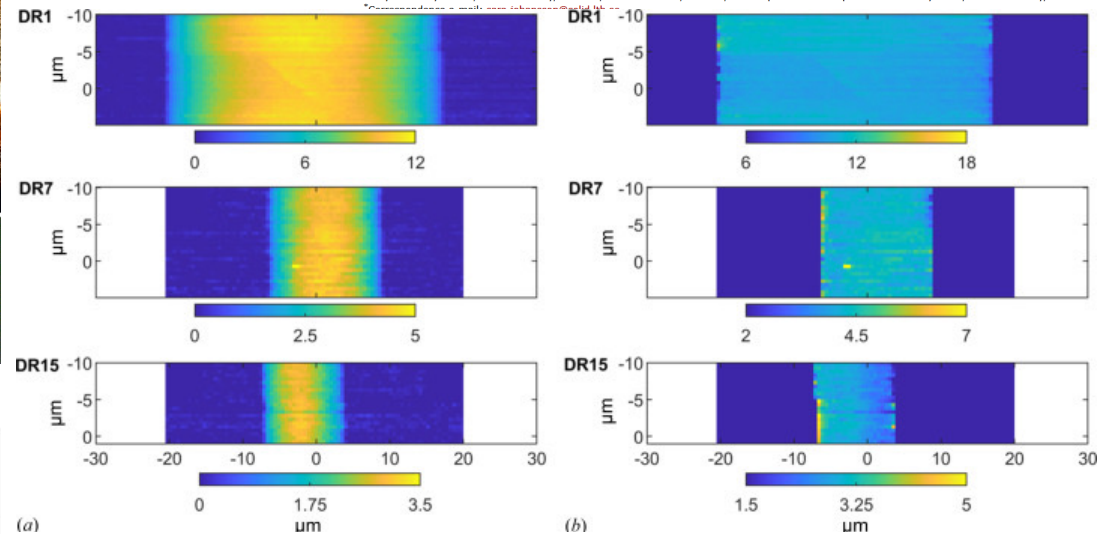
Volume 11 | Part 4 | July 2024 | Pages 570-577
<https://doi.org/10.1107/S205225252400383X>
OPEN ACCESS Cited by 2



Scanning WAXS microscopy of regenerated cellulose fibers at mesoscopic resolution

Sara Johansson,^{a,*} Francesco Scattarella,^b Sebastian Kalbfleisch,^c Ulf Johansson,^c Christopher Ward,^{c,d} Crispin Hetherington,^e Herbert Sixta,^f Stephen Hall,^g Cinzia Giannini,^h and Ulf Olsson^d

^aDivision of Solid Mechanics, Lund University, Box 118, Lund SE-22100, Sweden, ^bInstitute of Crystallography, National Research Council, Via Amendola 122/O, Bari 70126, Italy, ^cMAX IV Laboratory, Lund University, PO Box 118, Lund SE-22100, Sweden, ^dPhysical Chemistry, Lund University, Box 124, Lund SE-22100, Sweden, ^eNational Center for High Resolution Electron Microscopy, Centre for Analysis and Synthesis, Lund University, Box 124, Lund SE-22100, Sweden, and ^fDepartment of Bioproducts and Biosystems, Aalto University, PO Box 16300, Helsinki 00076, Finland



Scan maps after line-by-line background subtraction.

(b) Scan maps after radial path-length normalization of the background data [in Fig. 2 (a)]. The visualized range of values between the minimum and maximum are the same for each sample in (a) and (b).

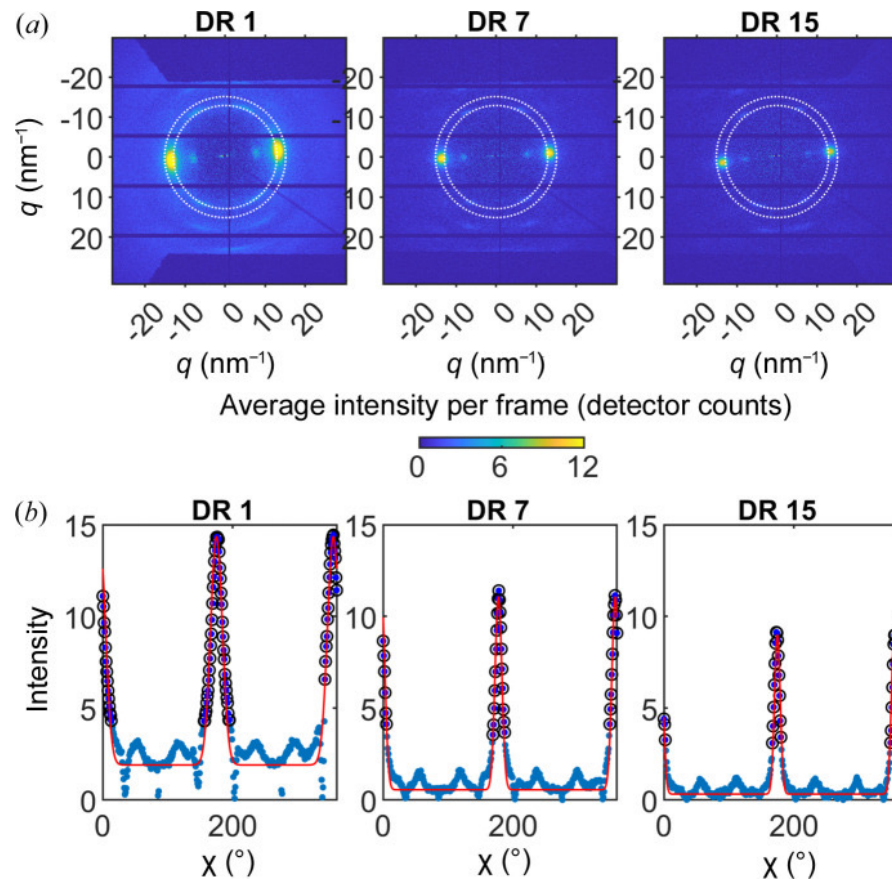


Ulf Olsson



Scanning **WAXS** microscopy on **CELLULOSE FIBERS**

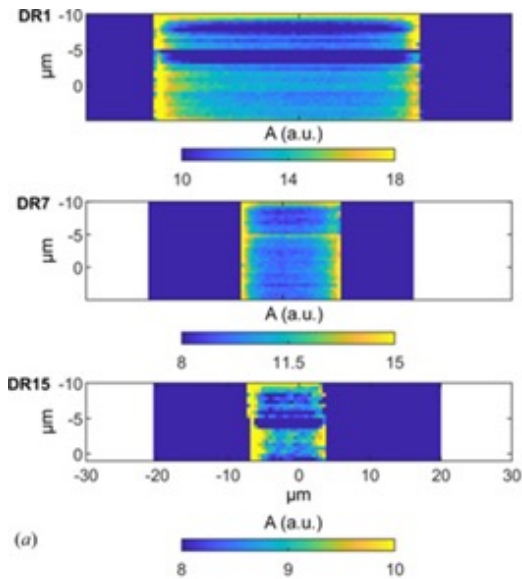
International SILS School
Muggia, 01 / 10 / 2026



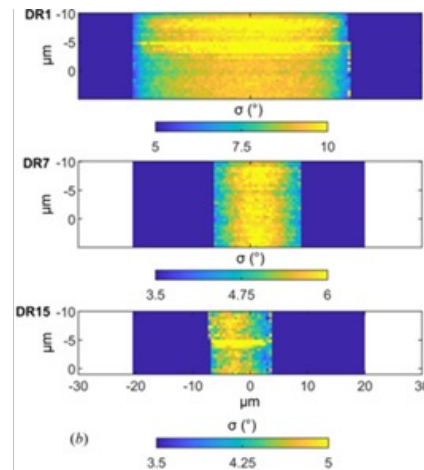
- (a) Average frame diffraction pattern after background normalization and path-length normalization. Dotted white circles on the patterns indicate the q -band selected for azimuthal integration.
- (b) Gaussian function fitted to the average frame diffraction patterns. Blue points show the azimuth integrated data within the selected q -band. Black circles mark the data used to fit the Gaussian functions (red lines).

Scanning **WAXS** microscopy on CELLULOSE FIBERS

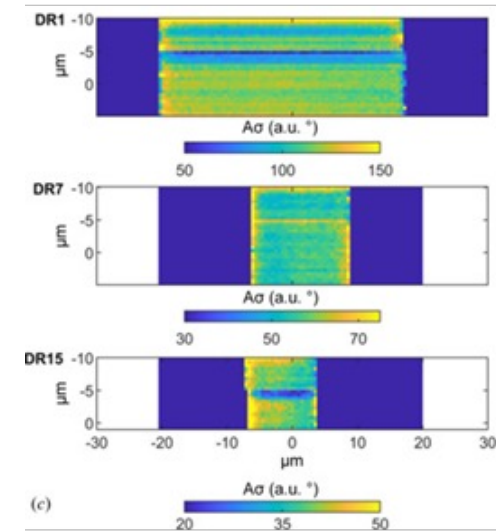
International SILS School
Muggia, 01 / 10 / 2026



Scan maps showing the variation of peak amplitude, A .



Scan maps showing the variation of standard deviation, σ .

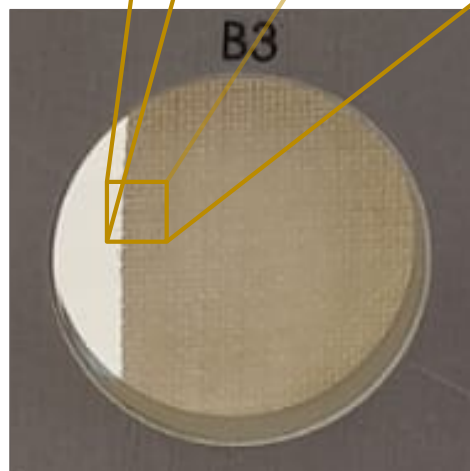
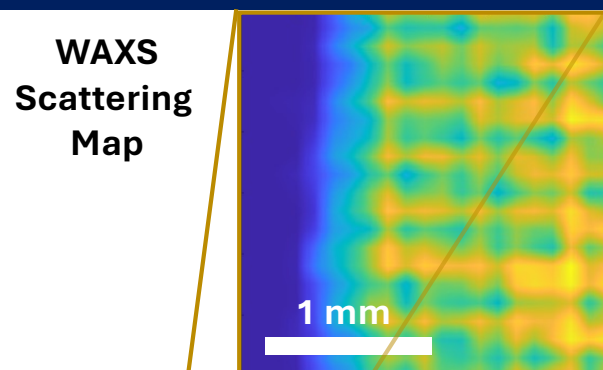


Scan maps of the product $A\sigma$, being a measure of the fiber crystallinity.

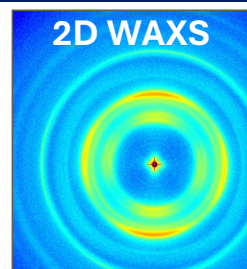
Fibers were found to be **homogeneous** in terms of crystallinity, but with a **radial gradient** in the crystal orientation here quantified as a **standard variation $\sigma(r)$** of an assumed **Gaussian distribution**. $\sigma(r)$ were found to be maximum in the center of the fibers ($r = 0$) and monotonically decrease towards the fiber surface. We attribute this to the **velocity gradients in the sample from the extrusion and from the drawing**. This additional structural information, on the sub-micrometre length scale, may be of use in the design of optimal spinning conditions and in the theoretical modeling of the fiber mechanical properties.

WAXS microscopy on CELLULOSE FIBERS

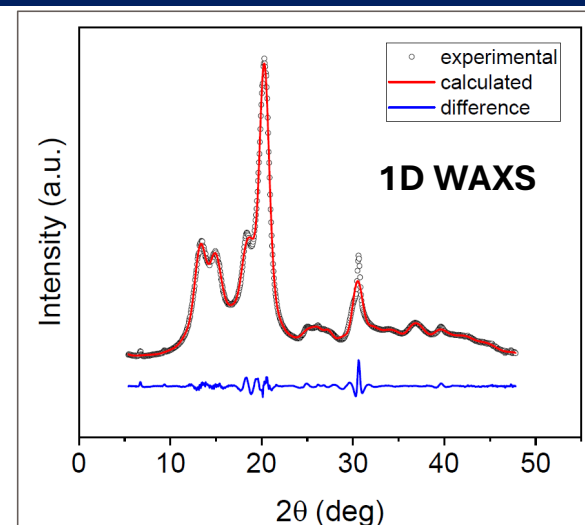
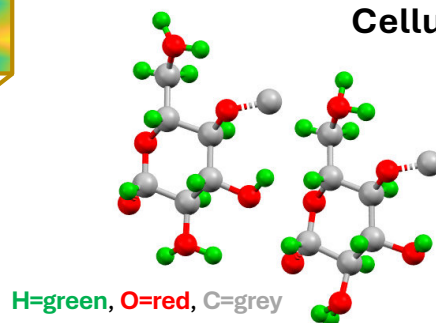
International SILS School
Muggia, 01 / 10 / 2026



Linen Fiber



Cellulose - β

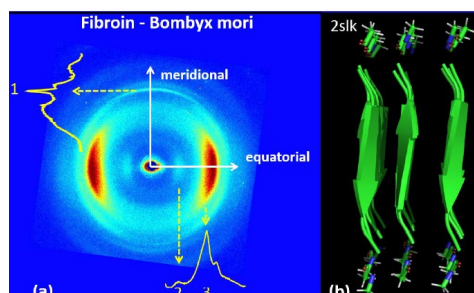


Cell Parameters					
a (Å)	b (Å)	c (Å)	α (°)	β (°)	γ (°)
7.78400	8.20100	10.38000	90.0	90.0	96.550
Cell Volume					
658.30 (Å ³)					

WAXS on SILK / COLLAGEN FIBERS

International SILS School
Muggia, 01 / 10 / 2026

FIBROIN



ACS OMEGA

Raman, WAXS, and Solid-State NMR Characterizations of Regenerated Silk Fibroin Using Lanthanide Ions as Chaotropic Agents

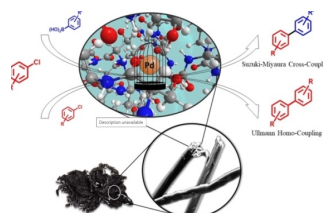
Giorgio Rizzo, Valentina Petrelli, Teresa Sibillano, Liberato De Caro, Maria Michela Giangregorio, Marco Lo Presti, Fiorenzo G. Omenetto, Cinzia Giannini,^{*} Piero Mastrorilli,^{*} and Gianluca M. Farinola^{*}

FULL PAPER

Macromolecular
Chemistry and Physics
www.mcp-journal.de

Silk Fibroin Processing from CeCl₃ Aqueous Solution: Fibers Regeneration and Doping with Ce(III)

Giorgio Rizzo, Marco Lo Presti, Cinzia Giannini, Teresa Sibillano, Antonella Milella, Giusy Matzeu, Roberta Musio,^{*} Fiorenzo G. Omenetto,^{*} and Gianluca M. Farinola^{*}



UNIVERSITÀ
DEGLI STUDI DI BARI
ALDO MORO

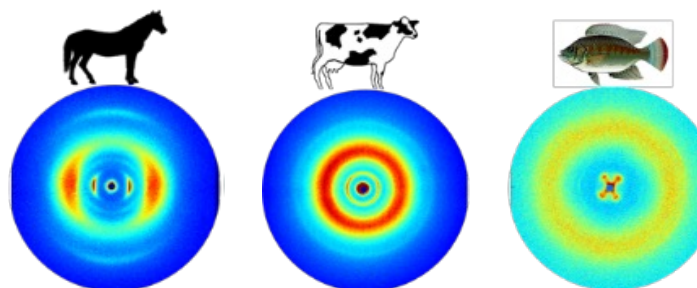
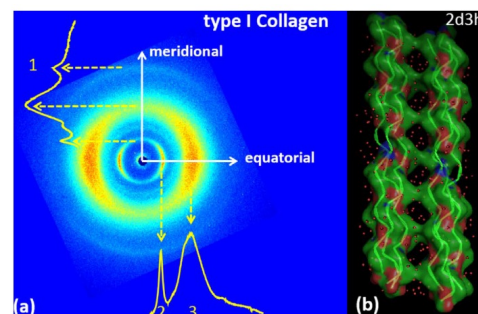


Gianluca Farinola



Giorgio Rizzo

COLLAGEN



Alessandro Sannino



Alberta Terzi

UNIVERSITÀ
DEGLI STUDI DI BARI
ALDO MORO

SCIENTIFIC REPORTS

OPEN Effects of processing on structural, mechanical and biological properties of collagen-based substrates for regenerative medicine

A. Terzi¹, E. Storti^{1,2}, S. Bettini¹, I. Sibillano¹, D. Altamura¹, J. Salvatore¹, M. Madaghiele¹, A. Sannino¹, F. Sibillano¹, M. Luciani¹, L. De Caro¹, A. Giannini¹, L. Velluto¹, A. Sannino¹, C. Giannini¹

frontiers in Bioengineering and Biotechnology

Investigations of Processing-Induced Structural Changes in Horse Type-I Collagen at Sub and Supramolecular Levels

Alberta Terzi¹, Nunzia Gallo¹, Simona Bettini¹, Teresa Sibillano¹, Davide Altamura¹, Lorenza Capobianco¹, Maria Lucia Natali^{1,2}, Luca Salvatore^{1,2}, Maria Madaghiele¹, Liberato De Caro¹, Ludovico Valli¹, Alessandro Sannino¹ and Cinzia Giannini^{1*}

REVIEW

Macromolecular
Bioscience
www.mbs-journal.de

Sub- and Supramolecular X-Ray Characterization of Engineered Tissues from Equine Tendon, Bovine Dermis, and Fish Skin Type-I Collagen

Alberta Terzi¹, Nunzia Gallo¹, Simona Bettini¹, Teresa Sibillano¹, Davide Altamura¹, Maria Madaghiele¹, Liberato De Caro¹, Ludovico Valli¹, Luca Salvatore¹, Alessandro Sannino¹, and Cinzia Giannini^{1*}

materials

Travelling through the Natural Hierarchies of Type I Collagen with X-rays: From Tendons of Cattle, Horses, Sheep and Pigs

Alberta Terzi¹, Nunzia Gallo^{1,2}, Teresa Sibillano¹, Davide Altamura¹, Annalisa Masi¹, Rocco Lassandro¹, Alessandro Sannino¹, Luca Salvatore^{1,2}, Oliver Bunk^{1,2}, Cinzia Giannini¹ and Liberato De Caro^{1,2}

crystals

WAXS and SAXS Investigation of Collagen-Rich Diet Effect on Multiscale Arrangement of Type I Collagen in Tilapia Skin Fed in Aquaponics Plant

Alberta Terzi¹, Teresa Sibillano^{1,2}, Liberato De Caro^{1,2}, Davide Altamura¹, Nunzia Gallo^{1,2}, Maria Lucia Natali^{1,2}, Alessandro Sannino¹, Luca Salvatore^{1,2}, Federica Stella Blasi¹, Angelo Corallo¹ and Cinzia Giannini¹

polymers

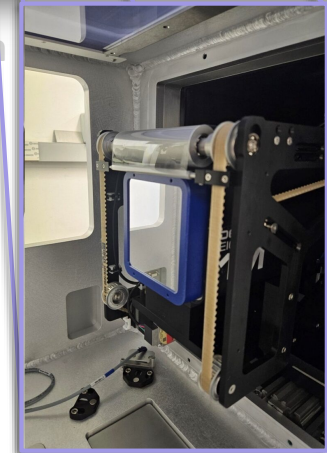
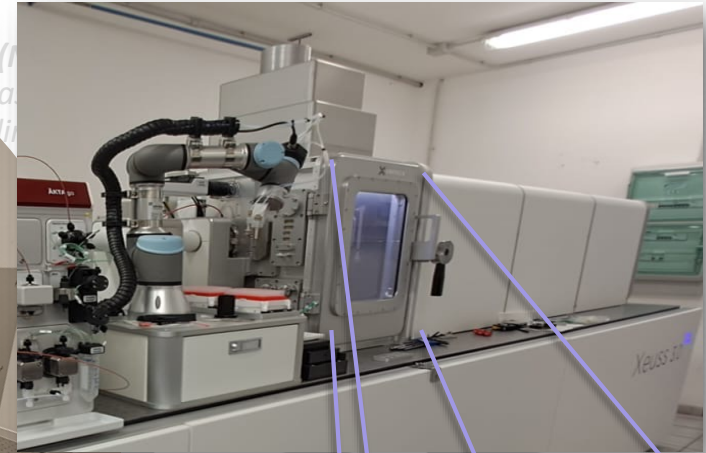
Aquaponics-Derived Tilapia Skin Collagen for Biomaterials Development

Nunzia Gallo^{1,2}, Maria Lucia Natali^{1,2}, Alessandro Quarta^{1,2}, Antonin Gahleitner¹, Alberta Terzi¹, Teresa Sibillano¹, Cinzia Giannini¹, Giuseppe Egilio De Benedetto¹, Paola Lunetti¹, Loredana Capobianco¹, Federica Stella Blasi¹, Alessandro Sicuro¹, Angelo Corallo¹, Alessandro Sannino¹ and Luca Salvatore^{1,2}

XEUSS 3.0 HR LABORATORY

<https://www.itaca-sb.it/biosaxs/>

- Motorized **dual source** (Ga Excillum liquid jet source and Cu micro-source)
- **Detector** Eiger 2R (1M), moving in vacuum from 42.5 mm to 1800 mm (SDD)



- Size-exclusion **chromatography coupled with SAXS (SEC-SAXS)** using an AKTA goTM protein purification system

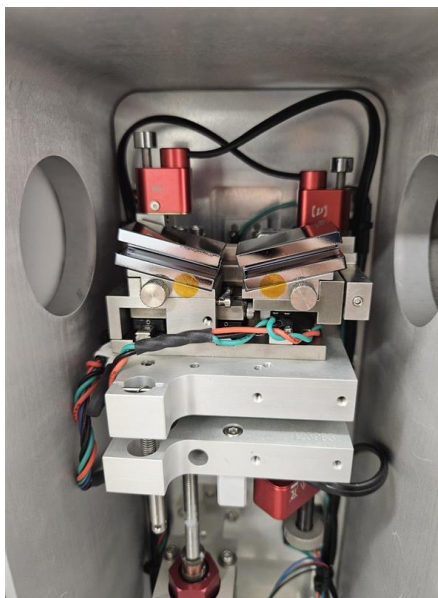
XEUSS 3.0 HR LABORATORY

International SILS School
Muggia, 01 / 10 / 2026

- **Biocube System**



- Bonse-Hart 4-bounce Si (111)
channel cut crystals **monochromator**
and analyzer for **USAXS**



XEUSS 3.0 HR LABORATORY

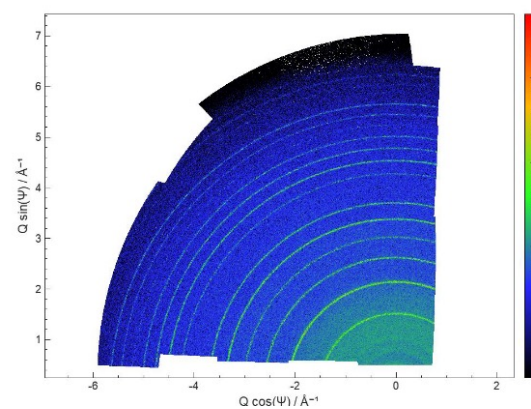
International SILS School
Muggia, 01 / 10 / 2026



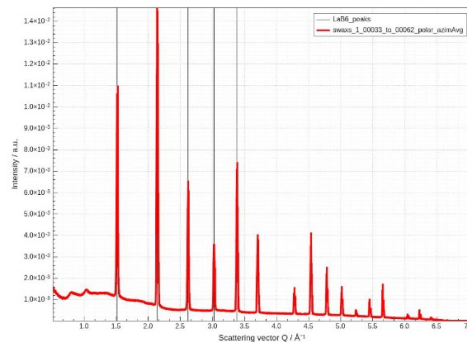
Models	Xeuss 3.0 C	Xeuss 3.0 HR	Xeuss 3.0 UHR
Footprint [Width x Length]	1 m x 3 m	1 m x 5 m	1 m x 8 m

4-Axis motorized **SWAXS** module with EIGER2 R 500K

International SILS School
Muggia, 01 / 10 / 2026



Up to $2\theta = 90^\circ$ for out-of-plane scattering



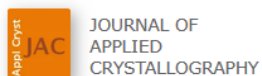
Up to $2\theta = 90^\circ$
for in-plane scattering

Sample Environment/Holder

International SILS School
Muggia, 01 / 10 / 2026

- Capillaries
- Solids
- Gels
- GISAXS/GIWAXS
- Shear Cell
- Reactivity Chamber

laboratory notes



JOURNAL OF
APPLIED
CRYSTALLOGRAPHY

ISSN: 1600-5767

Volume 59 | Part 5 | October 2026 |
<https://doi.org/10.1107/S1600576726007132>

OPEN ACCESS Cited by 1

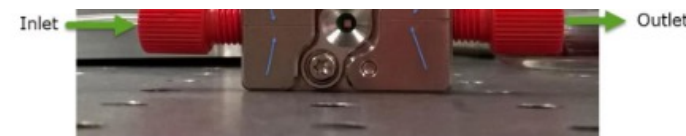
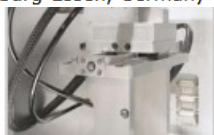
The BioSAXS laboratory of the ITACA.SB Research Infrastructure

Francesco Scattarella,^{a,ib} Davide Altamura,^{a,ib} Vincenzo Mangini,^{a,ib} Teresa Sibillano,^{a,ib} Erika Manicone,^{a,b} Rocco Lassandro,^a Liberato De Caro,^a Gemma Newby^c and Cinzia Giannini^{a*}

^aIstituto di Cristallografia, Consiglio Nazionale delle Ricerche, via Amendola 122/O, Bari, 70126, Italy, ^bDipartimento di Chimica, Università degli Studi di Bari, Via Edoardo Orabona 4, Bari, 70126, Italy, and ^cXenocs SAS, Allée du Nanomètre, Grenoble, 38000, France

*Correspondence e-mail: cinzia.giannini@cnr.it

Edited by S. Disch, Universität Duisburg-Essen, Germany (Received 17 March 2026; accepted 10 July 2026; online 24 August 2026)



(-20° to 150°C)



Potentiating the Italian Capacity for Structural Biology Services in Instruct-ERIC



THANKS!

International SILS School
Muggia, 01 / 10 / 2026



Brazilian Synchrotron
Light Laboratory



GRAZIE !



Cinzia Giannini
Istituto di Cristallografia, Italia 70126 Bari via Amendola 122/O
✉ cinzia.giannini@cnr.it ☎ +39 080 5929167

