Using synchrotrons to study structure-property relationships in complex nanostructured materials

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You could have access to a synchrotron!

Through the LAAAMP 2023 FaST grant program

Goal of this talk:

1. What could you do to study complex material structure-property relationships if you had access to a synchrotron
2. What capabilities are present at NSLS-II at Brookhaven National Laboratory and Diamond Light source in UK

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Brookhaven National Laboratory

National Synchrotron Light Souce-II (NSLS-II) =>

- XPD beamline
- Coherence
- Small beams
- High energy resolution
- Resonant scattering

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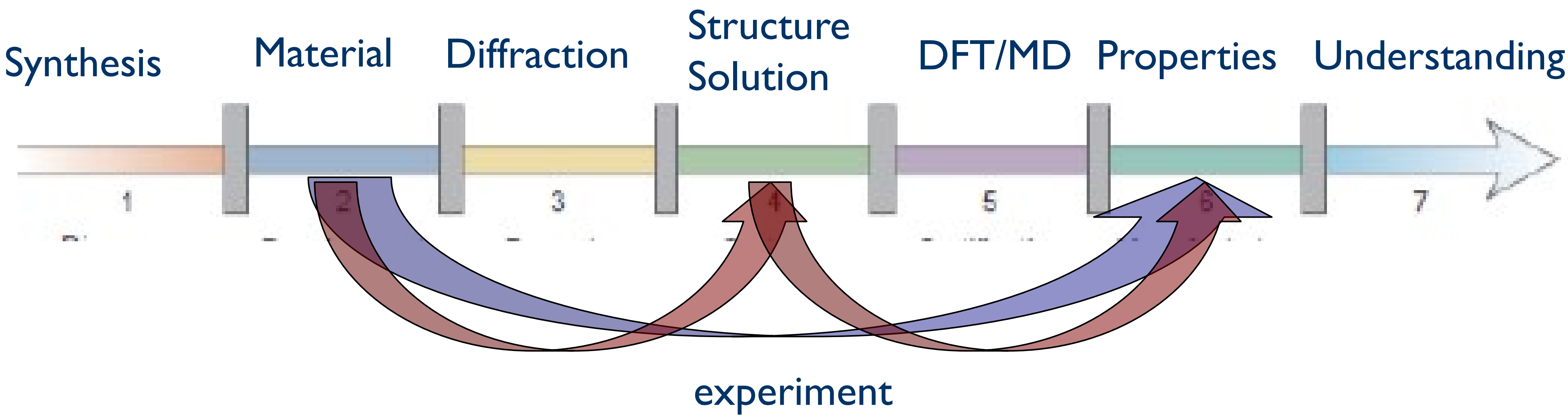
Current paradigm for materials discovery



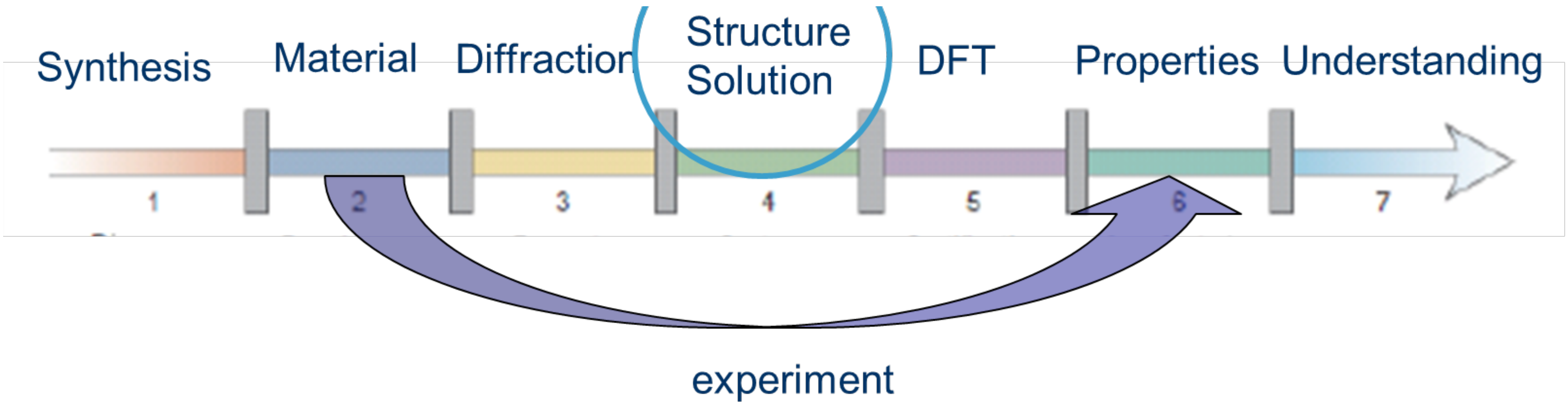
- Key step is Diffraction -> Structure solution. Understanding flows from that.
- The structure solution step is crystallography
  - According to IUCr, 48 nobels associated with crystallography (some loosely!)
- Crystals are idealizations of real material structure

Real Materials: more complex than ideal crystals

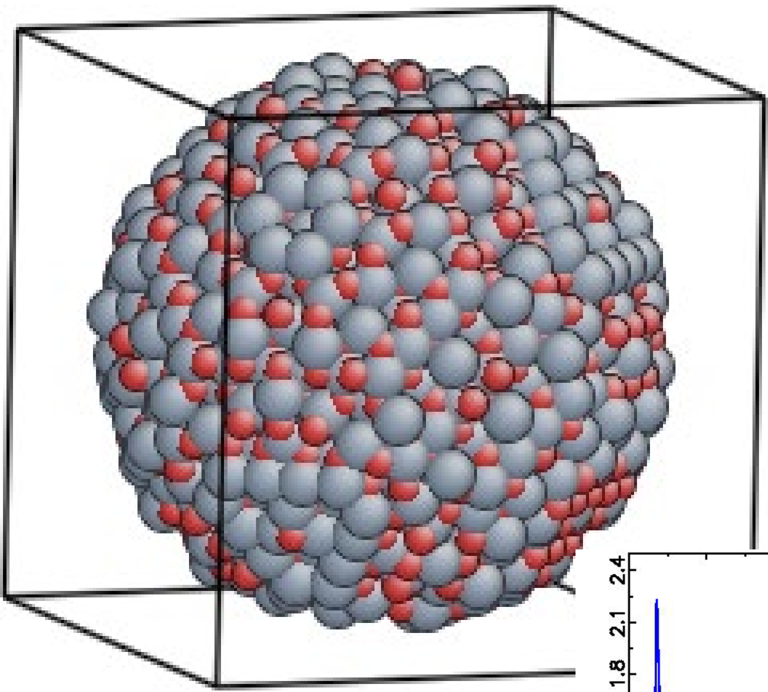
- Real-Material Structure model:
  - Crystal structure (if there is one)
  - Morphology (could be nano)
  - Surface reconstruction
  - Surface termination/dressing (ligands etc.)
  - Interfaces
  - Heterogeneities, phase separation
  - Point defects
  - Extended defects
  - Chemical short-range order
  - Distortive short-range order
  - …

Real material properties depend sensitively on crystalline imperfections

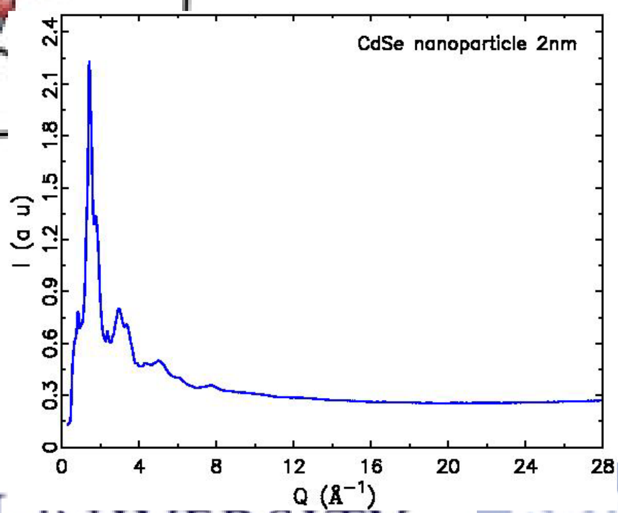
Real Materials



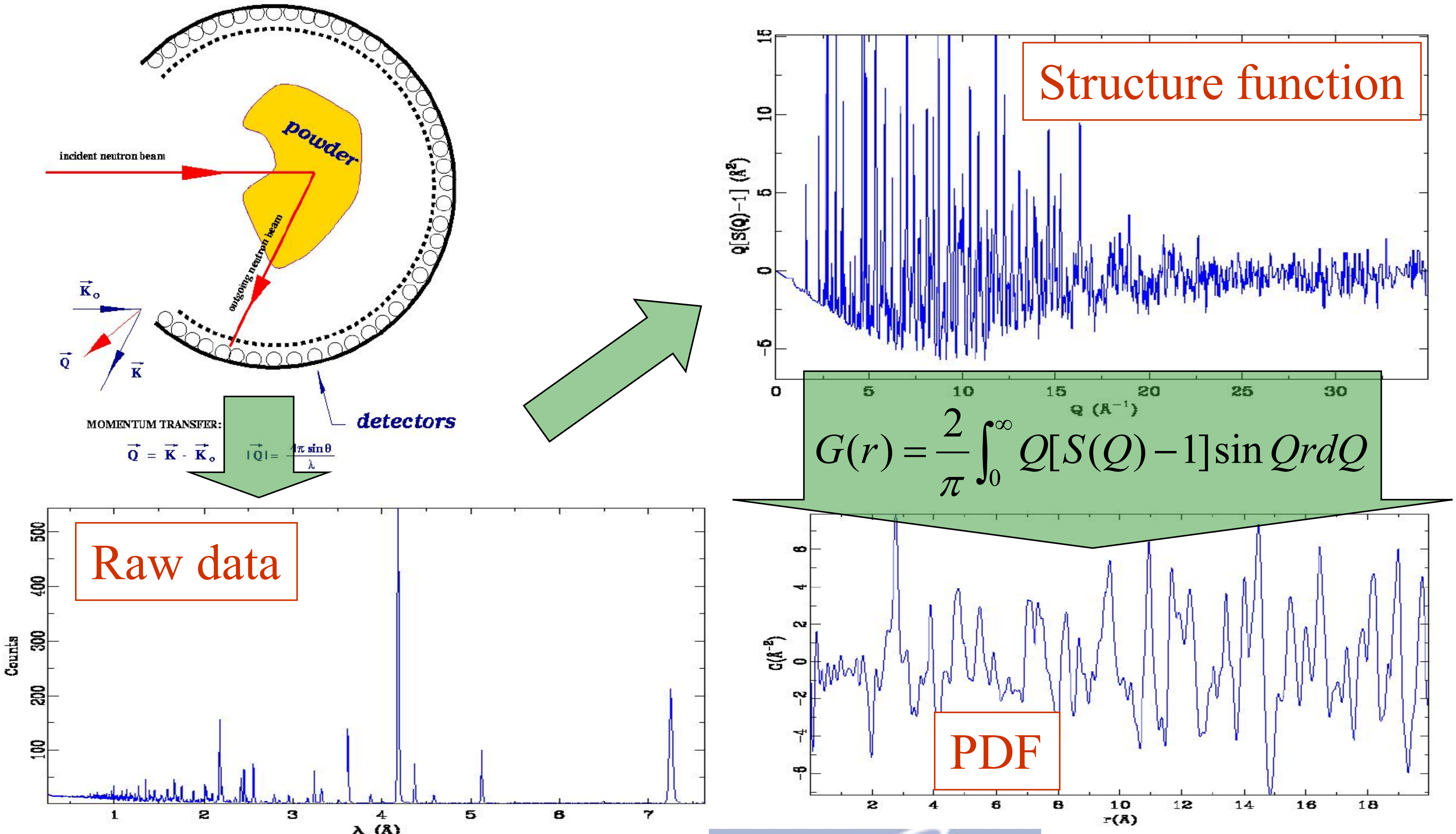
The Nanostructure Problem



- **Problem:**
  - Here is a nanoparticle, what is its structure?
- **Solution:**
  1. Give it to your grad student
  2. She puts it on the x-ray machine
  3. …Pushes the button



The atomic Pair Distribution Function



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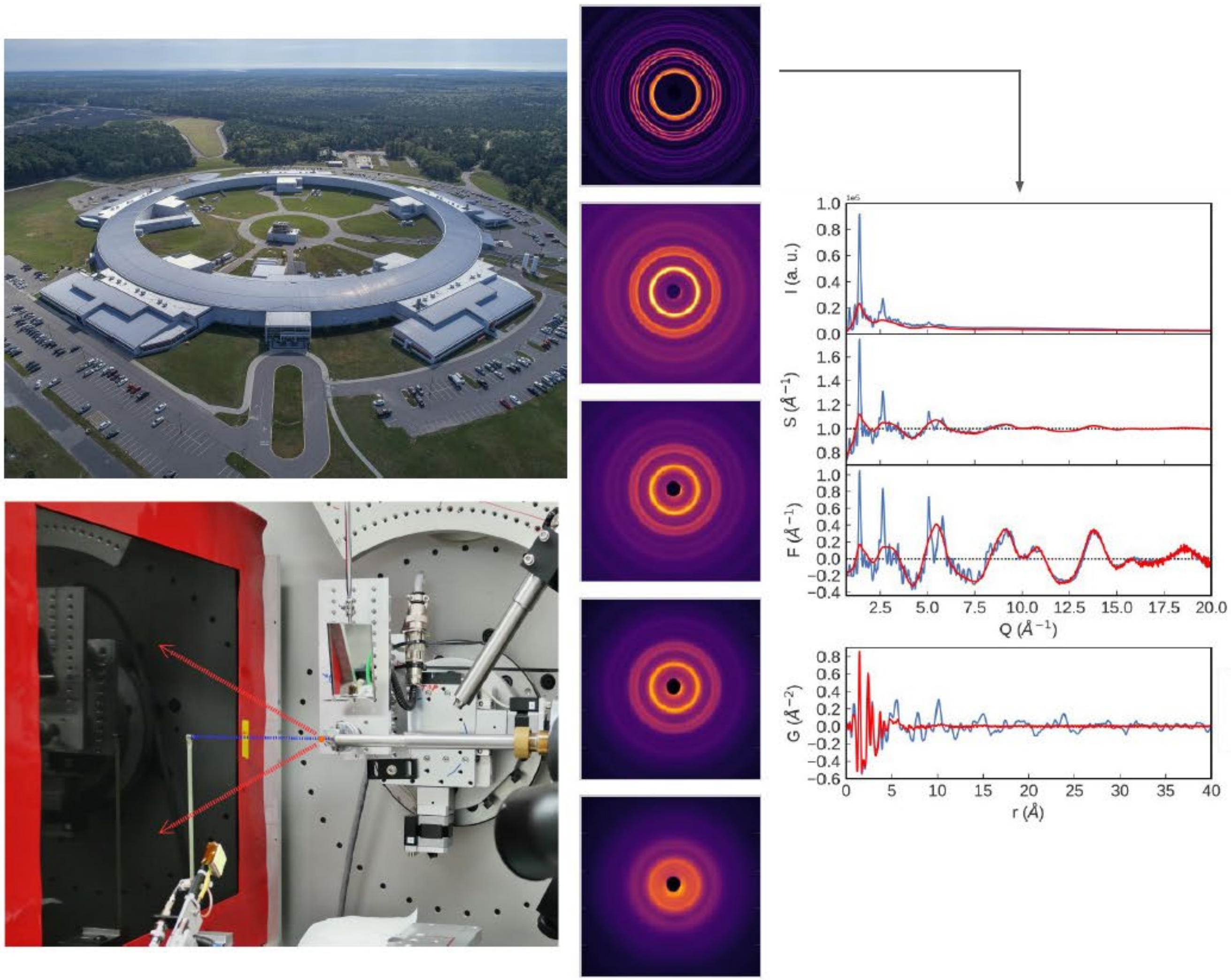

$$S(Q)-1=\frac{I(Q)}{N\langle f\rangle^2}-\frac{\langle f^2\rangle}{\langle f\rangle^2}$$

Normalize by scattering cross section

$$F(Q)=Q[S(Q)-1]$$

Transform to physical units

$$G(r)=\frac{2}{\pi}\int_{Q_{min}}^{Q_{max}} F(Q)\sin(Qr)\,dQ$$

Fourier transform

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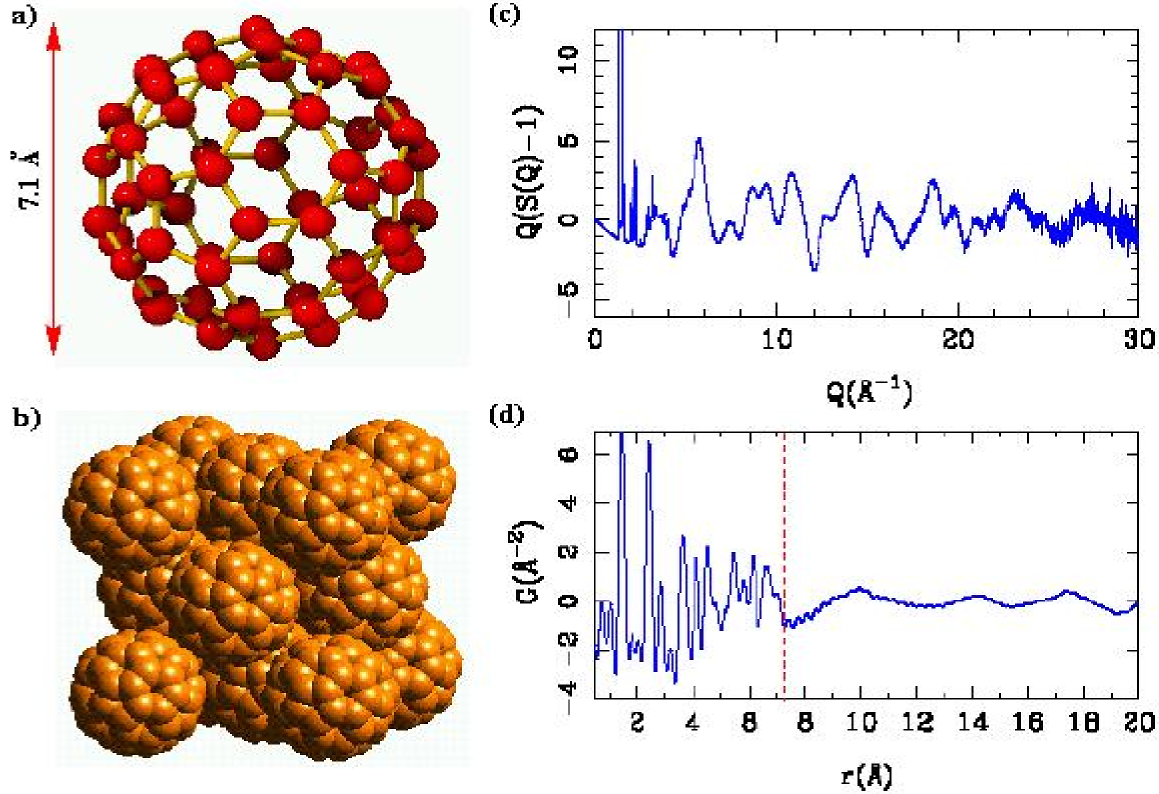
Sample is C60 Buckey balls

- Sit on an atom and look at your neighborhood
- *G(r)* gives the probability of finding a neighbor at a distance *r*
- PDF is experimentally accessible
- PDF gives the local structure



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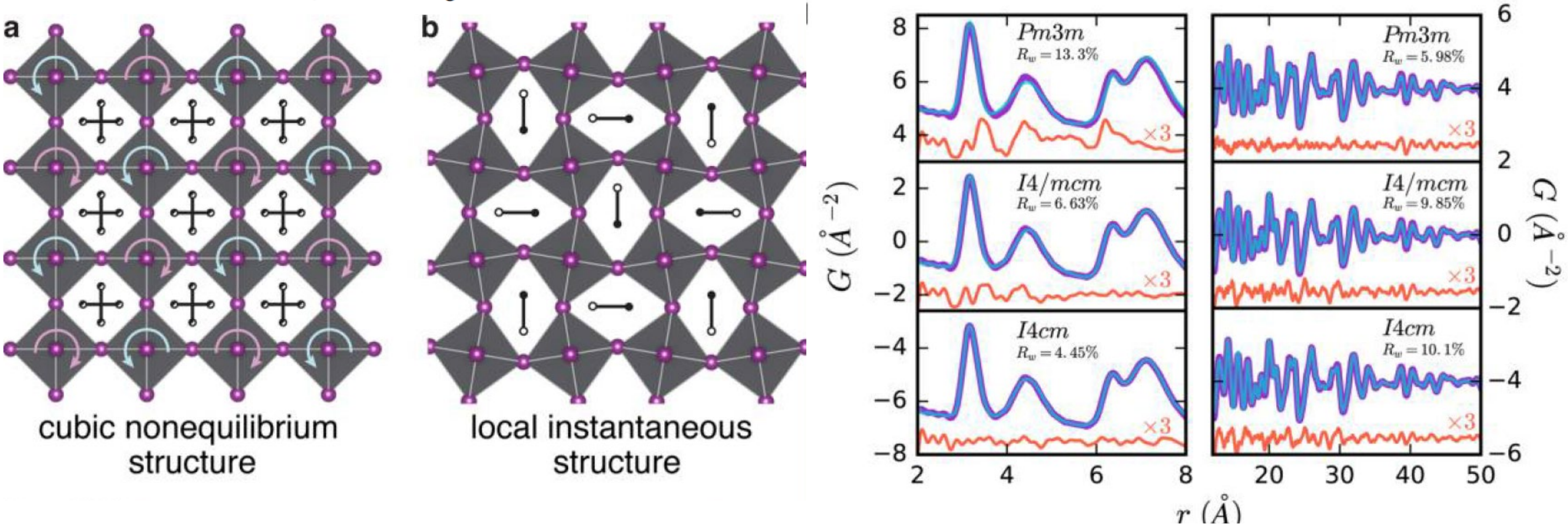
LETTER

Direct Observation of Dynamic Symmetry Breaking above Room Temperature in Methylammonium Lead Iodide Perovskite

Alexander N. Beecher,[†,⊥] Octavi E. Semonin,[†,⊥] Jonathan M. Skelton,[‡] Jarvist M. Frost,[‡] Maxwell W. Terban,[¶] Haowei Zhai,[¶] Ahmet Alatas,[§] Jonathan S. Owen,[†] Aron Walsh,[‡] and Simon J. L. Billinge[*,¶,‖]



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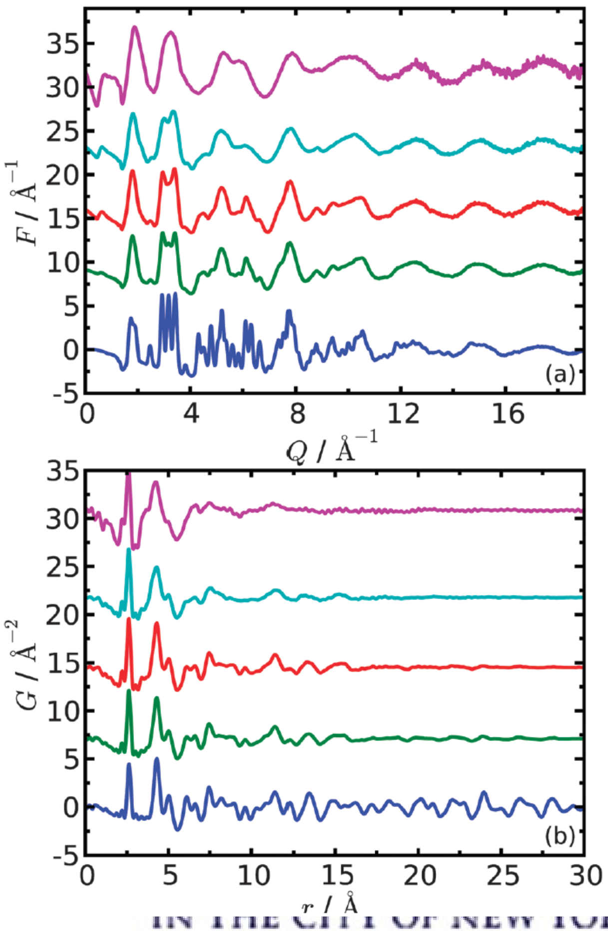
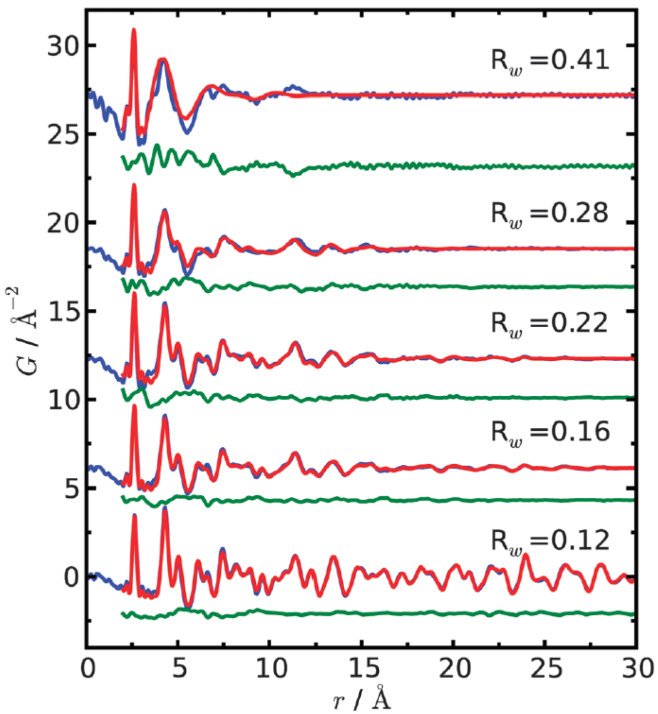
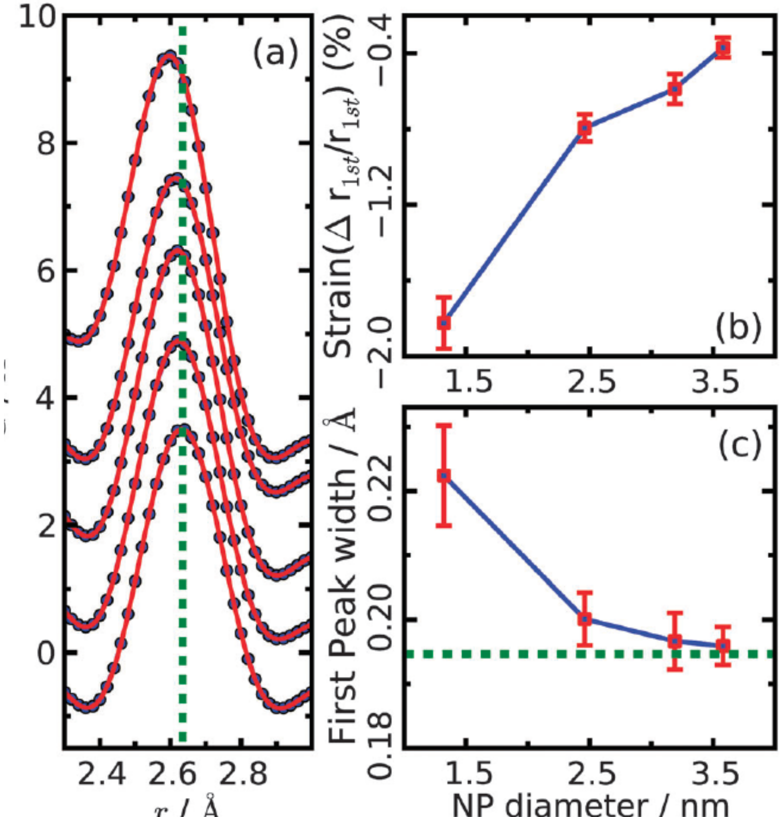
Confirmation of disordered structure of ultrasmall CdSe nanoparticles from X-ray atomic pair distribution function analysis

Cite this: *Phys. Chem. Chem. Phys.*, 2013, **15**, 8480

Xiaohao Yang,[a] Ahmad S. Masadeh,[b] James R. McBride,[c] Emil S. Božin,[d] Sandra J. Rosenthal[c] and Simon J. L. Billinge*[ad]







nature COMMUNICATIONS

ARTICLE

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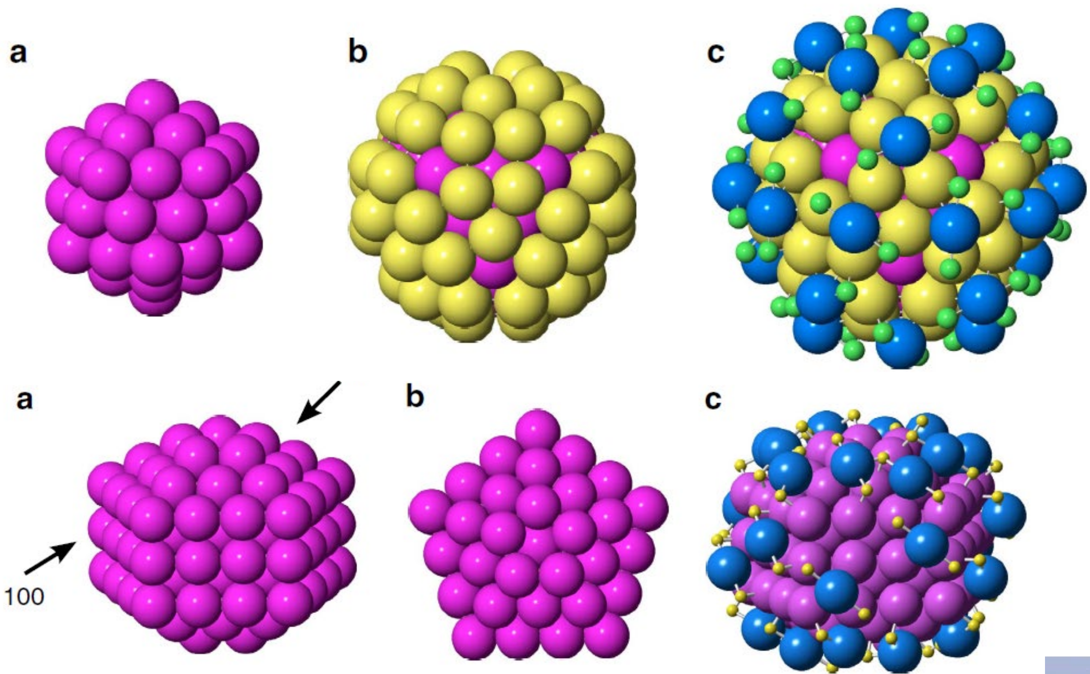
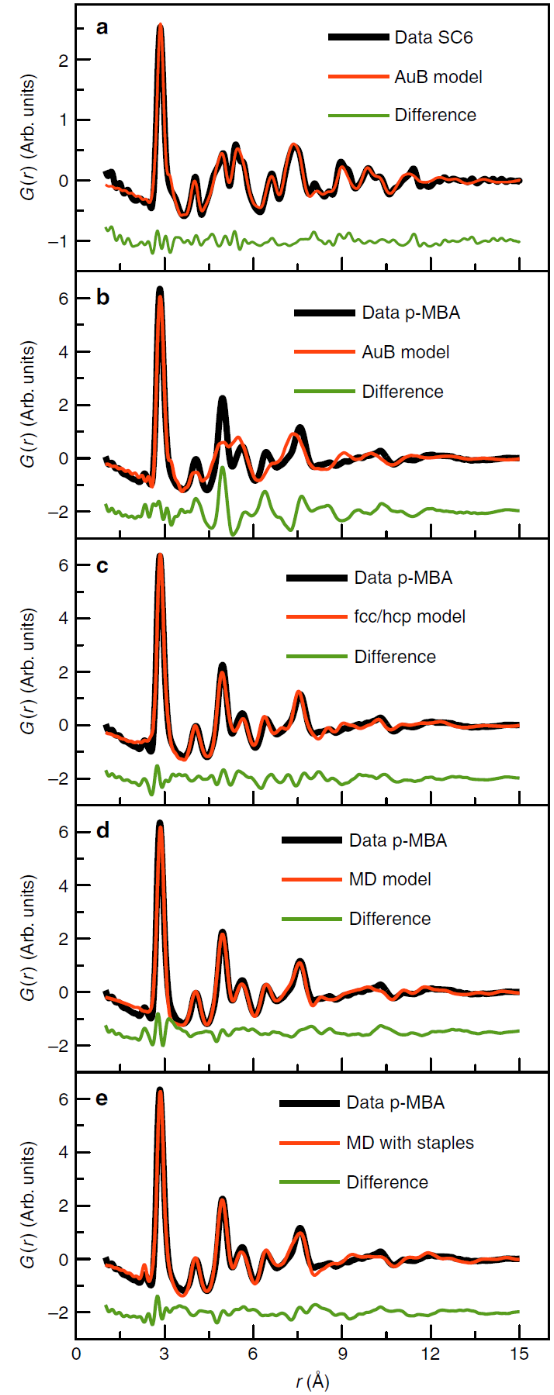
OPEN

Polymorphism in magic-sized $Au_{144}(SR)_{60}$ clusters

Kirsten M.Ø. Jensen[1,*], Pavol Juhas[2,*], Marcus A. Tofanelli[3], Christine L. Heinecke[3], Gavin Vaughan[4], Christopher J. Ackerson[3] & Simon J.L. Billinge[1,2]





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Inorganic Chemistry
Article
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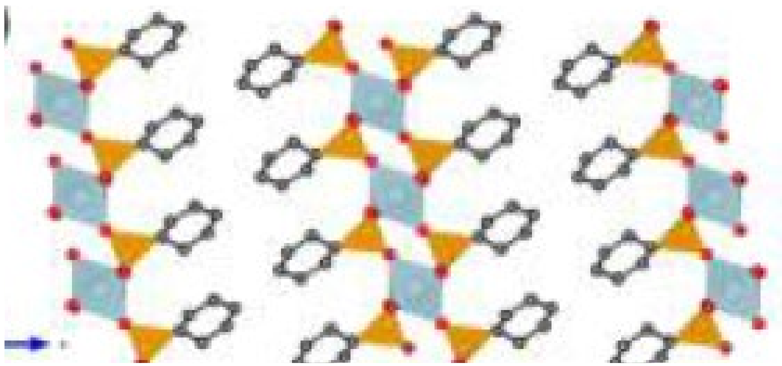
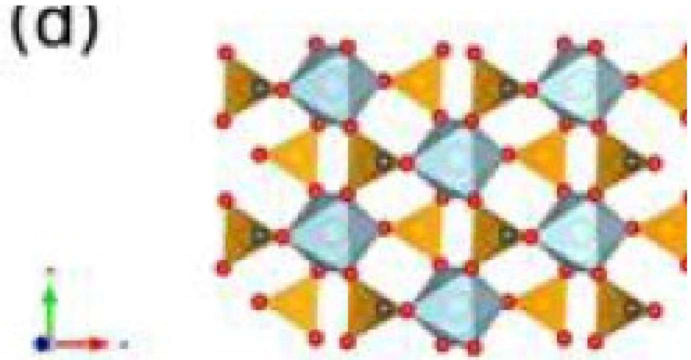
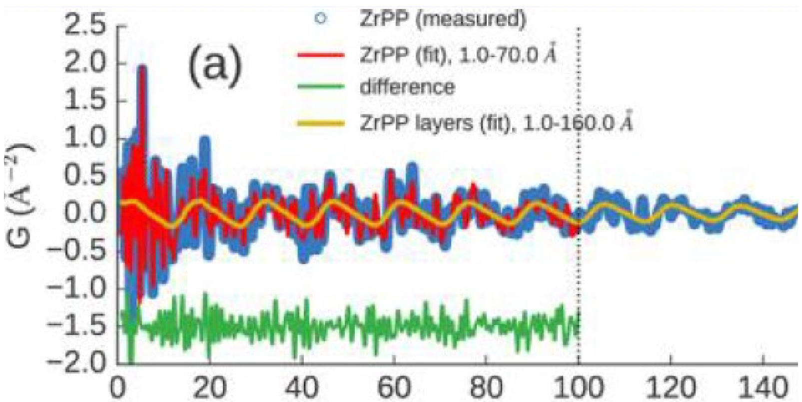
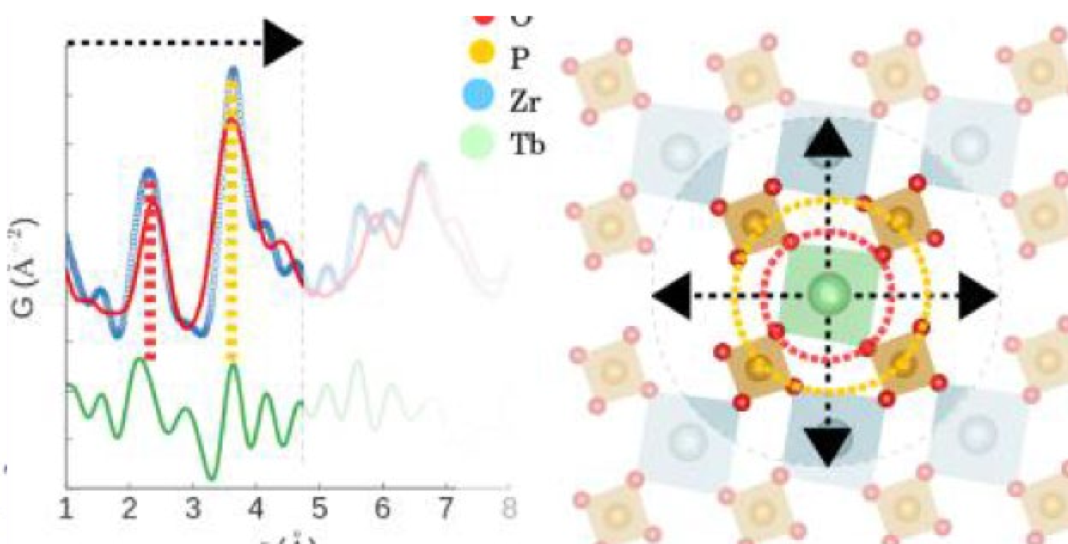
Local Environment of Terbium(III) Ions in Layered Nanocrystalline Zirconium(IV) Phosphonate–Phosphate Ion Exchange Materials

Maxwell W. Terban,[#,†] Chenyang Shi,[#,†] Rita Silbernagel,[‡] Abraham Clearfield,[‡] and Simon J. L. Billinge[*,†,⊥]









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CHEMICAL REVIEWS

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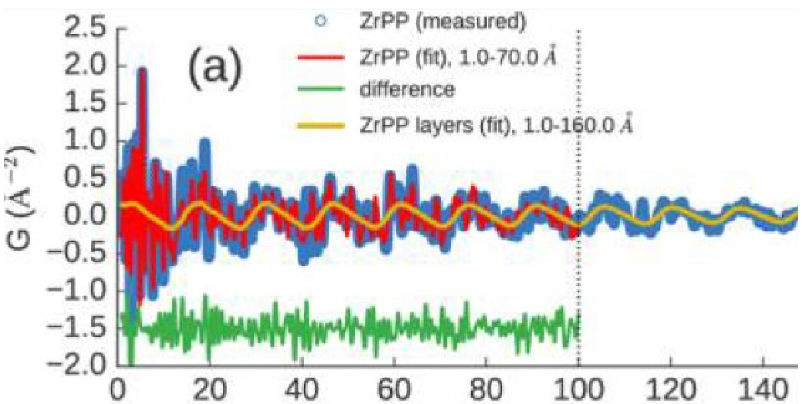
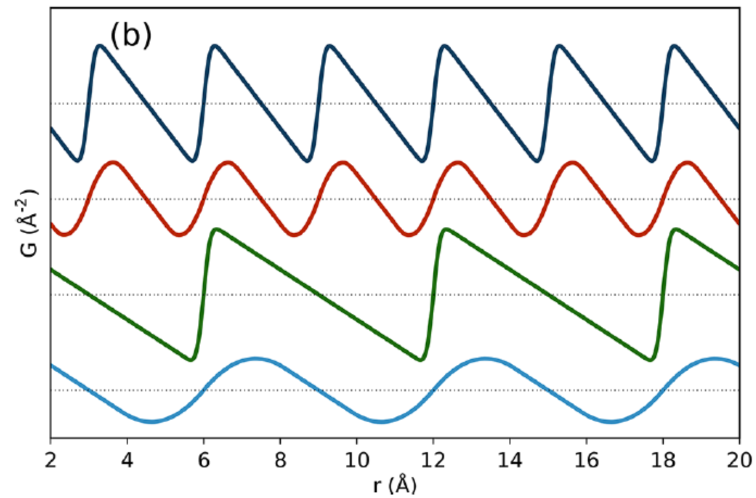
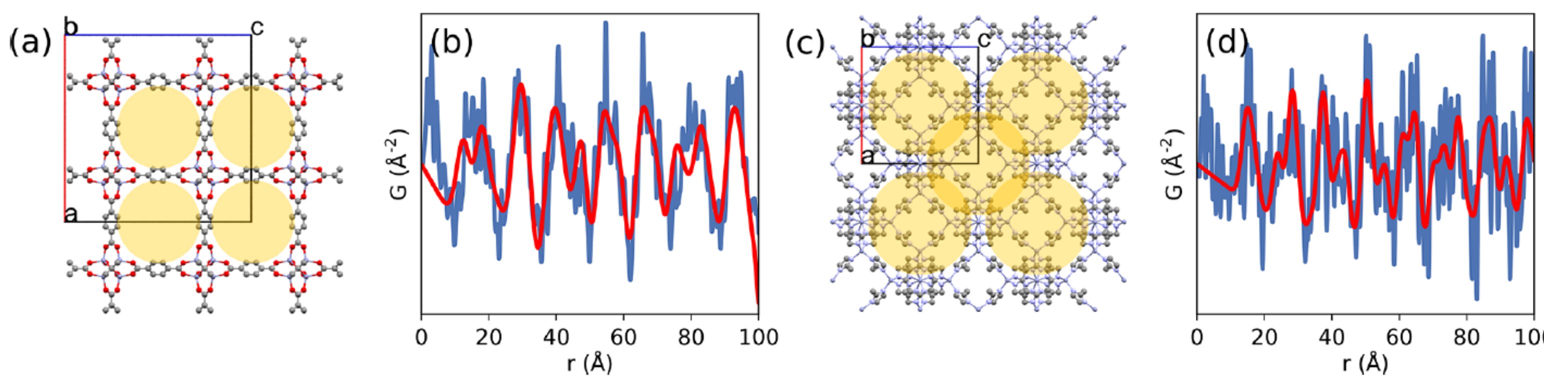
Review

Structural Analysis of Molecular Materials Using the Pair Distribution Function

Maxwell W. Terban* and Simon J. L. Billinge*







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Nanoscale

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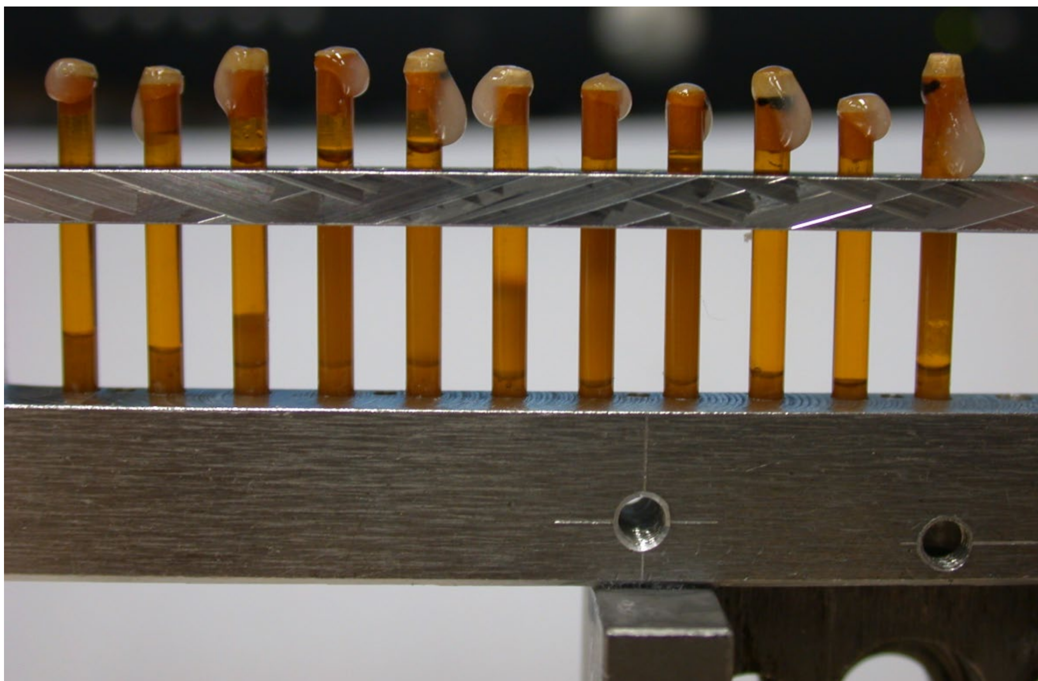
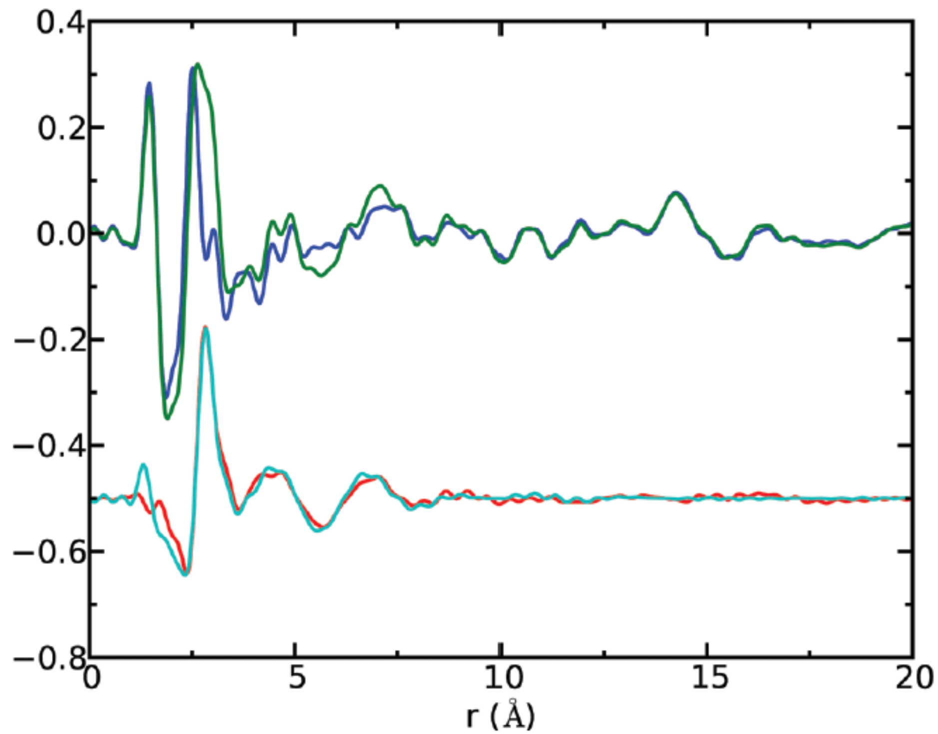
Cite this: *Nanoscale*, 2015, **7**, 5480

Detection and characterization of nanoparticles in suspension at low concentrations using the X-ray total scattering pair distribution function technique

Maxwell W. Terban,[a] Matthew Johnson,†[b] Marco Di Michiel[c] and Simon J. L. Billinge*[a,d]





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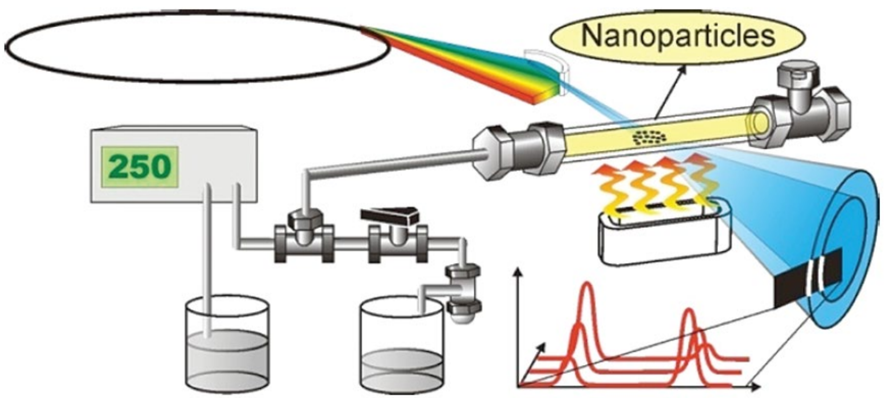
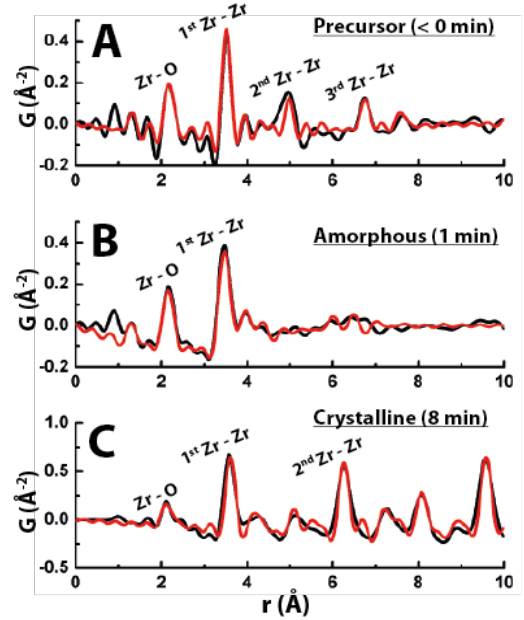
research papers

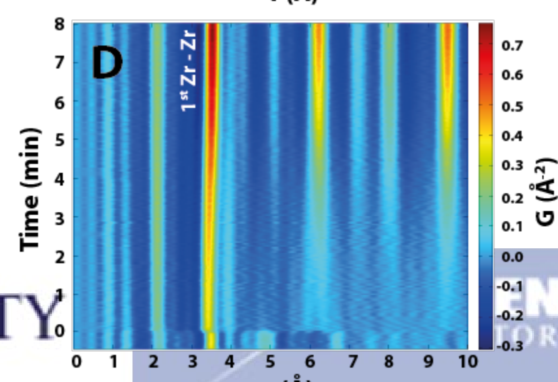
IUCrJ
ISSN 2052-2525
CHEMISTRY | CRYSTENG

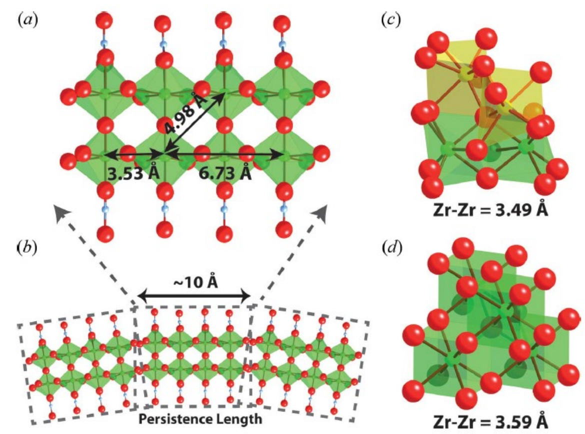
Evolution of atomic structure during nanoparticle formation

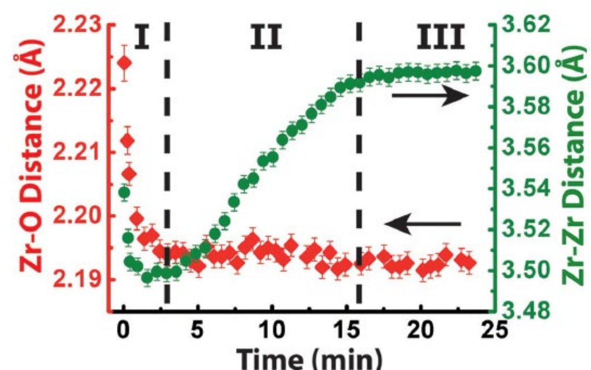
Christoffer Tyrsted,[a] Nina Lock,[a,b] Kirsten M. Ø. Jensen,[a,c] Mogens Christensen,[a] Espen D. Bøjesen,[a] Hermann Emerich,[d] Gavin Vaughan,[e] Simon J. L. Billinge[c,f]* and Bo B. Iversen[a]*











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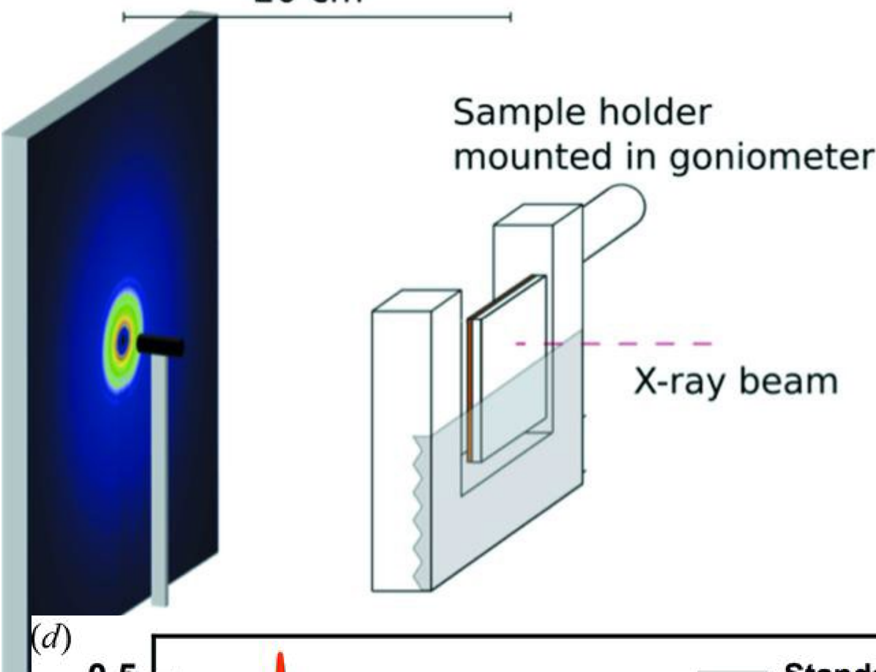
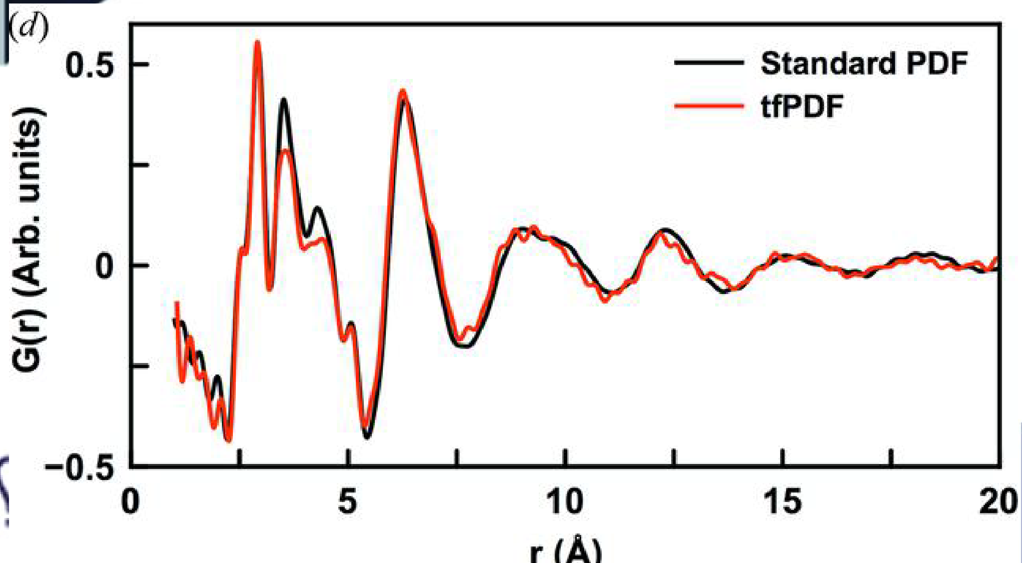
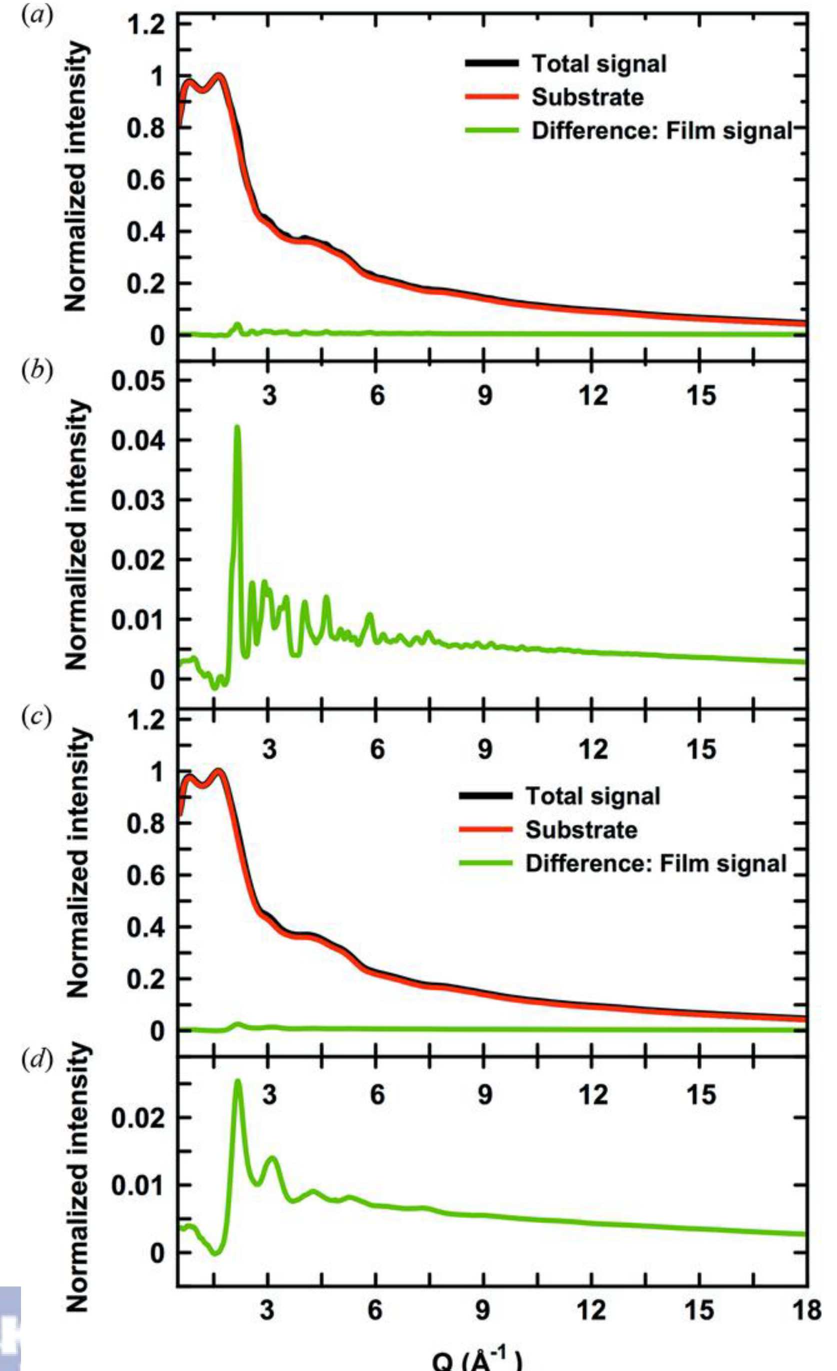
Thin film PDF at normal incidence



Jensen, Iversen, Johnson, Dooryhee SJLB et al. *IUCrJ* (2015)





nature COMMUNICATIONS

ARTICLE

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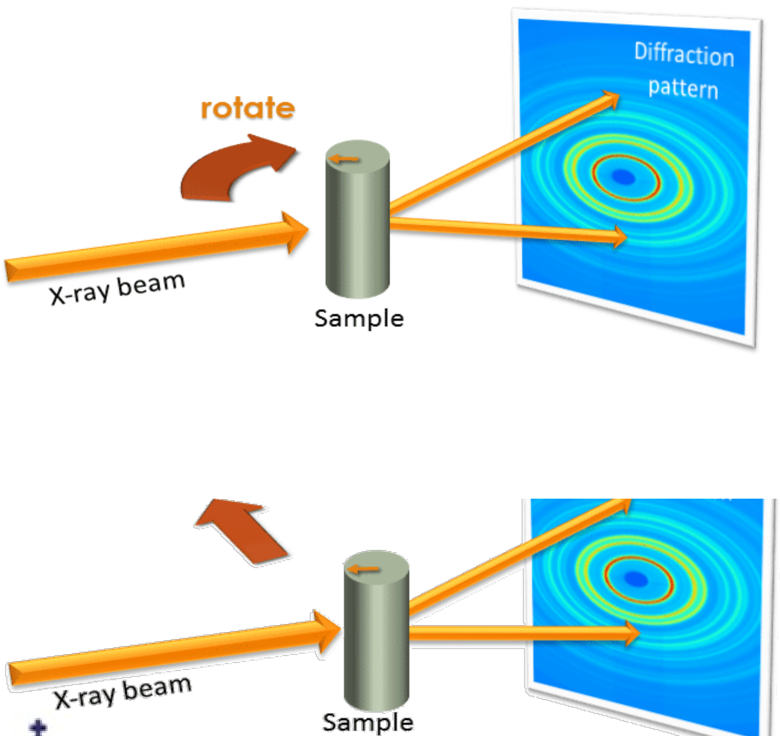
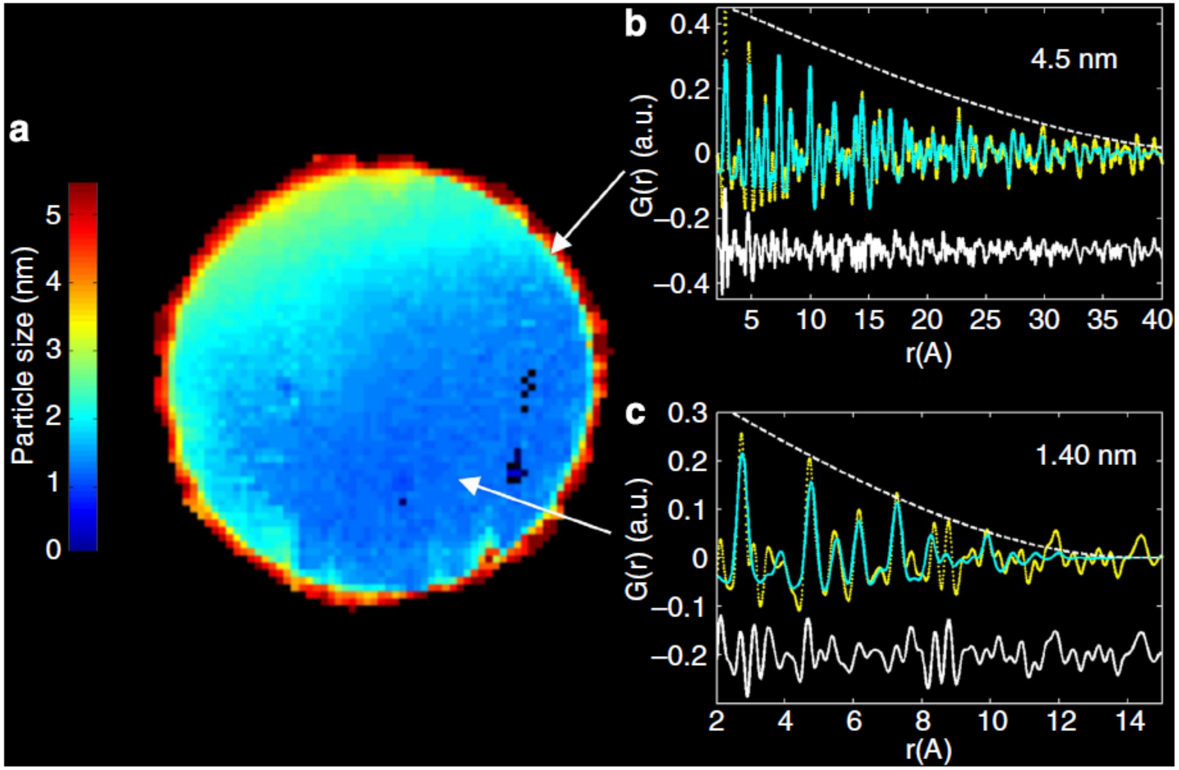
DOI: 10.1038/ncomms3536

Pair distribution function computed tomography

Simon D.M. Jacques[1,2], Marco Di Michiel[3], Simon A.J. Kimber[3], Xiaohao Yang[4], Robert J. Cernik[1], Andrew M. Beale[2,5,6] & Simon J.L. Billinge[4,7]





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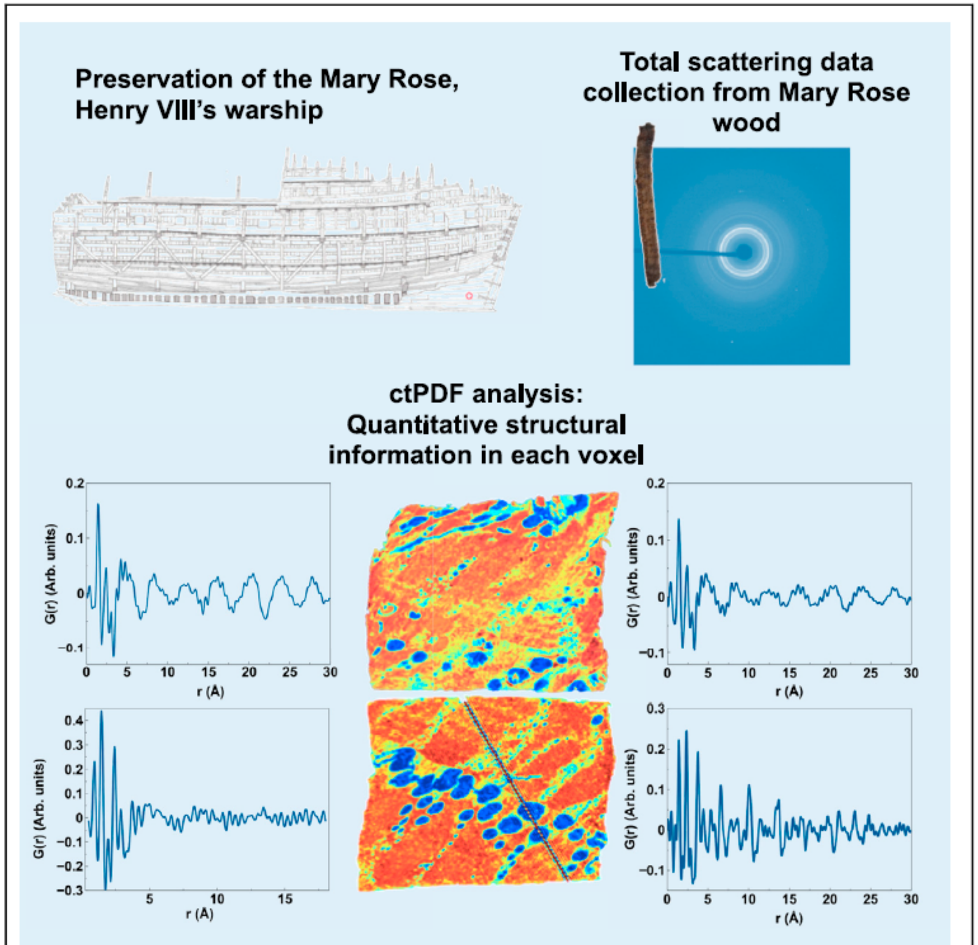
Matter

CellPress

Article

Location and characterization of heterogeneous phases within *Mary Rose* wood



Kirsten M.Ø. Jensen, Esther Rani Aluri, Enrique Sanchez Perez, ..., Eleanor J. Schofield, Simon J.L. Billinge, Serena A. Cussen

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kirsten@chem.ku.dk (K.M.Ø.J.)
e.schofield@maryrose.org (E.J.S.)
sb2896@columbia.edu (S.J.L.B.)

Highlights

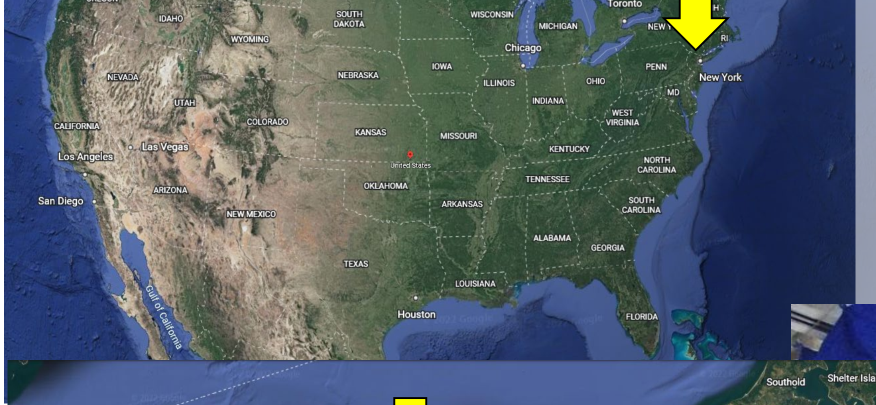
Wood from the *Mary Rose* is characterized with computed tomography total scattering

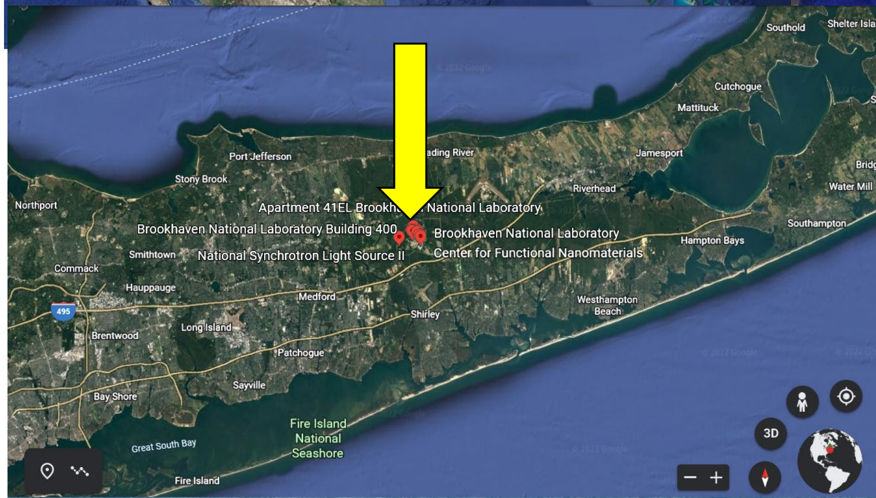
Polyethylene glycol from previous conservation treatments is identified and mapped

Five-nanometer zinc sulfide nanoparticles are identified in the waterlogged wood

Total scattering analysis shows position-dependent structure of the nanoparticles

XPD (28ID-2) @ NSLS-II





Google “XPD NSLS II”

TY

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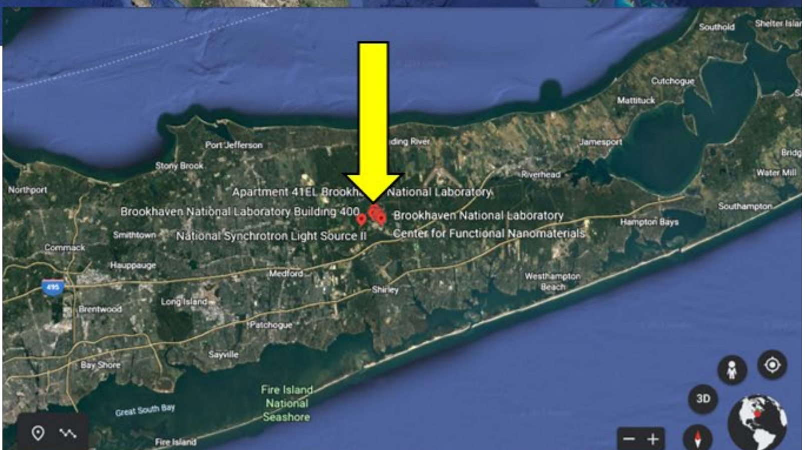
PDF (28ID-1) @ NSLS-II



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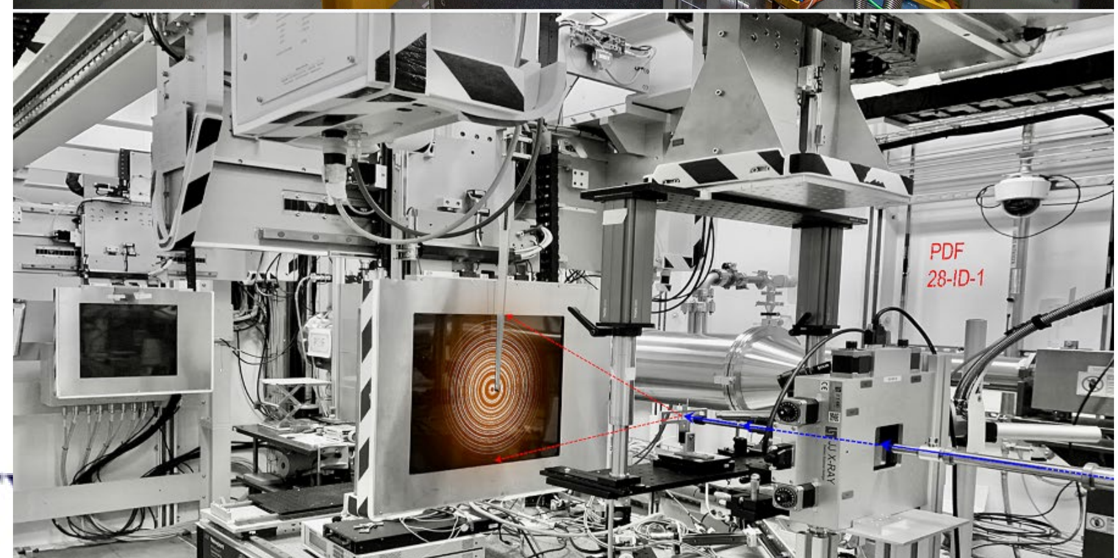
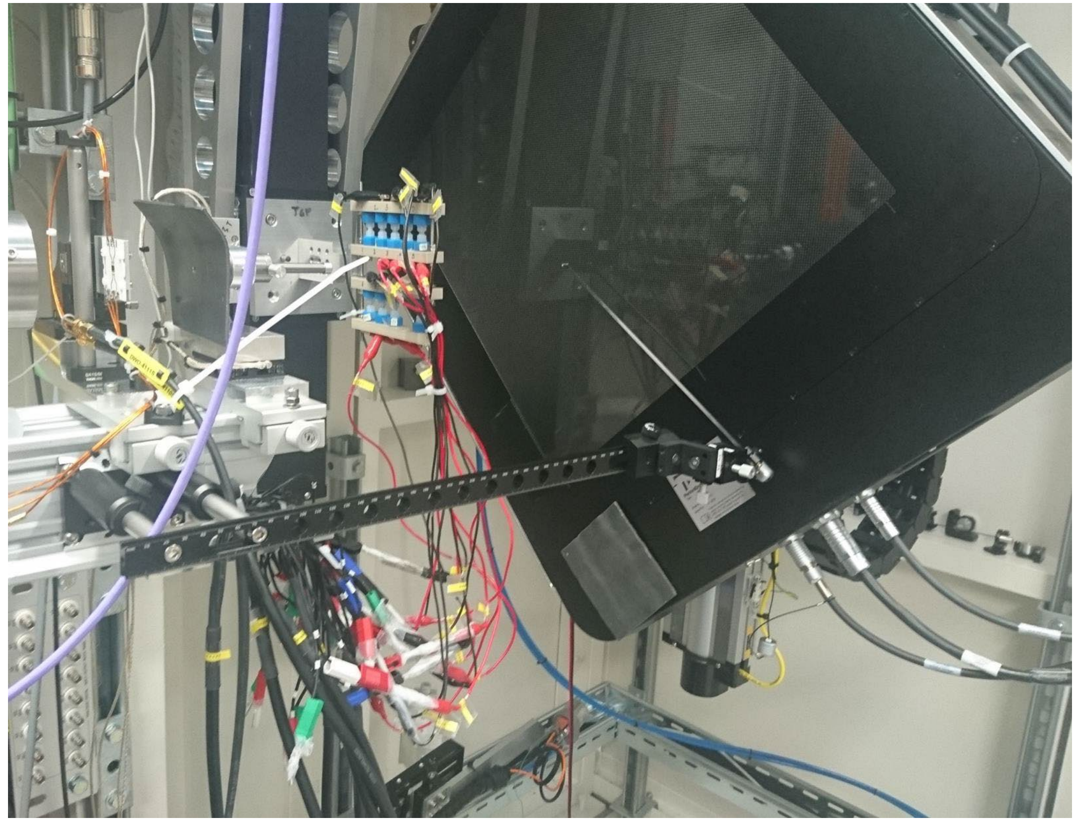
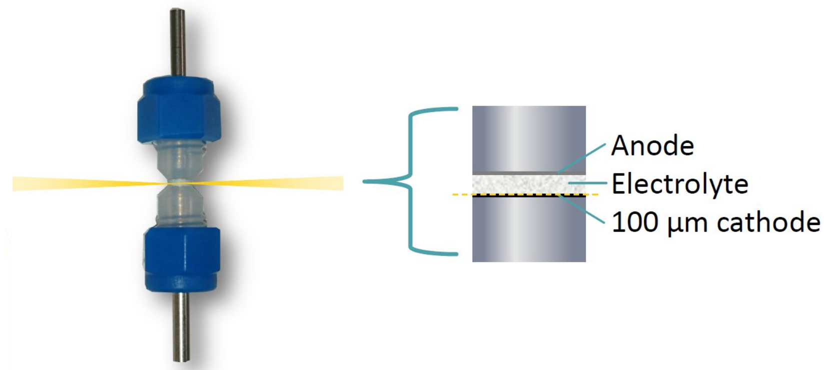


XPDF (I15-1) @ Diamond LS (UK)

Google "i15-1 Diamond"





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Mail in programs

- Opportunity to get Synchrotron powder diffraction data without traveling to the facility
- Search for "mail in programs"
  - Beamline 11BM at APS (search "11BM APS")
  - Beamline PDF at NSLS-II (search "PDF NSLS II")(future capability)
  - Easy Access at I15-I at Diamond (search "i15-1 easy access")
  - Email me (Simon Billinge, sb2896@Columbia.edu)
- Workflow
  - Submit a short proposal
  - Get approval
  - Receive info about how to prepare your samples
  - Make samples and send to facility by DHL/Fedex
  - Receive data from the facility
  - Carry out the analysis yourself (get help from collaborators if needed)

Help getting started with PDF

- For help getting started with PDF
- For advice and help with LAAAMP FaST proposal development
- Please reach out to me by email

Prof. Simon Billinge

Email: sb2896@columbia.edu