The University of Manchester
Photon Science Institute

MANCHESTER 1824

Functional materials development using accelerator-based light sources:

current capabilities and future prospects

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The University of Manchester, UK

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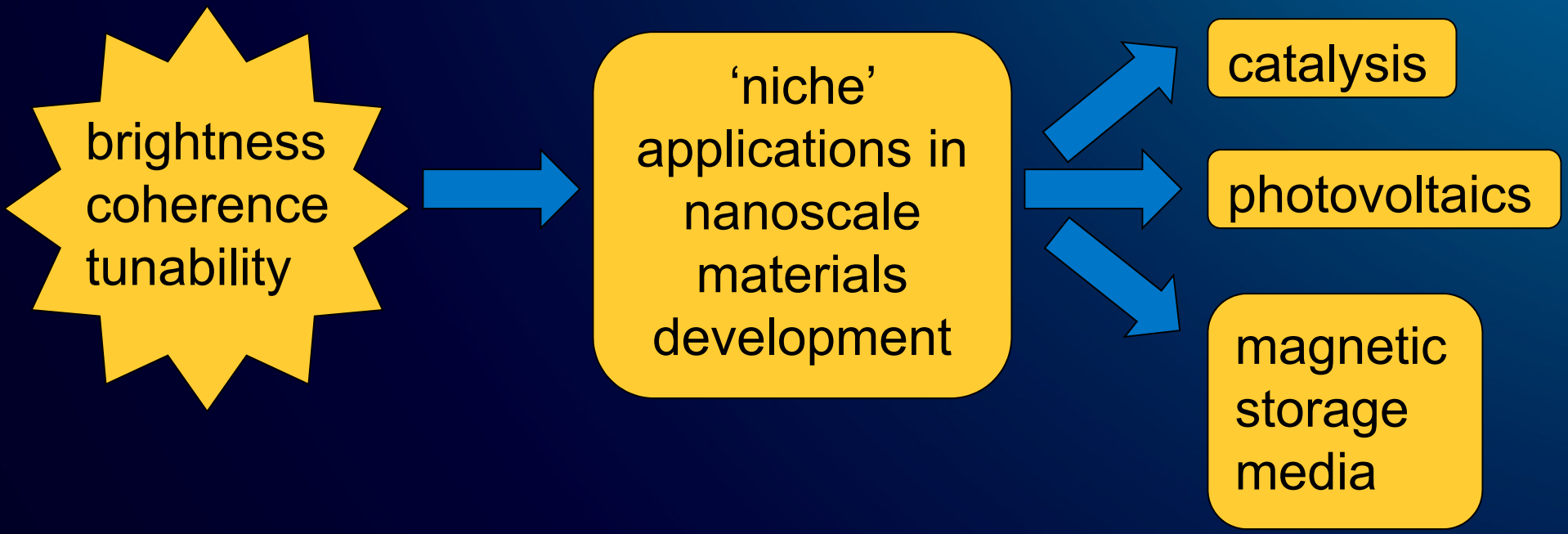
Accelerators for America's Future

… 'opportunities and challenges for particle beams in energy and environment, medicine, industry, national security and discovery science..'

- recognises the importance of electron synchrotrons and FELs for X-ray and photon science…
- but unsuitable for industrial materials processing…..

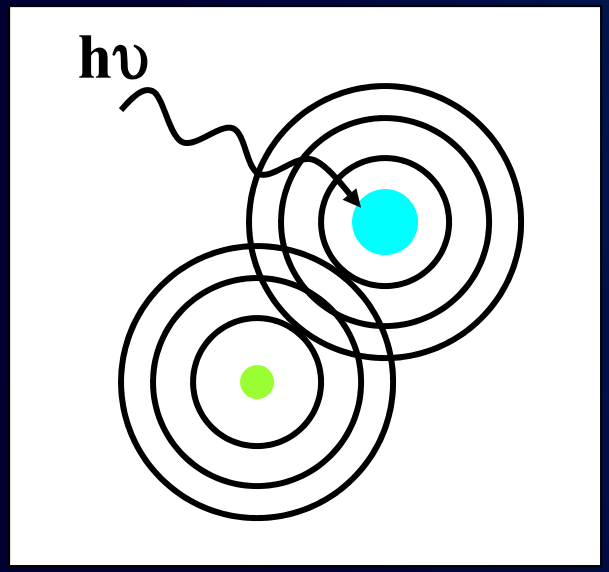
So I could end the talk here…….

- There are very few applications of synchrotron radiation for industrial scale materials production or processing
- but 3rd and 4th generation SR offers unique capability from

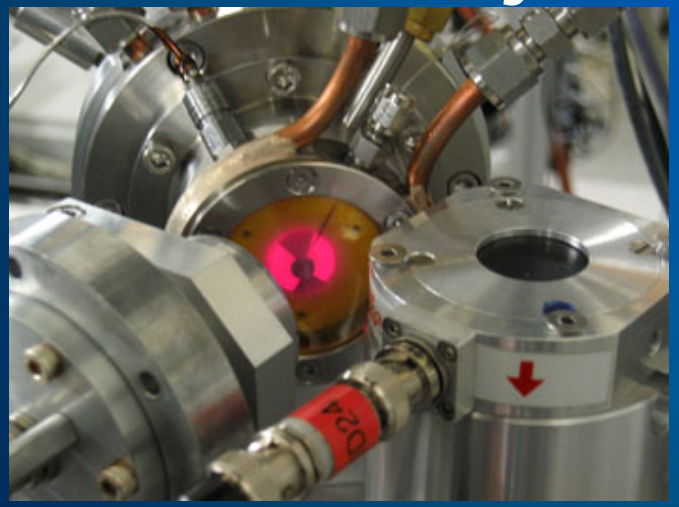


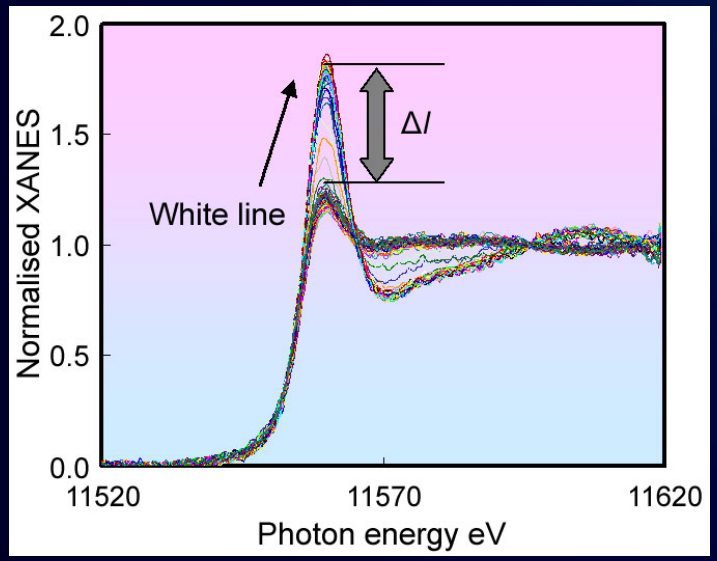
- So I will aim to illustrate some examples of real impact…..

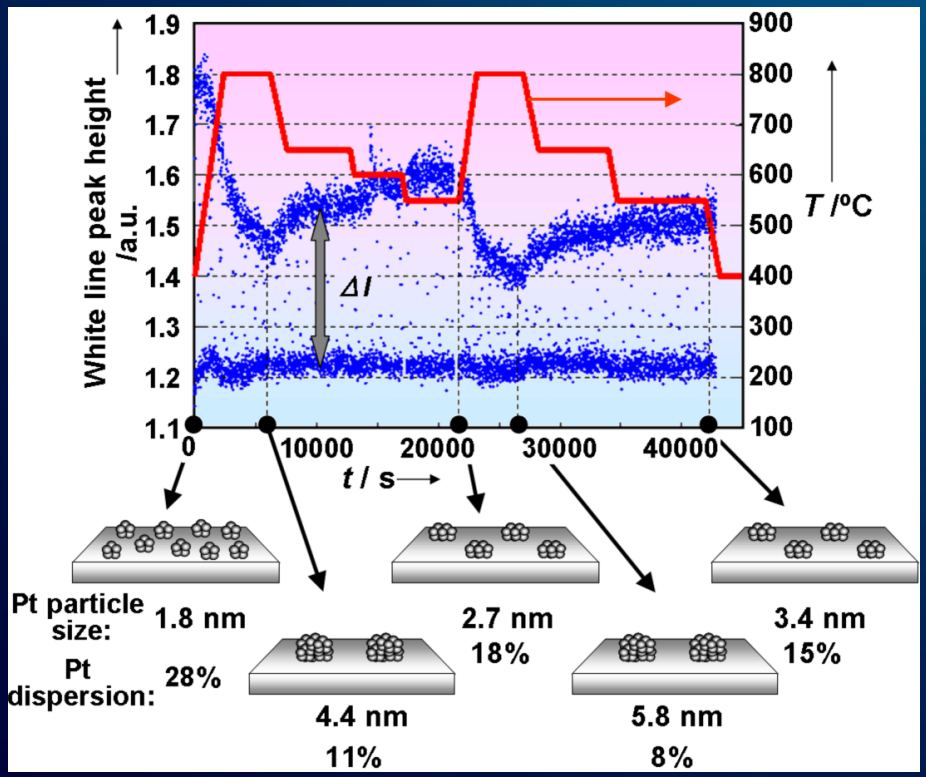
Improving the lifetime of car exhaust catalysts



- EXAFS 'white line' intensity allows relationship between particle size and catalyst temperature to be monitored *in situ*.
- Can be incorporated into 'on board' control.





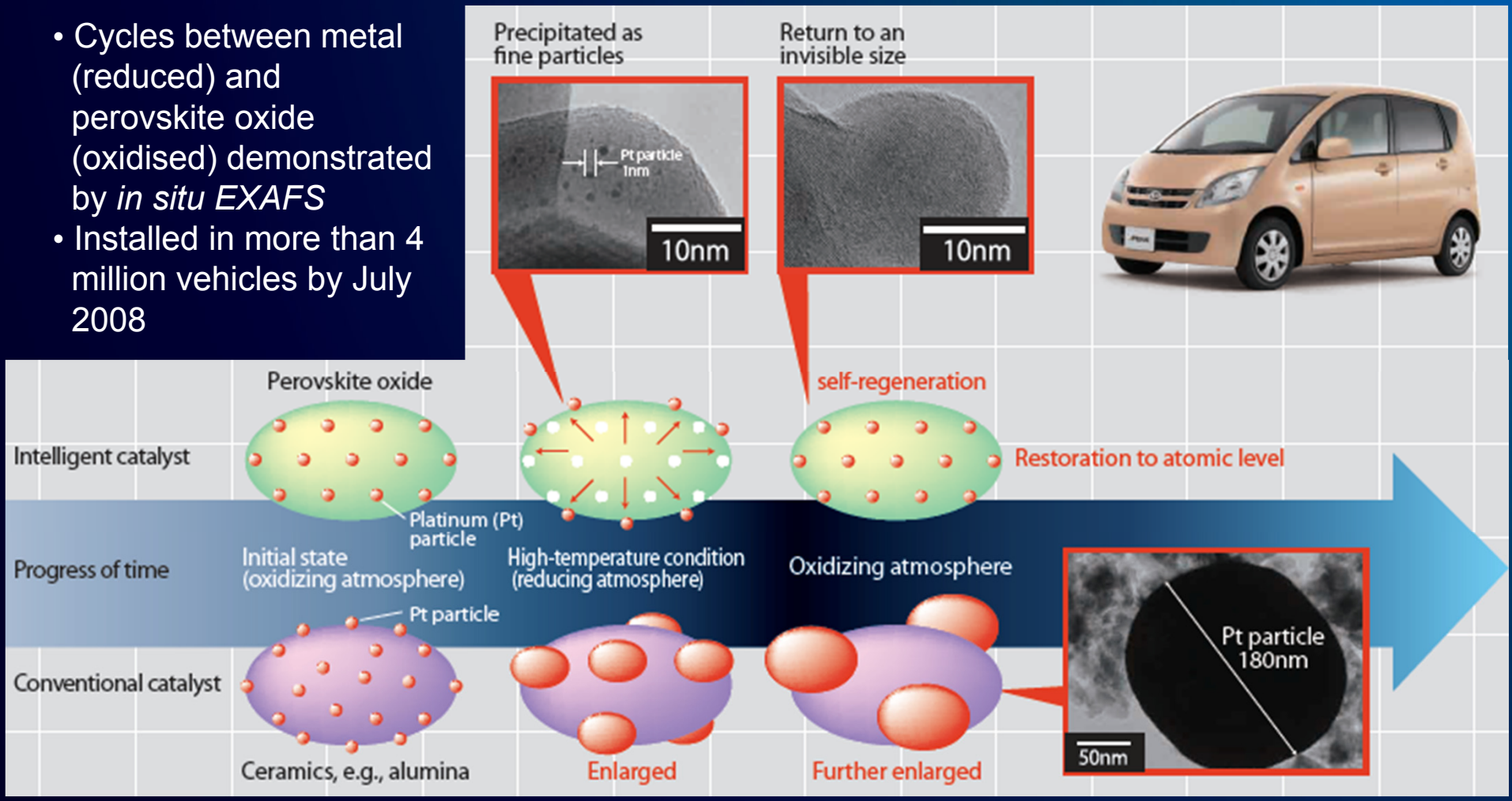


ESRF TOYOTA

Yasutaka Nagai et al., Angewandte Chemie 47 9303 (2008)

Intelligent catalysts for car exhaust control

- Cycles between metal (reduced) and perovskite oxide (oxidised) demonstrated by *in situ EXAFS*
- Installed in more than 4 million vehicles by July 2008



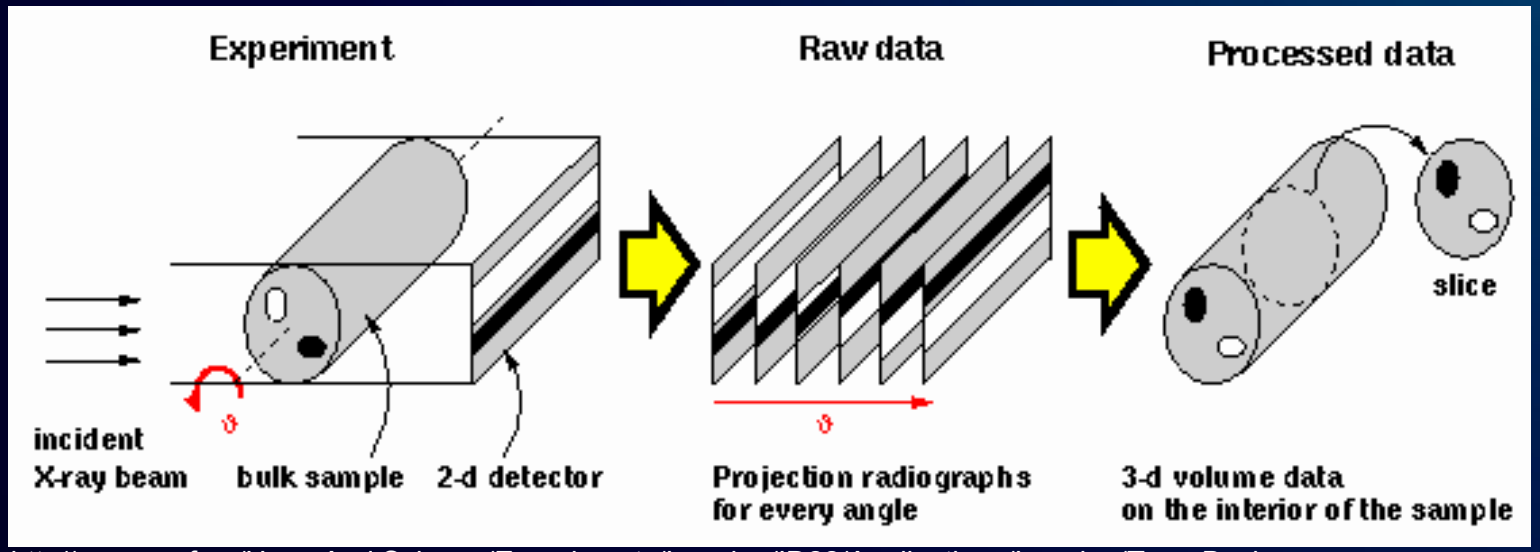
Hirohisa Tanaka et al., Angewandte Chemie 45 5998 (2006), Mari Uenishi et al., Catalysis Communications 9 311 (2008), Successful Results of Industrial Applications at SPring-8, p.4; http://www.spring8.or.jp/pdf/en/indu_appli/p04-05.pdf

JAEA DAIHATSU SPring-8

X-ray microtomography

- Uses SR brilliance, small source size
- Penetration depths 10 μm - 10 cm (soft - hard X-rays)
- Resolution 100 μm -100 nm
- Non-destructive
- Yields chemical information when combined with X-ray fluorescence

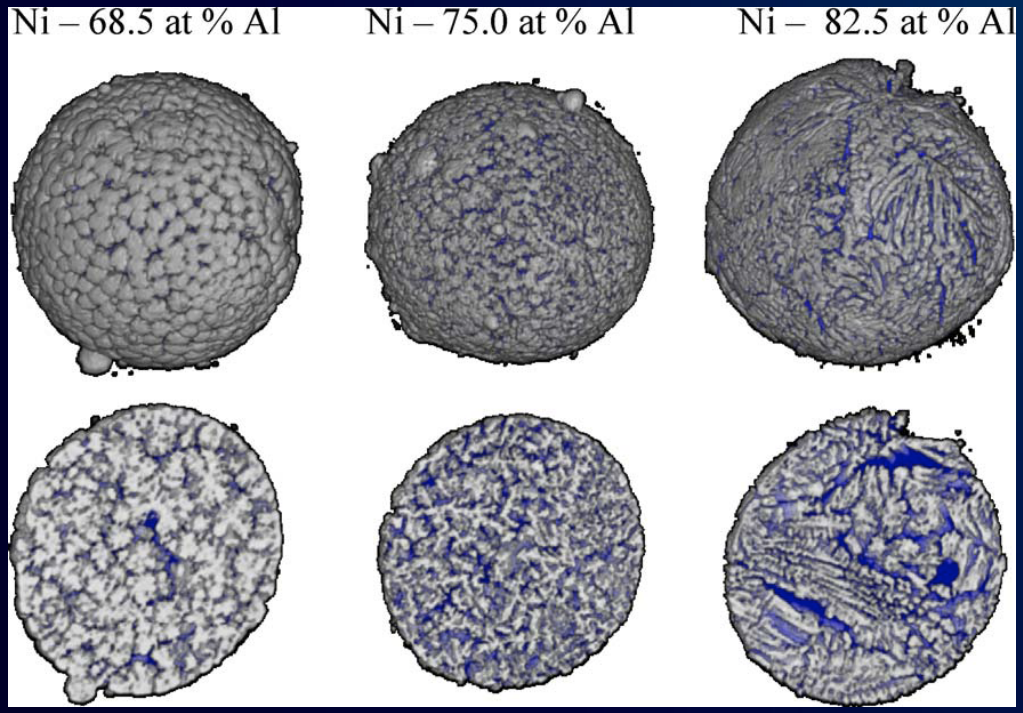
100 million-year old pollinators! (May 2012)



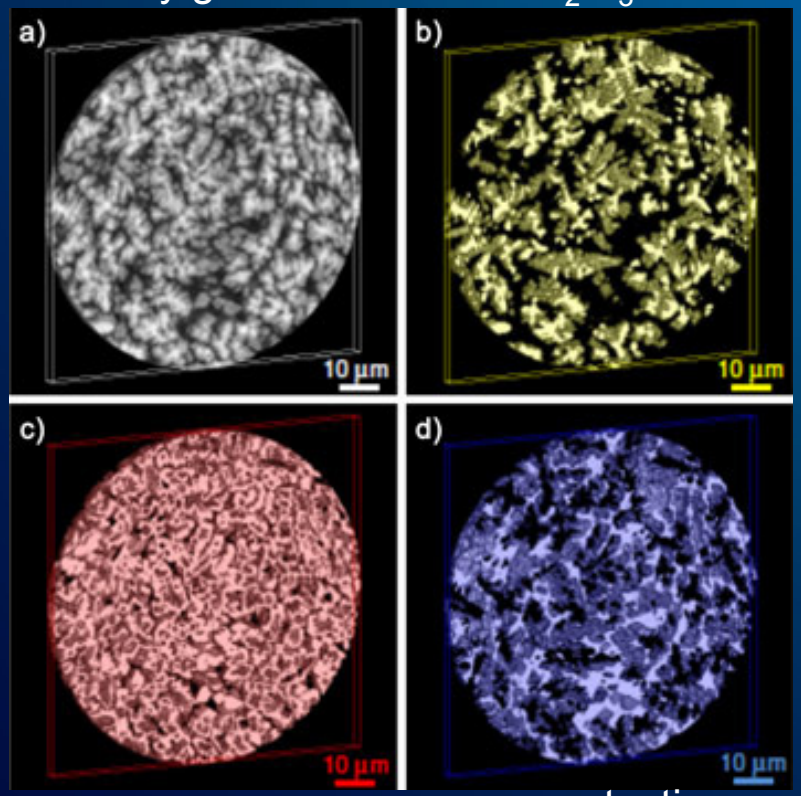
http//www.esrf.eu/UsersAnd Science/Experiments/Imaging/ID22/Applications/Imaging/TomoBasics
E. Peñalver et al., PNAS Early Edition, 14 May 2012.

Redesigning hydrogenation catalysts

- X-ray tomography used to establish porosity and phase composition as function of preparation conditions
- Activity doubled compared with conventional Raney Ni catalyst



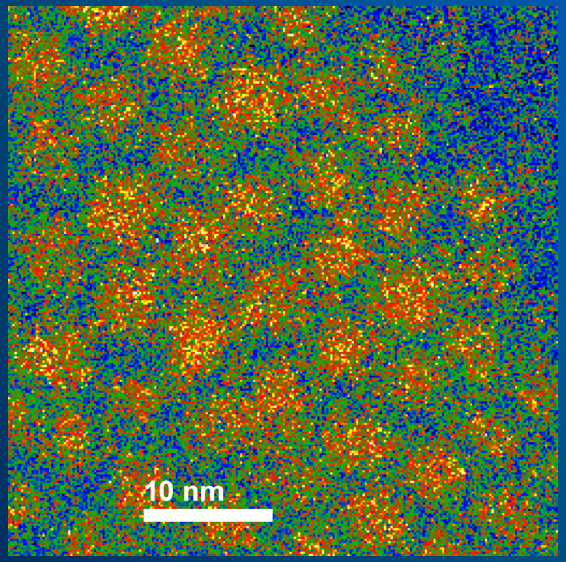
After leaching, dendritic Ni_2Al_3 network remains

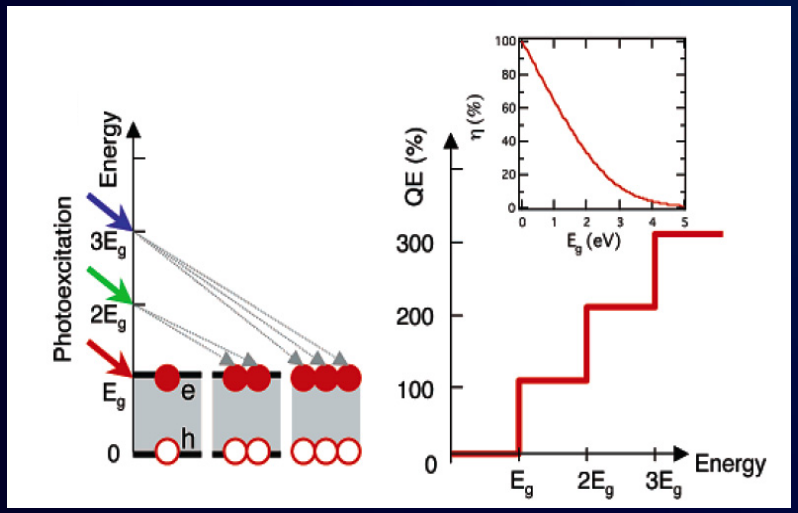


F Devred, *et al.*, Catalysis Today 163 (2011) 13;
http://www.esrf.eu/UsersAndScience/Experiments/Imaging/ID19

Characterising Colloidal Quantum Dots

- Uses in displays and next-generation solar cells
- Potential for significant efficiency improvement from carrier multiplication
- Issues: how to characterise complex ‘core-shell’ structure, surface degradation





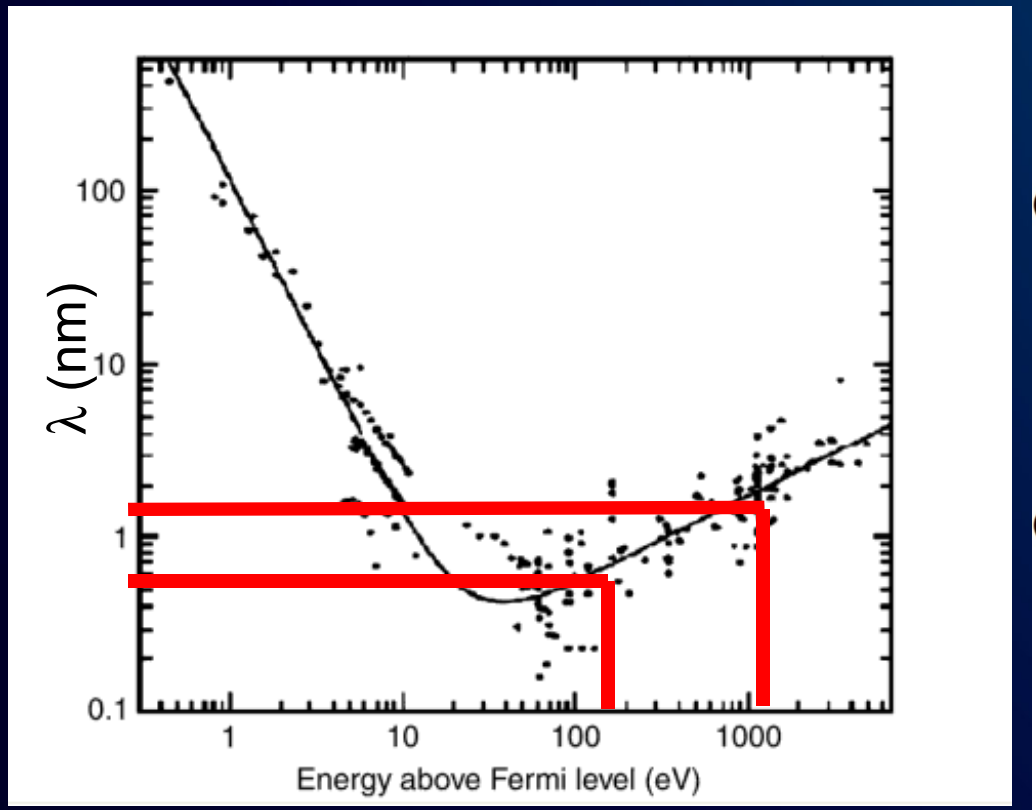
Schaller *et al.*, Nano Lett. **6**, 424, (2006)

Photo courtesy of Nanoco Technologies Ltd.

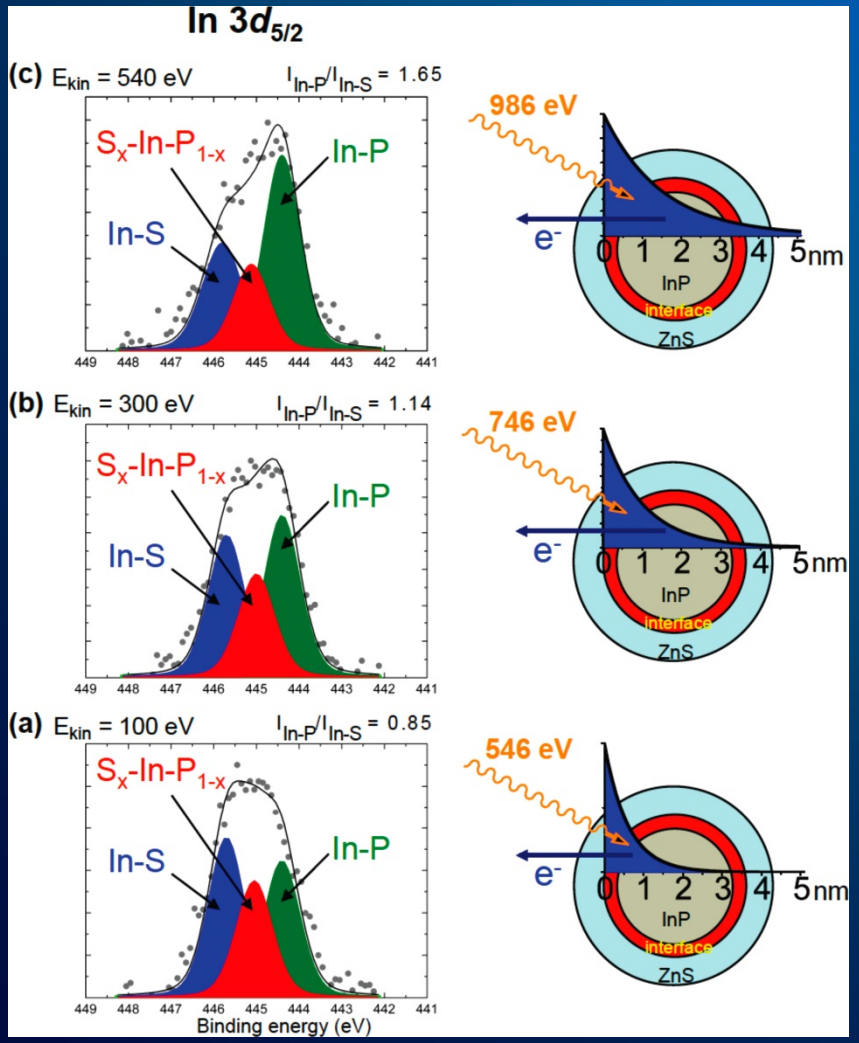
NANOCO TECHNOLOGIES

Non-destructive depth-profiling by SR

Inelastic Mean Free Path Curve

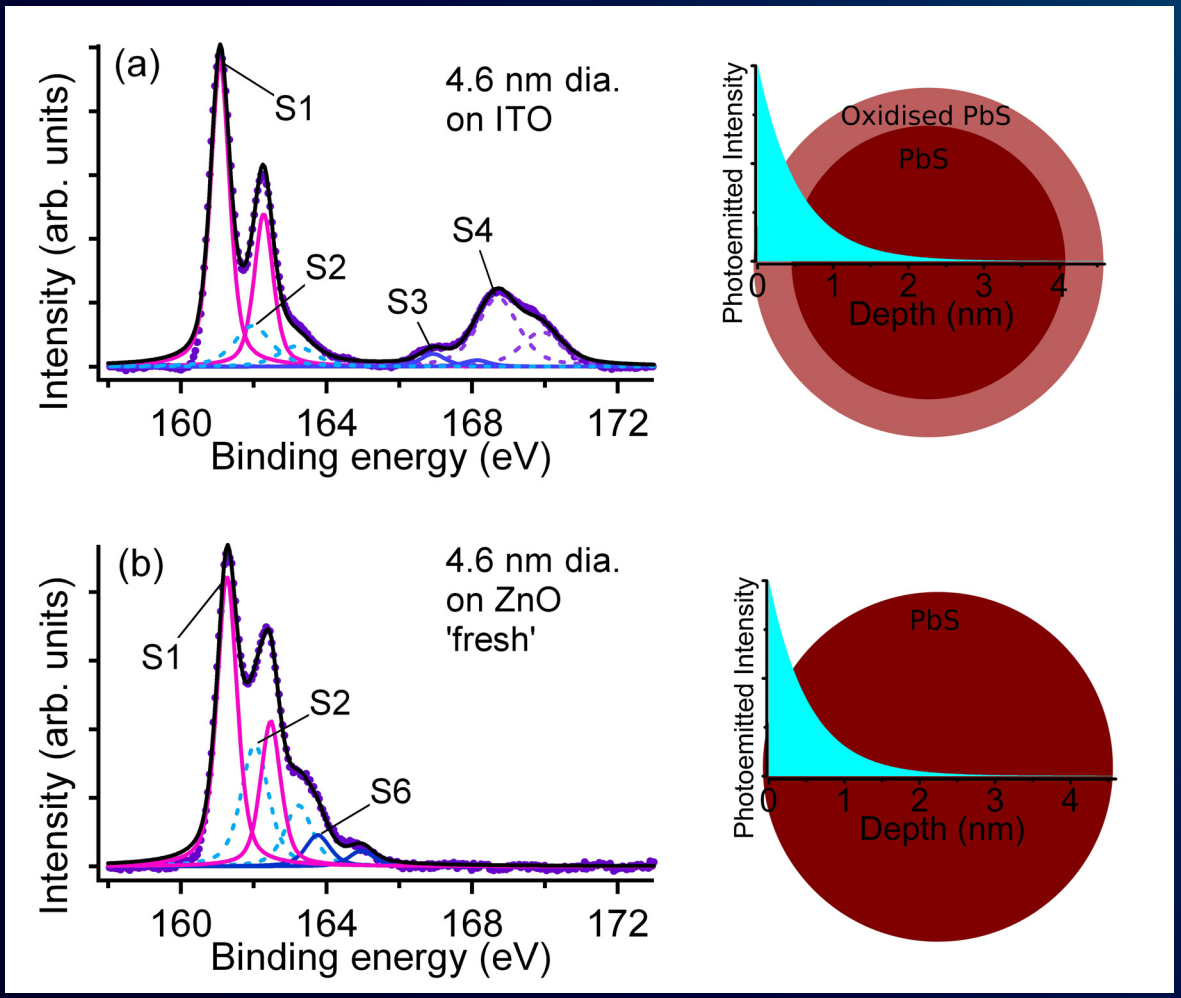


M.P. Seah and W.A. Dench, Surf. Interf. Anal. 1, 2, 1979

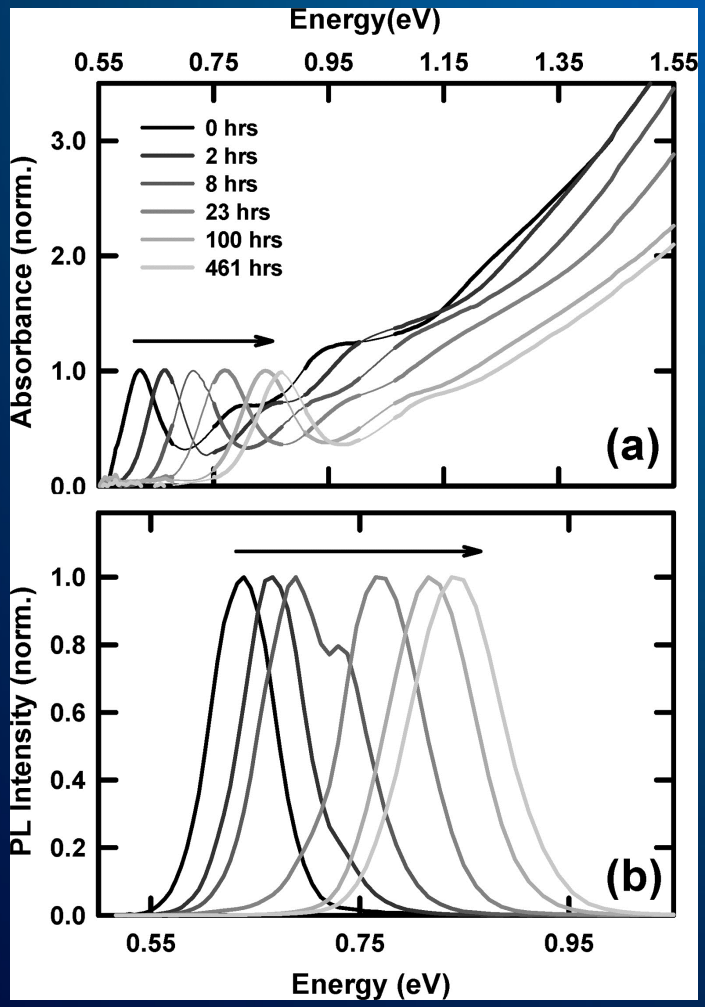


K Huang *et al.*, ACS Nano, 4 4799 (2010)

A handle on surface ageing



HR XPS reveals surface oxidation of PbS QDs



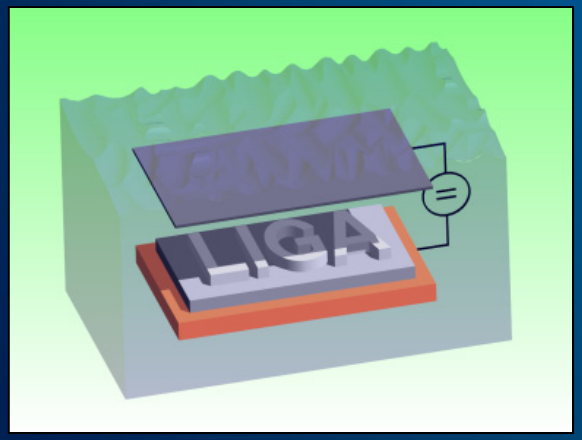
Blue-shift with degradation in PbSe QDs

S J O Hardman *et al*., Phys Chem Chem Phys, 13 20275 (2011)
M Sykora *et al.*, ACS Nano, 4 2021 (2010)

SOLEIL SYNCHROTRON elettra

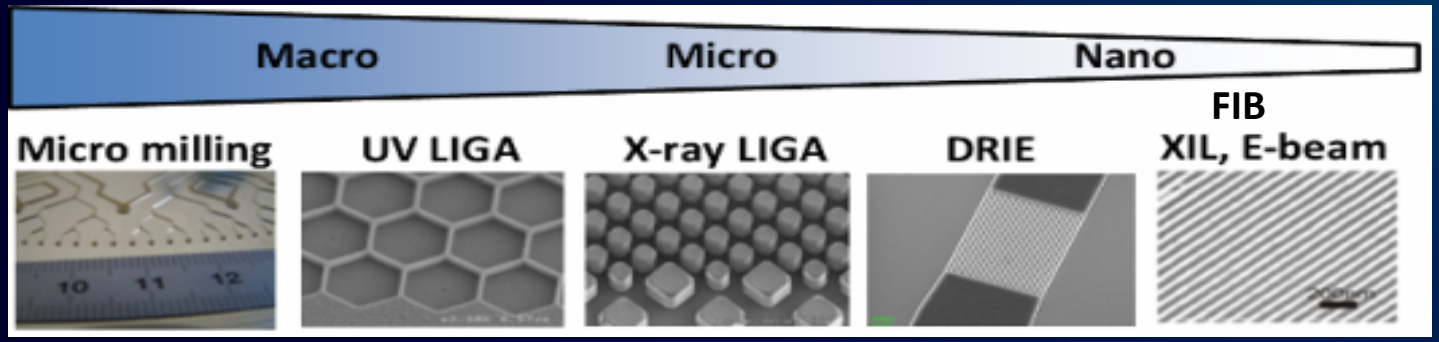
LIGA: niche areas for SR?

LI → lithography
- coating
- exposing
- developing



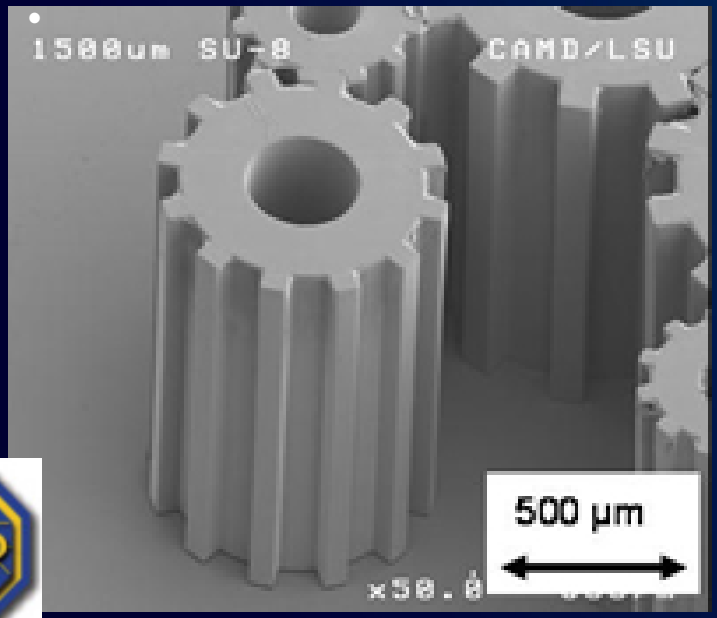
GA → electroplating (in German ‚Galvanik')
- electro-chemical deposition of a metal from a salt solution

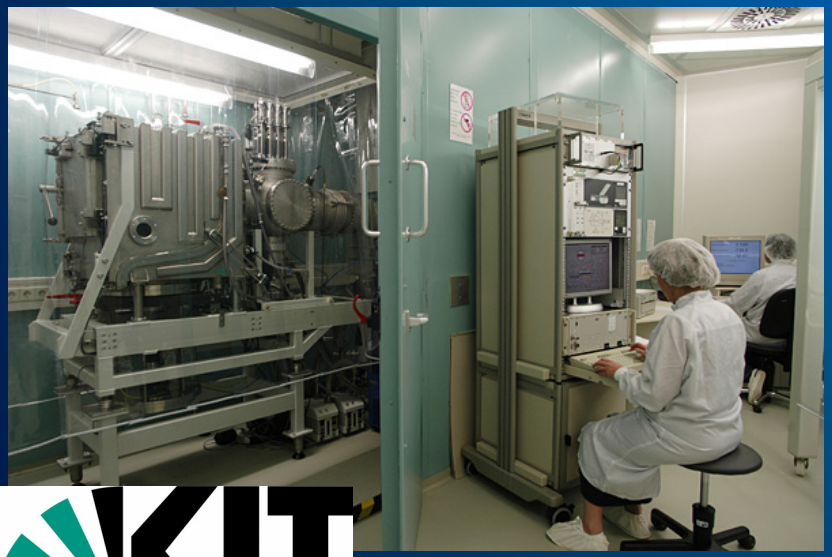
→ Metallic components through filling up of polymer patterns

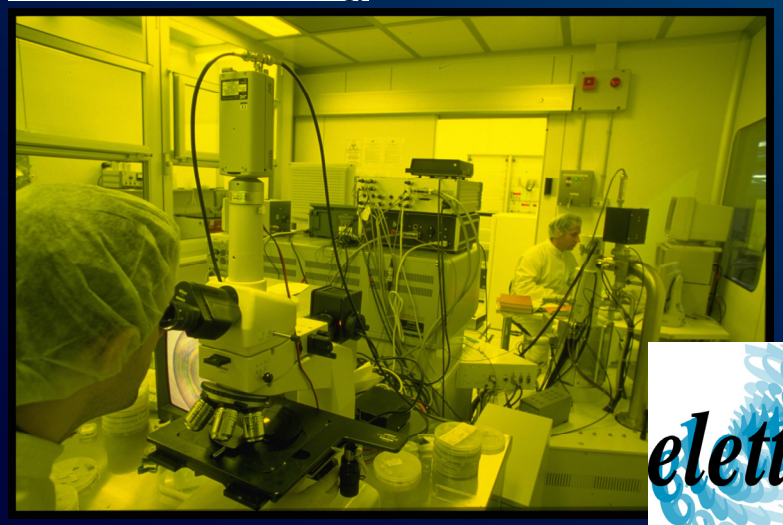


X-ray lithography for microfabrication

•Widely implemented at specialised sources and at national facilities, largely for R&D

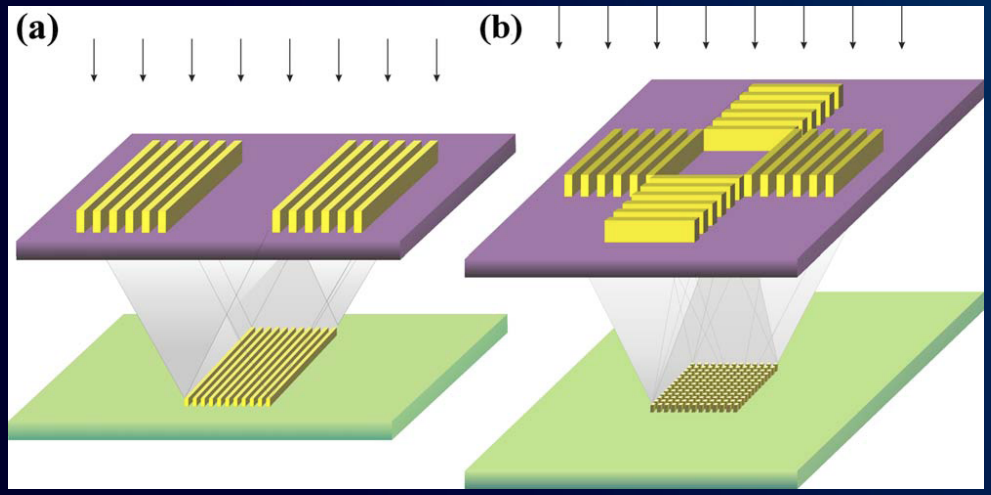






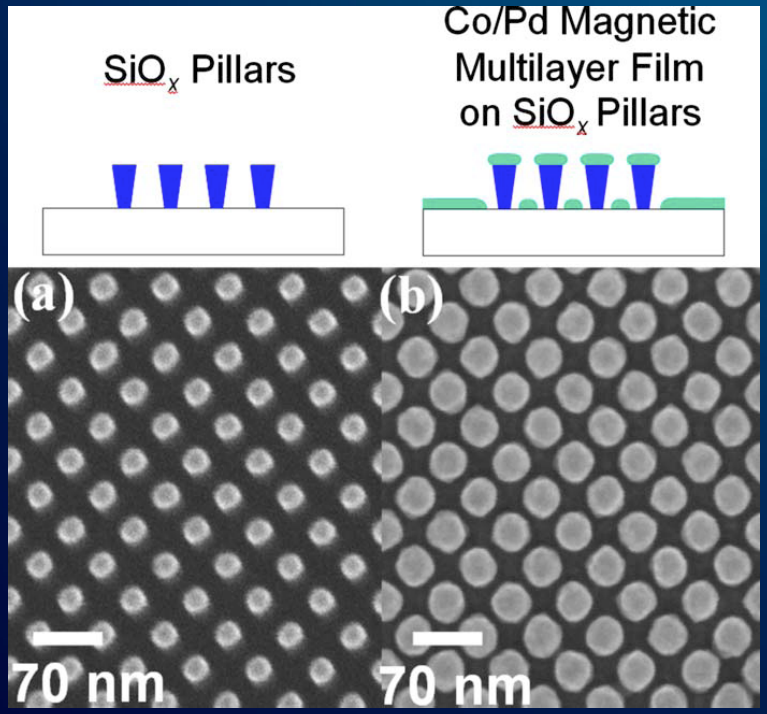
….and many more

Bit-patterned media prepared by EUV-IL



- Uses coherence of SR
- 2 and multi-beam interference schemes generate lines and dots
- High resolution and high throughput
- Much smaller proximity effects than EBL

- Used to produce 50 nm period magnetic islands
- Each bit of information is stored in an individual island
- Over 20 x 20 μm^2 area with narrow size distribution
- Data storage density of > 1 Tbit/in^2 achieved



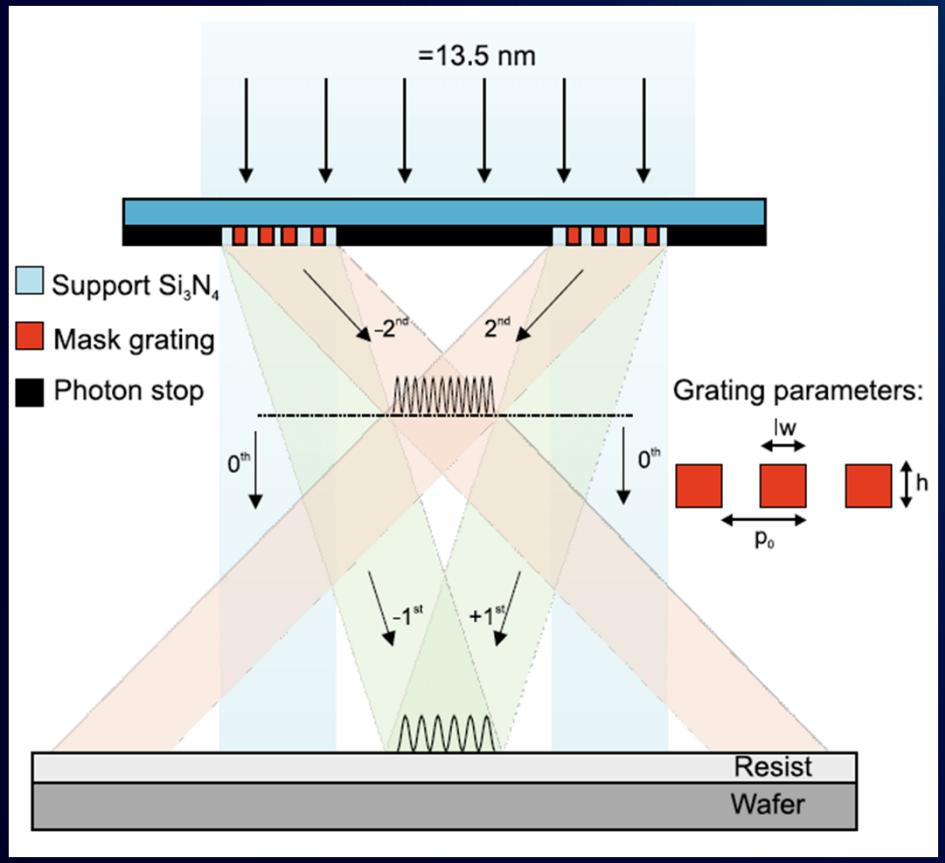
H H Solak, J Micro/Nanolith MEMS MOEMS 8 021204 (2009)
F Luo *et al.*, Appl. Phys. Lett. 92 102505 (2008)

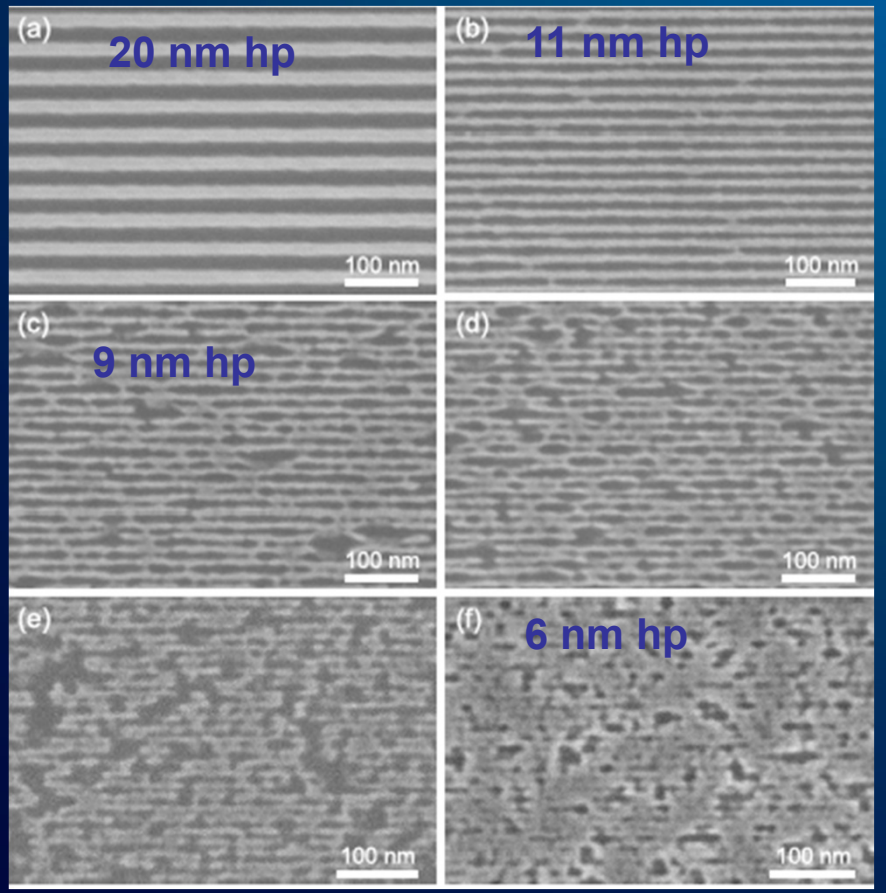
PAUL SCHERRER INSTITUT PSI HITACHI

Sub-10 nm features by EUV-IL

Aug 2011 Current record in photon-based lithography:
Top-down meets bottom-up?

- interference pattern from 2nd order diffraction
- Mo gratings fabricated by EBL, sub-50 nm scale





B Paivanranta *et al.,* Nanotechnology, 22, 375302 (2011)
EUV-IL: nanoetched Mo mask delivers sub-10 nm structures;
IOP Publishing; http://nanotechweb.org/cws/article/lab/47442

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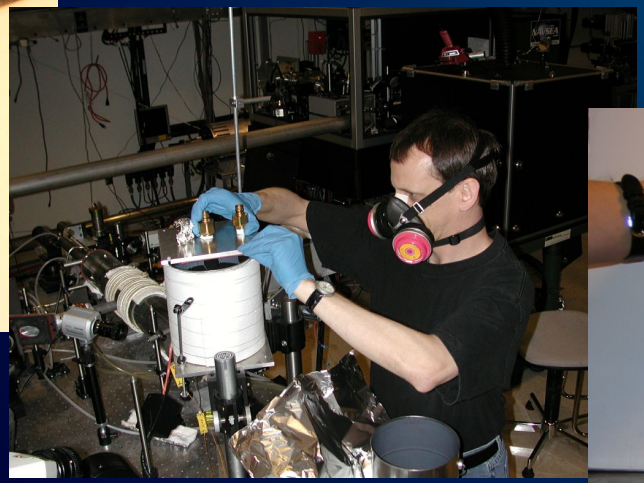
PSI

Prospects for FELs - synthesis

NASA/JLab Nanotube Synthesis since 2000 - Research to Production

New and used target

From target to product, 100% in-house



Product, ~1 hour of beam time

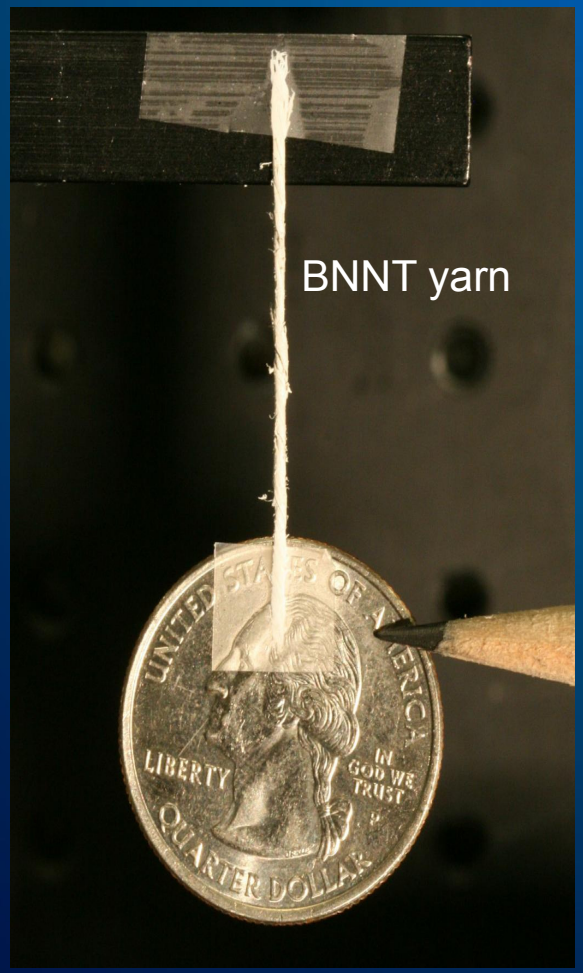
- Production with 750 W at 1.6 μm is routine.
- Production rate of 2-6 g/hour of high quality, "research grade" raw material cost competitive in $400/g market.
- Nanotube diameter is strong function of laser parameters - possibility of "designer" tubes
- Used for fibre-reinforced materials for aerospace applications

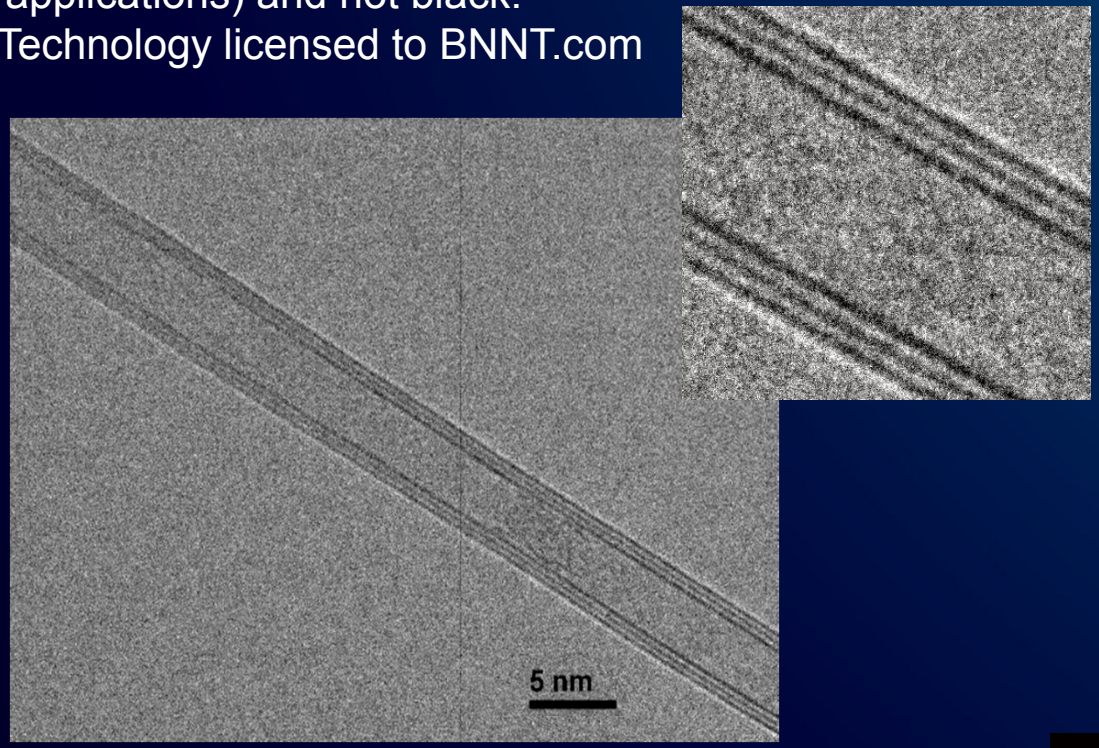
Mike Smith NASA LaRC

Jefferson Lab NASA

Boron nitride and hybrid nanotubes

- Pressurised vapour/condenser method (B vapour in N_2 gas, no catalyst)
- Few-walled NTs, aspect ratio > 10^5
- Basic technique developed at FEL, then continued with conventional laser
- Strong, tough (impact protection), good thermal conductivity, radiation shielding (aerospace applications) and not black.
- Technology licensed to BNNT.com



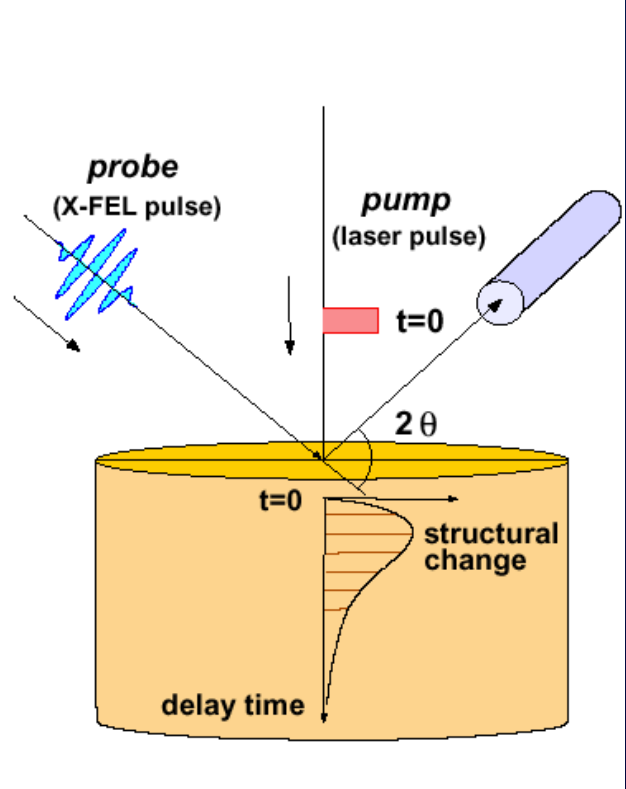


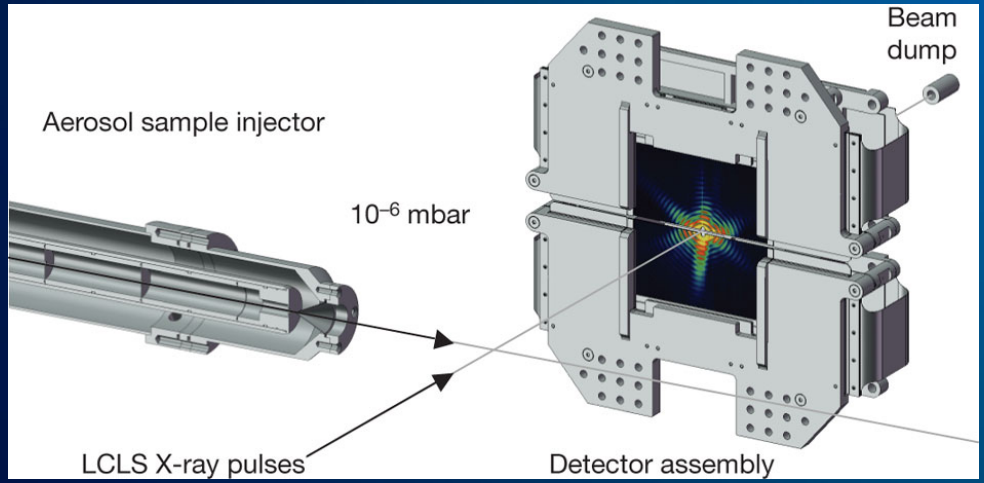
M W Smith *et al.,* Nanotechnology, 20, 505604 (2009)
J-W Kim *et al.,* Nanotechnology, 23, 035701 (2012)
M Zheng *et al.,* Nanotechnology, 23, 095703 (2012)

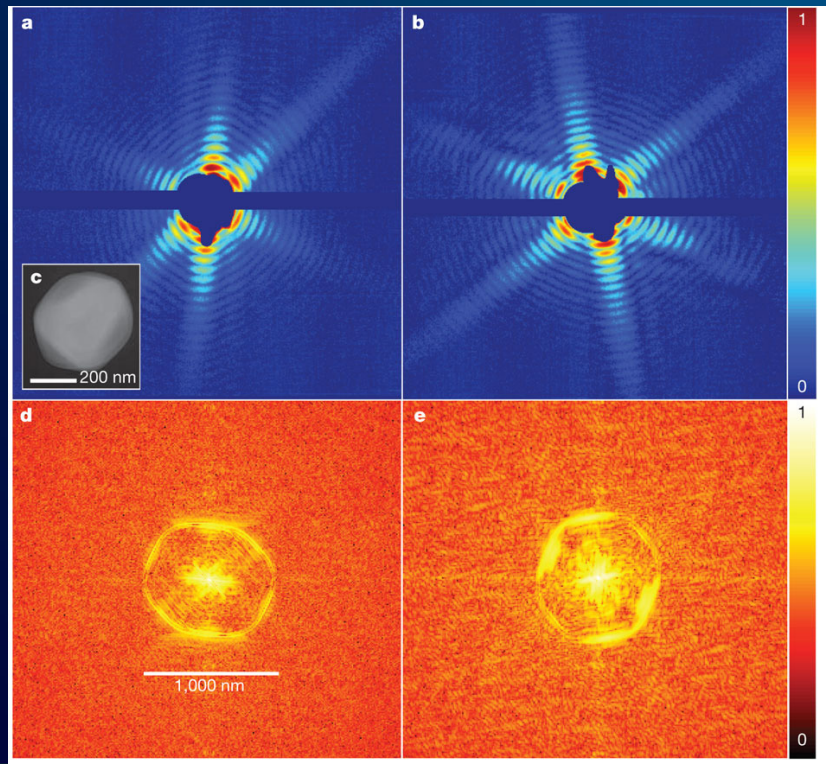
Jefferson Lab

NASA

Prospects for FELs: time-resolved diffraction





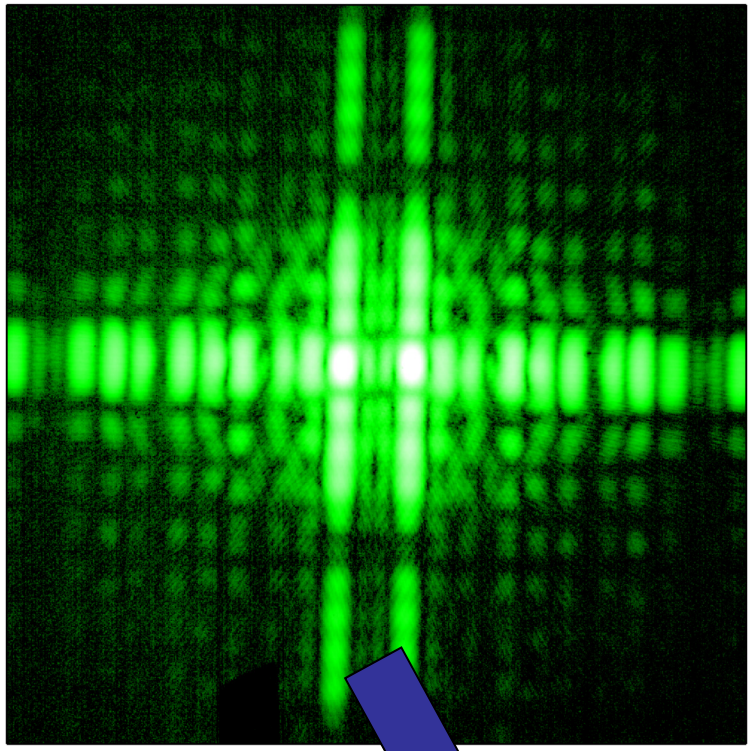


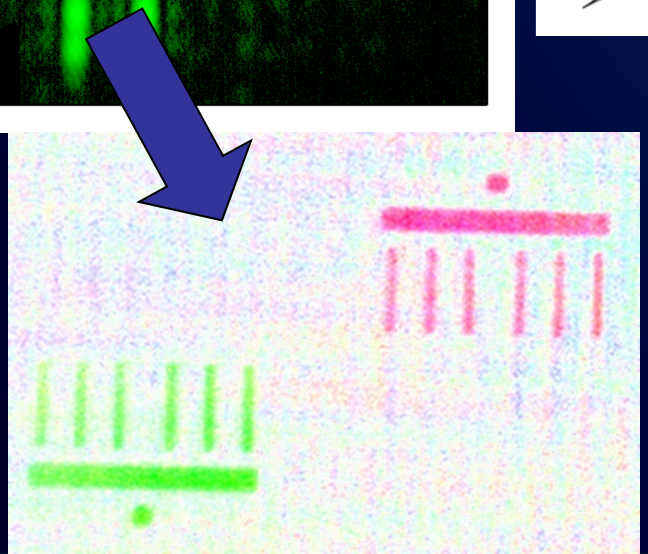
- Interpretable single shot diffraction obtained from single mimivirus particles at 32 nm resolution
- 15,000 shots recombined to give structure of Photosystem I at resolution down to 8.5 Å

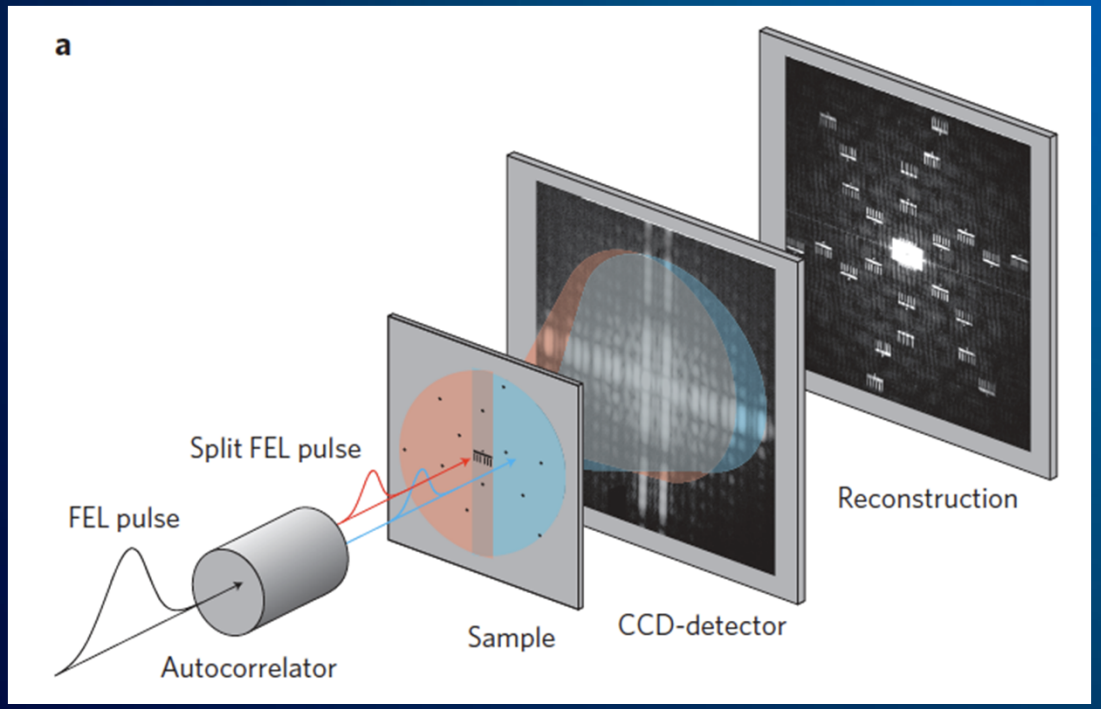
LCLS

M M Seibert *et al.,* Nature, 470 78 (2011)
H M Chapman *et al.,* Nature, 470 73 (2011)

Prospects for FELs: time-resolved diffraction



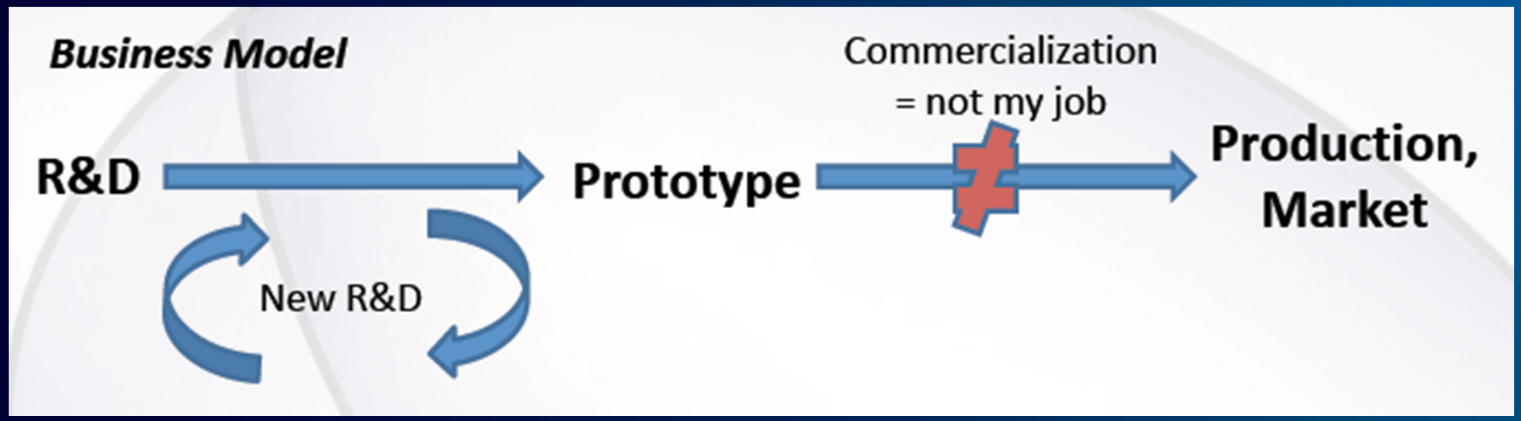




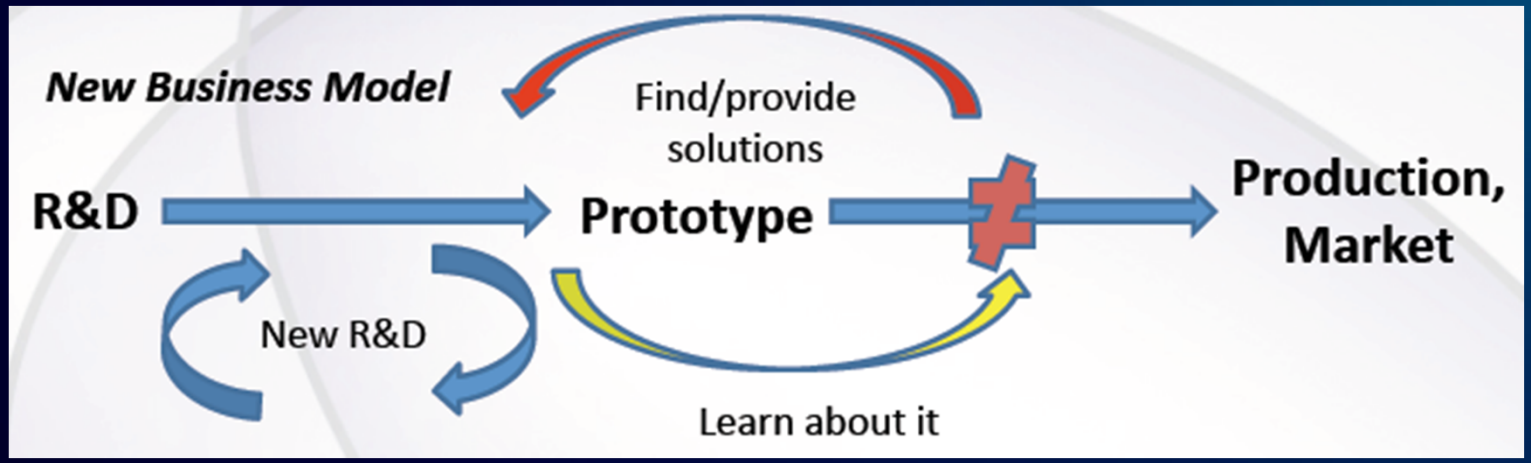
- two sequential images obtained at separation of 50 fs
- World's fastest movie! (Guinness BWR)
- 2 images from split pulse recorded as superimposed hologram

C M Günther *et al.*, Nature Photonics 5 (2011) 99
http://desy.de/information_services/press/pressreleases/@@news-view?id=2181&lang=eng

What's the issue with industrial exploitation of SR?



- Maybe OK for one-off specialist application (satellite, military); performance rather than unit cost

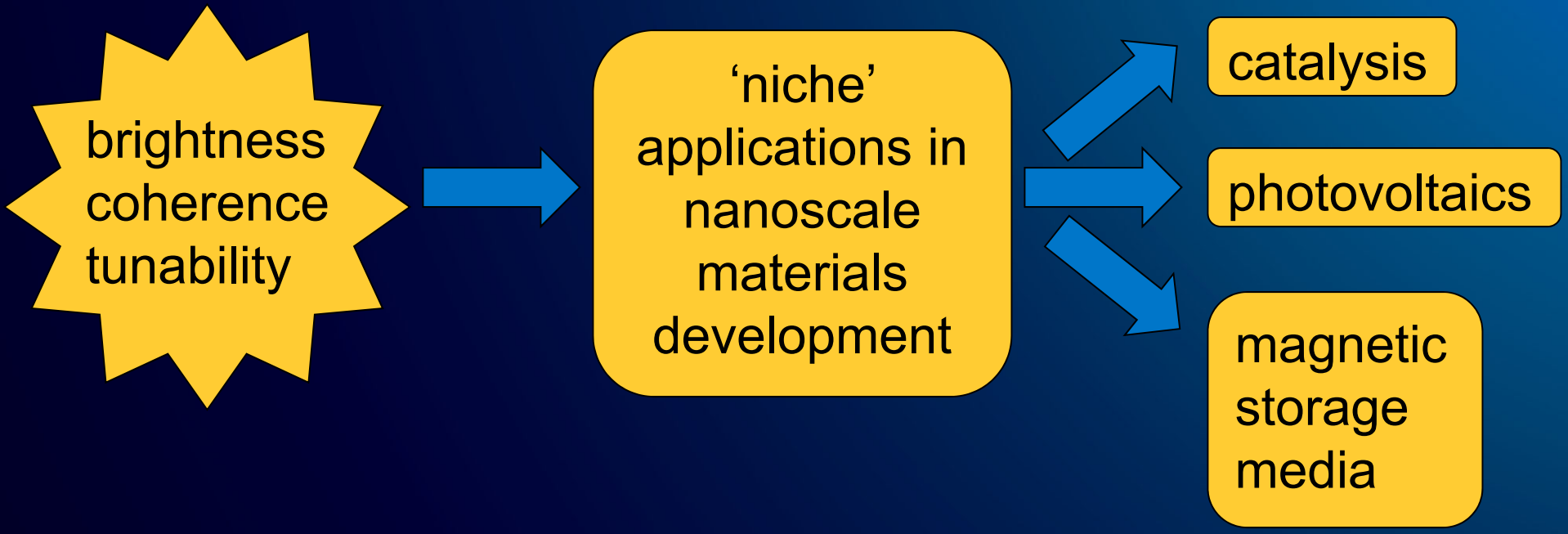


- process developments at FEL/SR transfer offline
- link-up with larger manufacturing concern
- licensing

Jost Goettert, CAMD, on LIGA

Conclusions

- 3rd SR offers unique capability from



- FELs offer the dynamic information to complete the picture, and synthetic capability that needs to be explored much further

- but SR scientists need help getting from prototype to production/market

Thanks to

- Yasutake Nagai, Toyota Central R&D Labs
- George Neil and colleagues at JLab
- Jost Goettert, CAMD
- Guillaume Reinhart, ESRF
- Nanoco Technologies Ltd
- Tom Thomson, University of Manchester
- Samantha Hardman, University of Manchester