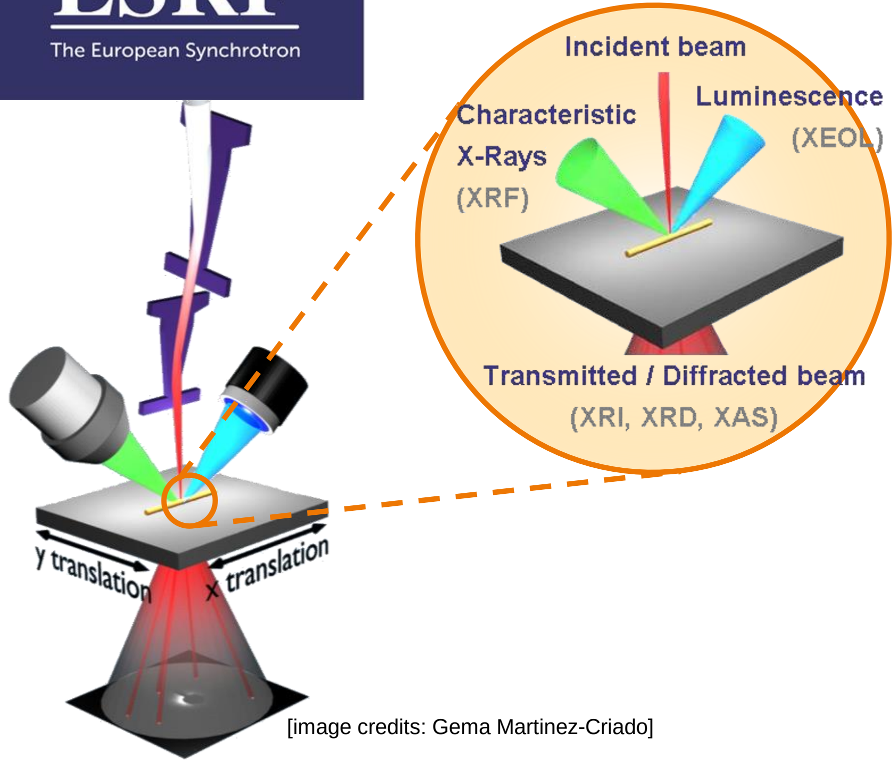
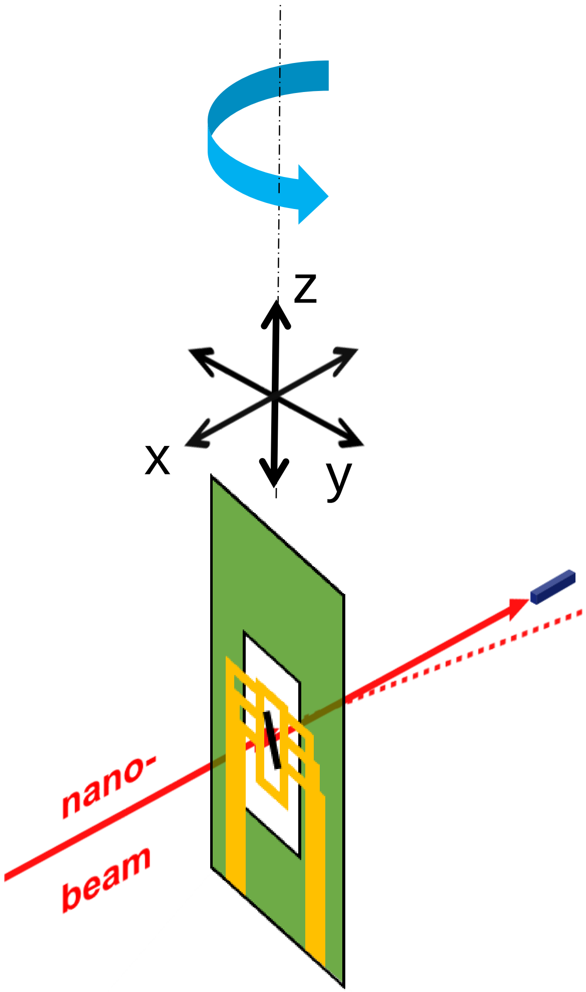


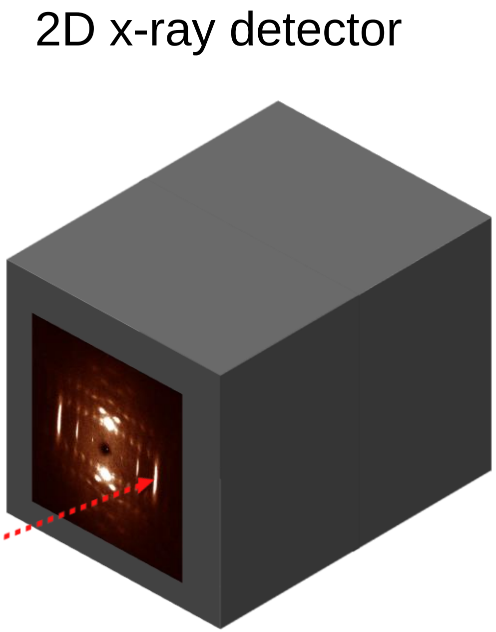
[image credits: Gema Martinez-Criado]

Synchrotron based nano-focused X-ray investigations

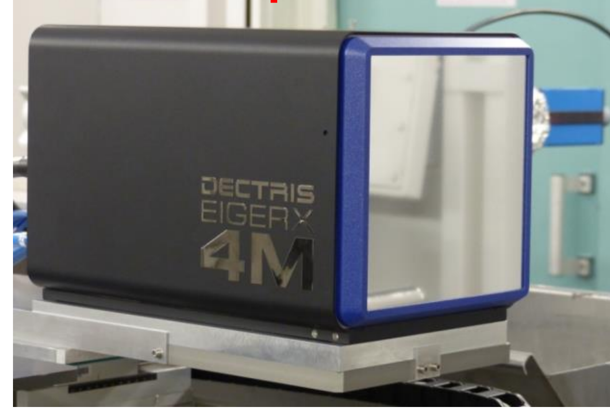
and
examples of X-Ray beam modification of samples at ID13

Andreas Johannes, Tilman Gruenewald, Martin Rosenthal, Manfred Burghammer





EIGER 4M pixel detector



- 750 Hz frame rate
- dynamic range ~10^6
- ~2167 x 2070 pixels
- single photon counting
- 75 µm pixel size

default: **lateral 2D-scans**

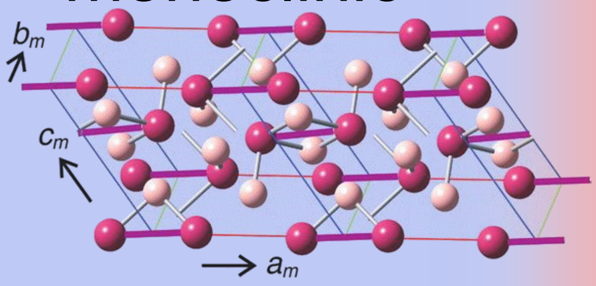
optionally combined with vertical rotation

→ in single crystals probe the Bragg reflex

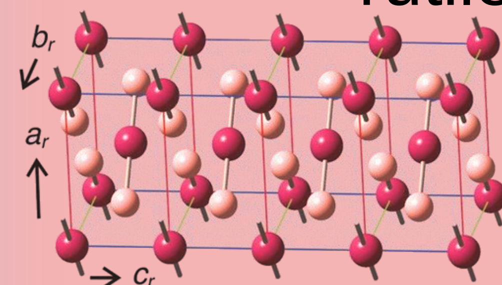
Sample: VO_2 microwire on heatable SiN foil chip

VANADIUM DIOXIDE PHASE TRANSITION

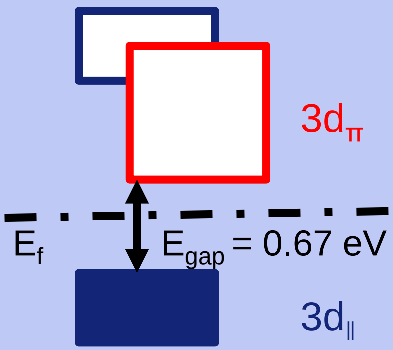
monoclinic



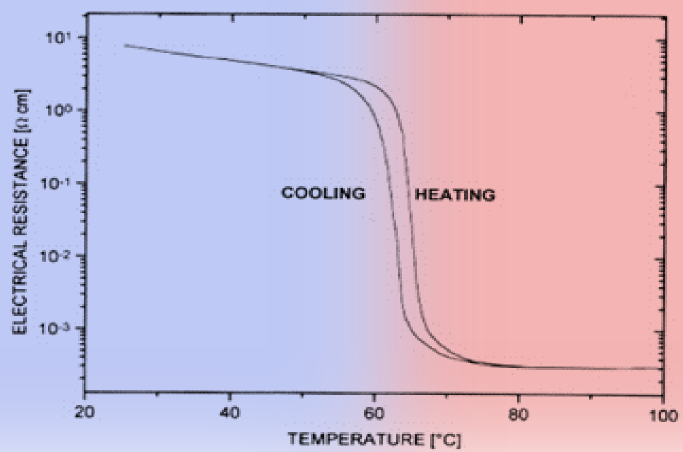
rutile



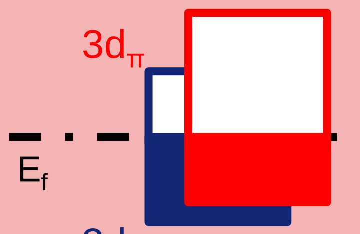
[1]



insulator



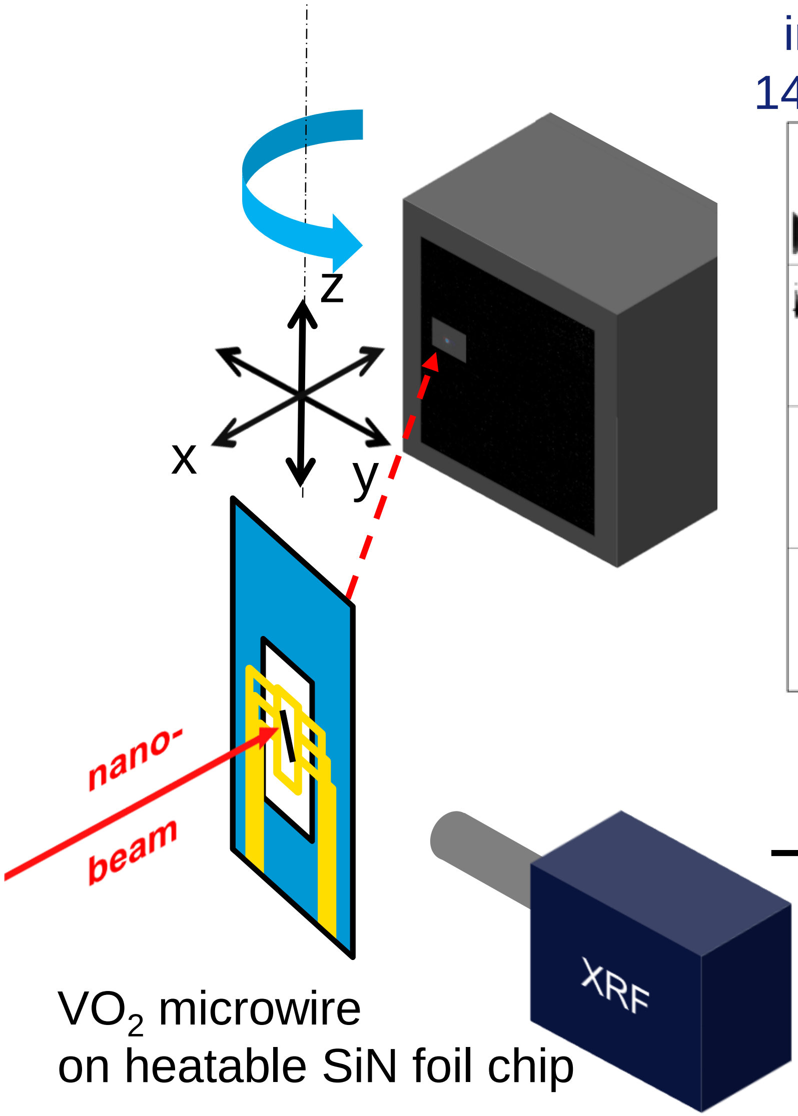
[2]



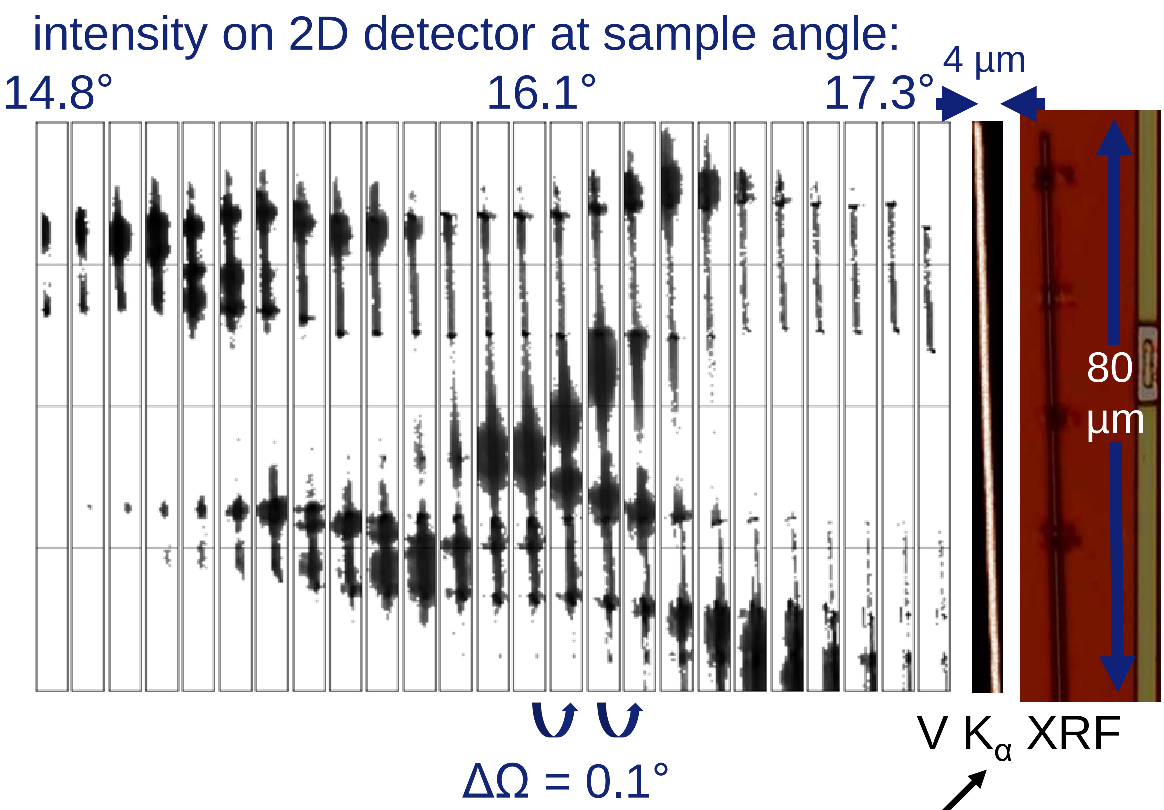
quasi - metal

1. Baum P, Yang D-S, Zewail AH. Science. (2007) 2. Joyeeta Nag and R F Haglund Jr J. Phys.: Condens. Matter (2008)

NANO-FOCUSED DIFFRACTION IMAGING

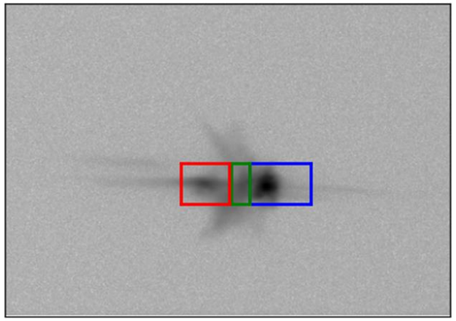


VO_2 microwire
on heatable SiN foil chip

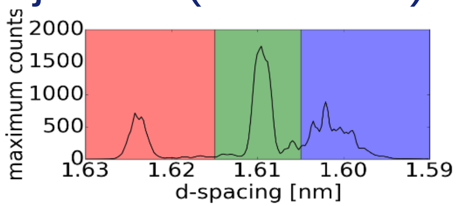


sample raster scanning:
4 µm × 80 µm × 2.5 °
12 × 240 × 25 steps

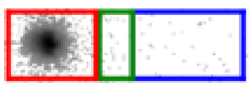
NANO-FOCUSED DIFFRACTION IMAGING

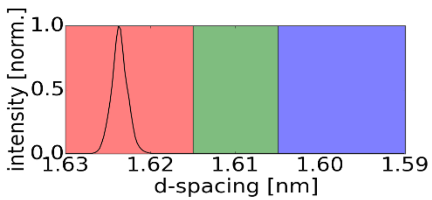


maximum projection (all frames):

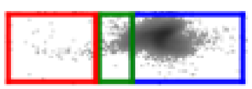


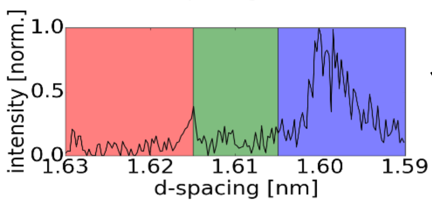
rutile:



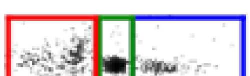


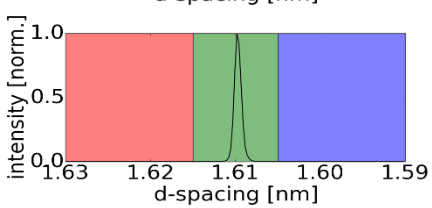
monoclinic 1:

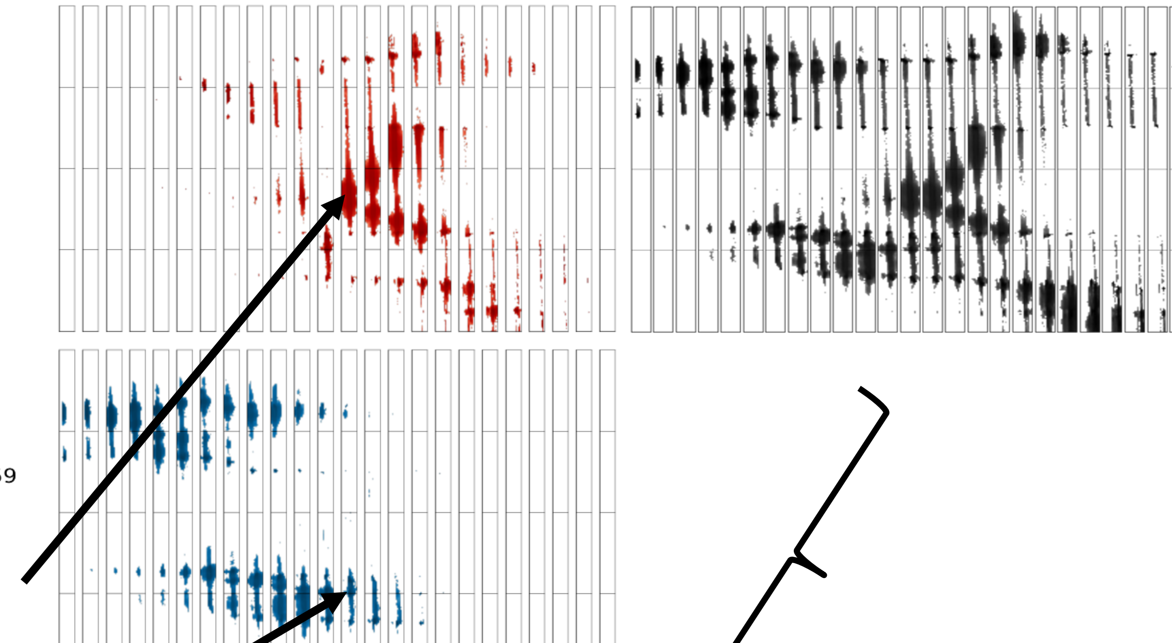


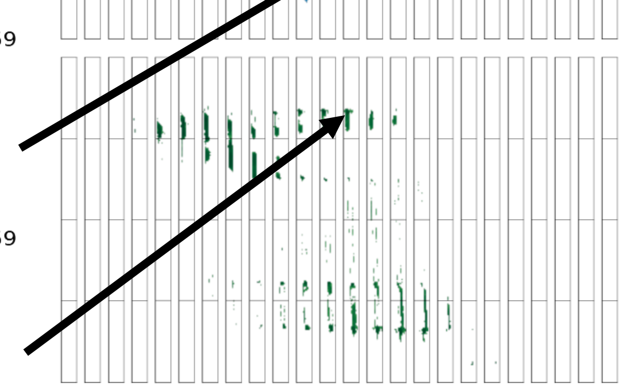


monoclinic 2:





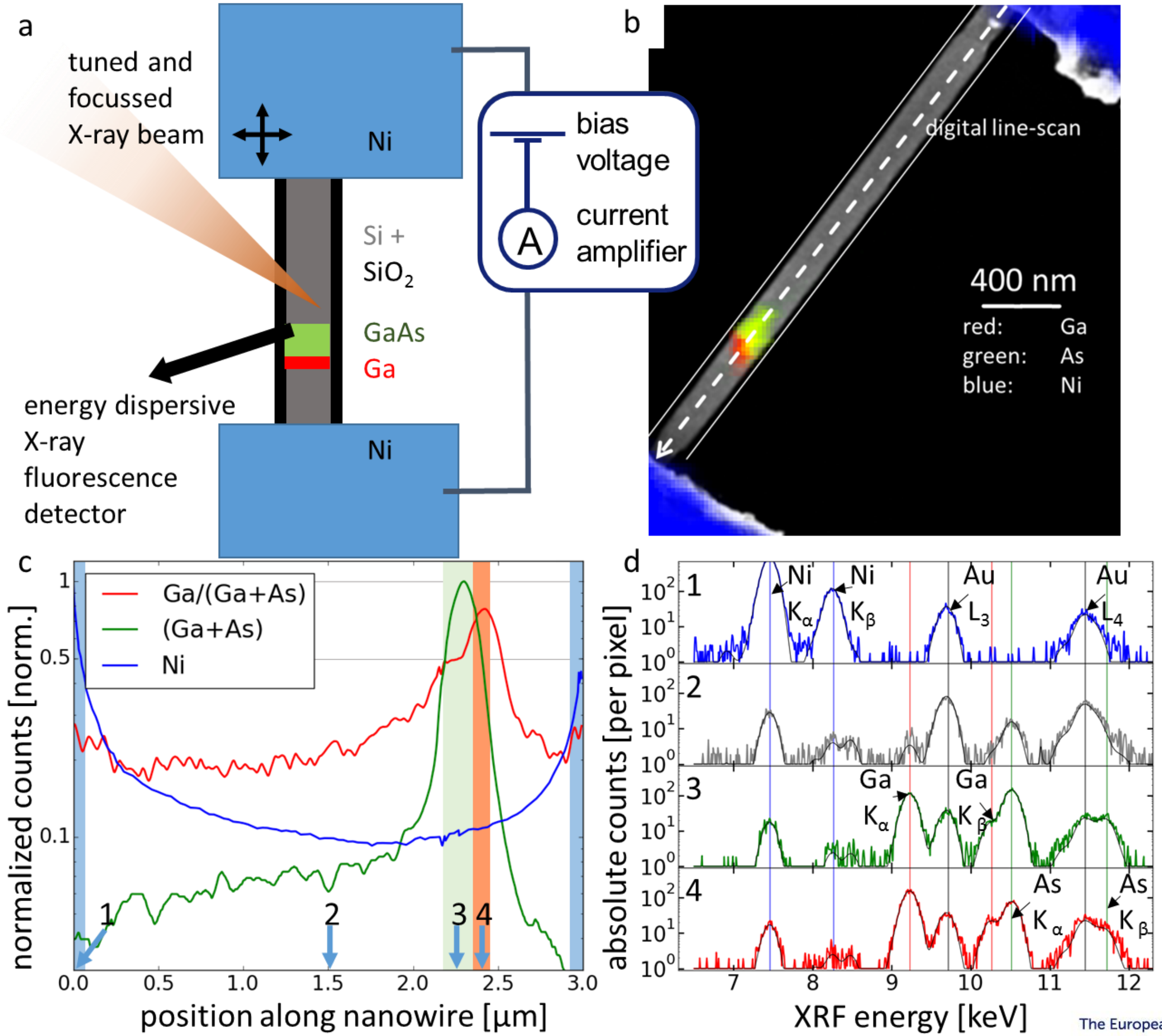




mapping of:
phases
and their strain

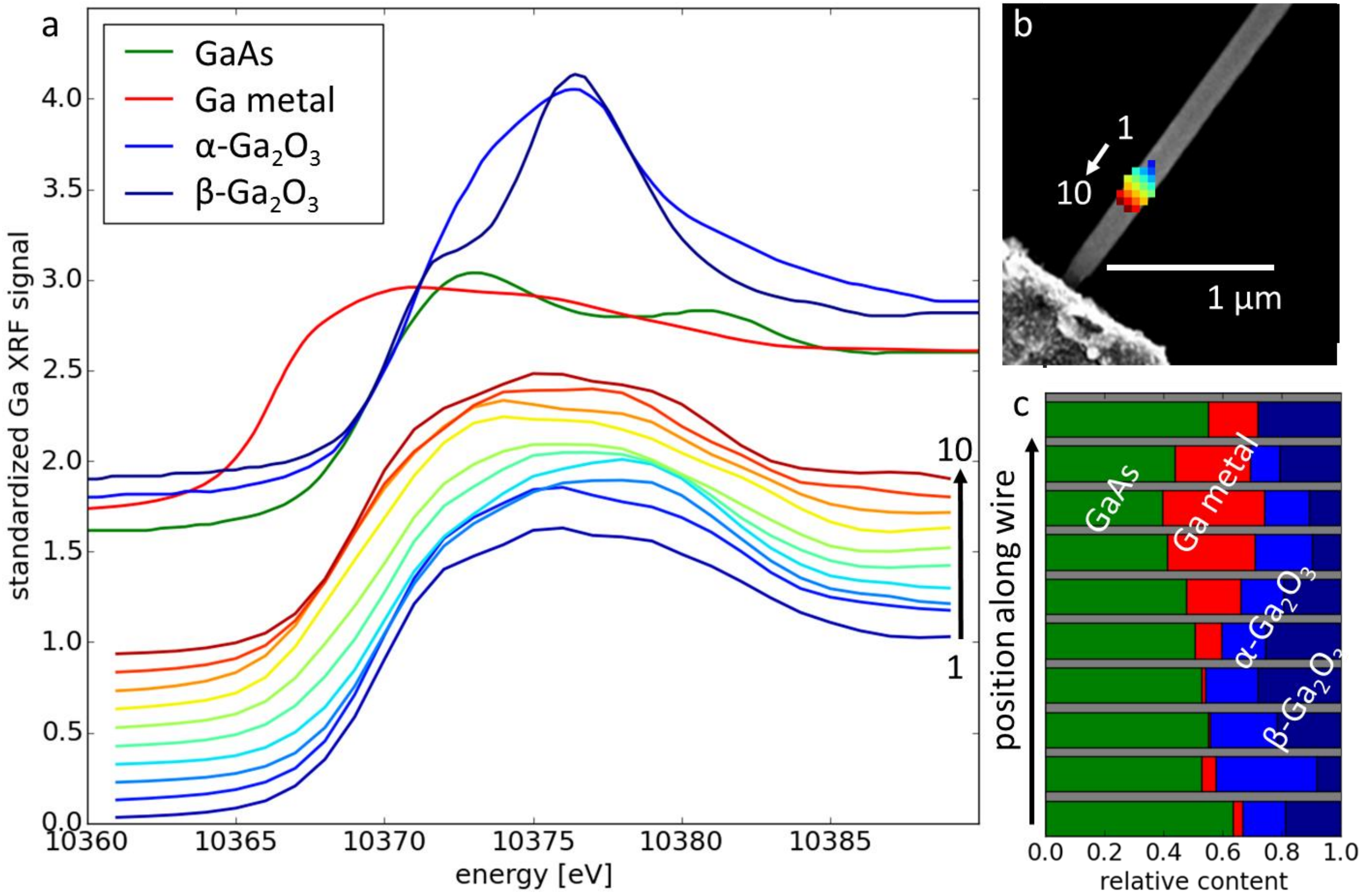
…
work in progress

XRF MAPPING



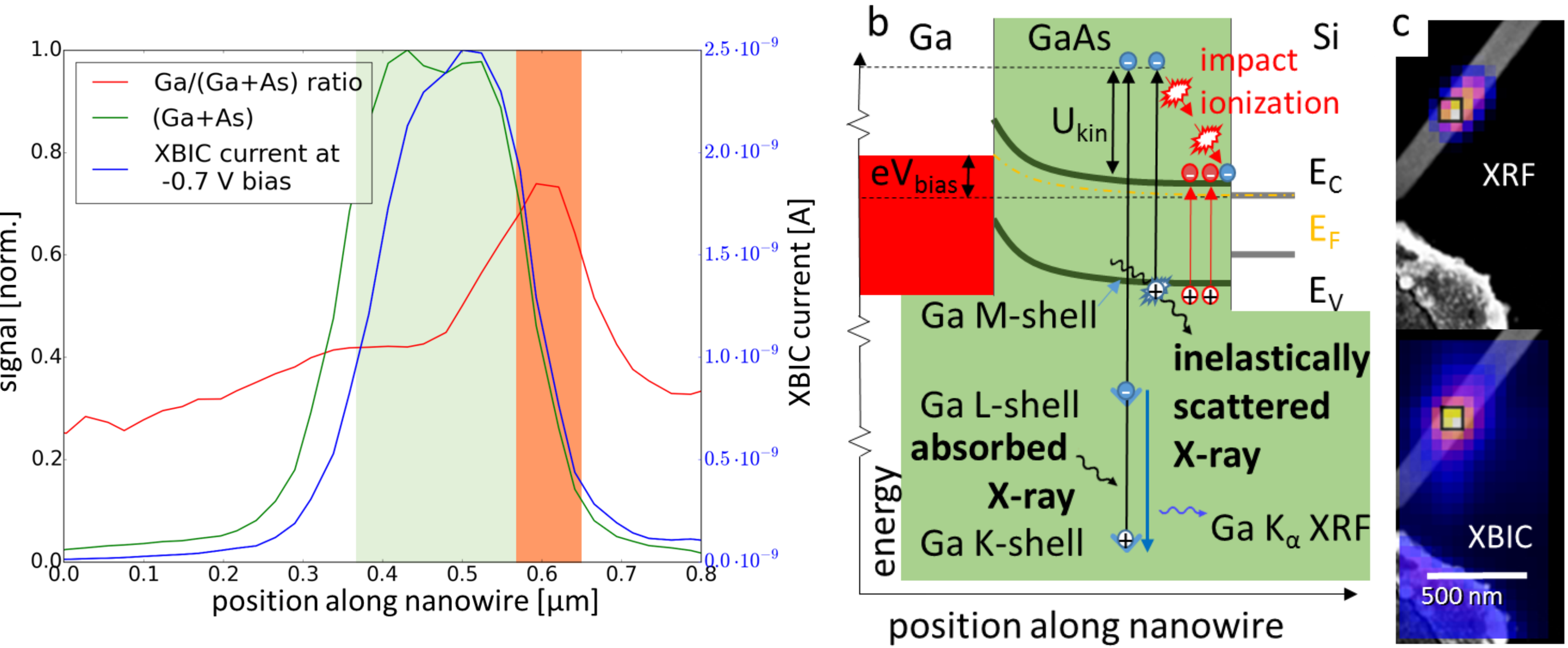
a
tuned and focussed X-ray beam
Ni
Si + SiO_2
GaAs
Ga
energy dispersive X-ray fluorescence detector
Ni
bias voltage
current amplifier
A
b
digital line-scan
400 nm
red: Ga
green: As
blue: Ni
c
Ga/(Ga+As)
(Ga+As)
Ni
normalized counts [norm.]
position along nanowire [µm]
1
2
3
4
d
Ni K_α
Ni K_β
Au L_3
Au L_4
Ga K_α
Ga K_β
As K_α
As K_β
absolute counts [per pixel]
XRF energy [keV]

NANO - XANES

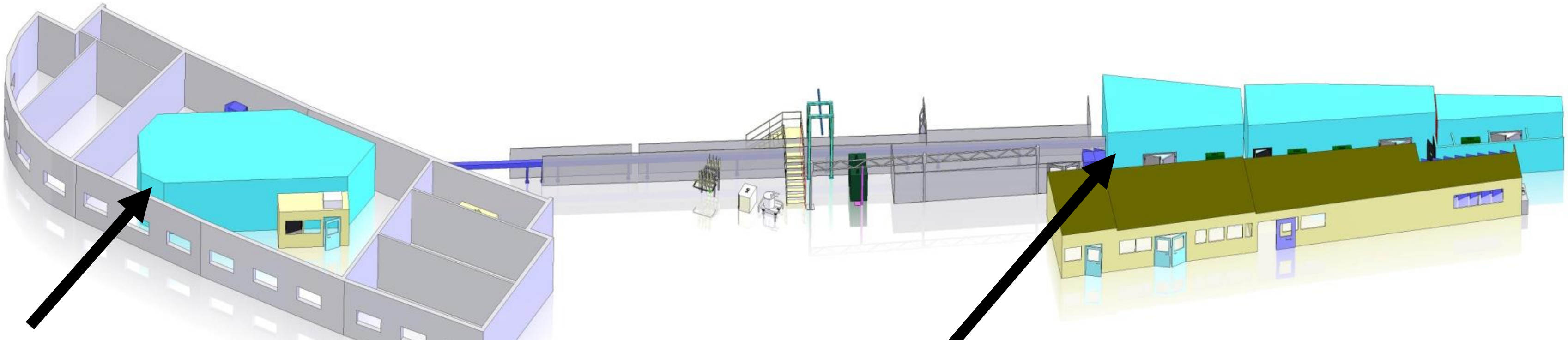


→ direct evidence of metallic Gallium

X-RAY BEAM INDUCED CURRENT - XANES



With a bias or intrinsic field
X-ray Beam induces a current (XBIC)



nanobranch

10^{10} ph/s

100 – 500 nm FWHM

beam @ 15 keV

Using Si-NFL, MLL, ZP

microbranch

10^{12} ph/s

1.5x2 μm^2

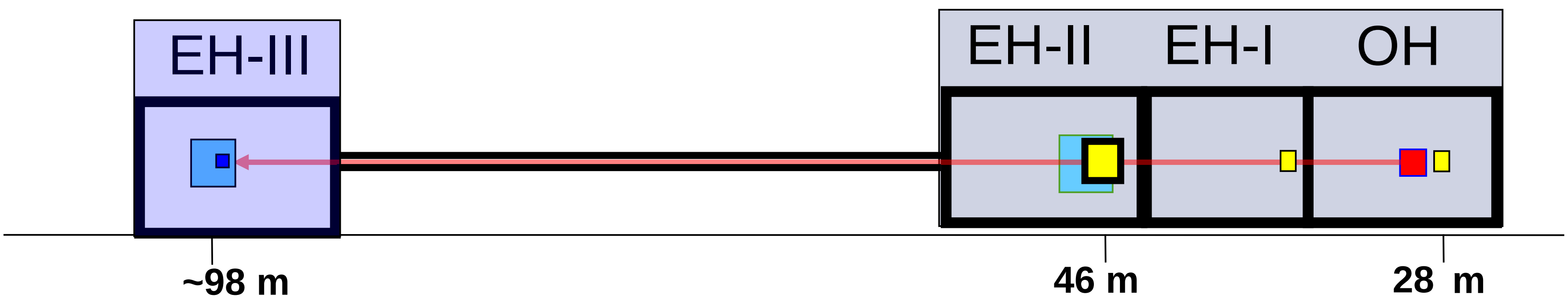
@ 13 keV

using Be-lenses

liquid N_2-cooled

Si_{111} channel-cut

monochromator



White beam
Be-CRL optics

TYPICAL BEAM SIZES

Microbeam (EH-II)

2 x 1.5 microns
2 x 10^{12} ph/sec
13 keV

Nanobeams (EH-III)

170 nm
(Si-NFL + prefocus)
1.5 x 10^{10} ph/sec
15 keV

80 nm (Si-NFL)
8 x 10^{8} ph/sec
15 keV

40 nm (MLL-lenses)
~10^{10} ph/sec
12.7 keV

HOW HOT IS THE BEAM?

$$10^{12} \text{ photons} / (\text{s } \mu\text{m}^2)$$

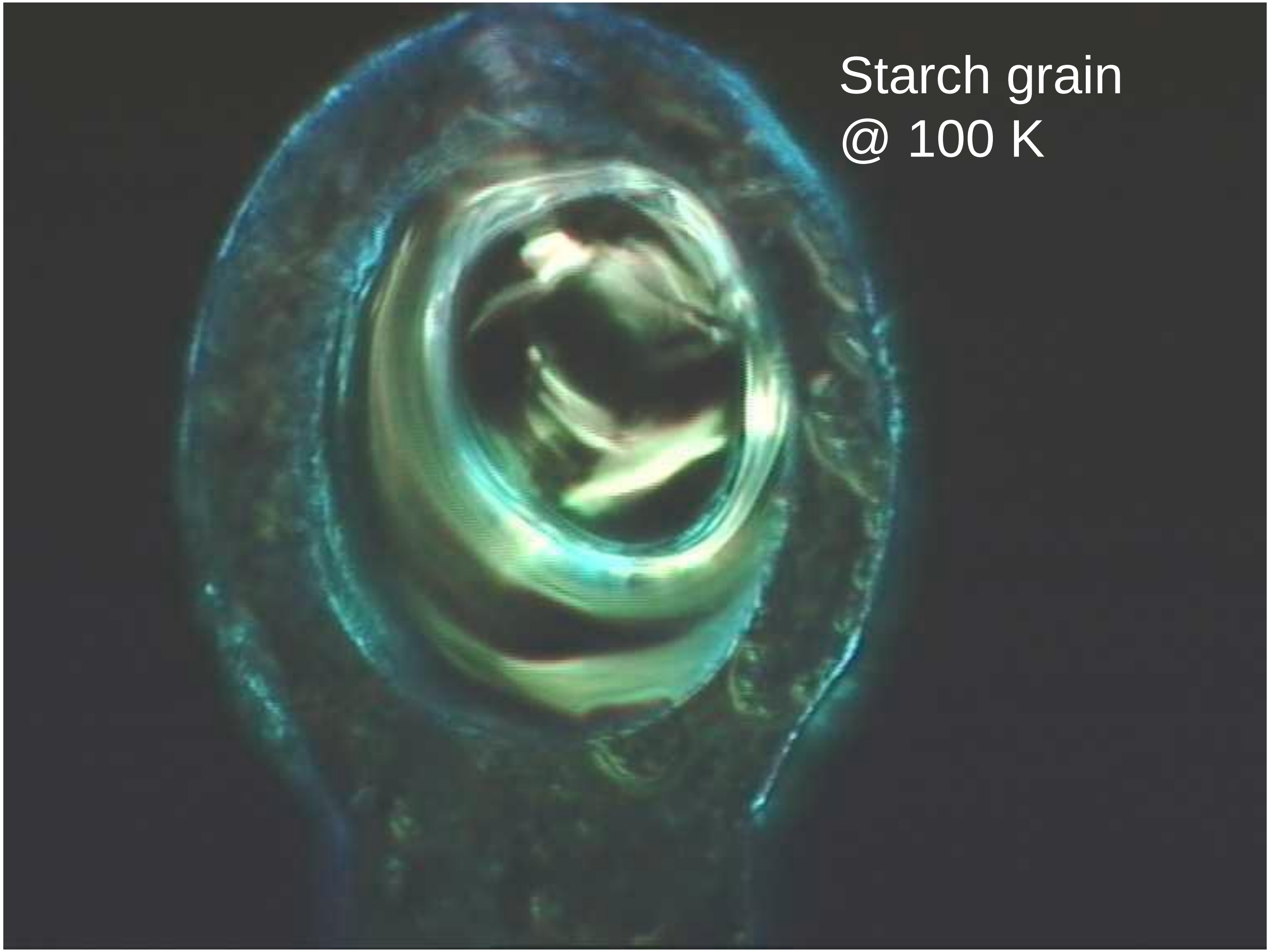
@ 10 keV
power density:
$(10^5 \cdot 10^{12})$ eV/ (s μm^2)
= 10^{17} eV/ (s μm^2)
= 16 mW / μm^2

assuming 10 µm = full absorption
= 1.6 mW / μm^3

at typical atomic density:
$5 \cdot 10^{22}$ / cm^3 = $5 \cdot 10^{16}$ / μm^3

that is 0.5 eV/s per atom!

RADIATION DAMAGE EXEMPLIFIED ON STARCH



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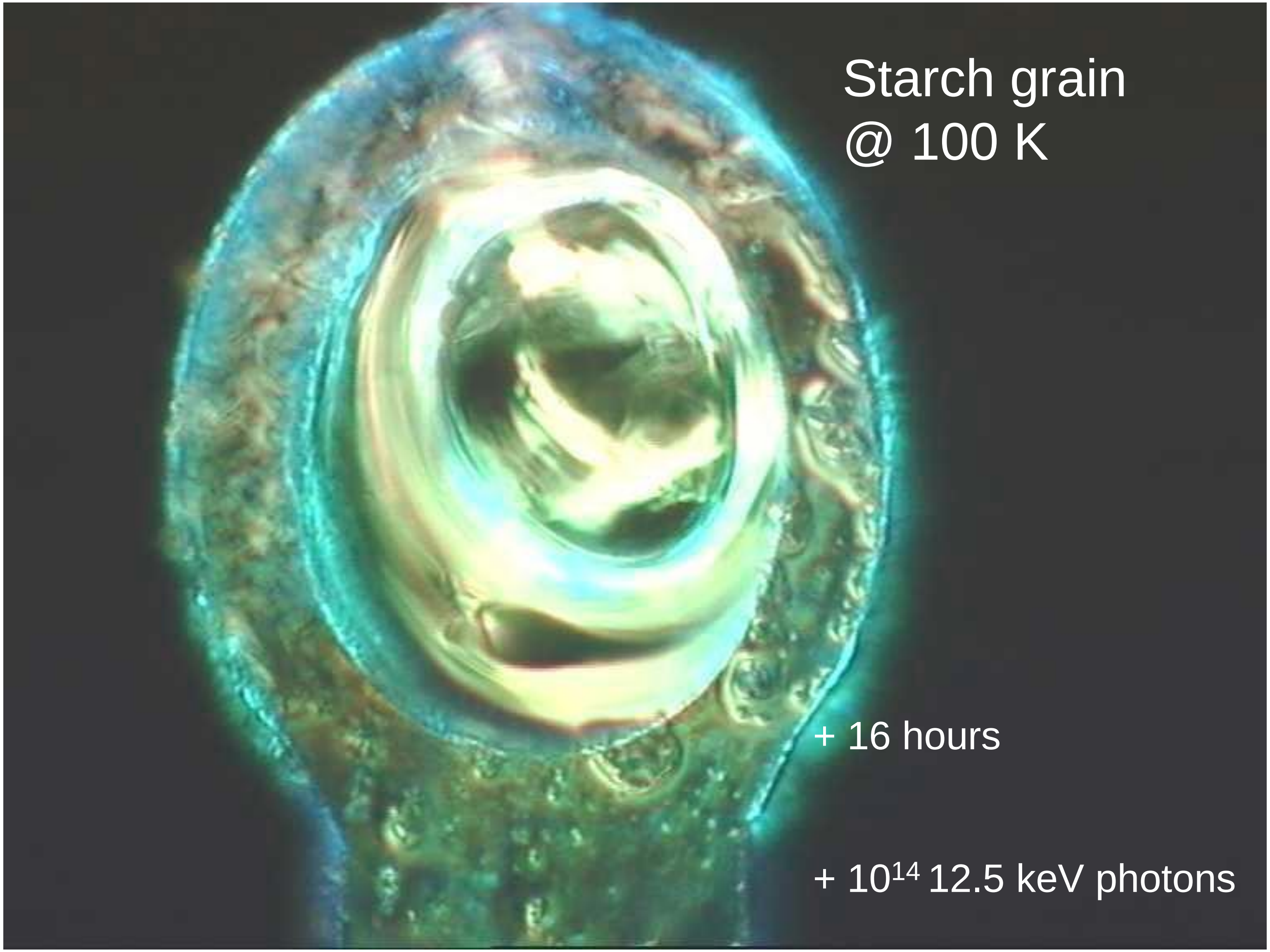

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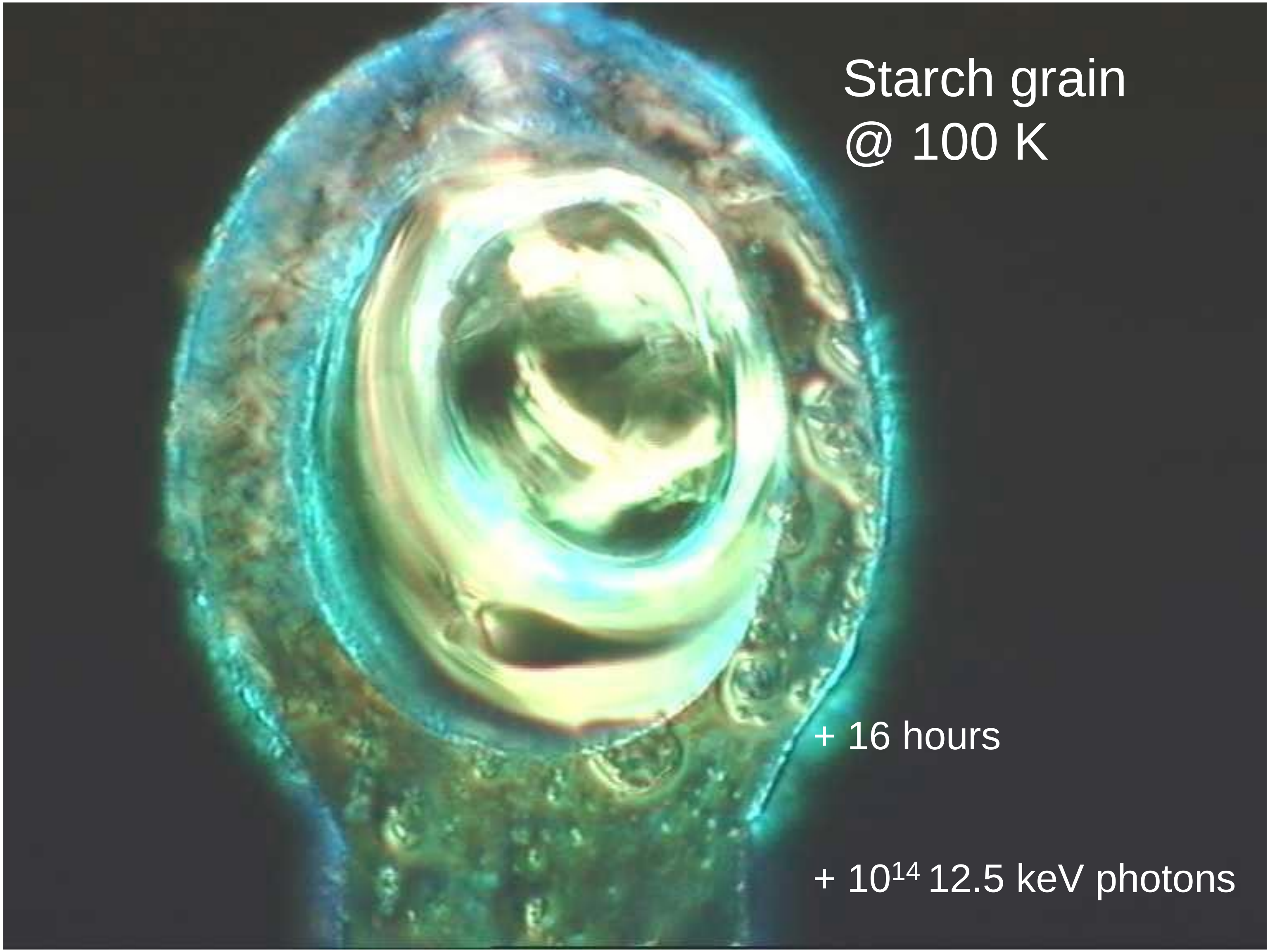

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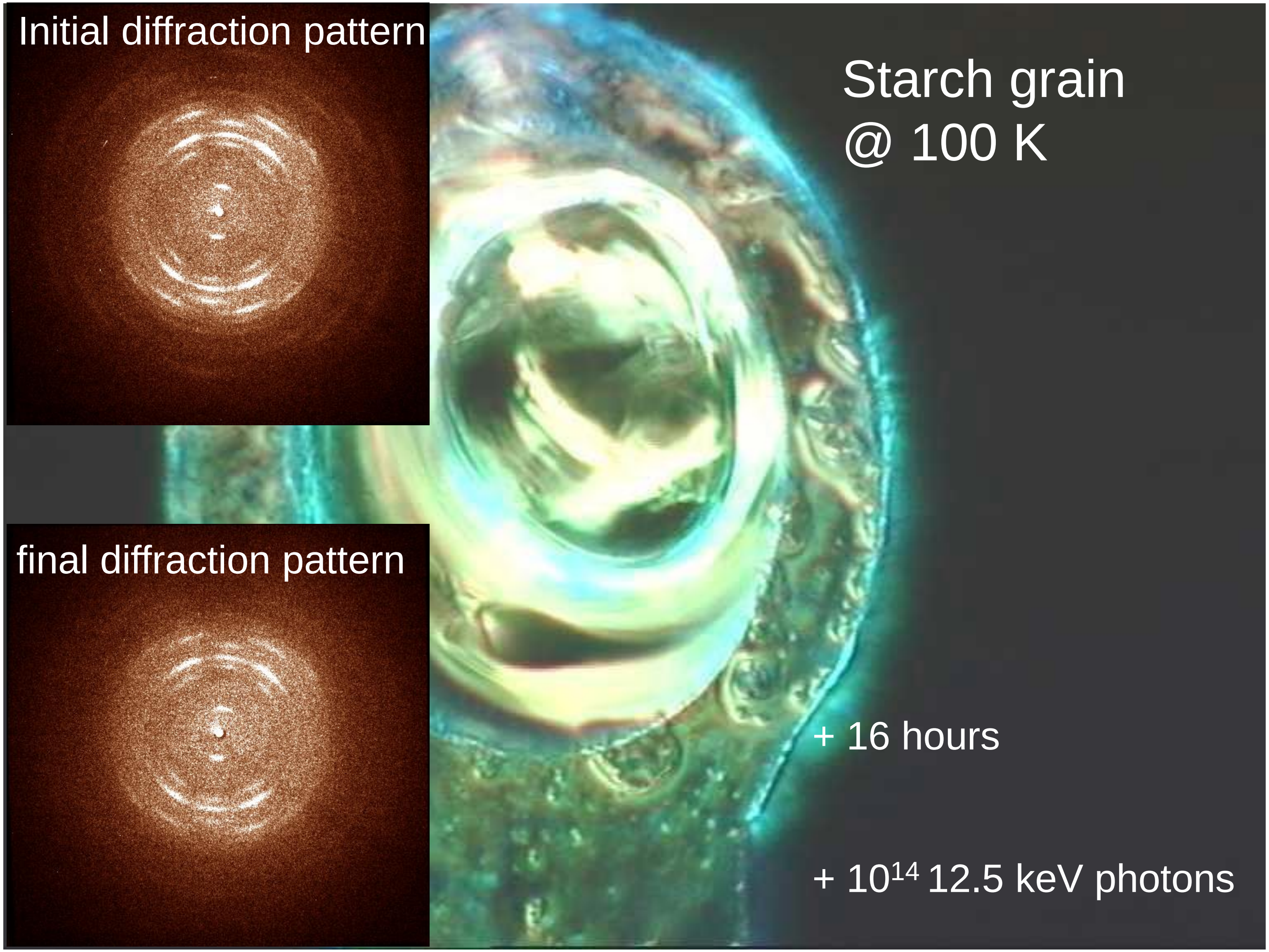

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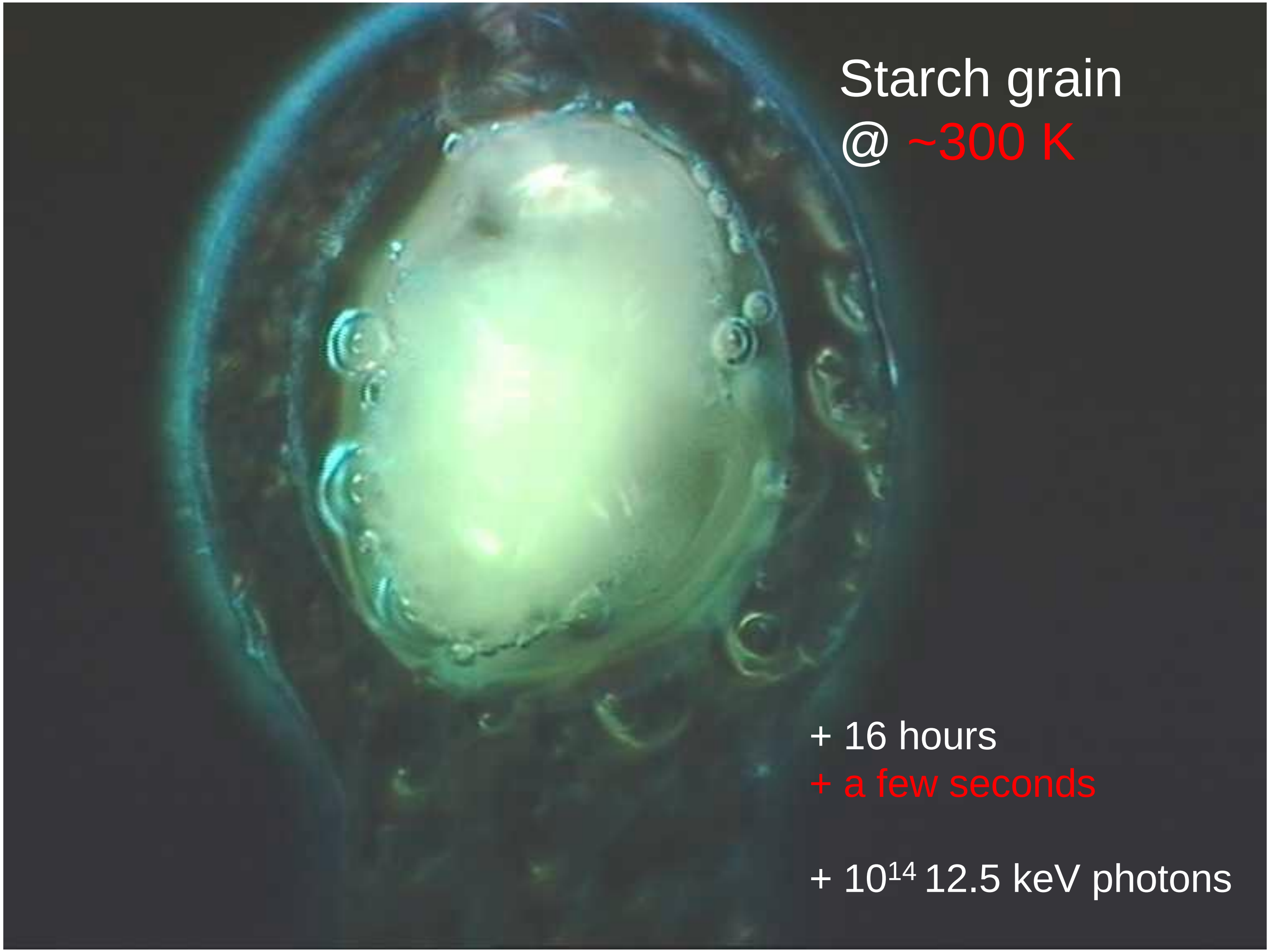
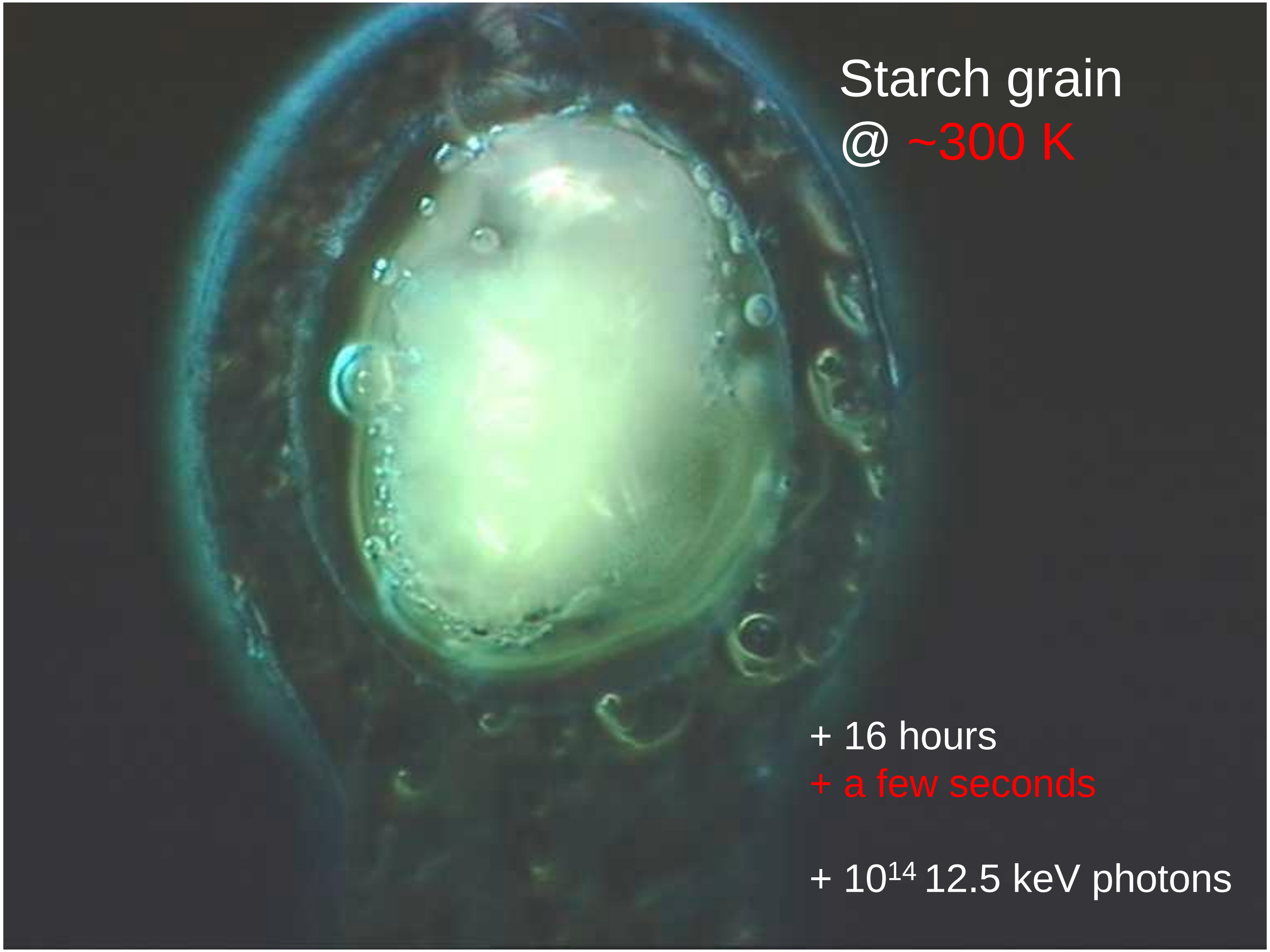
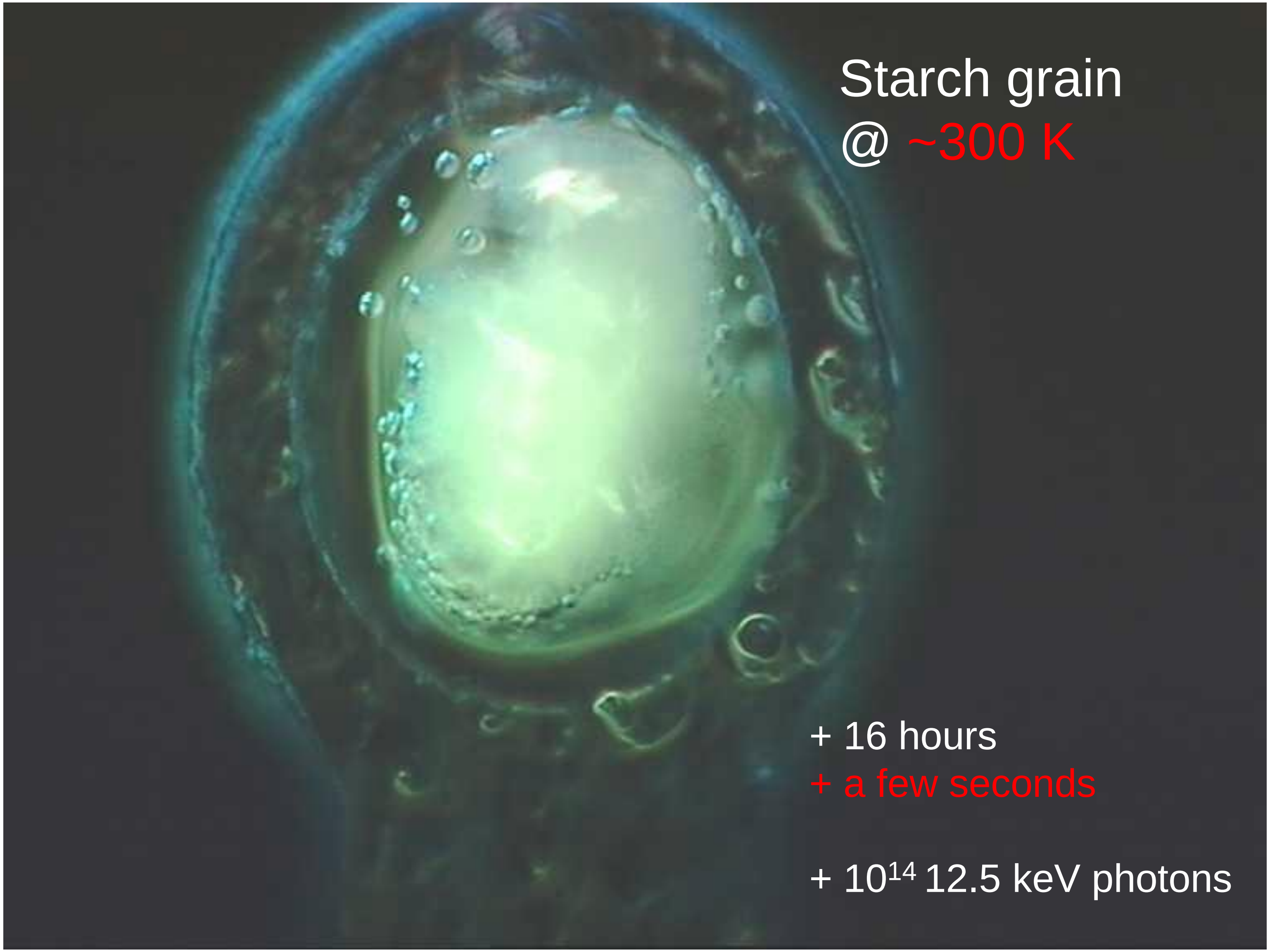


RADIATION DAMAGE EXEMPLIFIED ON STARCH



RADIATION DAMAGE EXEMPLIFIED ON STARCH



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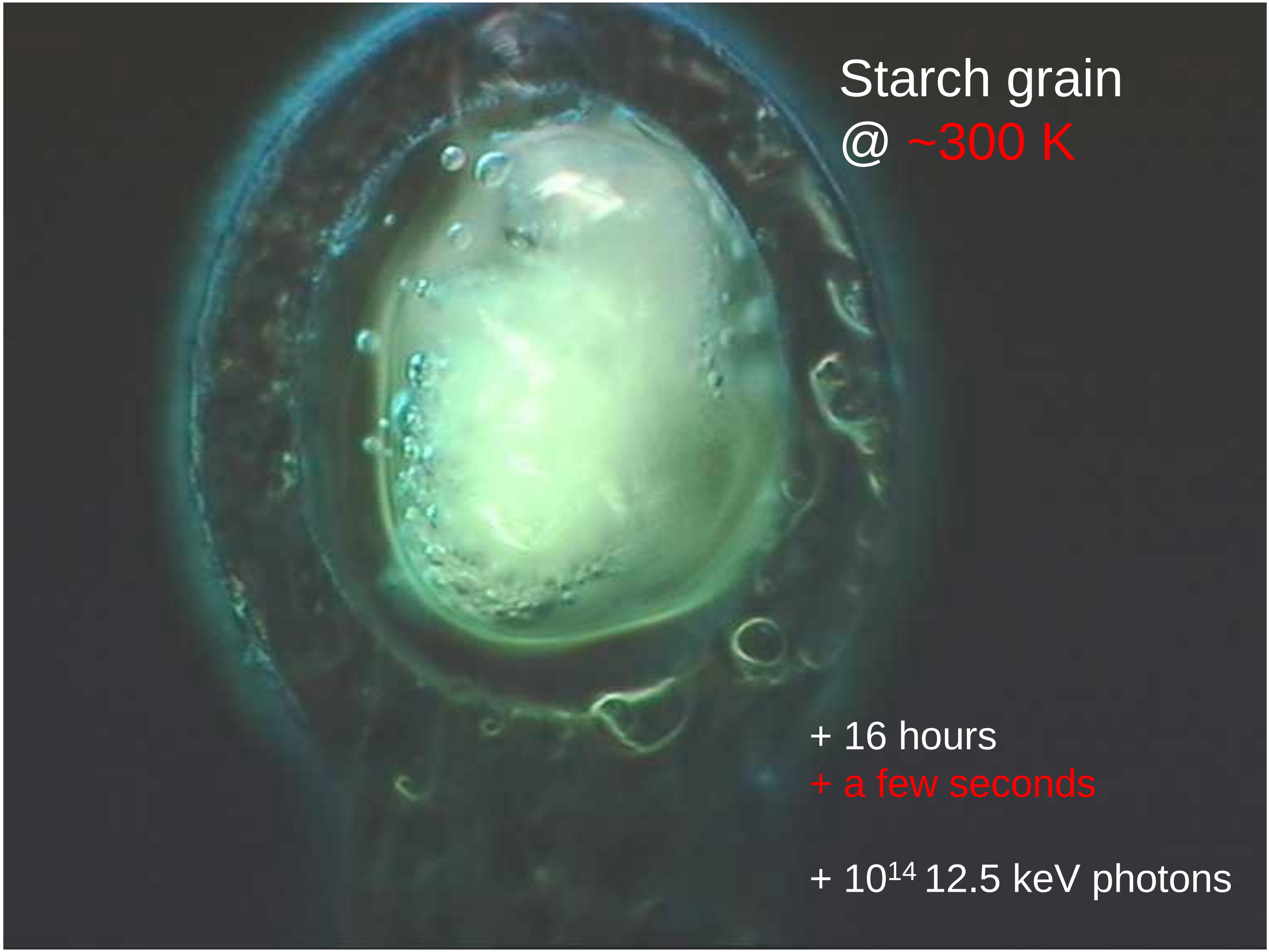
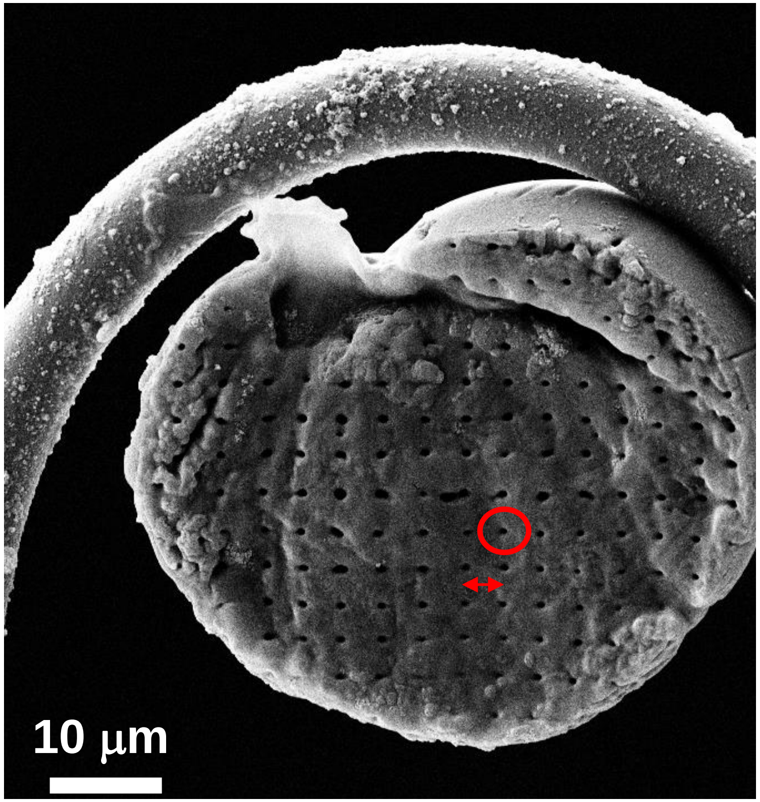


RADIATION DAMAGE EXEMPLIFIED ON STARCH



hydrated granule

scanned at 100 K

≈1*1 µm² beam, 4*4 µm² mesh

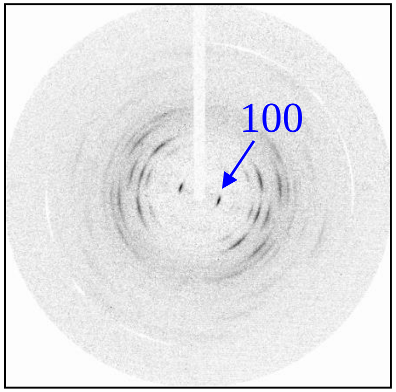
SEM at room temperature

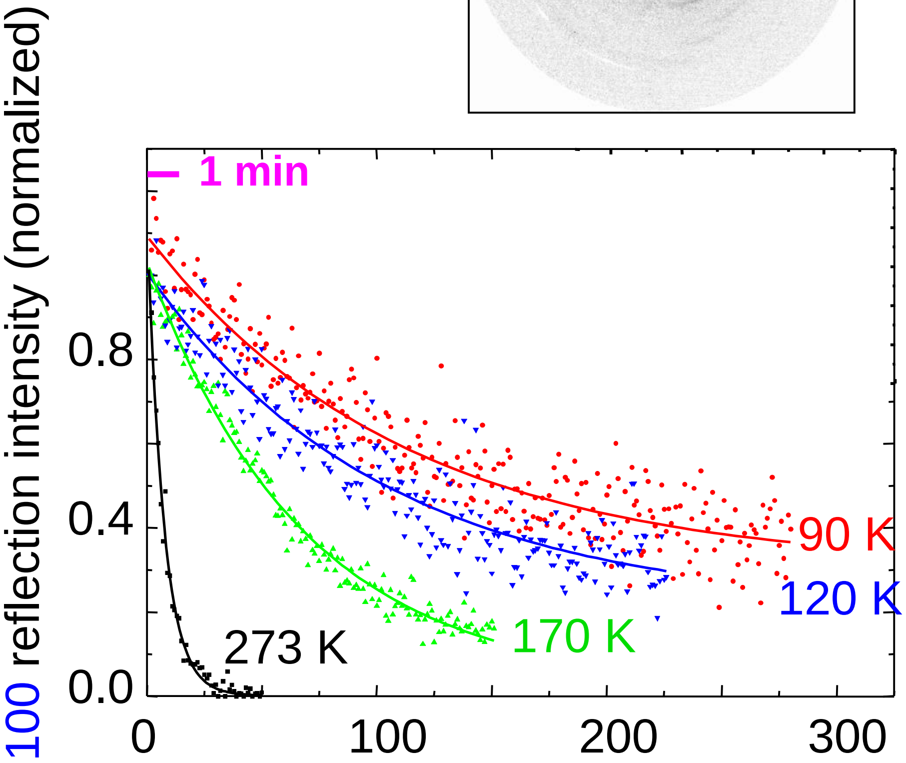
5 µm beam

0.5 sec exposure

+ 3.7 sec readout

~ 10^{11} ph/sec





[C. Riekel et al., **2010,** *JSR,* 7 (6) 743-750]

HOW HOT IS THE BEAM?

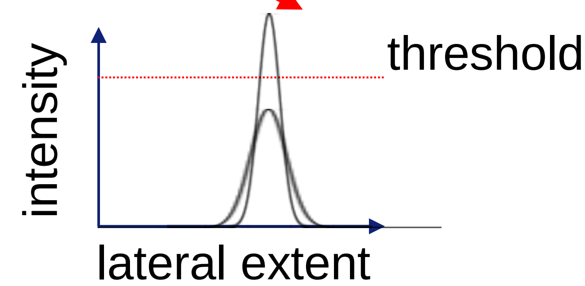
10^{12} photons / (s µm^2)

@ 10 keV
power density:
$(10^5 \cdot 10^{12})$ eV/ (s µm^2)
= 10^{17} eV/ (s µm^2)
= <u>16 mW / µm^2</u>

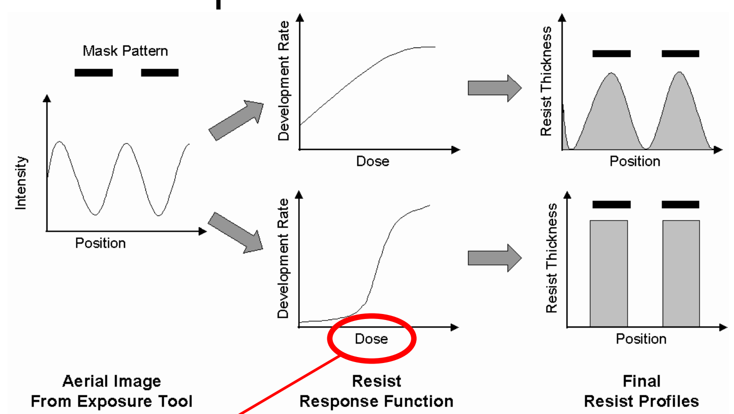
assuming 10 µm = full absorption
= 1.6 mW / µm^3

at typical atomic density:
$5 \cdot 10^{22}$ / cm^3 = $5 \cdot 10^{16}$ / µm^3

that is <u>0.5 eV/s per atom</u>!



known in photo and e-beam resists



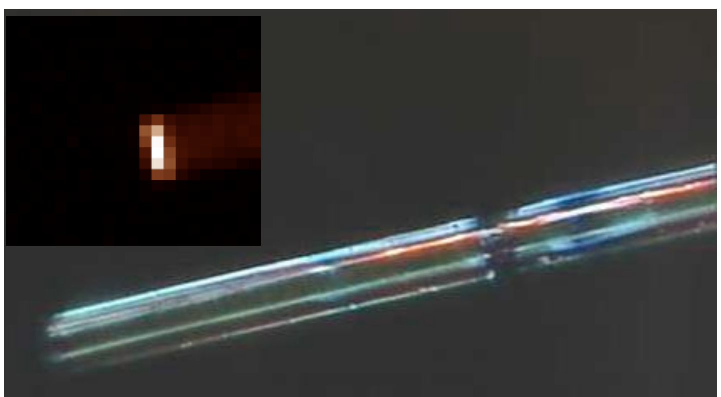
[Henderson Research Group GIT]

however:
indications that also doserate plays a role!

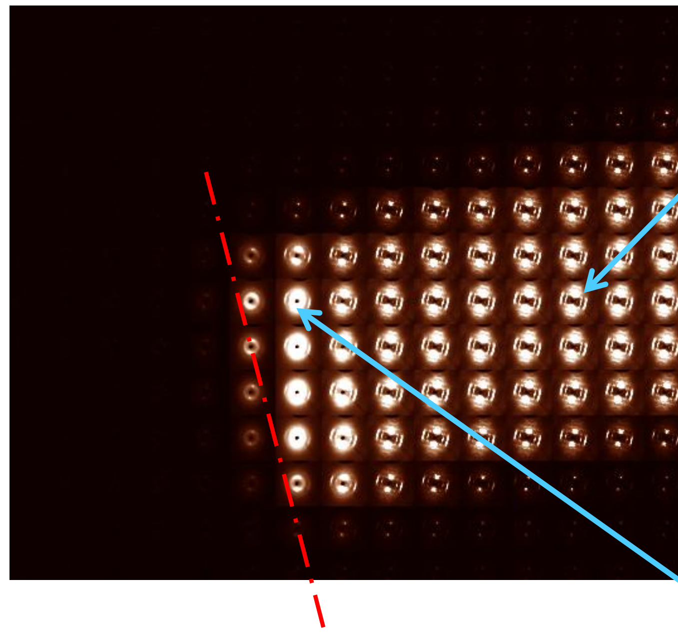
Repeated line scans across fiber

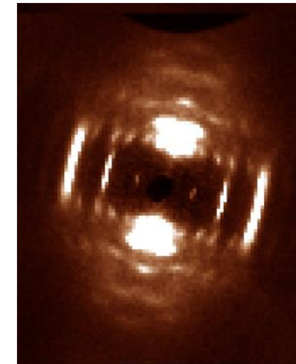
beam: ~2 µm
flux: 7 x 10^{11} ph/sec
Exptime/scanpoint: 2 sec

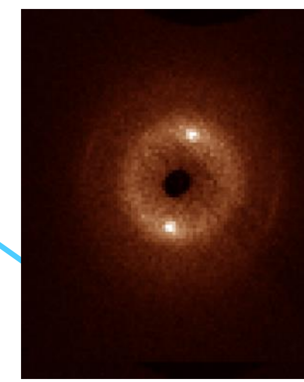
Transition to amorphous state induced by damage caused by the beam


Optical image of kevlar fiber cut

cumulative X-ray dose ~10^{14} ph/µm²


scan line


intact


damaged

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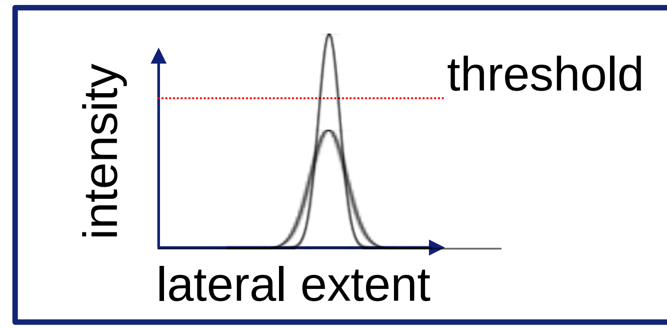
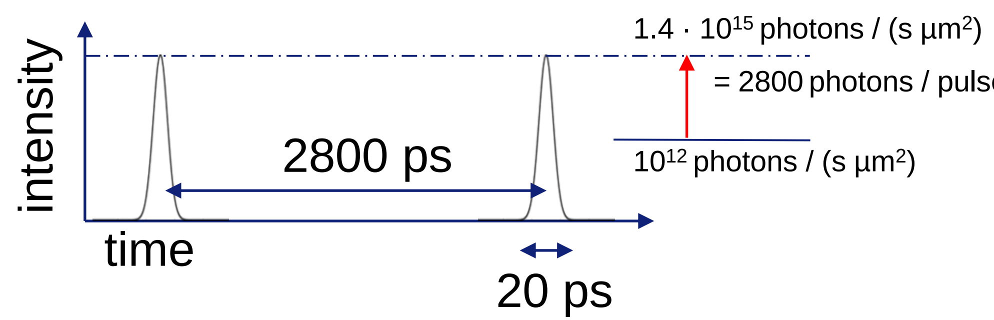
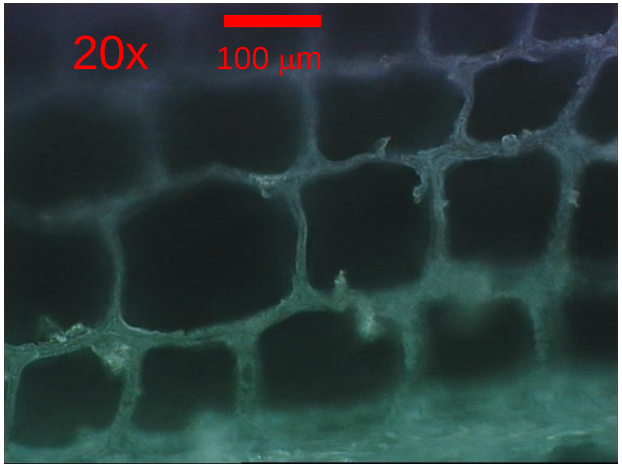


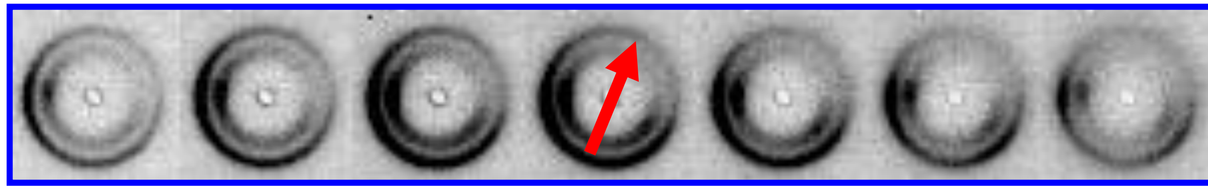
time structure of synchrotron radiation:
– 1000 bunches of 20 ps in 2.8 µs

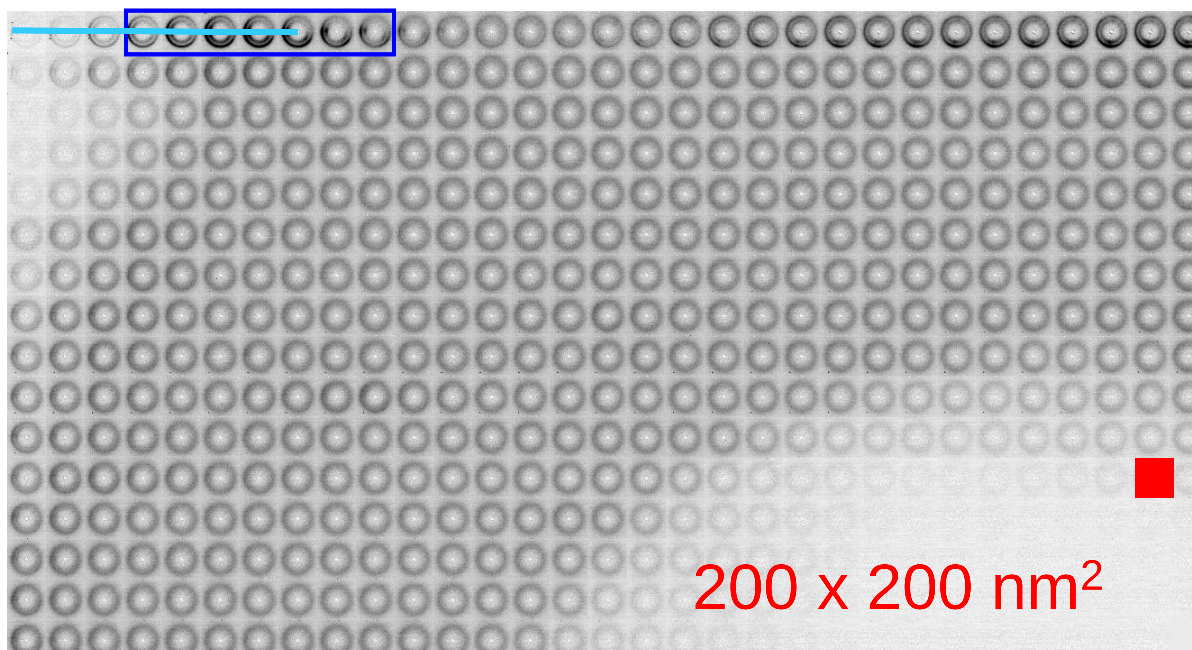


factor > 1000 in peak intensity

Damage propagation in spruce wood cell walls





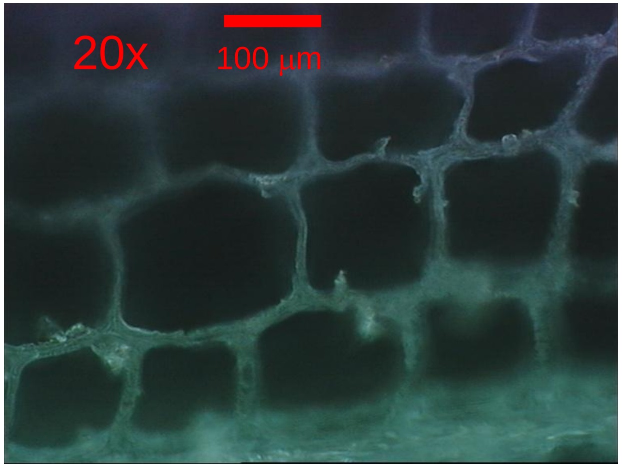


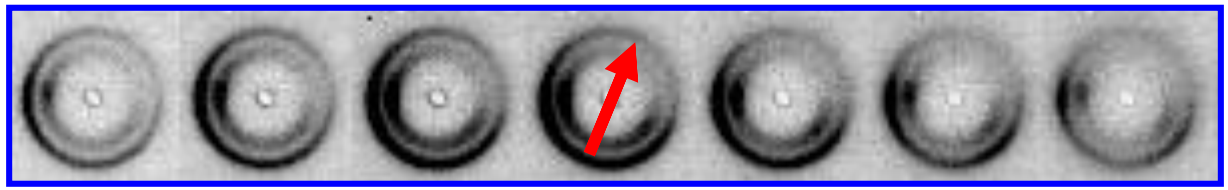
Cellulose WAXS patterns

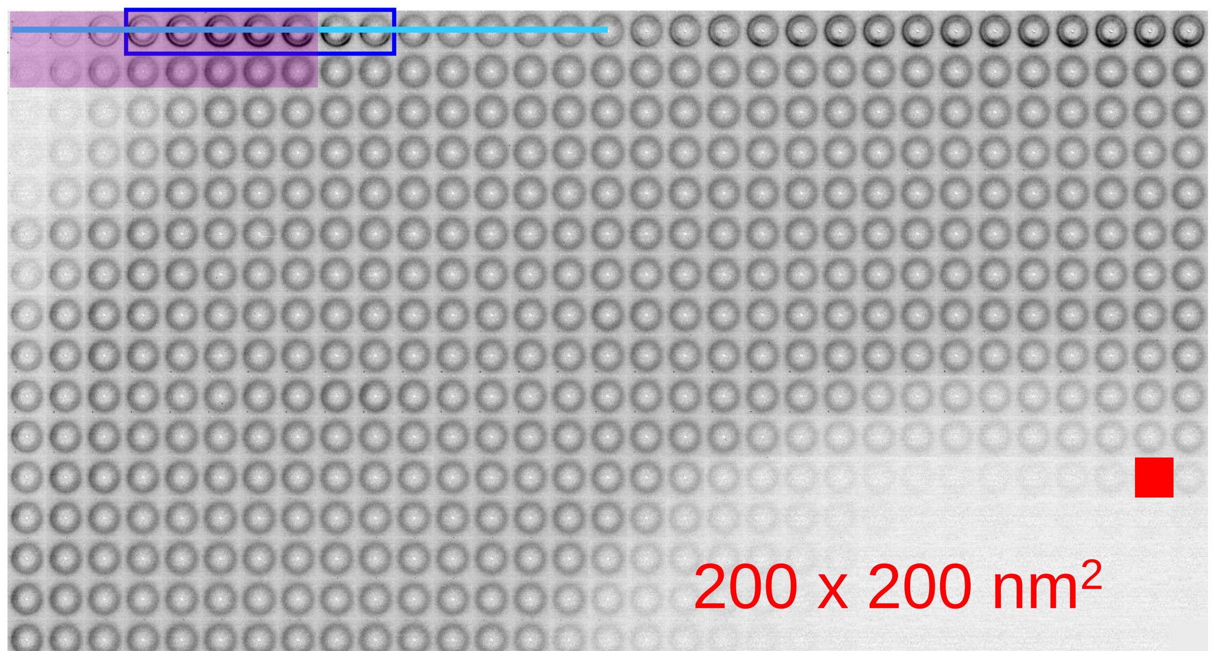
detector:	Frelon 4M
energy:	12.5 keV
exposure:	2 sec
grid:	31 x 16

Collaboration:
M. Mueller, Kiel University/GKSS Germany

Damage propagation in spruce wood cell walls







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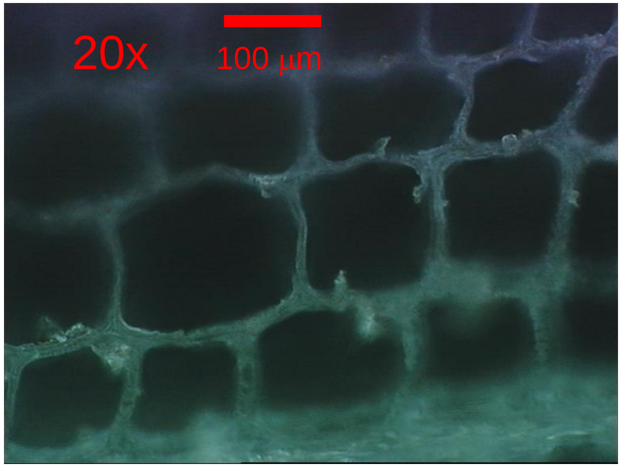

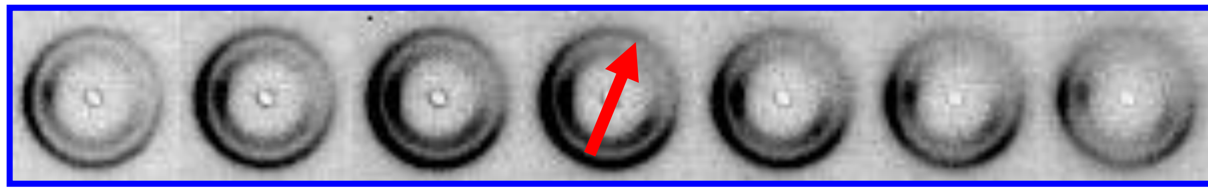

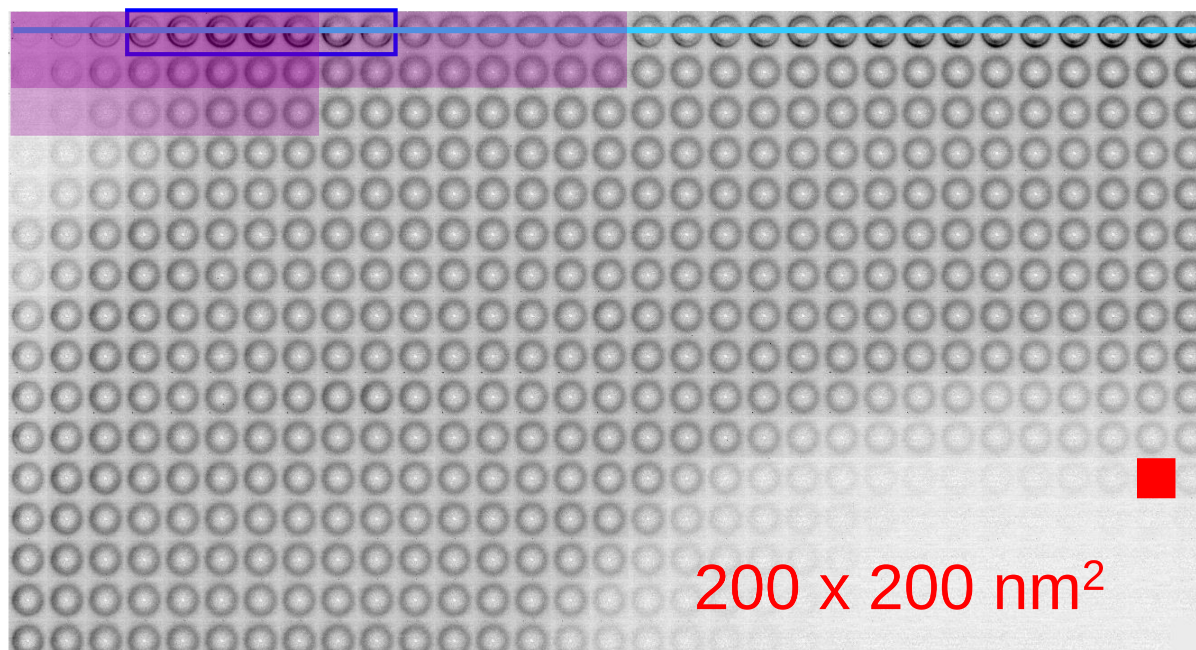

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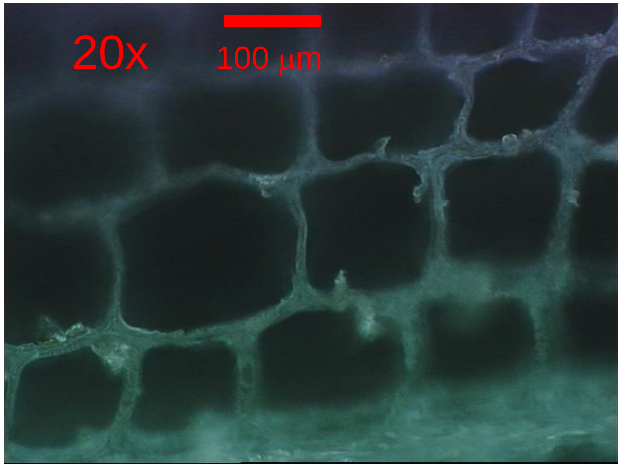

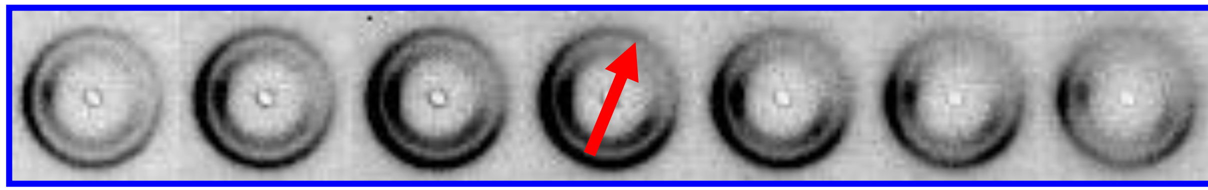

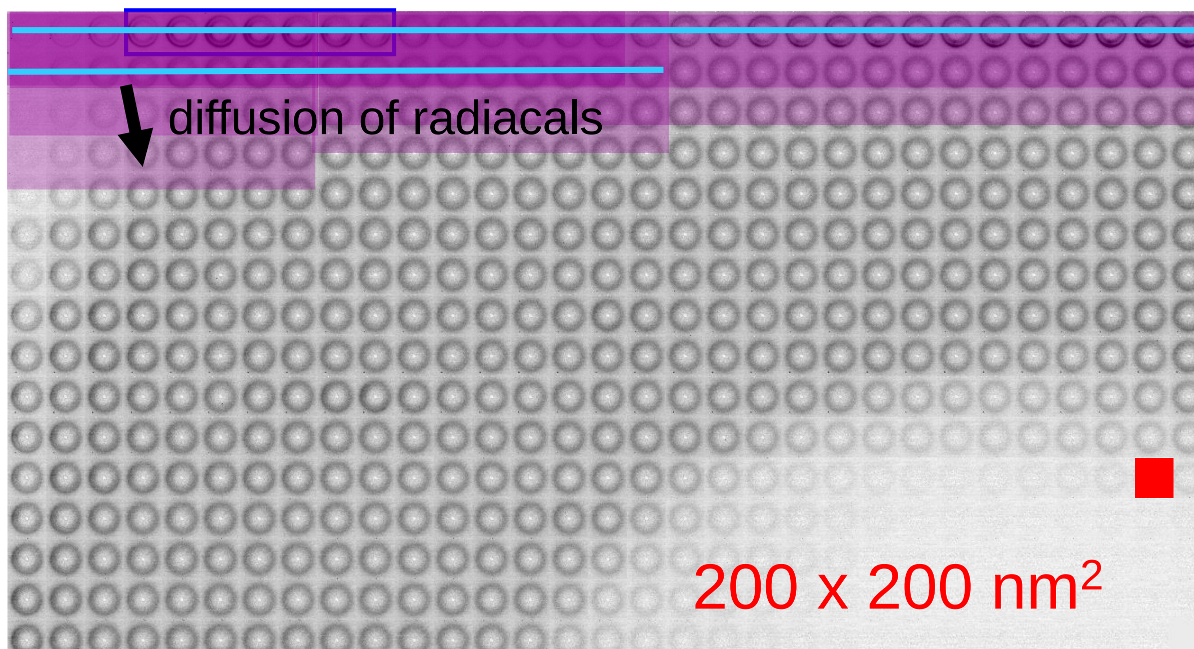

Cellulose WAXS patterns

detector:	Frelon 4M
energy:	12.5 keV
exposure:	2 sec
grid:	31 x 16

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ESRF EBS and similar X-ray sources, upgrades:

- Source x30 Gain in brilliance, undulator x10
- optimization of prefocussing x5
- pink beam x30 (full undulator harmonic + CRL monochromator)

>> conservative estimate of overall potential gain in flux:

45000

conservative estimate of overall potential gain in flux:

45000

1) management and investigation of radiation damage
future micro-/nano-beamlines will have many traits of today's nano-second time resolved end-stations

beam choppers, complex positioning devices
timing electronics, detectors adapted to count rates

…

conservative estimate of overall potential gain in flux:

45000

2) Experimental design has to improve:
today poor compared to highly automated MX beamlines
one key issue:
time lag of data acquisiton sequence:
i.e. reaction time of the system for automatic decision making and optimized experimental design

Fast does not necessarily mean quick !
example: on-board computation capacity of detectors would help

...

conservative estimate of overall potential gain in flux:

45000

3) - N)

sample preparation, data management, anticipating software needs

...

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