

A talk on X-ray imaging of small bubbles in water...



Katasushika Hokusai (1760-1849)



small bubbles actually

cavitation bubble = (quasi-) spherical gas phase in liquid
when hydrostatic pressure falls below vapor pressure

far from equilibrium -

created by laser pulse (optical breakdown)
of strong ultrasound field

Tim Salditt

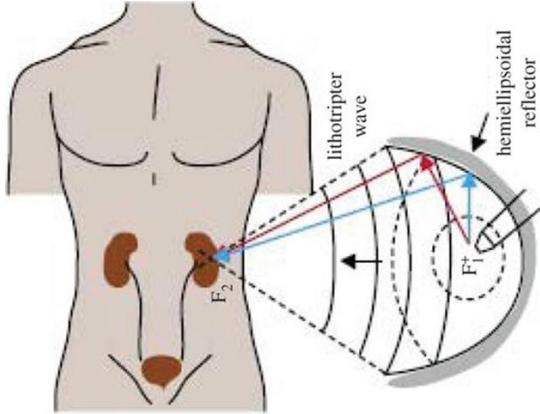
Institut für Röntgenphysik, Universität Göttingen

MID workshop, XFEL/DESY User Meeting, 24.1.2022



learn about cavitation

relevant for engineering, life sciences & medicine



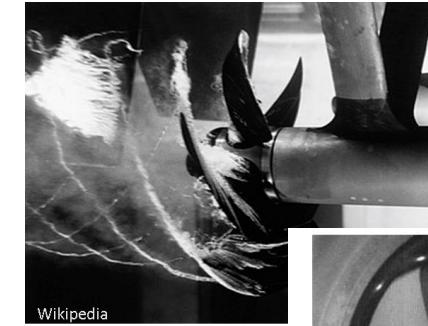
Different biomedical applications:

- Surgery / laser-tissue interaction
- drug delivery
- contrast enhancement in radiology

Lithotripter / phacoemulsification / ultrasound fat cavitation

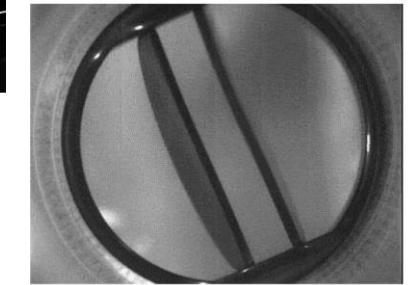
Cavitation limits performance of :

- ship propellers, blades
- pumps, motors



Cavitation enables:

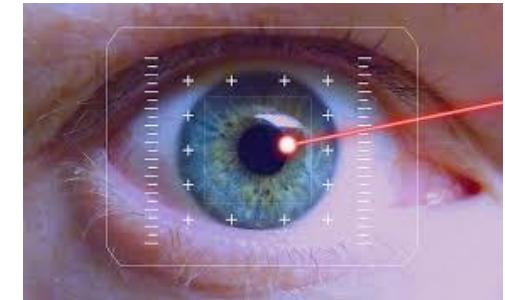
- ultrasonic cleaning
- Laser ablation for drilling & hardening
- nano-particle production



artificial heart valves

Surgery :

- Ophthalmology
(e.g. lens membrane destruction following cataract surgery)
- Urology and gastroenterology
(e.g., kidney and gall stone ablation and fragmentation)
- Cardiology and vascular surgery
(e.g., laser ablation, removal of fibro-fatty, calcified arterial plaque)

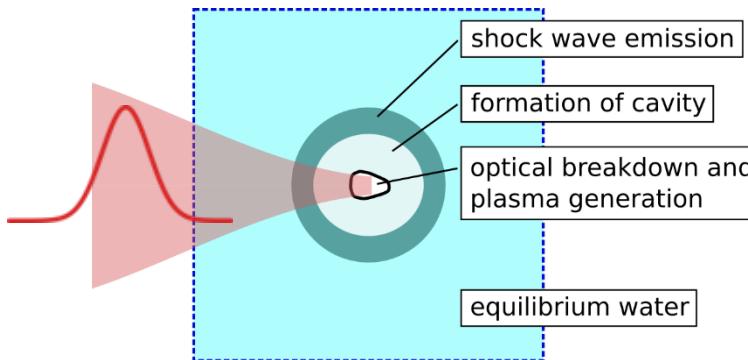


Laser-induced cavitation!

and learn about water under extreme conditions

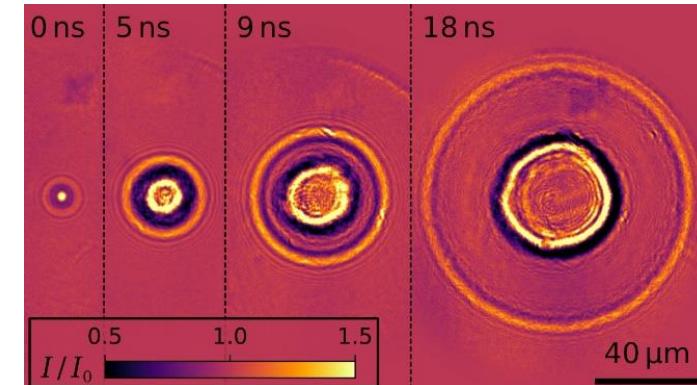


controlled dynamics!



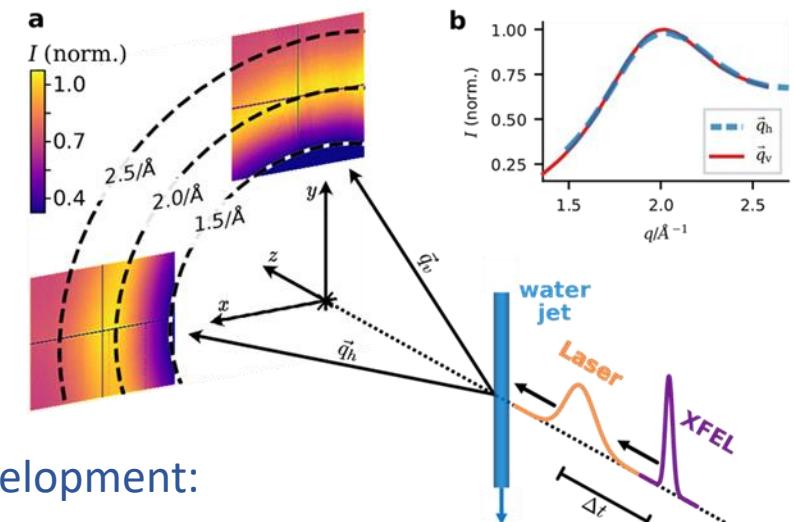
imaging & diffraction!

Single Pulse Full-field holographic X-ray imaging



&

high spatial & temporal resolution !



method development:

Osterhoff et al. *Nanosecond Timing and Synchronisation for Holographic Pump-Probe Studies at XFELs*. Journal of Synchrotron Radiation 2021

Hoeppke et al., in preparation

recent results obtained at MID:

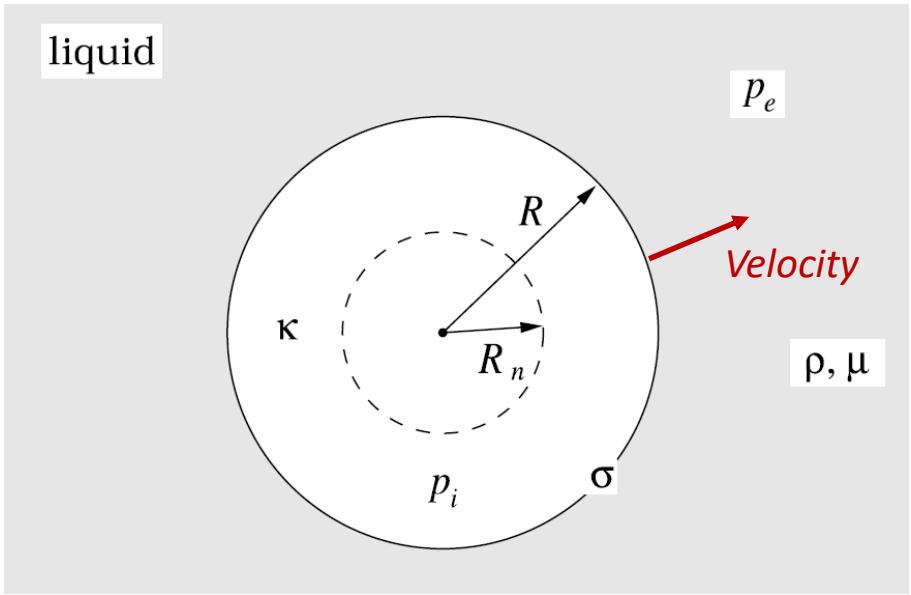
- shockwaves / equation of state @extreme conditions (p2207 & p2545)
- laser-induced breakdown (p2807)
- cavitation bubble collapse (p2807)

Vassholz et al., *Pump-probe X-ray holographic imaging of laser-induced cavitation bubbles with femto-second FEL pulses*. Nature Communications 2021

Vassholz et al., unpublished MS

Hagemann et al., *Single-pulse phase-contrast imaging at free-electron lasers in the hard X-ray regime*. Journal of Synchrotron Radiation 2021.

Physics of bubbles and bubble oscillations



R: radius

R_n : radius at equilibrium

p_i : internal pressure

p_e : external pressure

σ : surface tension

κ : adiabatic exponent

ρ : density

μ : (dynamics) viscosity

$$\rho R \ddot{R} + \frac{3}{2} \dot{R}^2 = p_i - p_e$$

Rayleigh equation (1917)

$$\rho R \ddot{R} + \frac{3}{2} \dot{R}^2 = p_{gn} \left(\frac{R_n}{R} \right)^{3\kappa} + p_v - p_{stat} - \frac{2\sigma}{R} - \frac{4\mu}{R} \dot{R} - p(t)$$

$$p - p_0 = B \left(\left(\frac{\rho}{\rho_0} \right)^m - 1 \right) \quad \text{Tait eq.}$$

$$B = K/m \quad m = c_p/c_v \simeq 7$$

Interesting and extreme
physical states and phenomena:

- bubble collapse: T ? p ? R_{min} ?
- sonoluminescence
- cavitation in artificial heart valves
- nature of the interfaces
- equation of state of H_2O : beyond Tait

*...a lot is known, but all based on bubble trajectory,
how about interior? Always a clear phase separation?*

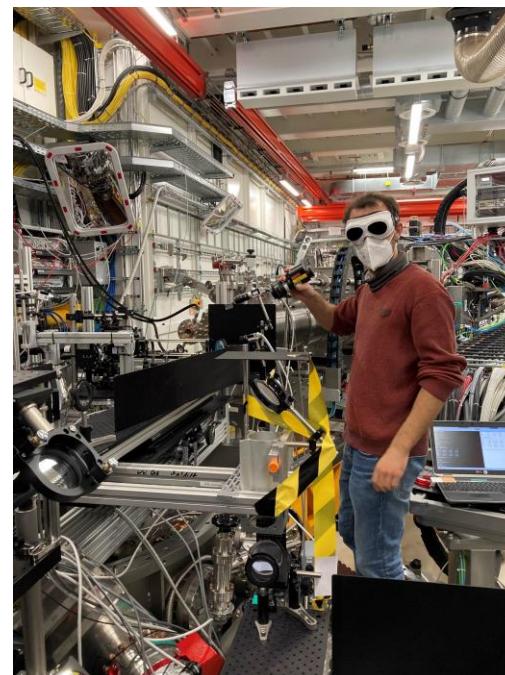
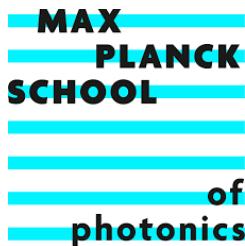
Small bubble collaboration & Acknowledgements

Hannes Hoeppé, Markus Osterhoff, Malte Vassholz, Juan Rosello, Atiyeh Aghelmaleki, Robert Mettin, Tim Salditt
Universität Göttingen, III-Phys. Inst.-Biophysik & Institut für Röntgenphysik

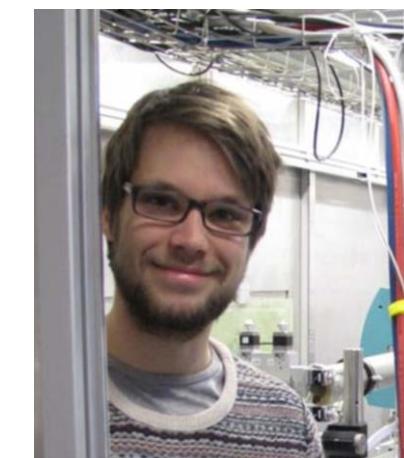
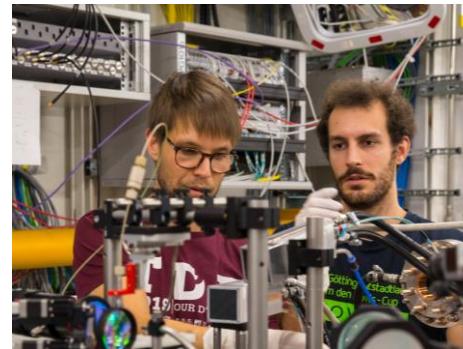
Johannes Hagemann, Frank Seiboth, Andreas Schropp, Christian G. Schroer, DESY Photon Science

Johannes Möller, Jörg Hallmann, Ulrike Boesenberg, Chan Kim, Markus Scholz, Alexey Zozulya, Wei Lu, Roman Shayduk, Robert Schaffer, Anders Madsen
MID / XFEL

UAC MID 06/2019 @14keV
Beamtime 10/ 2019 @17.8keV
(p2207 & p2545)
Beamtime 10/2021 @18keV
(p2807)



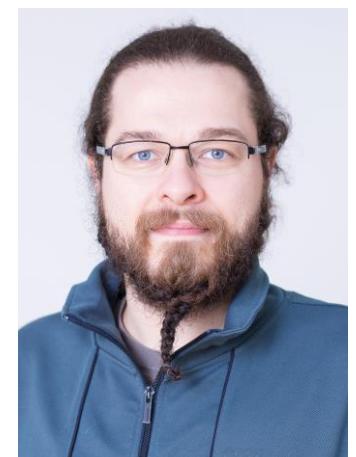
Hannes Paul Hoeppé



Malte Vassholz



Markus Osterhoff



Johannes Hagemann

Research questions

A) For laser-generated cavitation bubble:

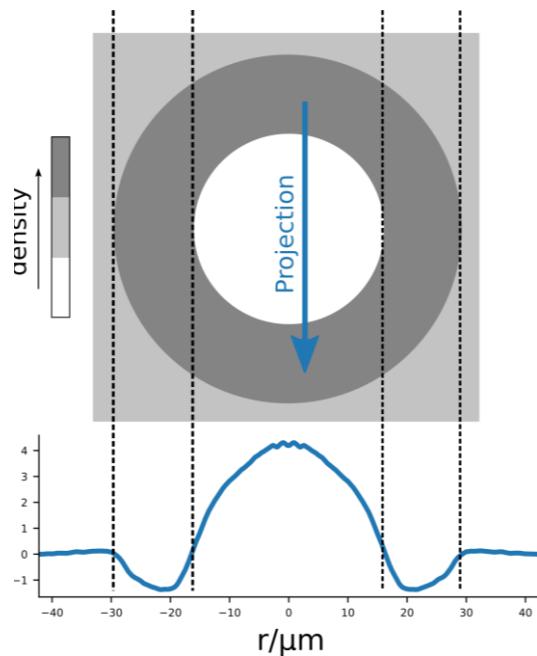
better understanding of **transition from plasma to cavitation bubble**

B) Shock wave effects can be useful or unwanted depending on applications

-> detailed knowledge of the shock emission processes and the **properties of the shock wave** required

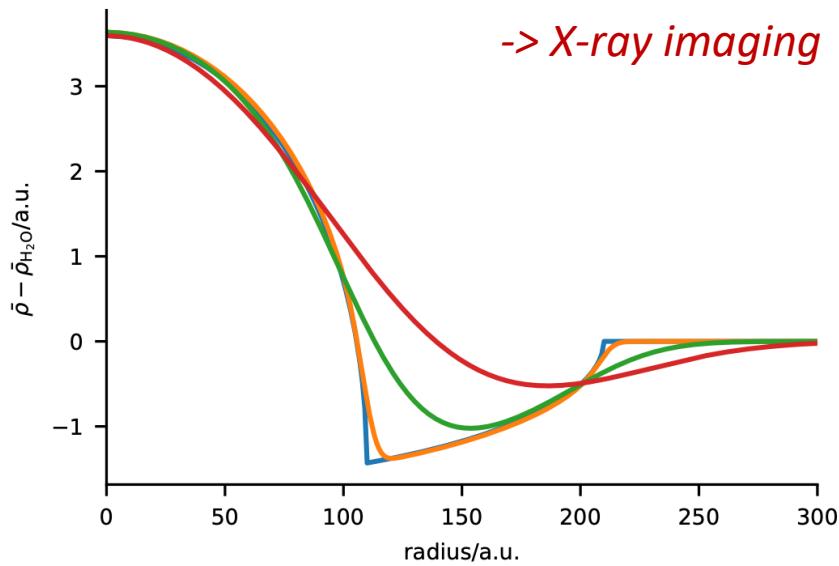
C) Fundamental physics: extreme conditions at **bubble collapse** (sonoluminescence, $T > 10^4$ K)

*Plasma generated by
Optical breakdown
Multi-photon absorption
 $I > 10^{12}$ W/cm²*



*Challenge: Spatio-temporal resolution, contrast
refraction, scattering, opacity, resolution –*

-> X-ray imaging

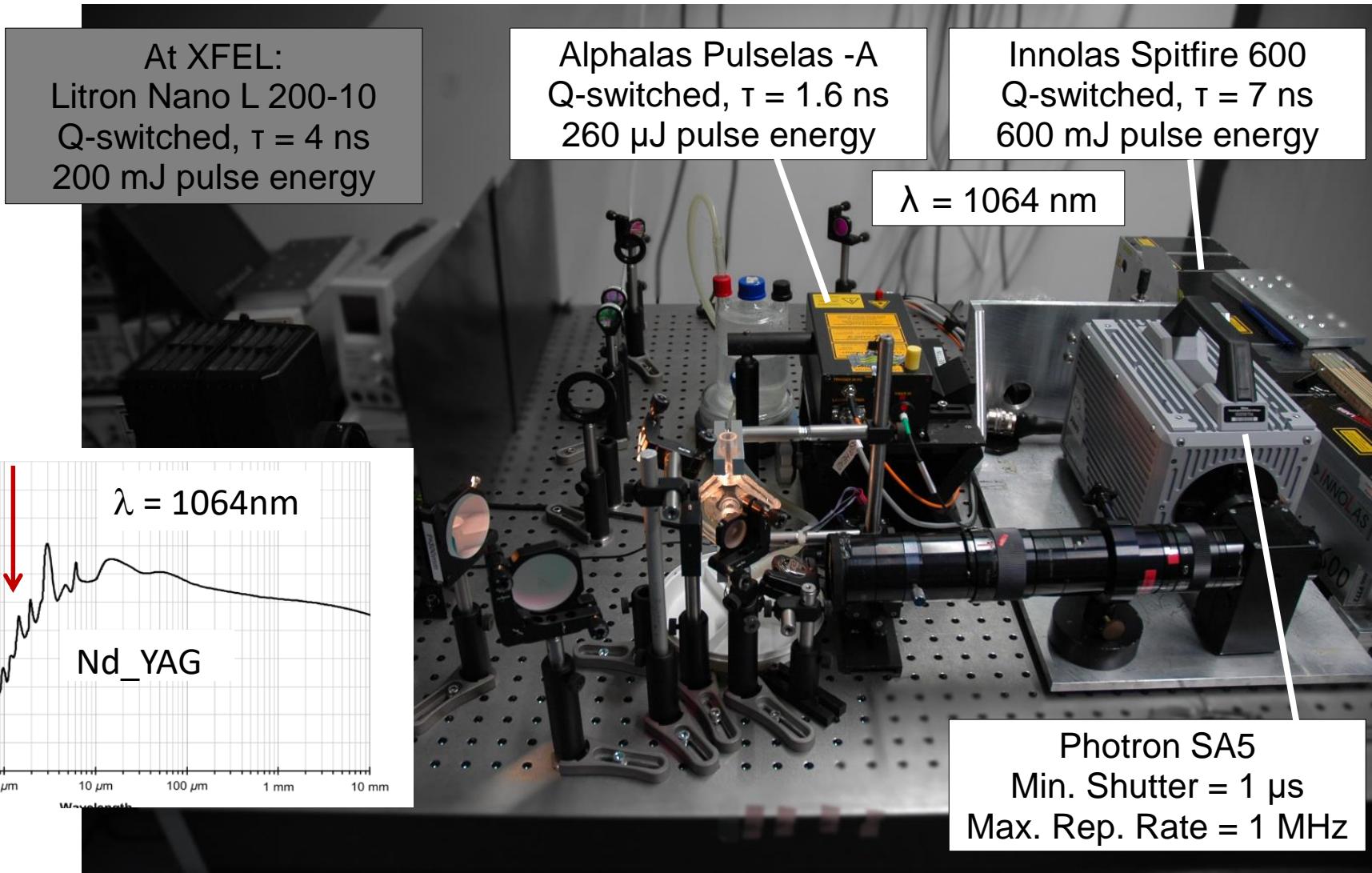
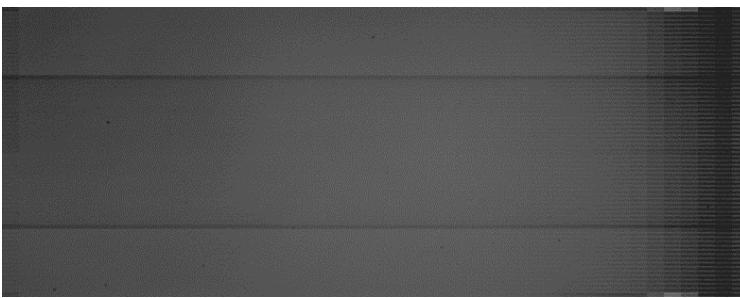


- bubble evolution
- density of the shockwave ?
- sharp interfaces gas/water ?

radial density profile !

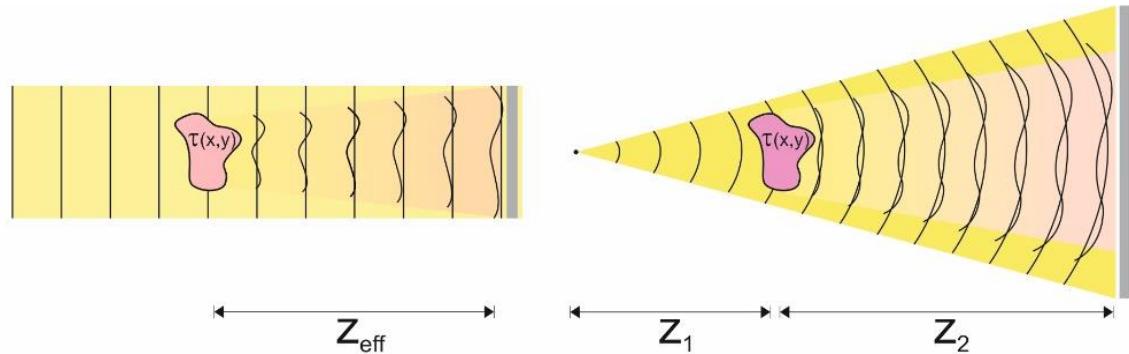
High speed optical imaging

Hannes Hoeppel,
Juan Rosello



Full-field X-ray imaging / (inline) holography

SR: Blurring of fringes & low signal in water

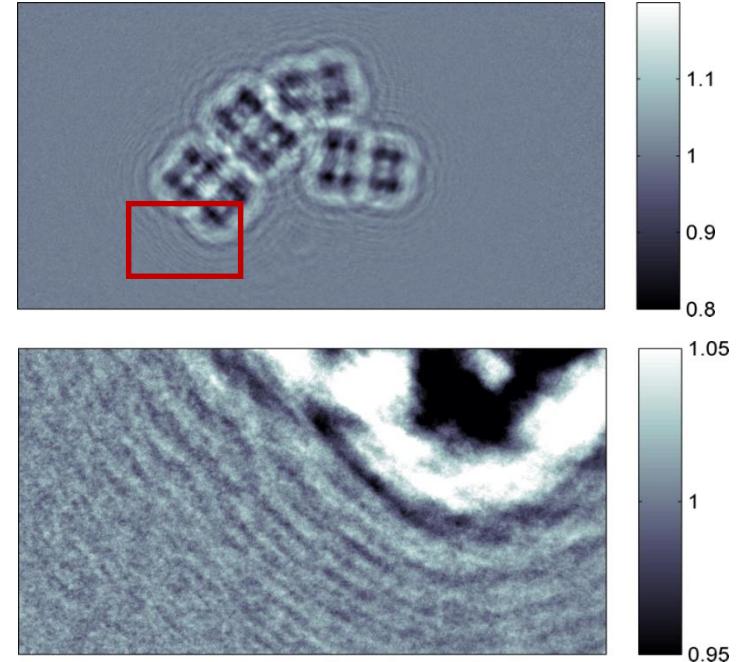
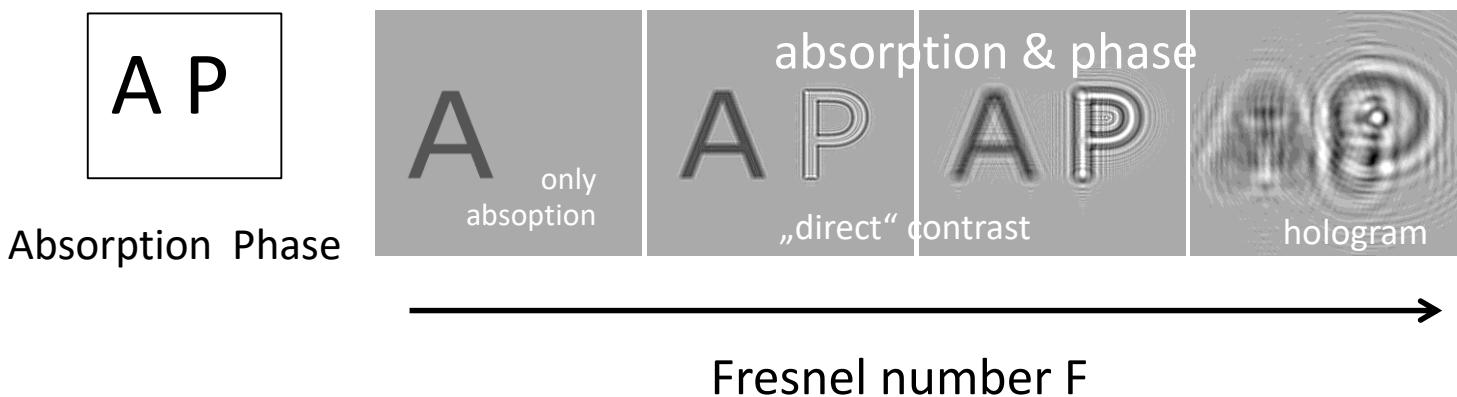


$$M = \frac{z_1 + z_2}{z_1}$$

$$z_{eff} = \frac{z_1 z_2}{z_1 + z_2}$$

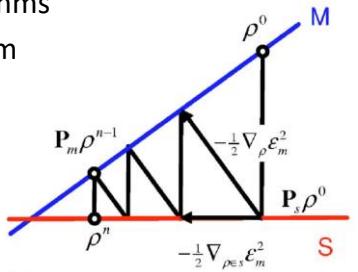
object with complex transmission function $\tau(x,y)$

$$\psi_z = FT^{-1} [\exp[iz\sqrt{k^2 - k_x^2 - k_y^2}] FT[\psi_0]]$$

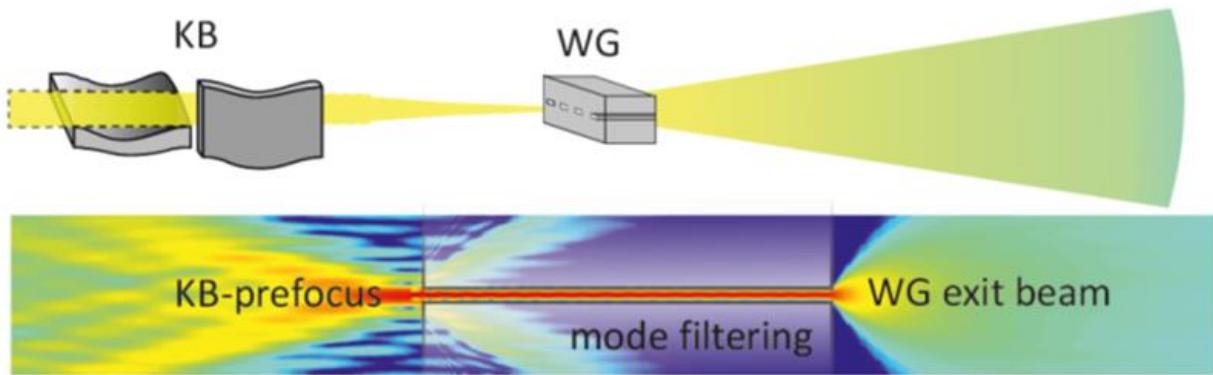


XFEL: no motional blurring, sharp holograms, outrun radiation damage!

iterative projection algorithms
to solve the phase problem



The empty beam problem in holography



Simultaneous probe and object reconstruction for the near-field
A.L. Robisch, K. Kröger, A. Rack, T.Salditt, N.J.Phys. 2015

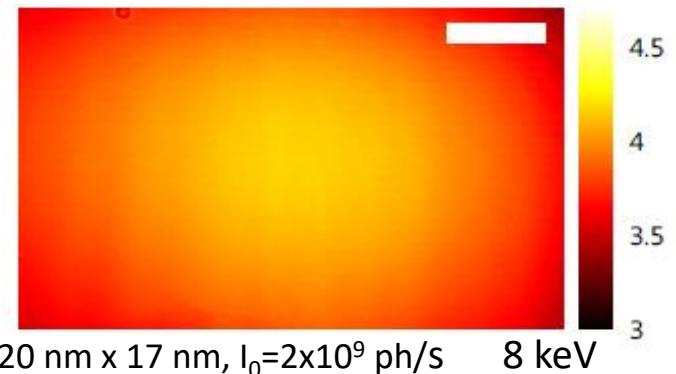
Reconstruction of wave front and object for inline holography
from a set of detection planes
J.Hagemann, A.L.Robisch et al., Optics express 2014

Validity of the empty-beam correction in near-field imaging
C. Homann, T.Hohage, J. Hagemann, A.L. Robisch, T.Salditt
Physical Review A 2014

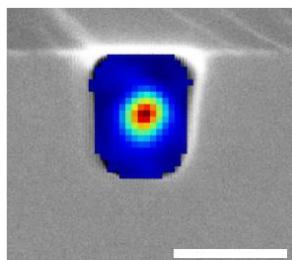
Divide and update: towards single-shot object and probe retrieval for near-field holography
J. Hagemann, and T. Salditt Opt.Expr. 2018

Holographic imaging with a hard x-ray nanoprobe: Ptychographic vs. conventional phase retrieval
A.-L. Robisch et al., J. Wallentin, A. Pacureanu, P. Cloetens, and T. Salditt Opt.Lett. 2016

waveguide farfield

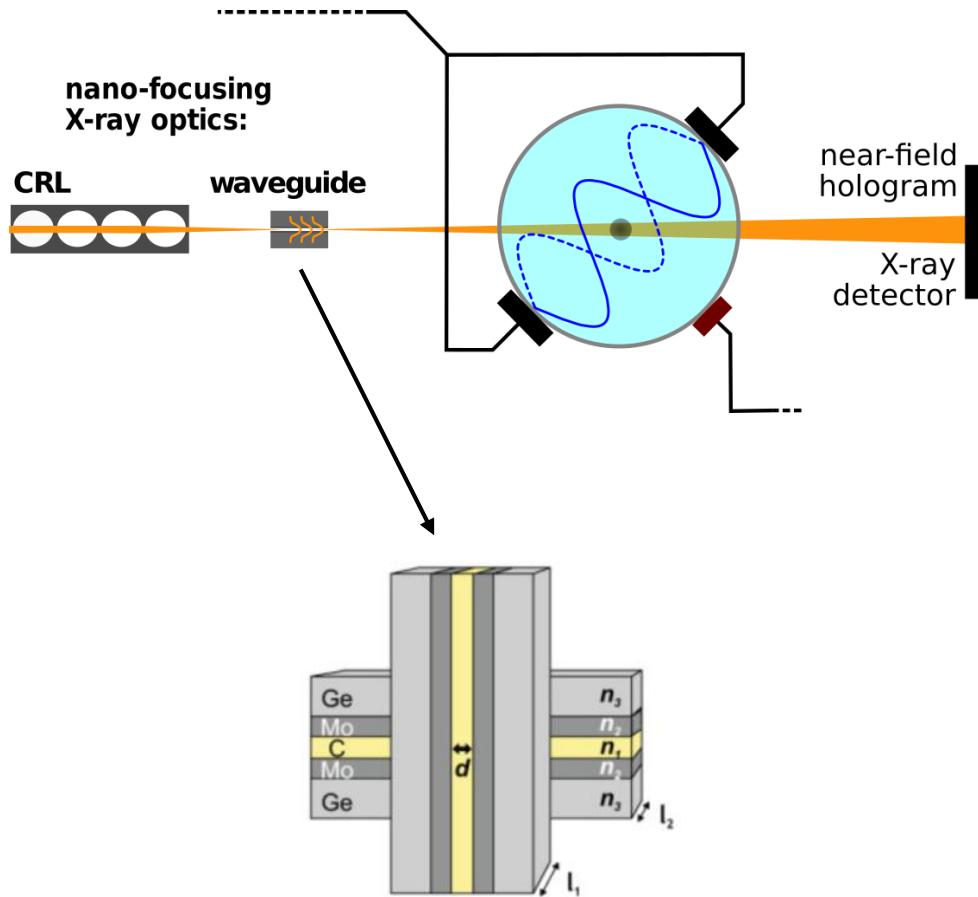


waveguide optics
to clean the probe !



in 10 Hz MID beam (after alignment) !

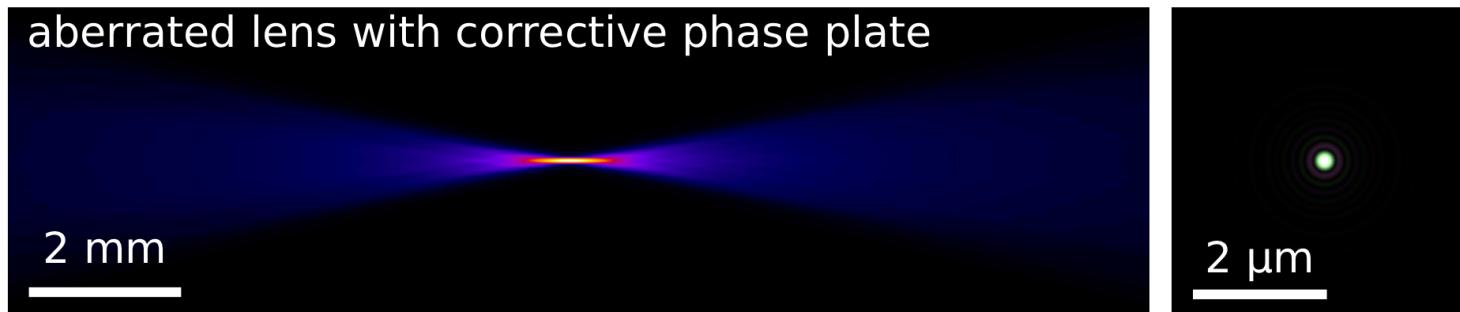
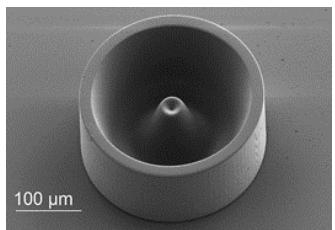
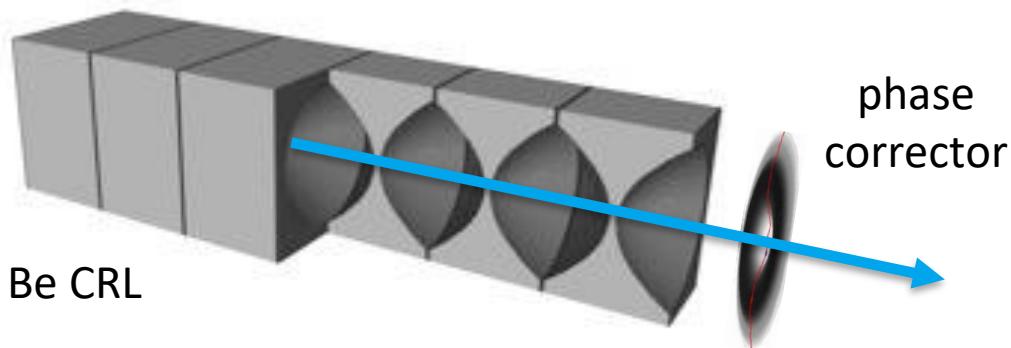
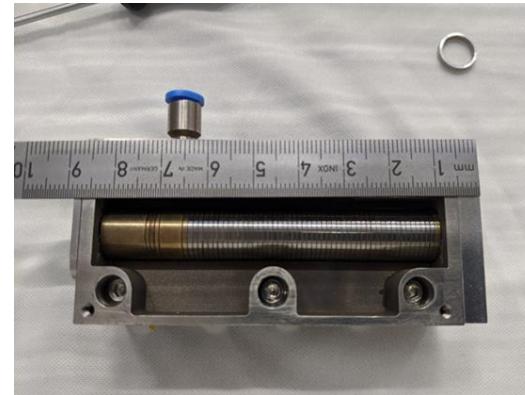
Waveguides at XFEL: first exposure



After alignment procedures

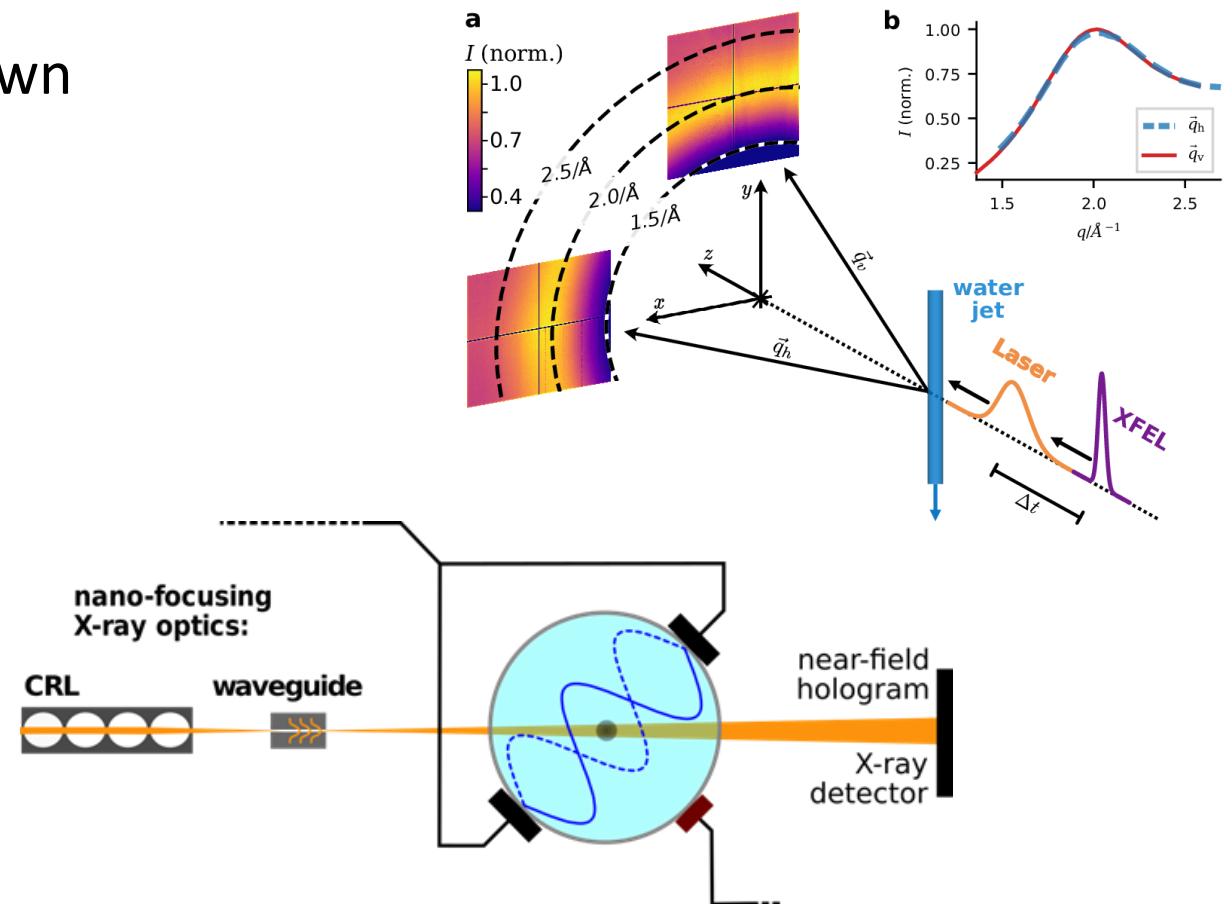
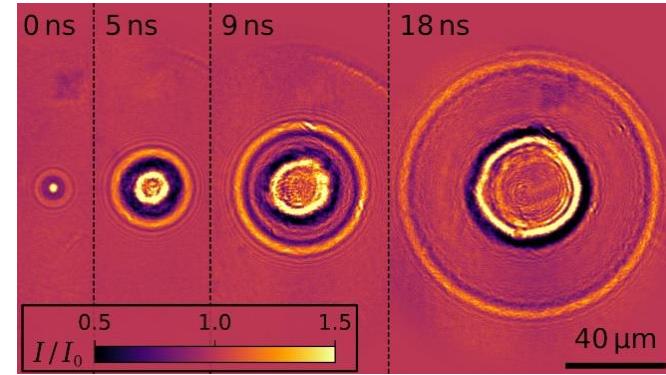
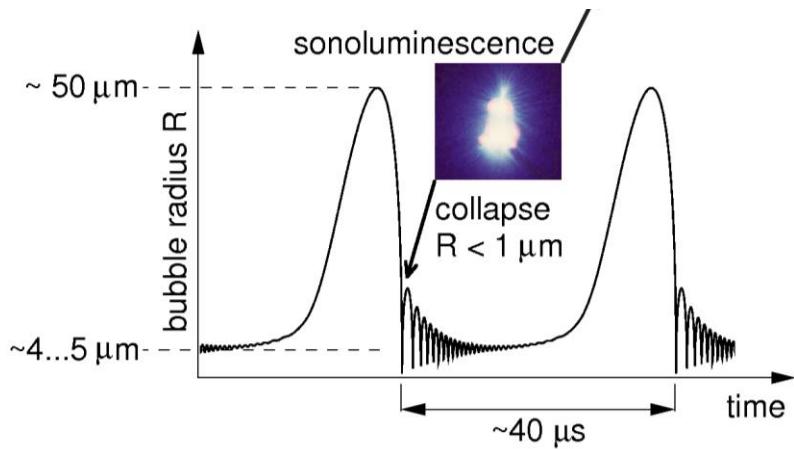
CRL (Be) focusing to ~80nm

- stack of 50 Be lenses, 300 μm aperture
- $f = 297 \text{ mm} @ 14 \text{ keV}$
- spot size $\sim 80 \text{ nm}$
- printed polymer phase plate
to correct aberrations

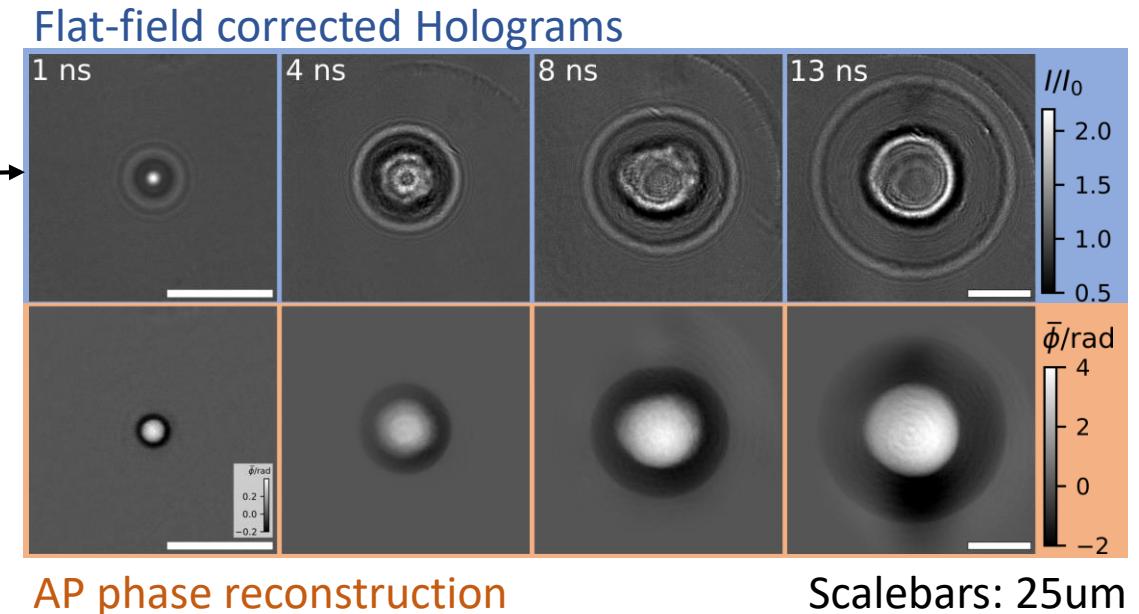
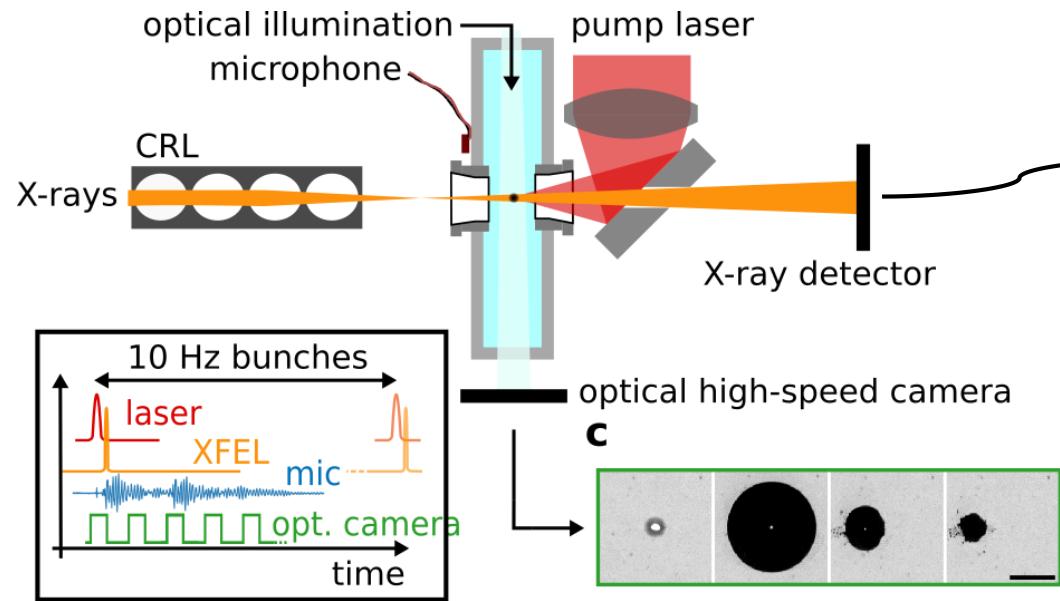


Outline

- 1) Recap 2019 experiments – *holography*
- 2) *diffraction from a shockwave*
- 3) Femtosecond laser-induced breakdown
- 4) Bubble collapse



1. X-ray FEL imaging of nanosecond laser-induced cavitation (2019)



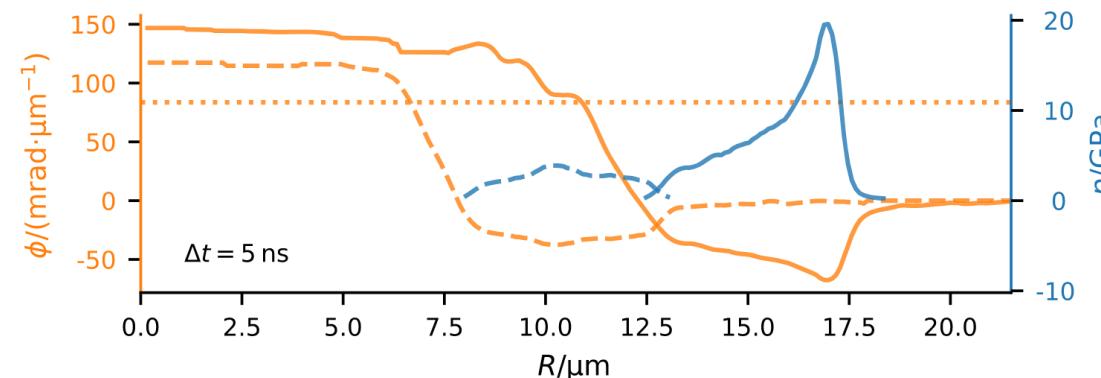
Mass density

$$\rho(R) = \rho_0 \left(1 - \frac{\phi(R)}{k \delta} \right)$$

Tait EOS

$$\frac{p(R) + B}{p_\infty + B} = \left(\frac{\rho(R)}{\rho_0} \right)^n$$

13



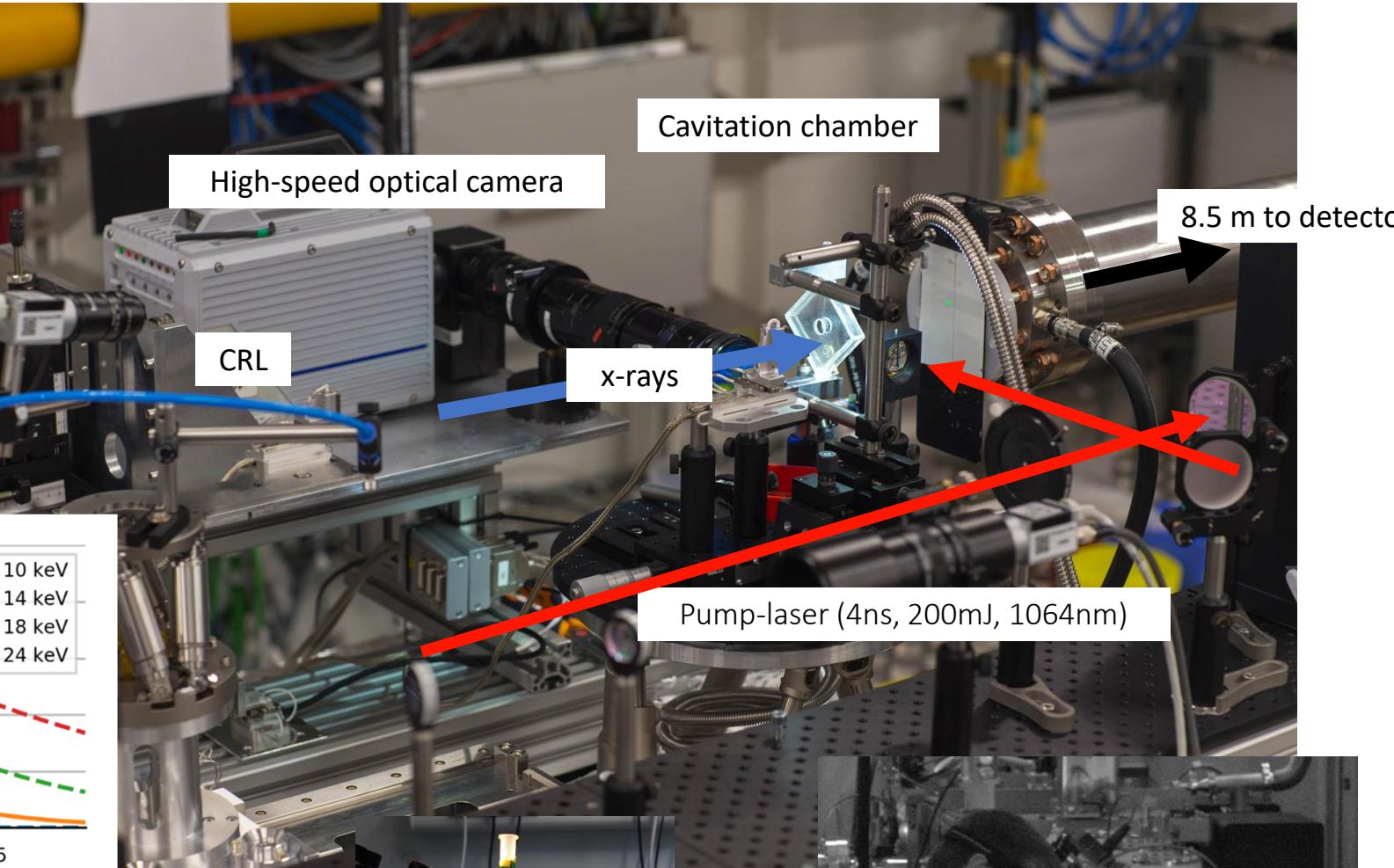
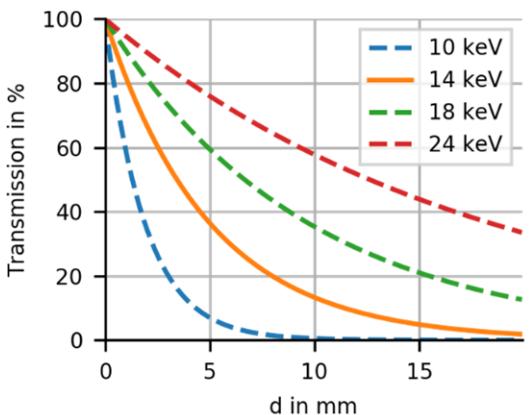
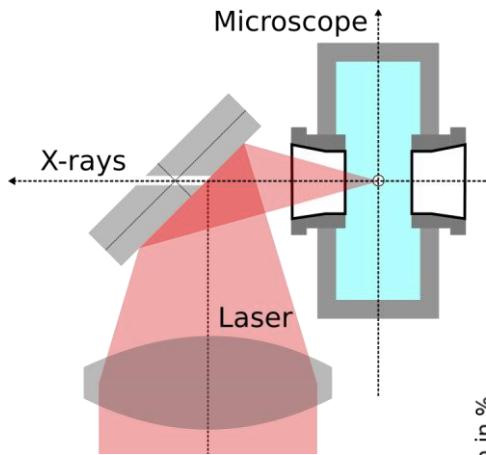
ARTICLE

<https://doi.org/10.1038/s41467-021-23664-1> OPEN

Pump-probe X-ray holographic imaging of laser-induced cavitation bubbles with femtosecond FEL pulses

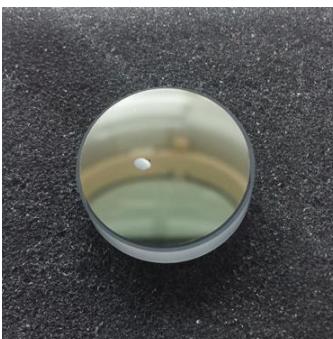
M. Vassholz¹, H. P. Hoeppen¹, J. Hagemann², J. M. Rosselló³, M. Osterhoff¹, R. Mettin³, T. Kurz³, A. Schropp², F. Seiboth², C. G. Schröer^{2,4}, M. Scholz⁵, J. Möller⁵, J. Hallmann⁵, U. Boesenbergs⁵, C. Kim⁵, A. Zozulya⁵, W. Lu⁵, R. Shayduk⁵, R. Schaffer⁵, A. Madsen⁵ & T. Salditt^{1,6*}

Cavitation cuvette x-ray compatible...

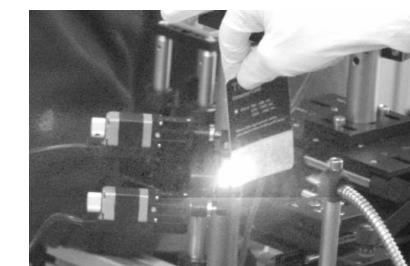
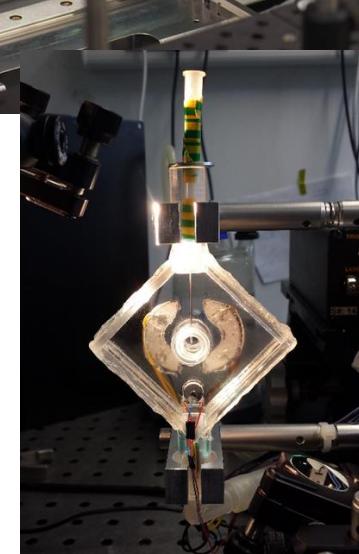


- compatible with XFEL defocused beam
- compatible with IR laser
- compatible with speed optical camera

-> anti-parallel XFEL and IR-beams

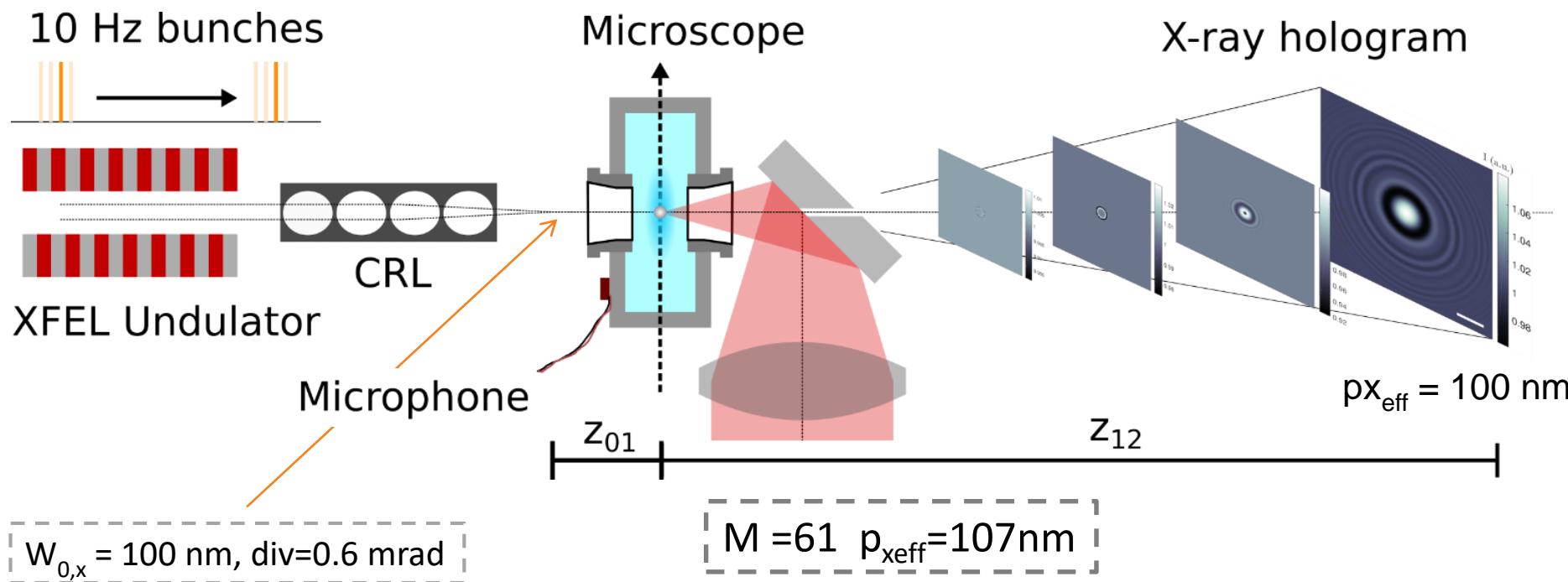
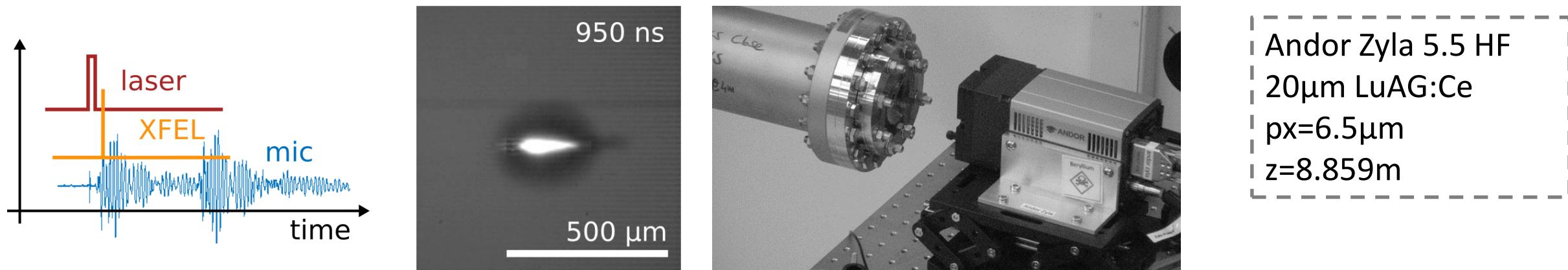


Mirror with drilled hole



IR- alignment

Settings at MID / exp. parameters



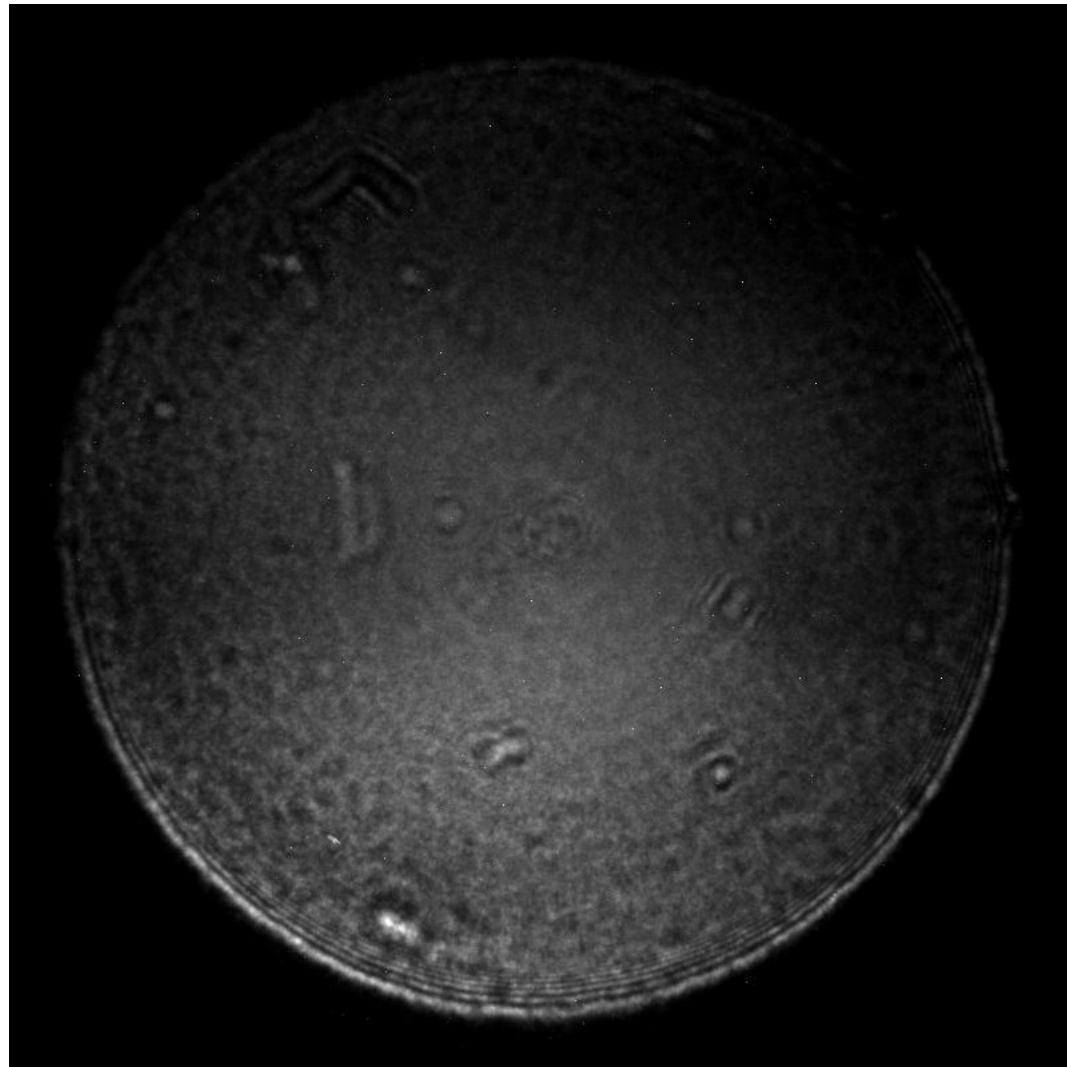
10 Hz
600 μJ / pulse
 $3 \cdot 10^{11} \text{ ph / pulse}$
 $\tau < 100 \text{ fs}$
 $E = 14 \text{ keV (UAC)}$
 $E = 18 \text{ keV (Oct.19)}$

And this is how it looks – *meet your probe !*

in terms of

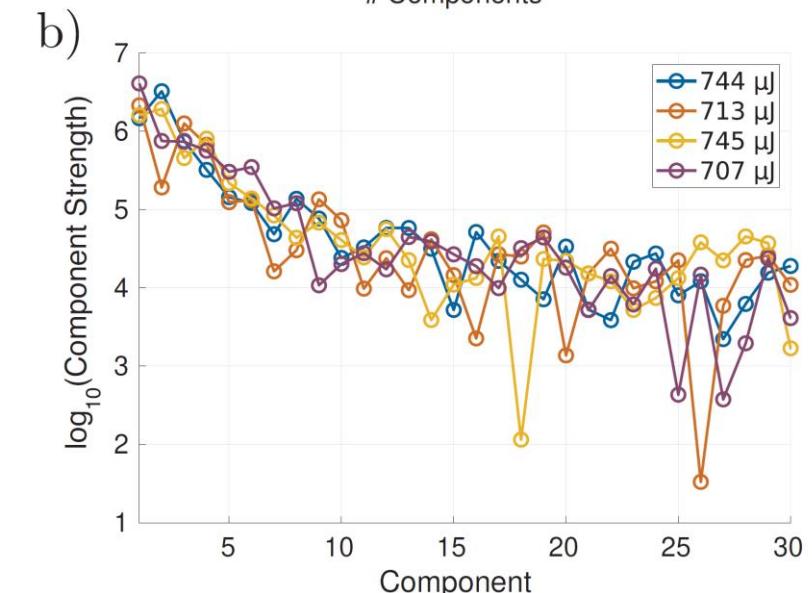
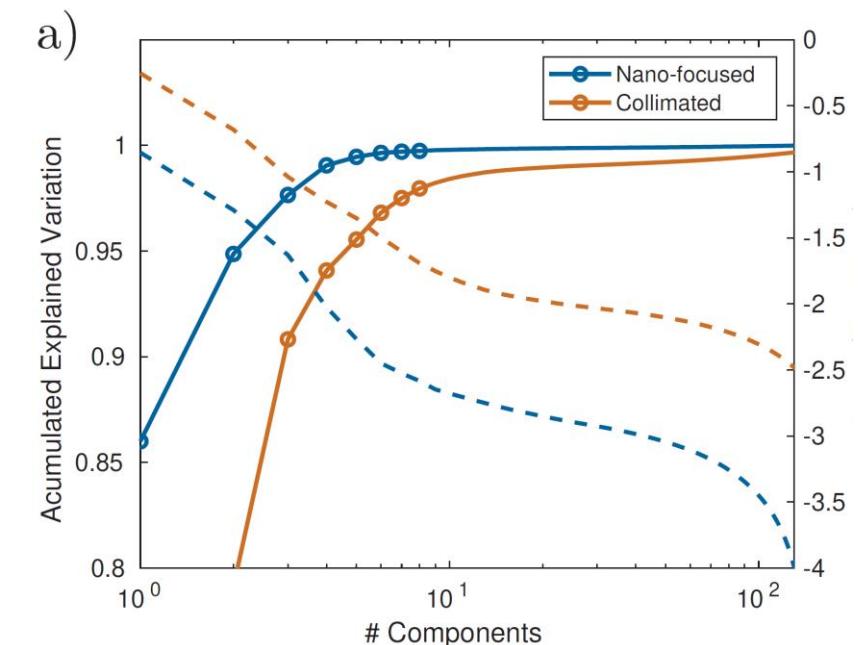
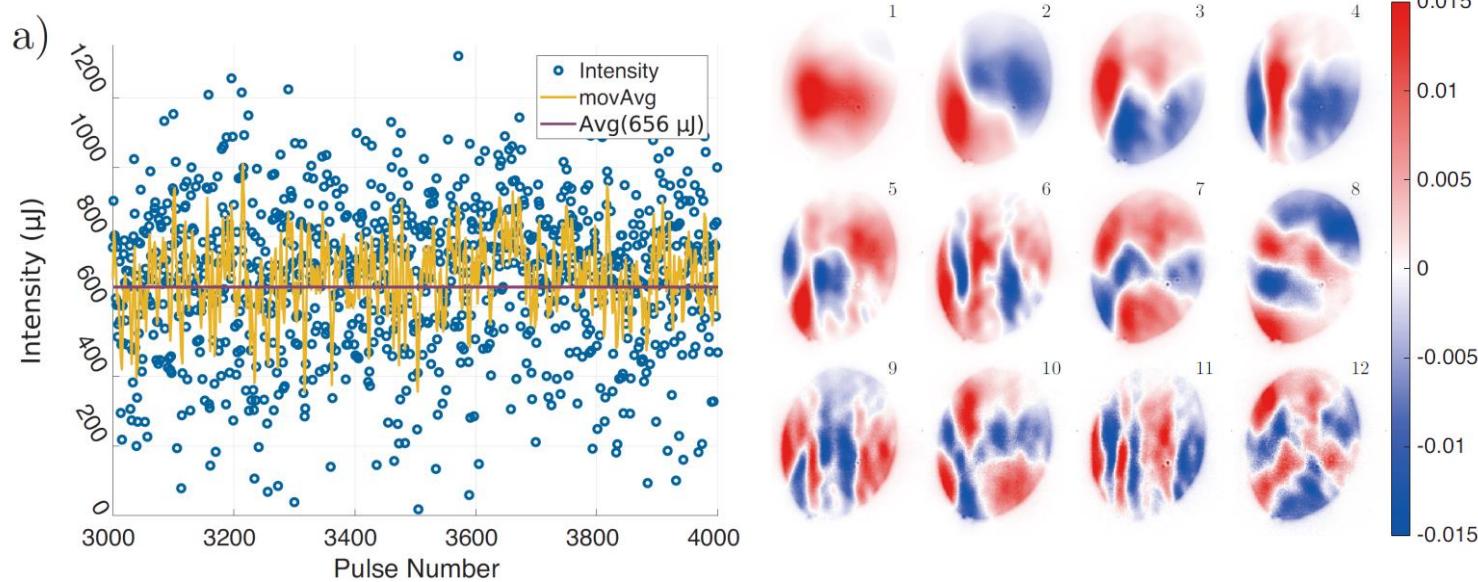
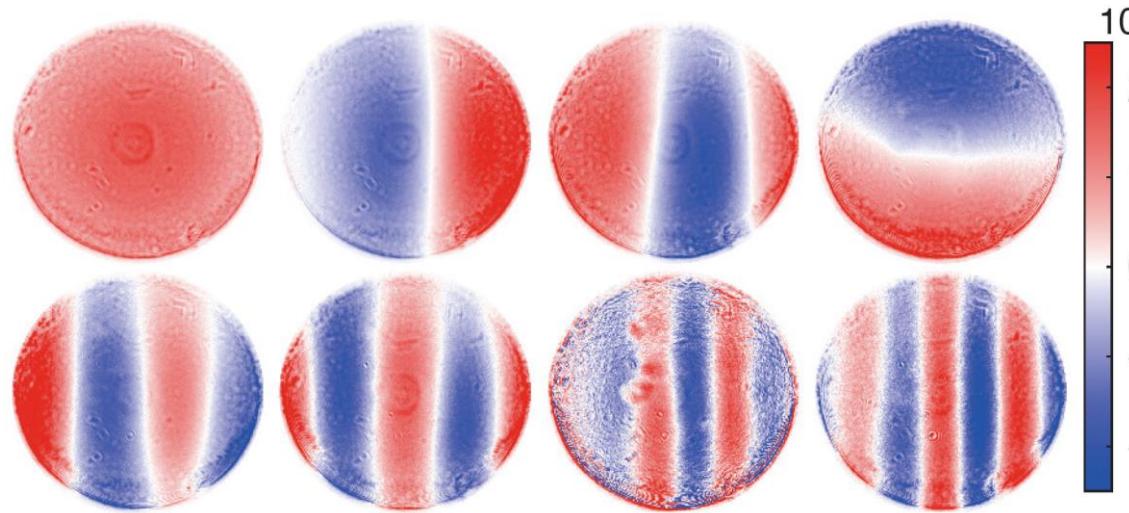
- *divergence,*
- *pointing stability*
- *signal*

*it is all actually
not too bad
for a single pulse!*



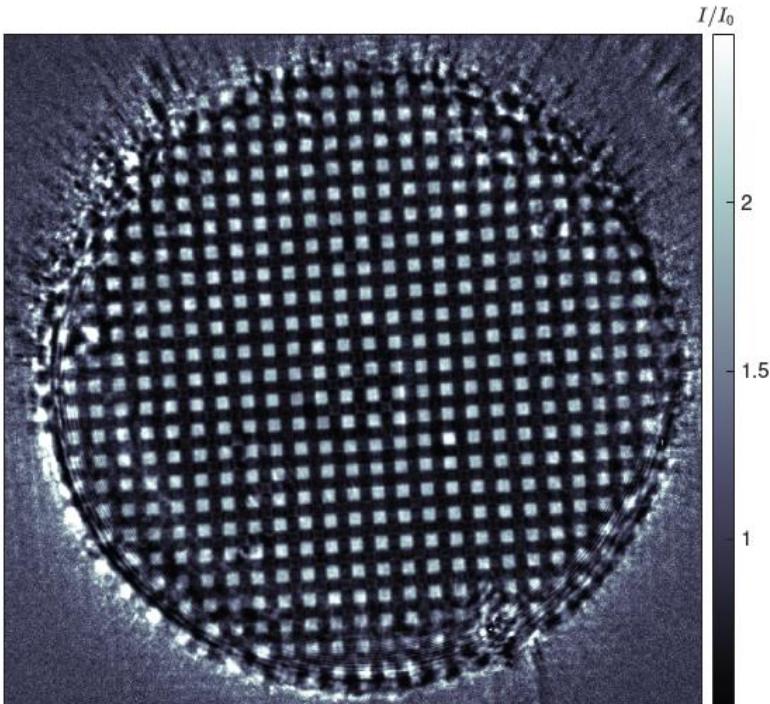
*But how to
perform the
flat field correction?*

Let's decompose (PCA analysis)



Let's decompose (PCA analysis)

now imaging works!

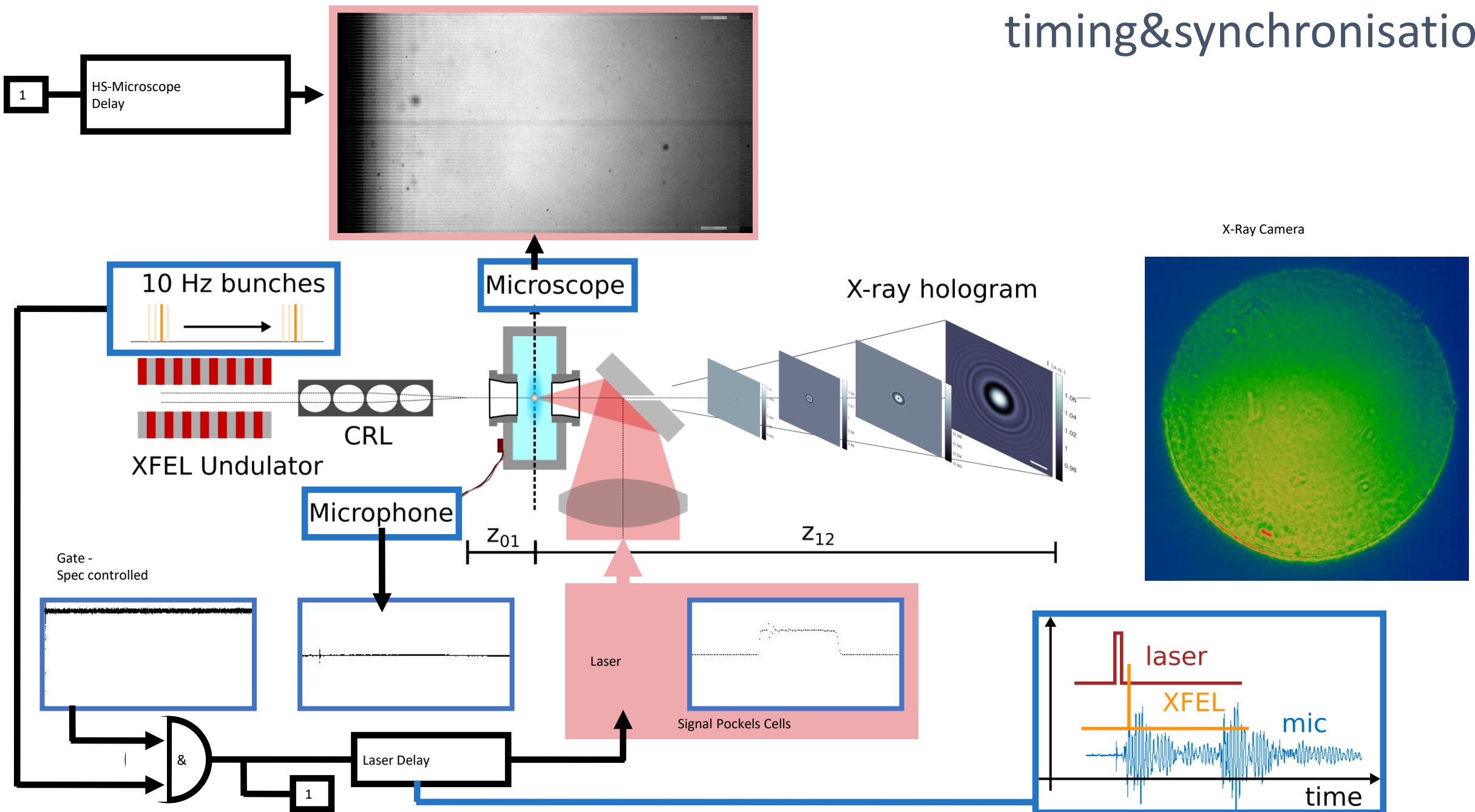


6 μ mAu mesh

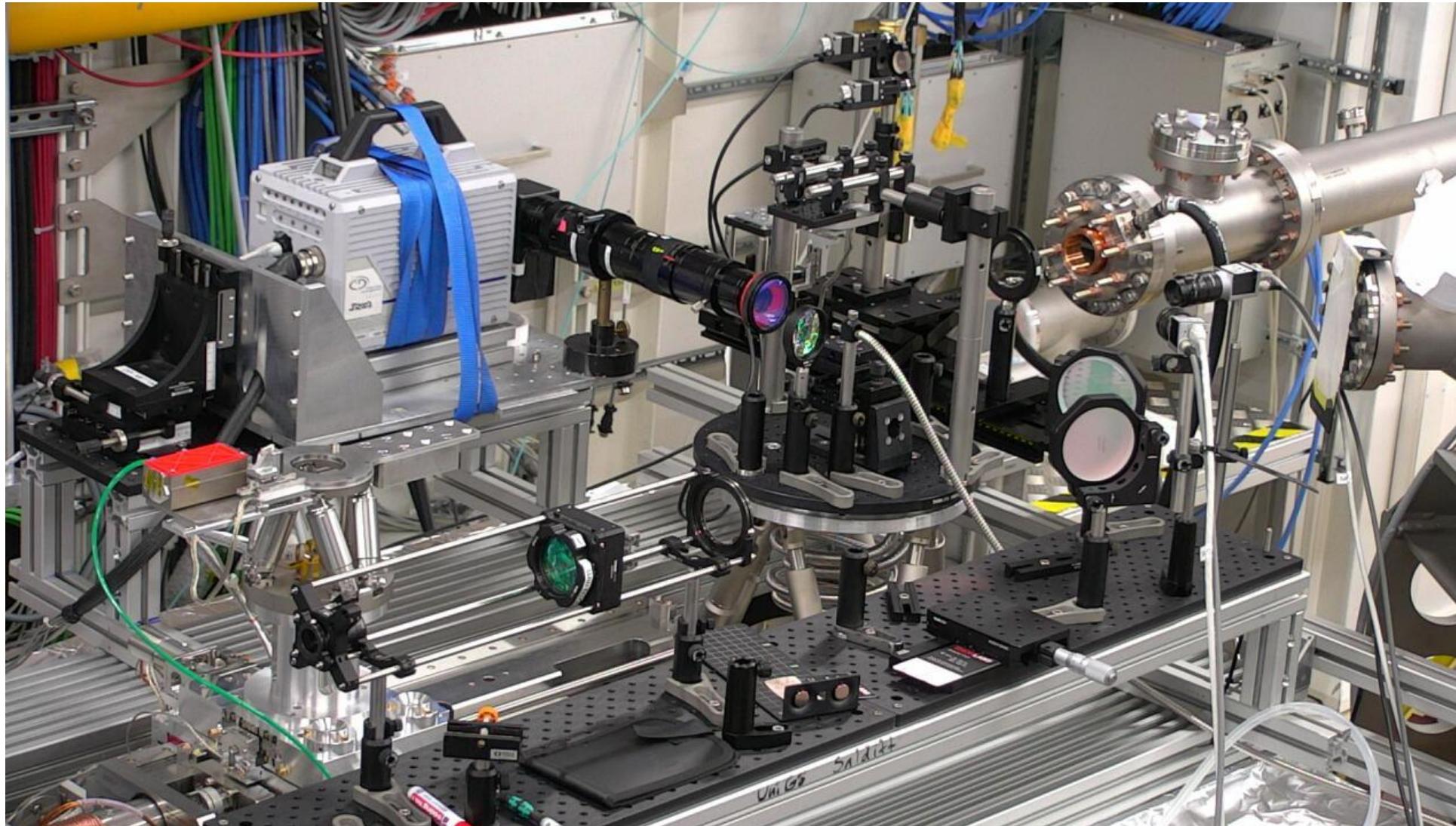
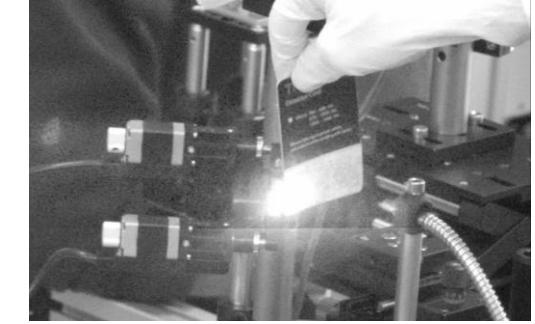


laminar water jet

timing&synchronisation



A good timing scheme is not sufficient ...

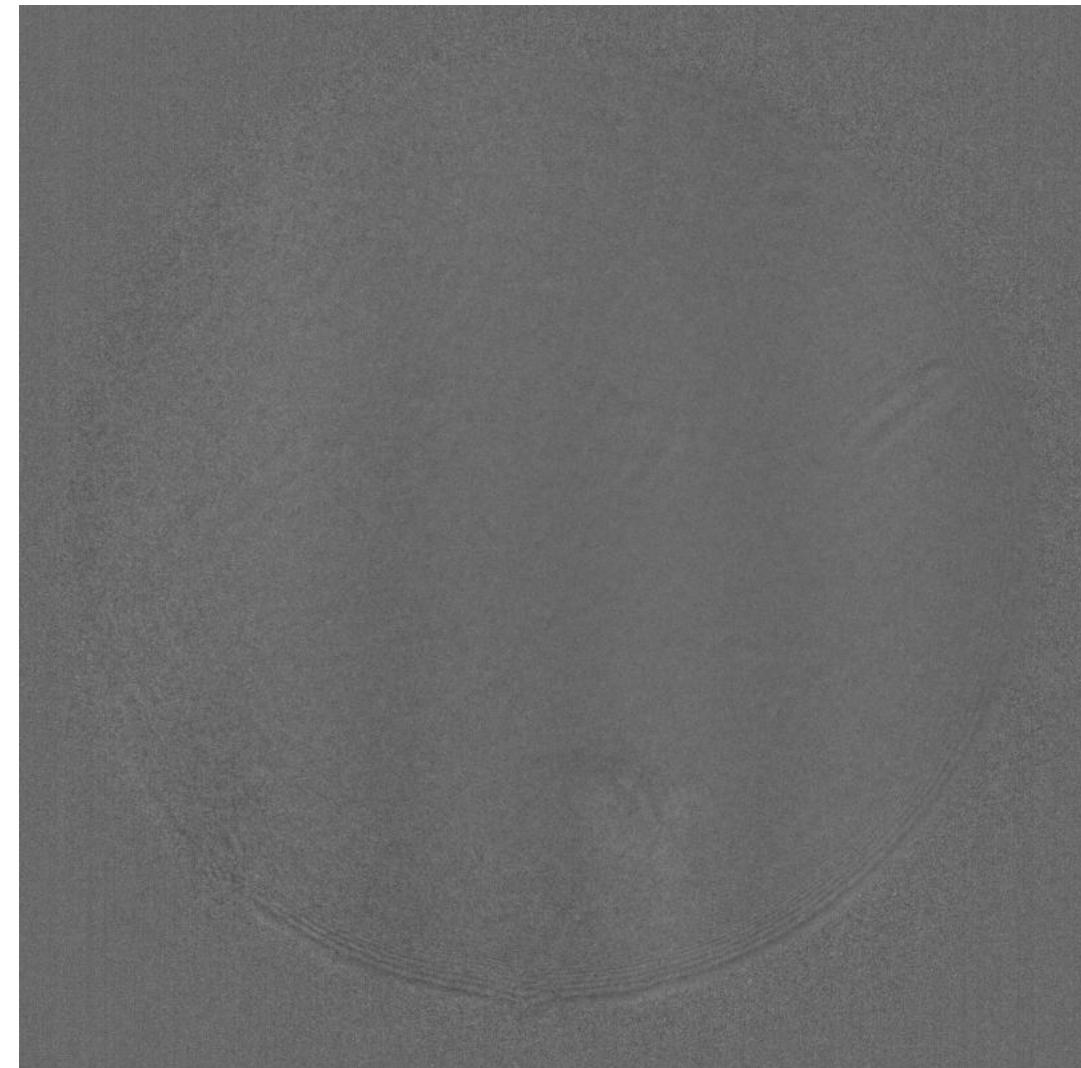
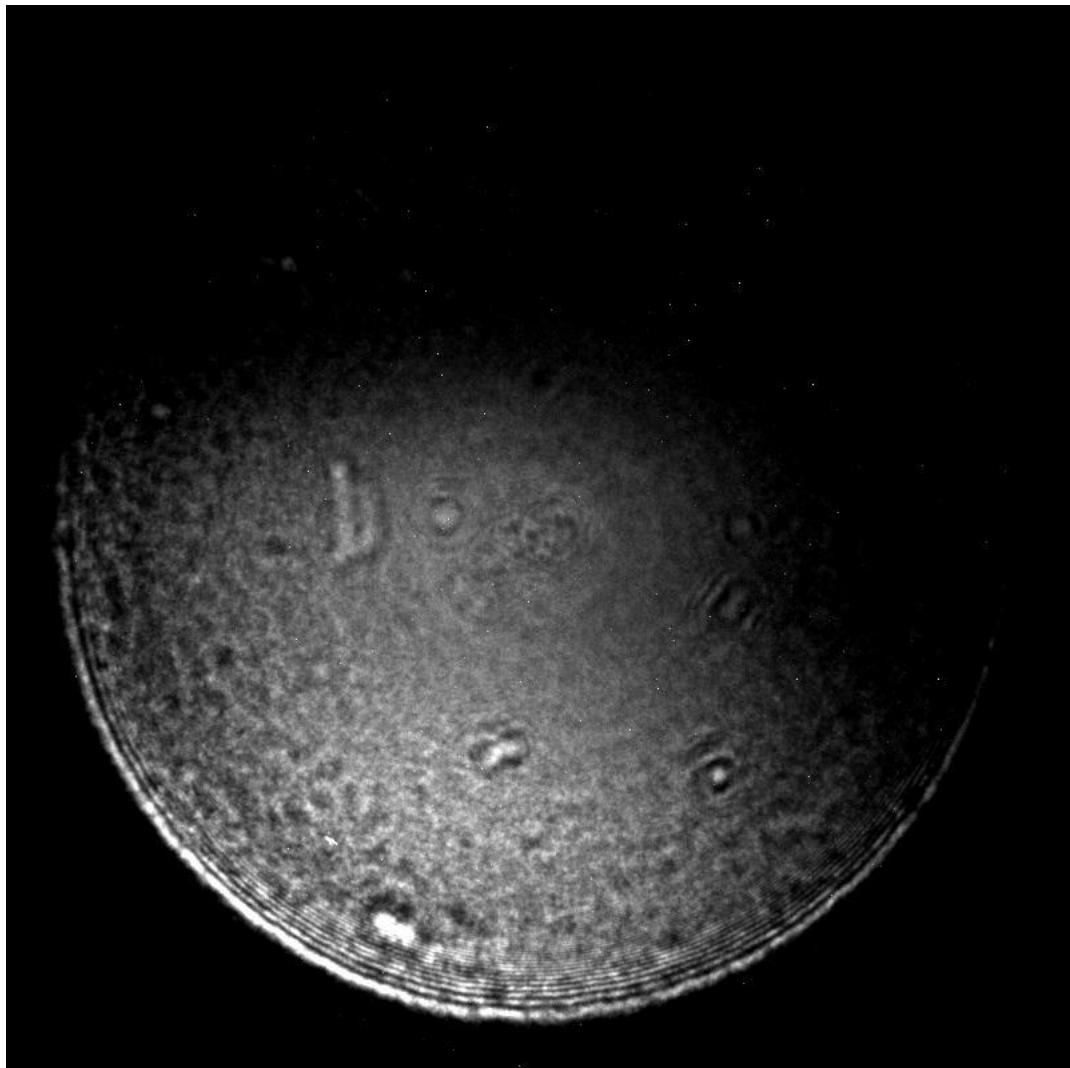


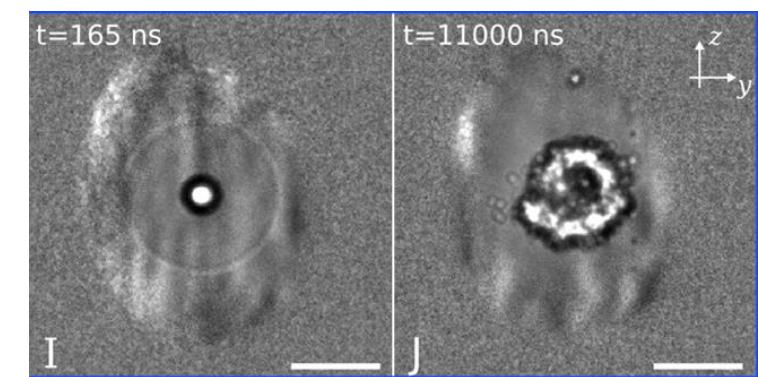
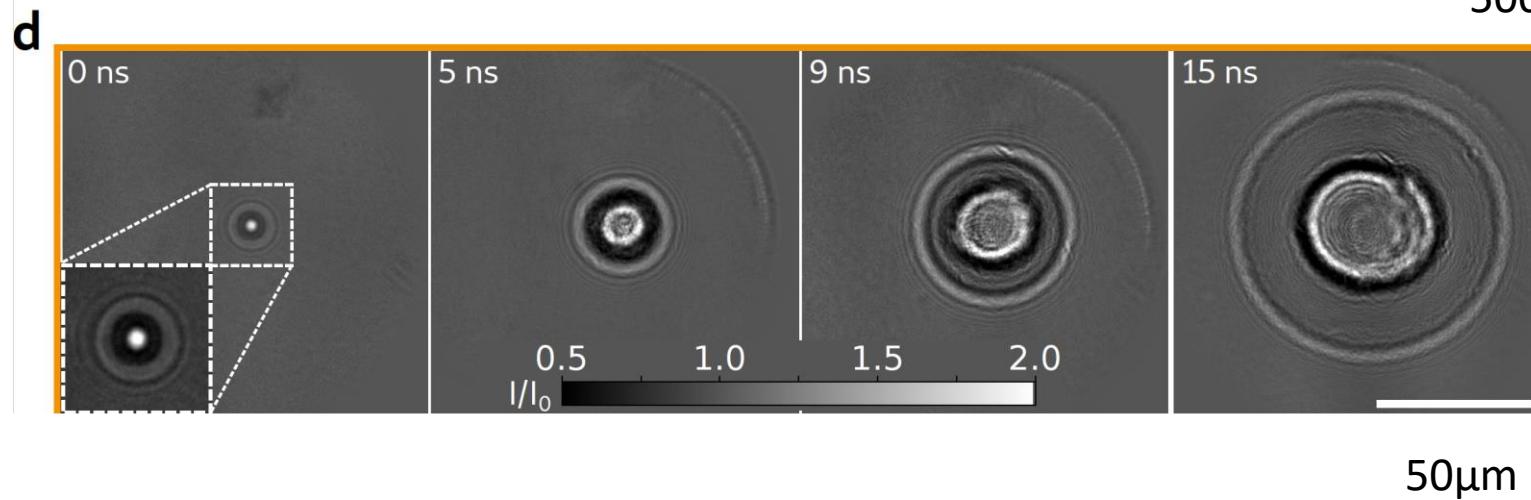
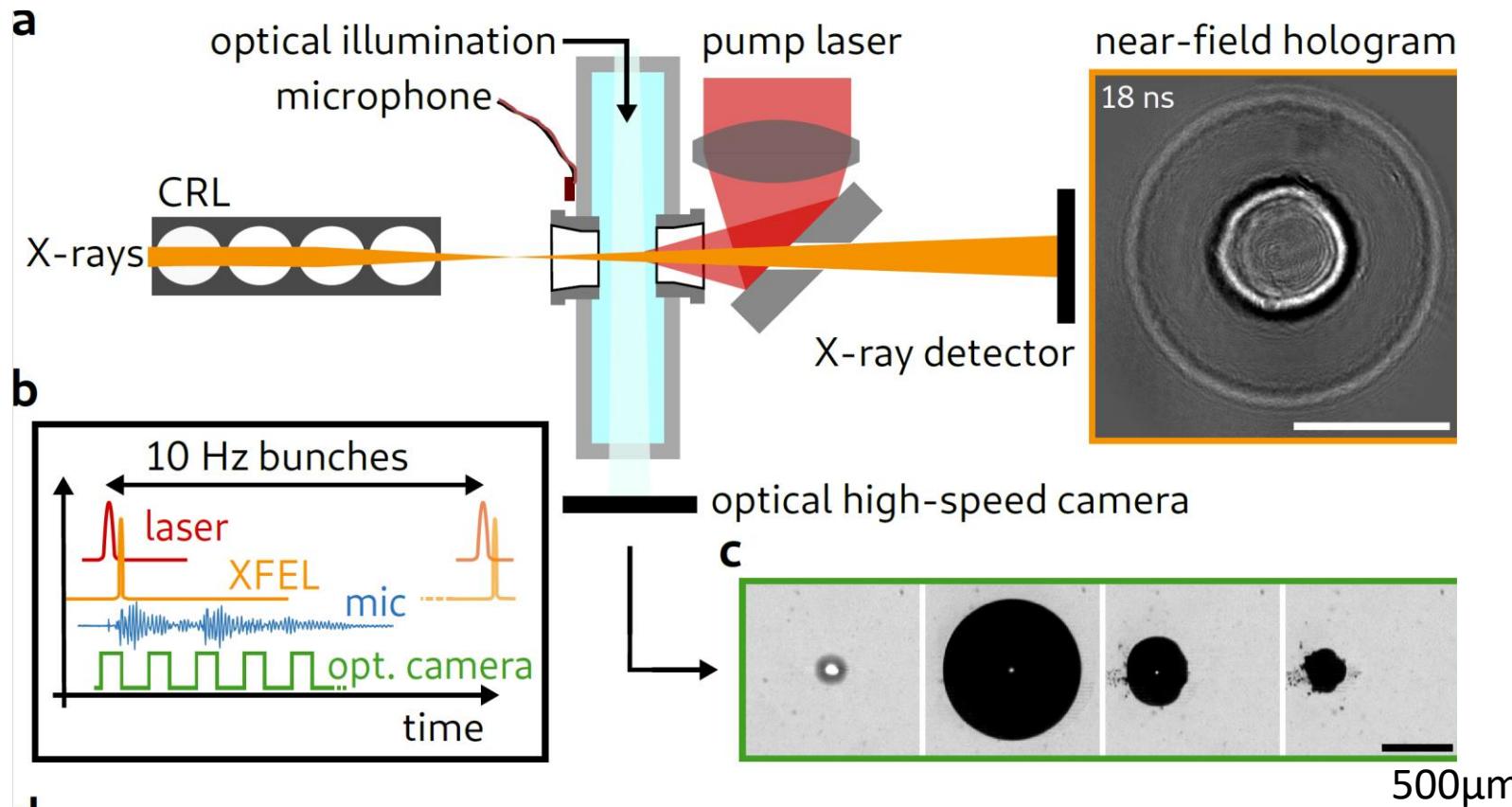
IR- alignment

*you have
to have
spatial-
temporal
overlap !*

Malte Vassholz
Hannes Hoeppel

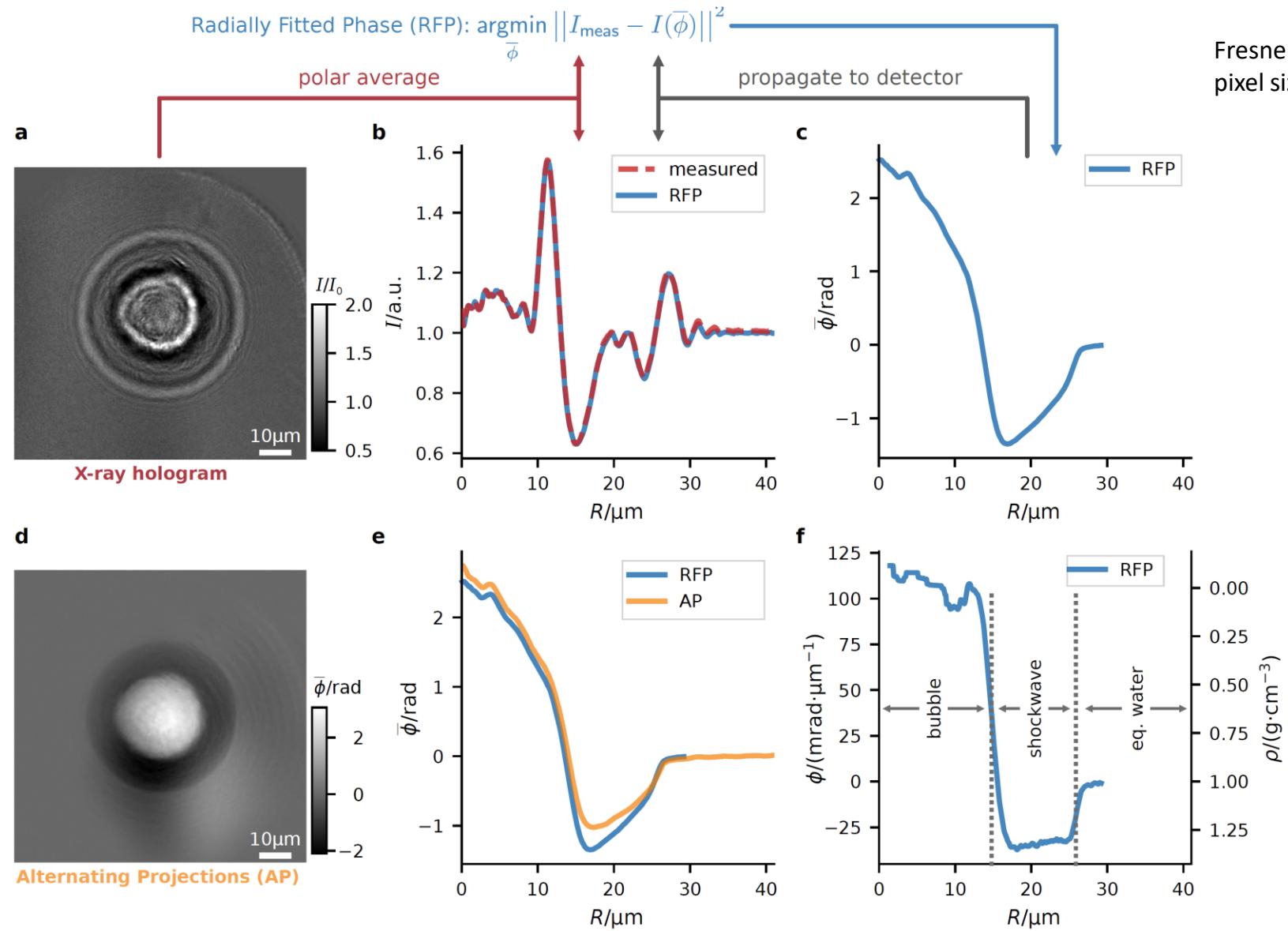
This is what we saw when we saw something (online analysis)



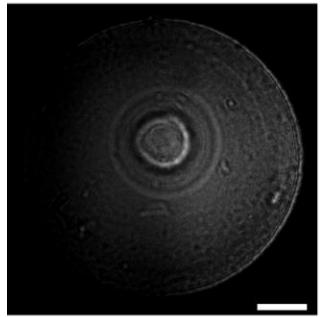


*But how to
analyze all
of this?*

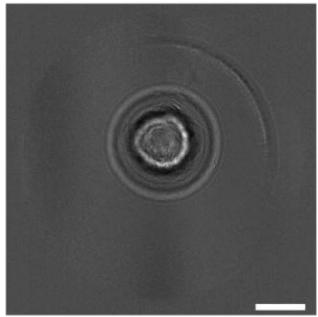
Phase retrieval



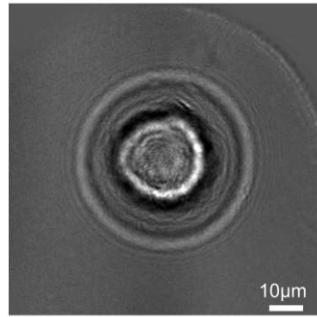
Phase retrieval in radial coordinates



raw
X-ray hologram



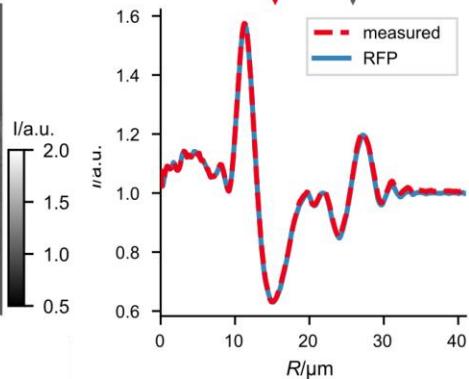
flat field corrected
X-ray hologram
(based on principal
component analysis of
a set of empty beams)



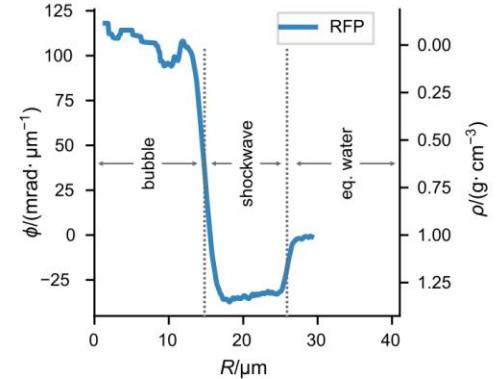
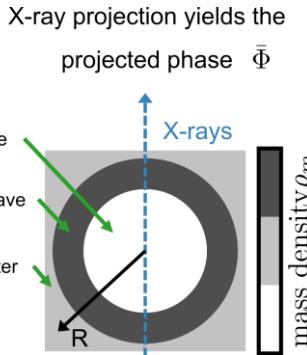
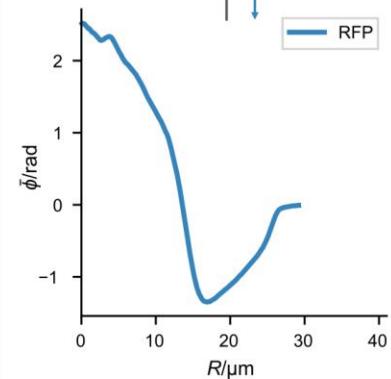
centered bubble
X-ray hologram

$$\text{Radially Fitted Phase (RFP): } \operatorname{argmin}_{\bar{\phi}} \| I_{\text{meas}} - I(\bar{\phi}) \|^2$$

polar average



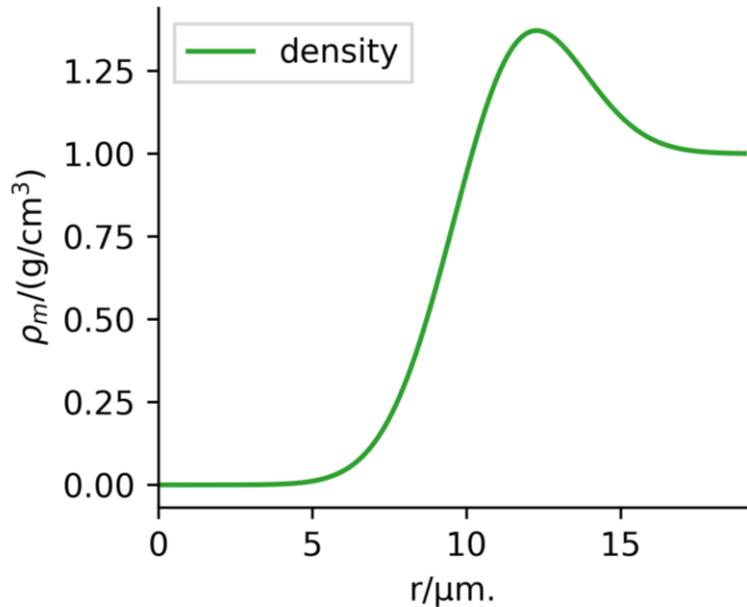
propagate to detector



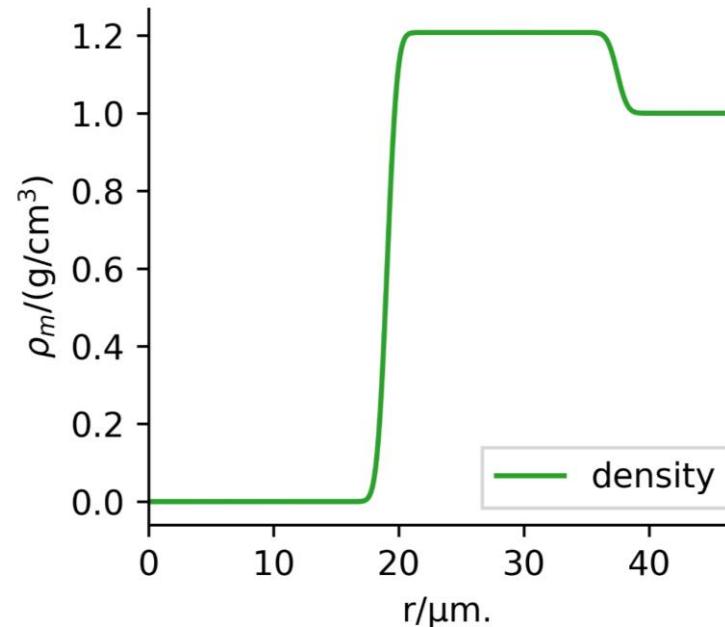
J. Hagemann et al., *Single-pulse phase-contrast imaging at free-electron lasers in the hard X-ray regime*.
Journal of Synchrotron Radiation, 28(1):52–63, 2021.

M. Vassholz et al., *Pump-probe X-ray holographic imaging of laser-induced cavitation bubbles with femto-second FEL pulses*.
Nature Communications, 12(1):3468, 2021.

$\tau=4\text{ns}$

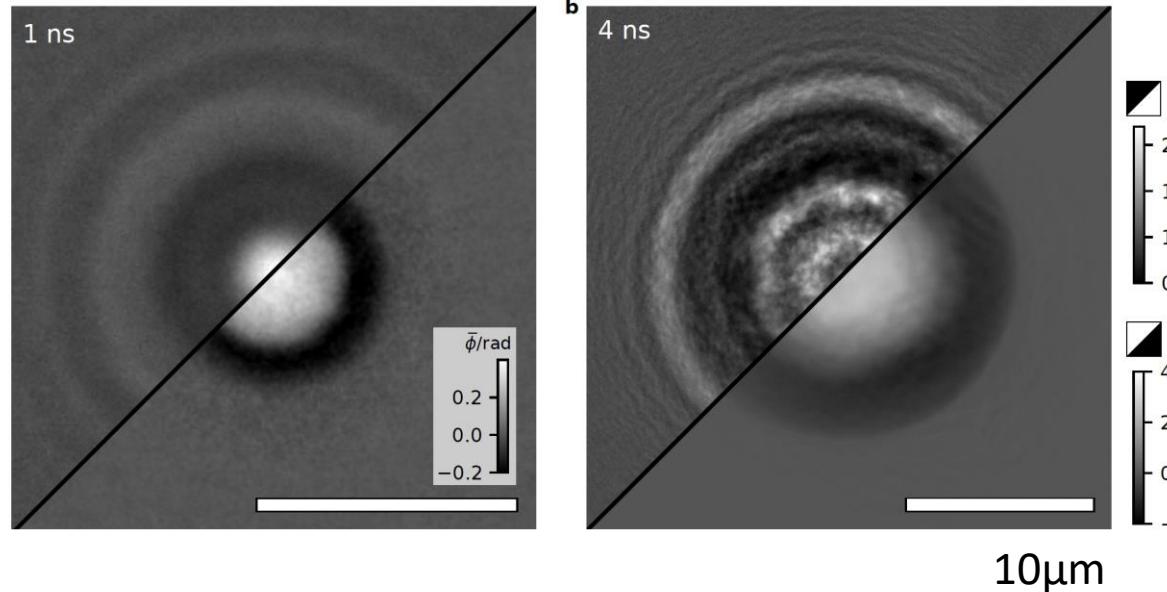


$\tau=13\text{ns}$



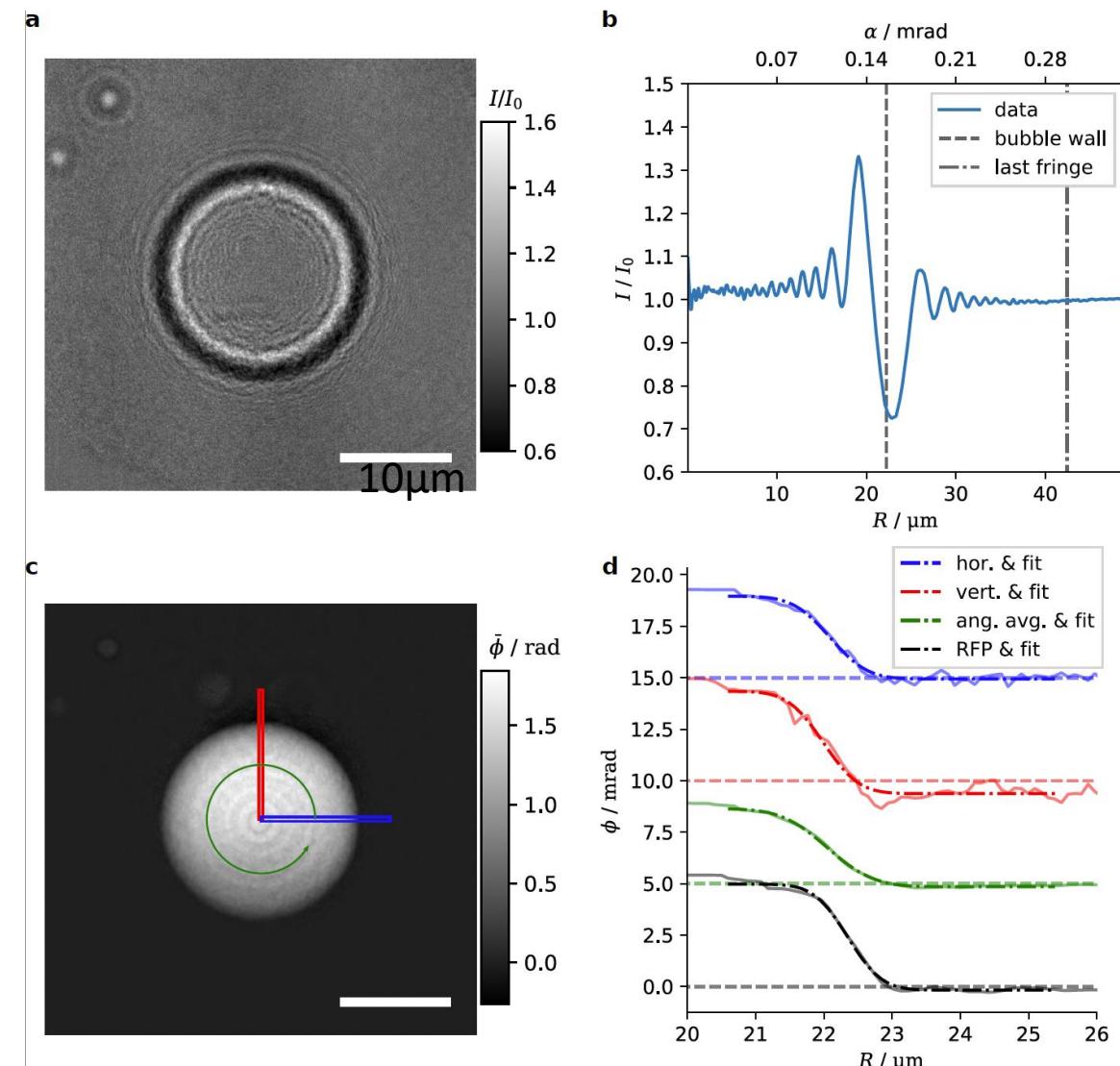
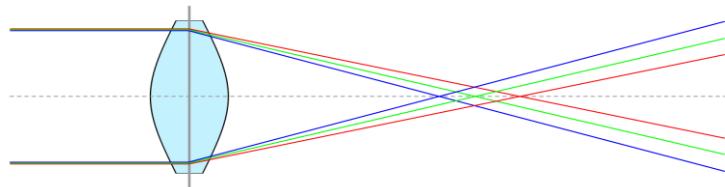
- *emergence of phase boundary / sharpening of the interface*
- *density of the shock wave -> equation of state*
- *Outlook: comparison with MD simulation*
- *Outlook: fs time scales / plasma dynamics / water structure in shock wave collapse of the bubble*

Phase retrieval /Resolution

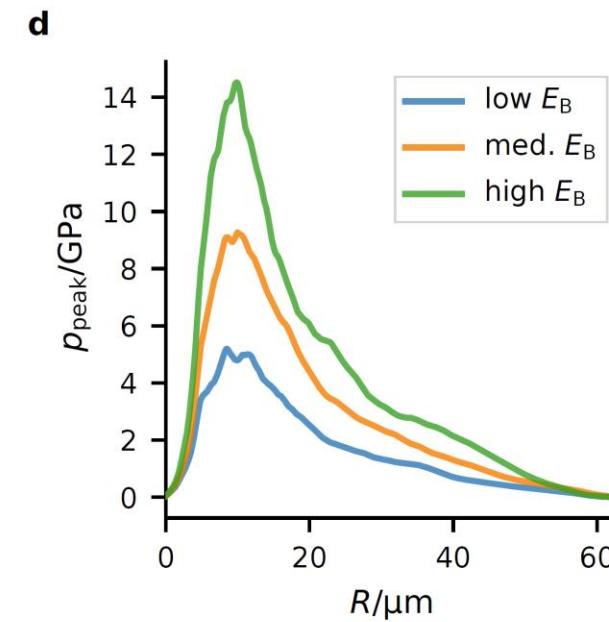
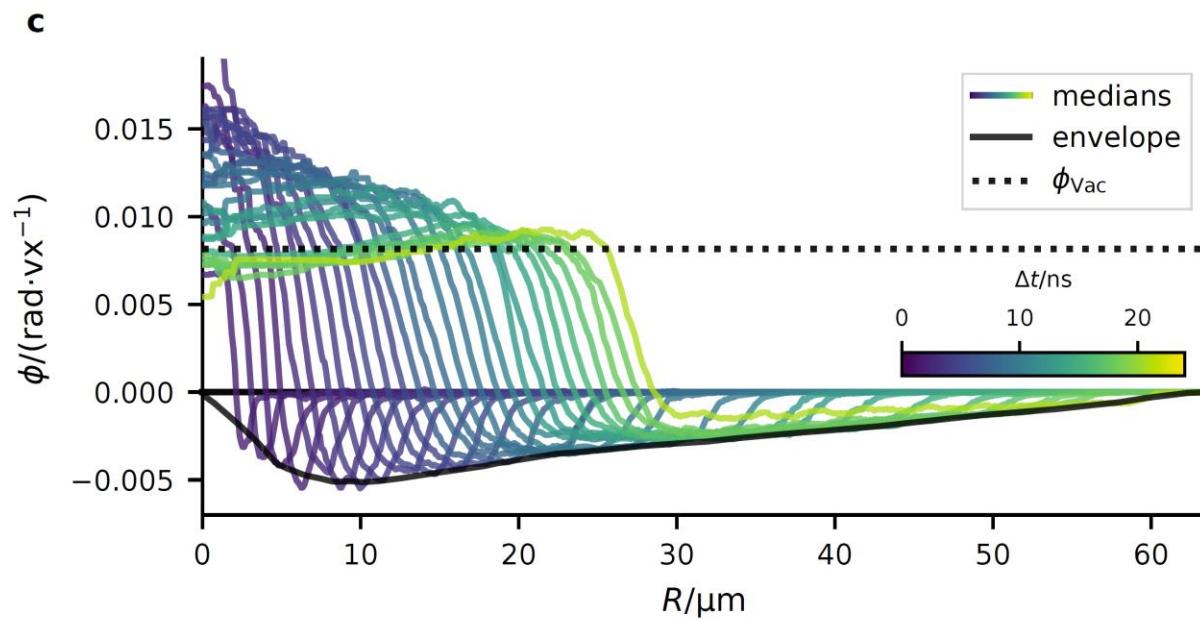
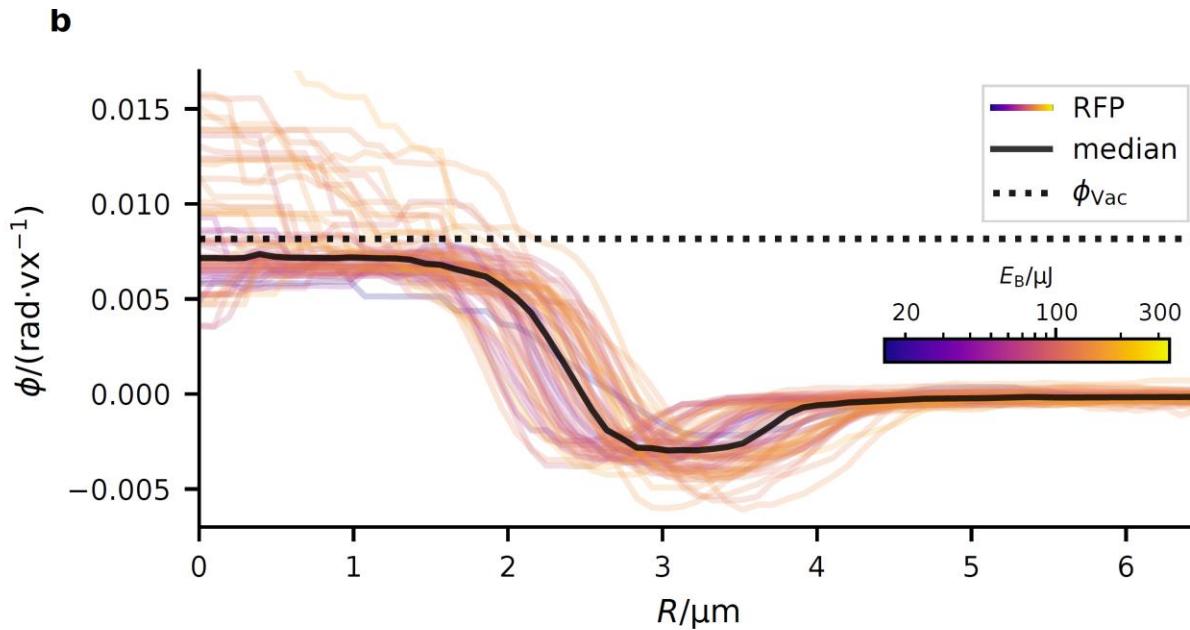
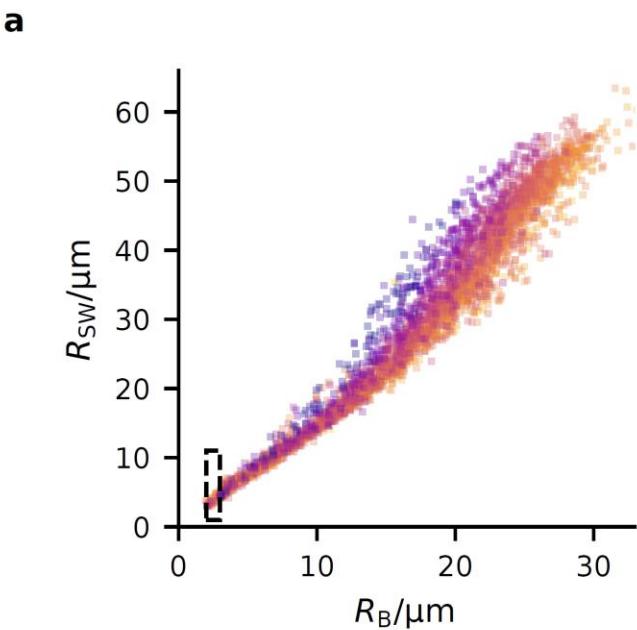


@ Fresnel number $\text{Fr} = 0.8 \cdot 10^{-3}$ (with respect to the pixel size)
 pixel size $\Delta x = 109 \text{ nm}$, FOV = 140 μm

contrast sensitivity limited by wavefront stability!

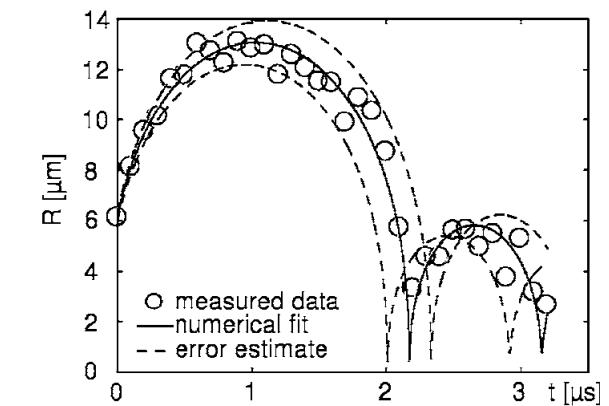
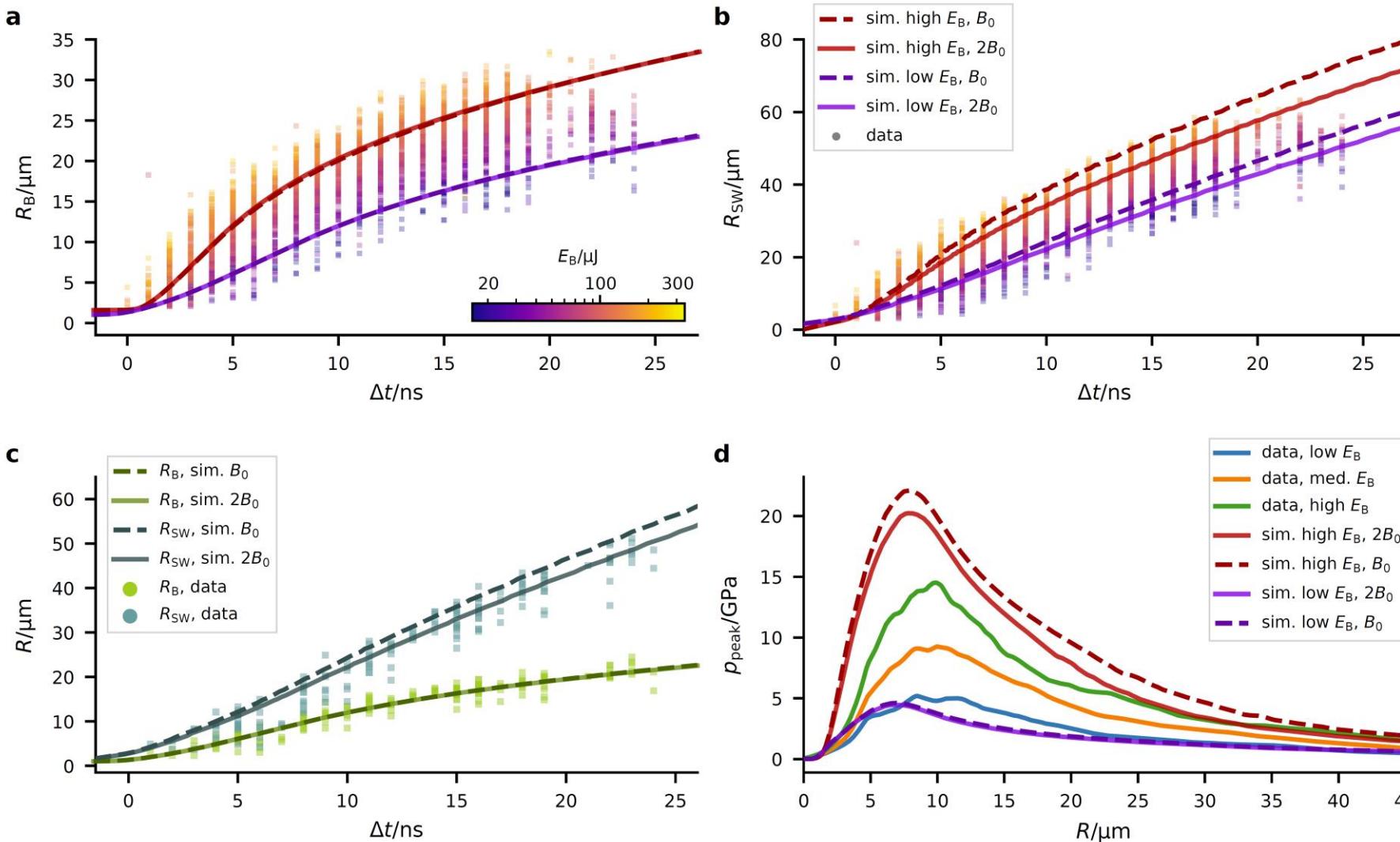


resolution limited by dispersive optics !



Working out the entire ensemble / sorting for E_B

- maximum radius (photon)
- life time (microphone)
- bubble energy from life time and/or maximum radius

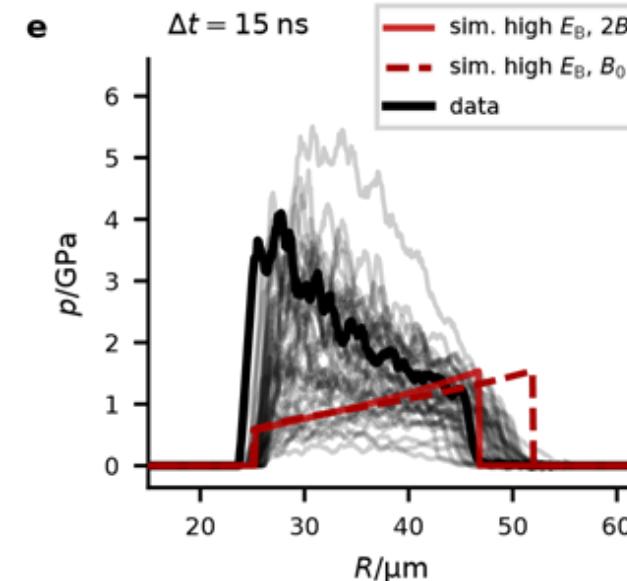
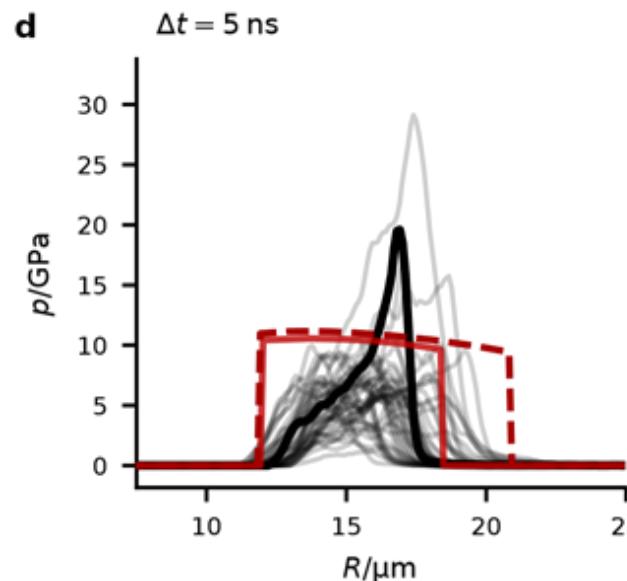
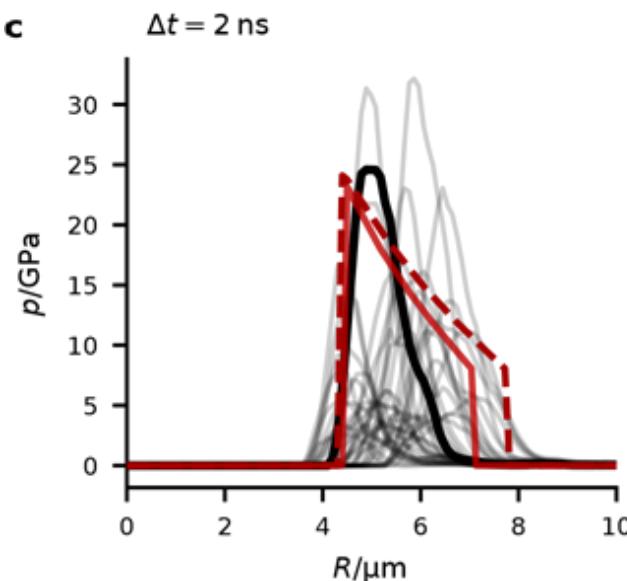
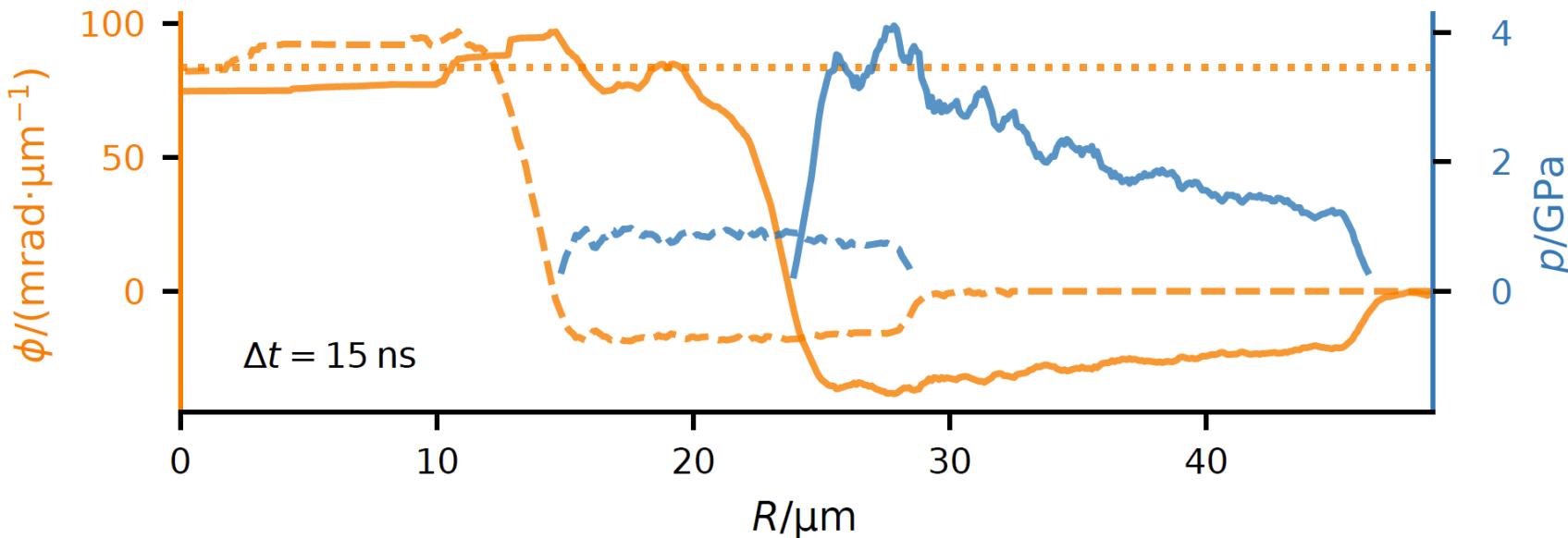


T. Kurz et al. Phys. Rev. E (2006)

*bubble energy determines
maximum radius*

$$E_B = \frac{3\pi}{4}(p_0 - p_v)R_{max}^3$$

Results: Pressure profile of shock wave



$$p - p_0 = B \left(\left(\frac{\rho}{\rho_0} \right)^m - 1 \right)$$

$$B = K/m \quad m = c_p/c_v \simeq 7$$

Tait eq.

Gilmore model

-> data puts current hydrodynamic models of shock wave into question !

The Gilmore model for cavitation dynamics

$$R\ddot{R}\left(1 - \frac{\dot{R}}{C}\right) + \frac{3}{2}\dot{R}^2\left(1 - \frac{\dot{R}}{3C}\right) = H\left(1 + \frac{\dot{R}}{C}\right) + \frac{\dot{R}}{C}\left(1 - \frac{\dot{R}}{C}\right)\frac{dH}{dR}$$

2. step: shock wave propagation based of the
Kirkwood-Bethe Hypothesis and method of characteristics

$G = r(h + u^2/2)$ invariant quantity

$$\dot{r} = u + c$$

$$\dot{u} = \frac{1}{c-u} \left((u+c) \frac{G}{r^2} - \frac{2c^2 u}{r} \right)$$

$$\dot{p} = \frac{\rho_0}{r(c-u)} \left(\frac{p+B}{p_0+B} \right)^{\frac{1}{n}} \left(2c^2 u^2 - \frac{c^2 + uc}{r} G \right)$$

The pressure profiles are cuts at constant time in the three-dimensional space of all characteristics.

ρ water density

$n = 7 \hat{=} \text{adiabatic index of water}$

$$B = 314 \text{ MPa} \hat{=} \frac{\rho_0 c_0^2}{n}$$

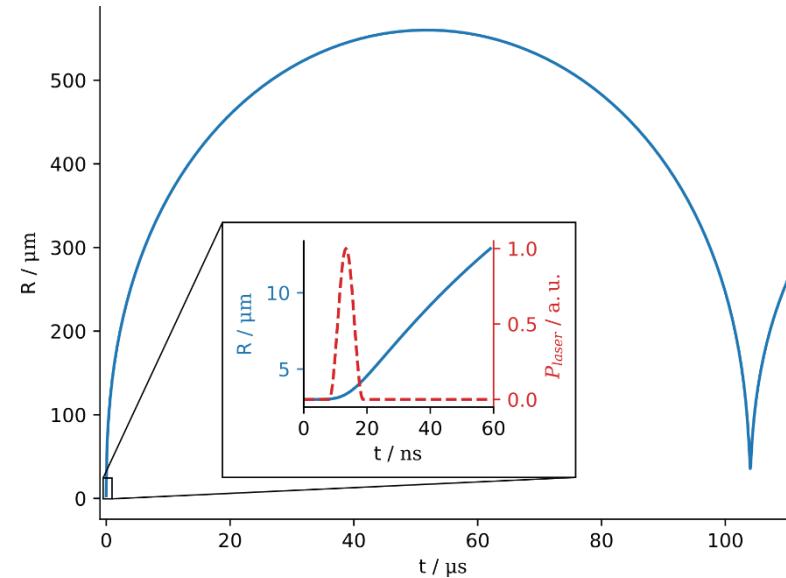
σ surface tension

κ adiabatic index of air

η dynamic viscosity

$$p(\rho) = \left(\frac{\rho}{\rho_0} \right)^m (p_\infty + B) - B$$

Modified Tait equation of state



The Gilmore model for cavitation dynamics

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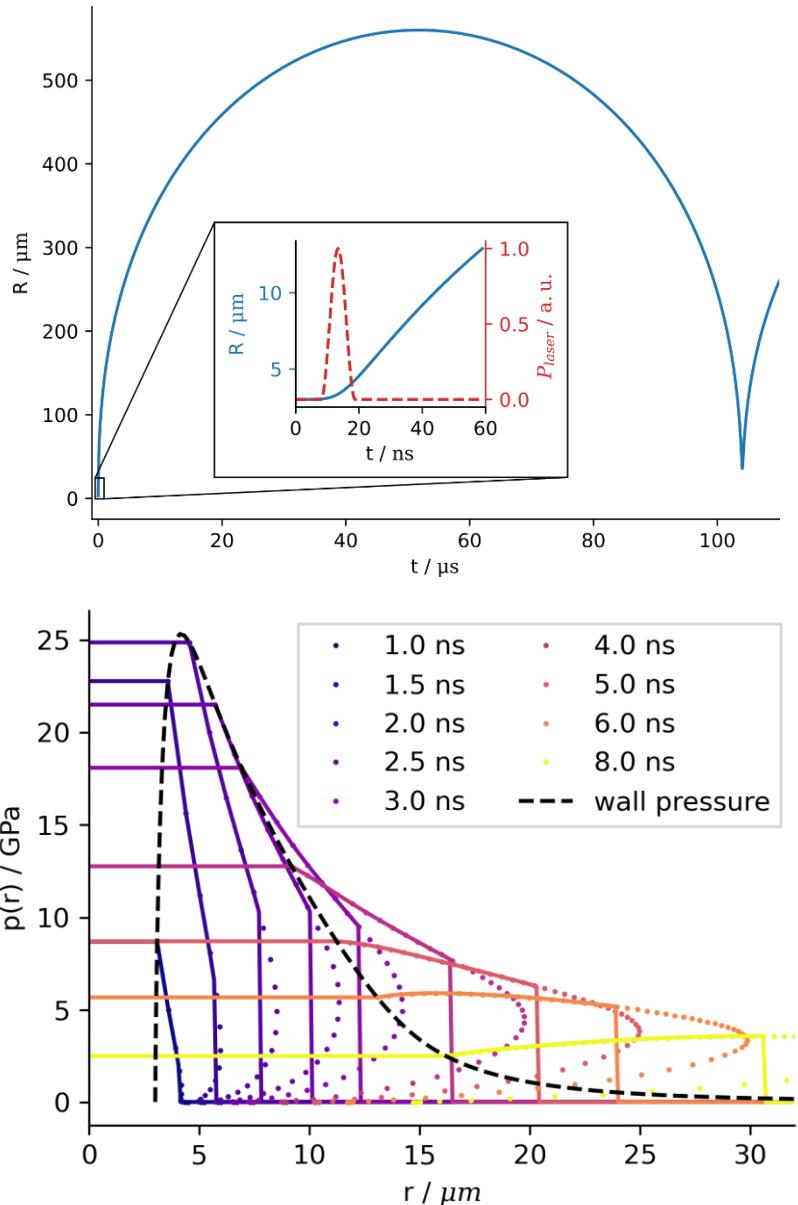
σ surface tension

κ adiabatic index of air

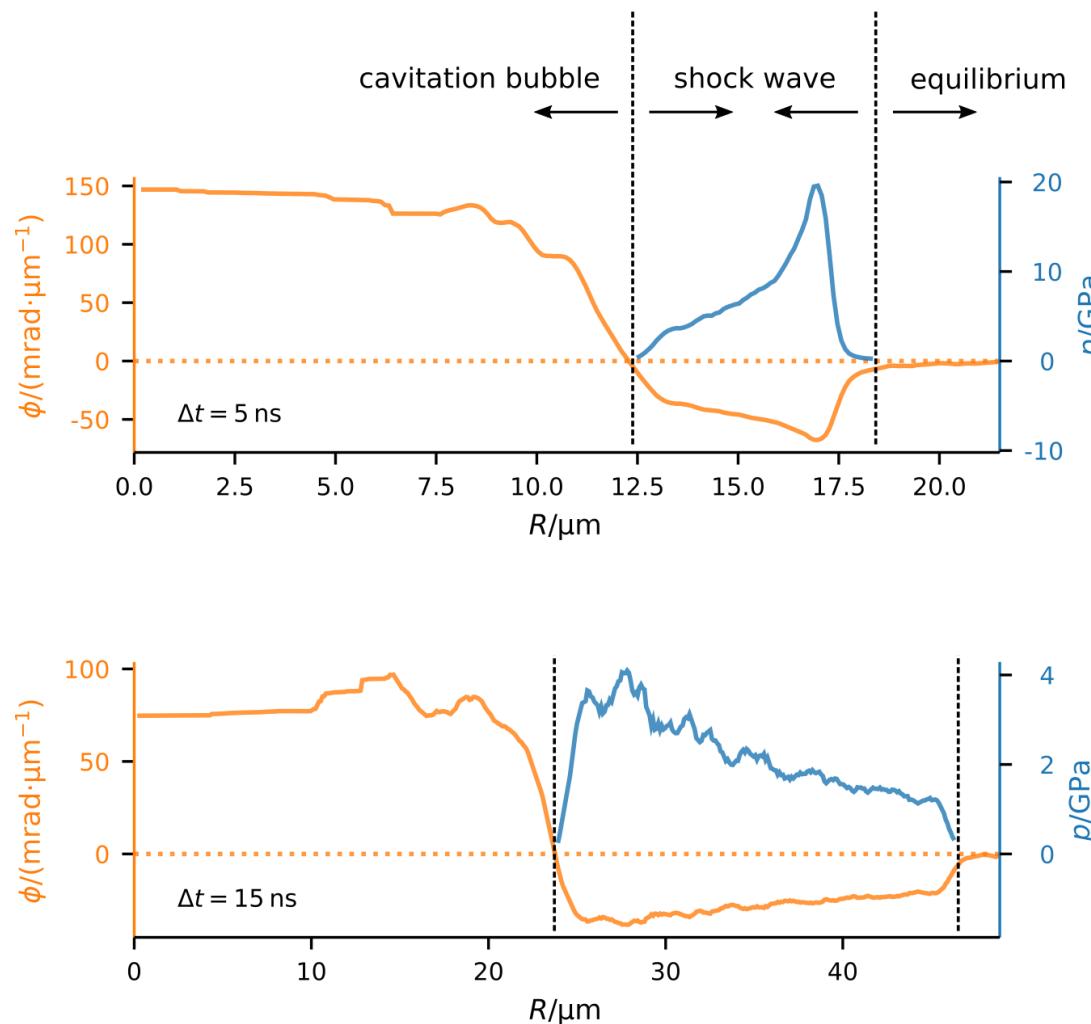
η dynamic viscosity

$$p(\rho) = \left(\frac{\rho}{\rho_0} \right)^m (p_\infty + B) - B$$

Modified Tait equation of state



3d radial Phase, density & pressure



mass density:

$$\rho(R) = \rho_0 \left(1 - \frac{\phi(R)}{k \delta} \right)$$

k X-ray wave number

δ real part of the refractive index

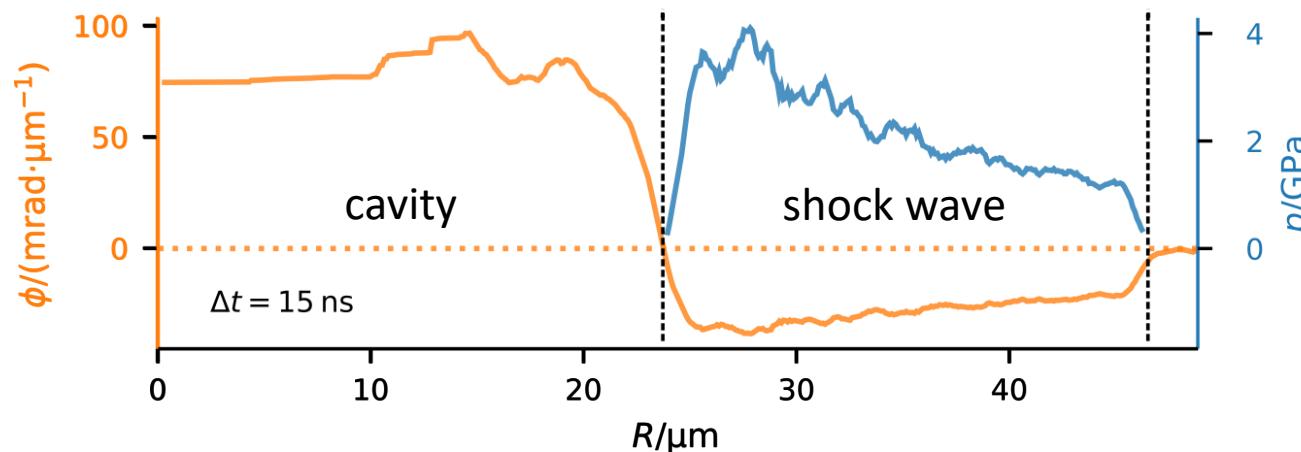
modified Tait Equation of state,
relates density to **pressure**:

$$p(\rho) = \left(\frac{\rho}{\rho_0} \right)^n (p_\infty + B) - B$$

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3d radial Phase, density & pressure



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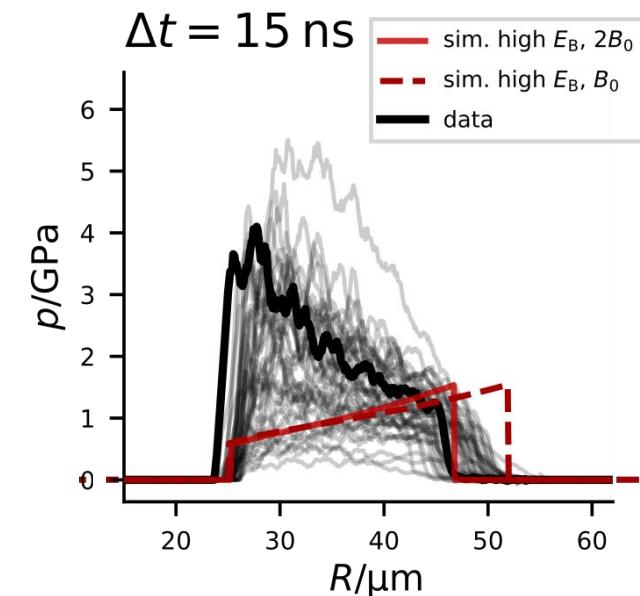
δ real part of the refractive index

modified Tait Equation of state (EOS),
relates density to **pressure**:

$$p(\rho) = \left(\frac{\rho}{\rho_0}\right)^n (p_\infty + B) - B$$

$n = 7 \doteq$ adiabatic index of water

$$B = 314 \text{ MPa} \doteq \frac{\rho_0 c_0^2}{n}$$



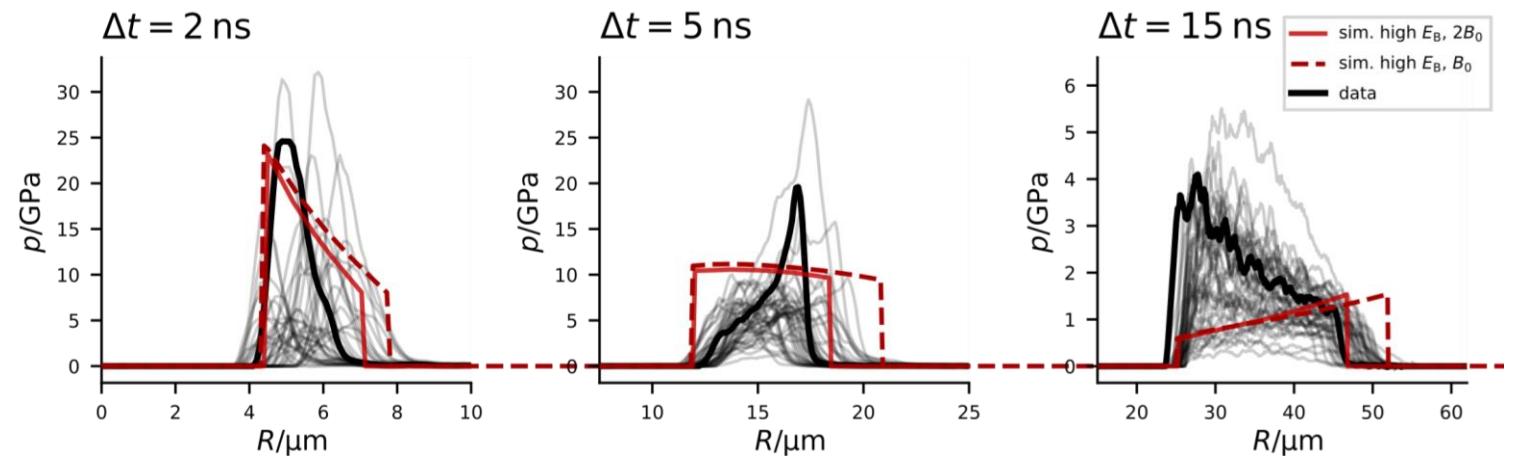
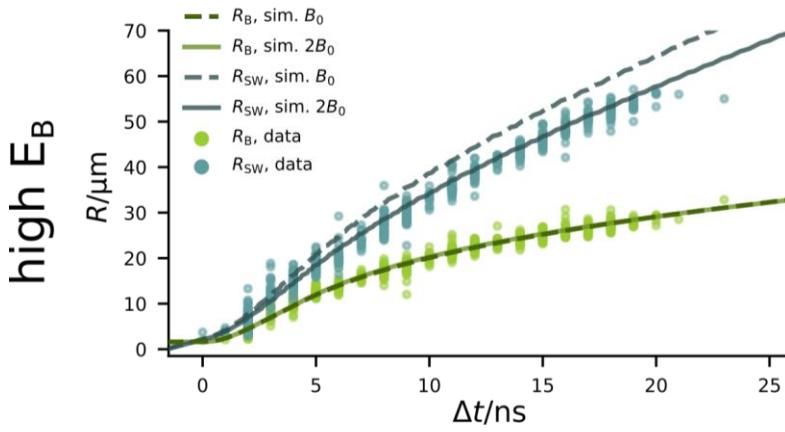
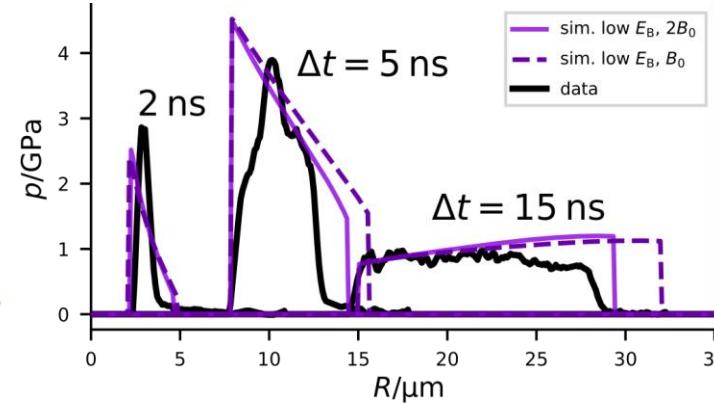
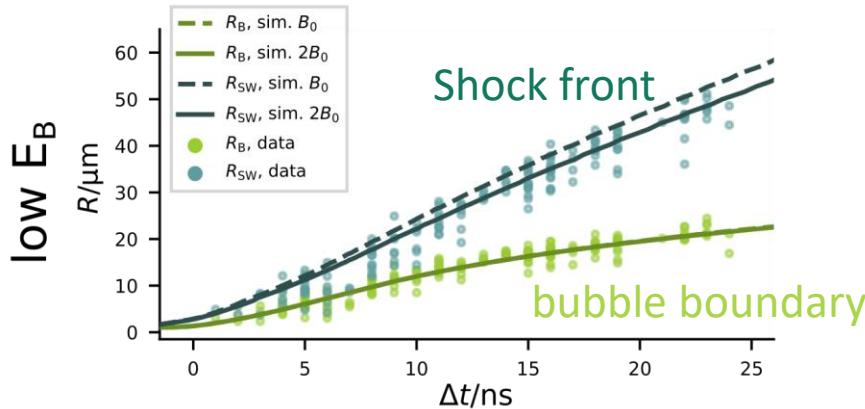
Comparing with typical hydrodynamic models:

- Gilmore Model for cavitation dynamics
- Kirkwood & Bethe-based shock propagation

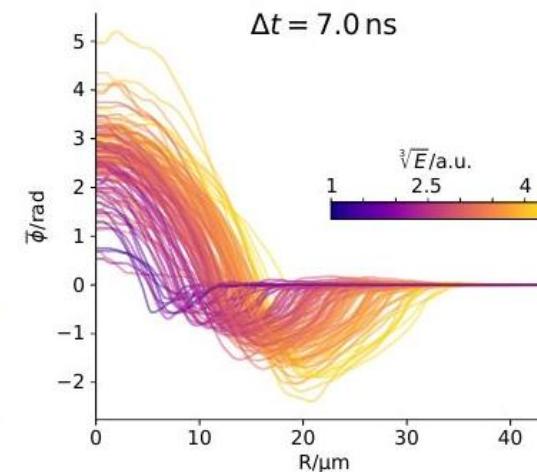
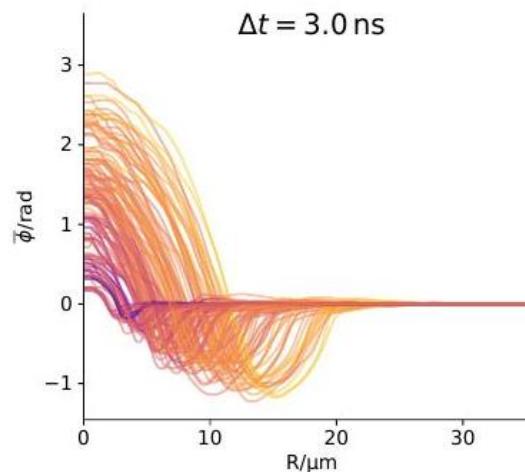
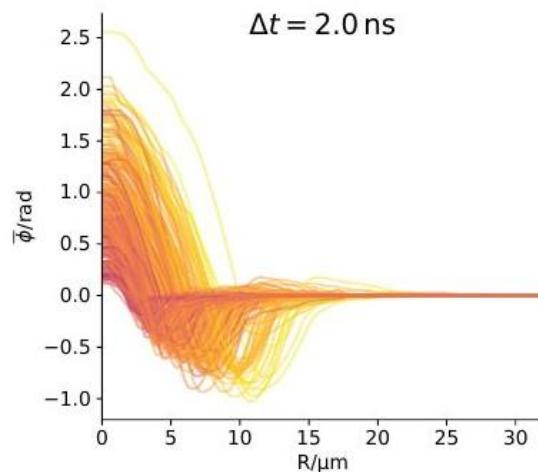
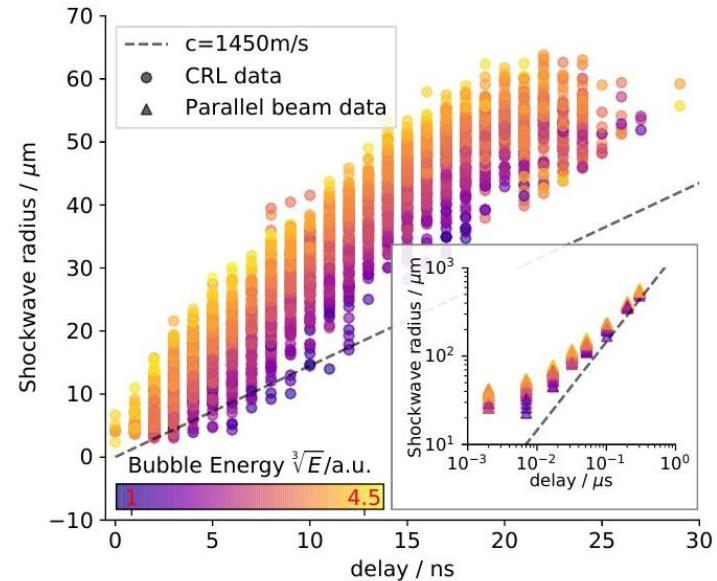
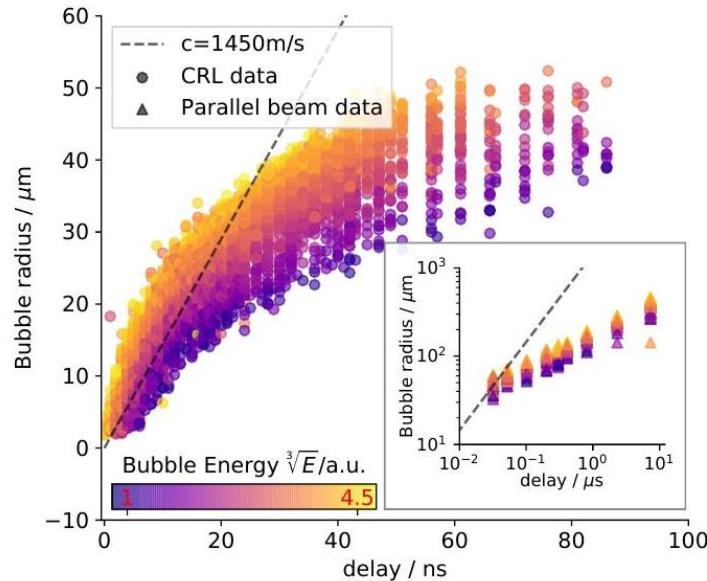
Discrepancies for high-energy events:

- Shape / slope
- peak pressures
- trajectory

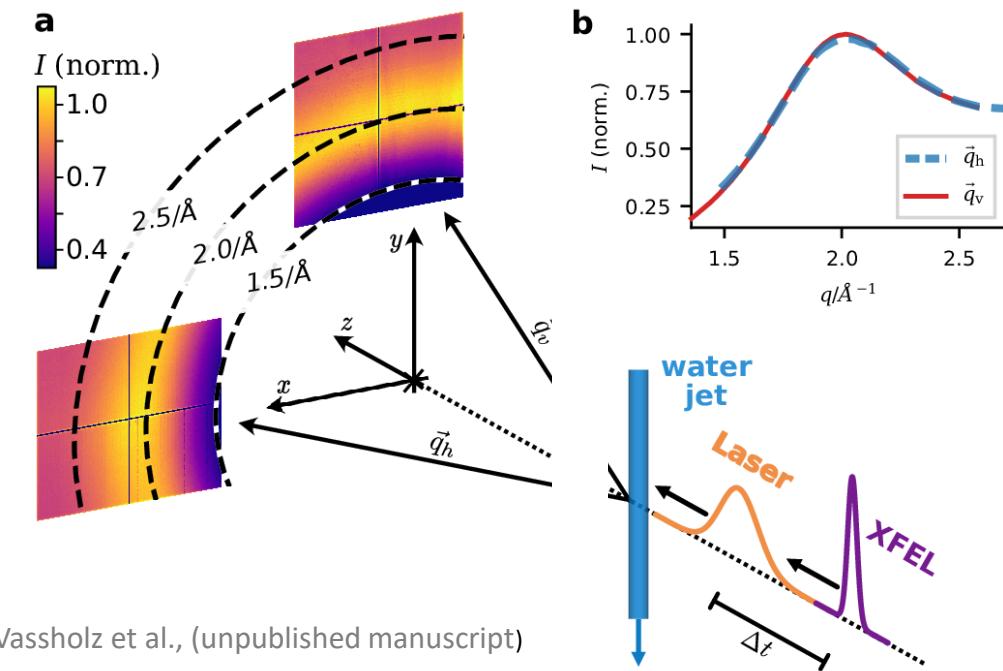
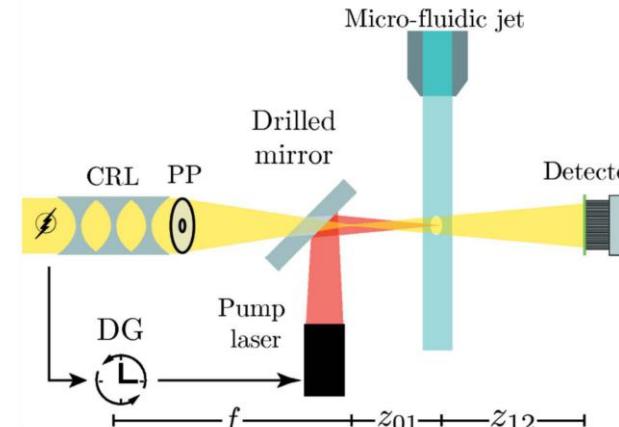
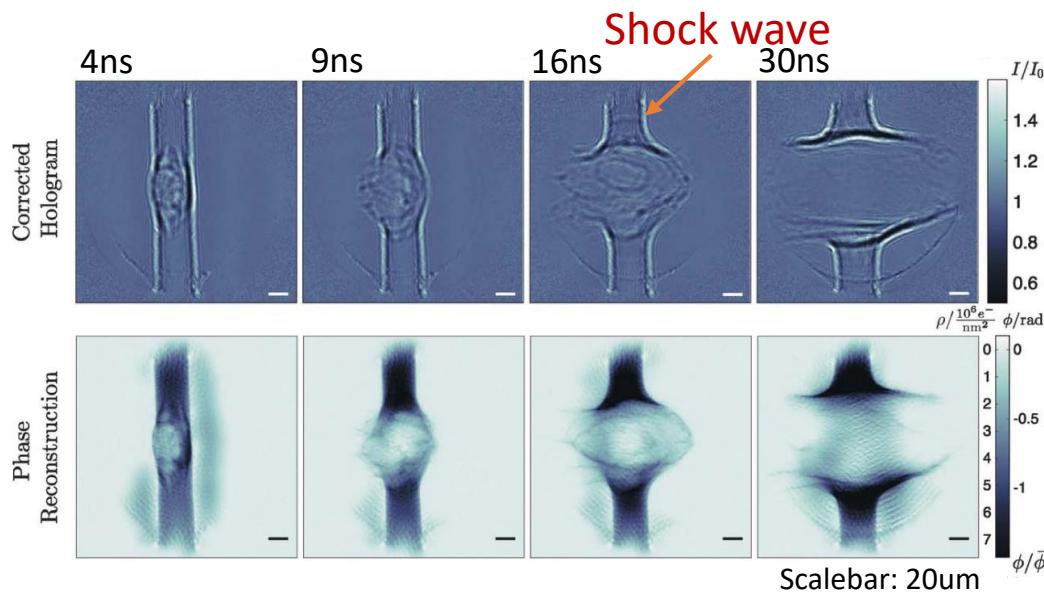
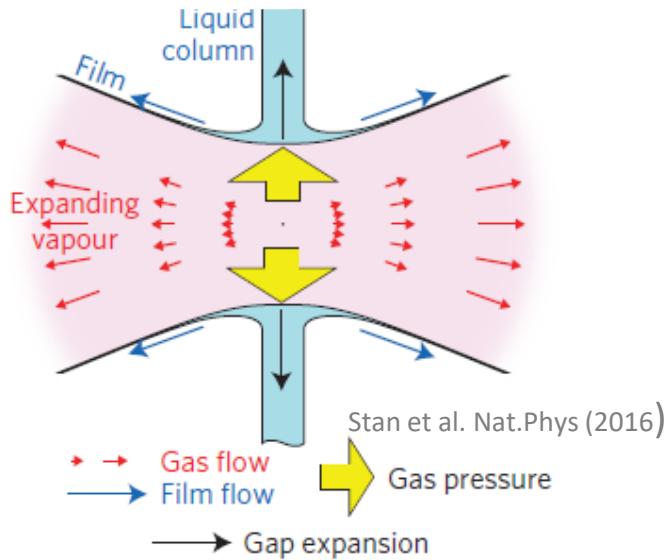
Shock wave structural dynamics, hydrodynamic simulations



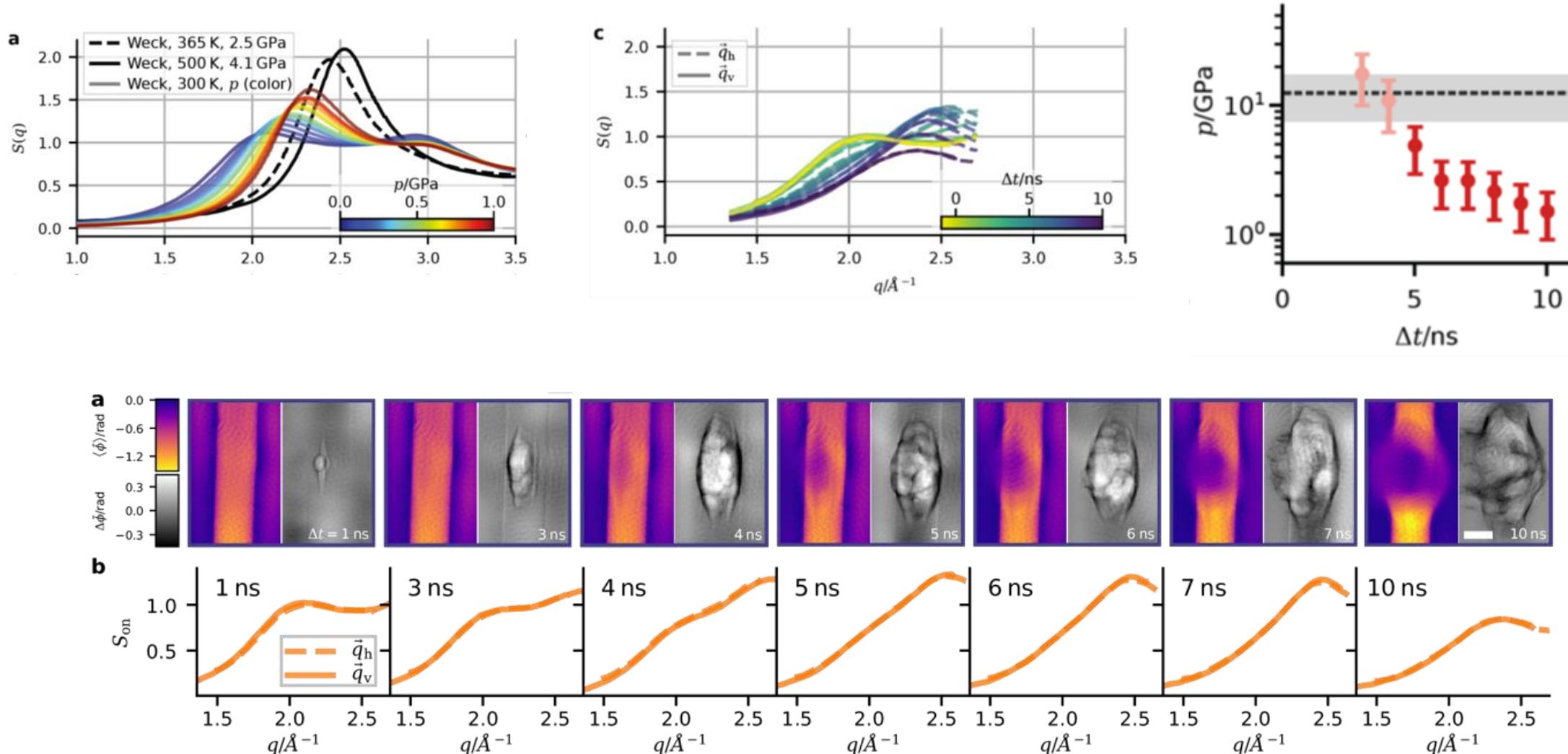
Shock wave structural dynamics, hydrodynamic simulations



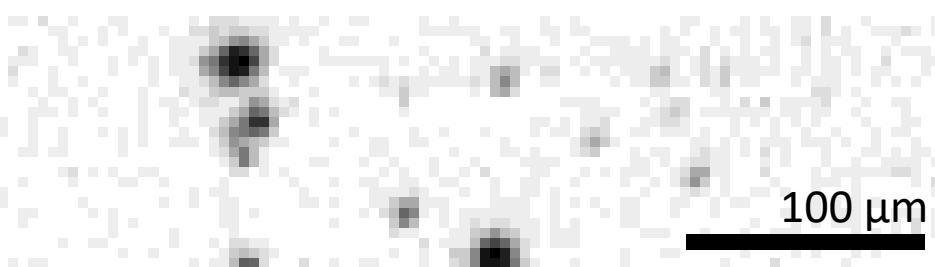
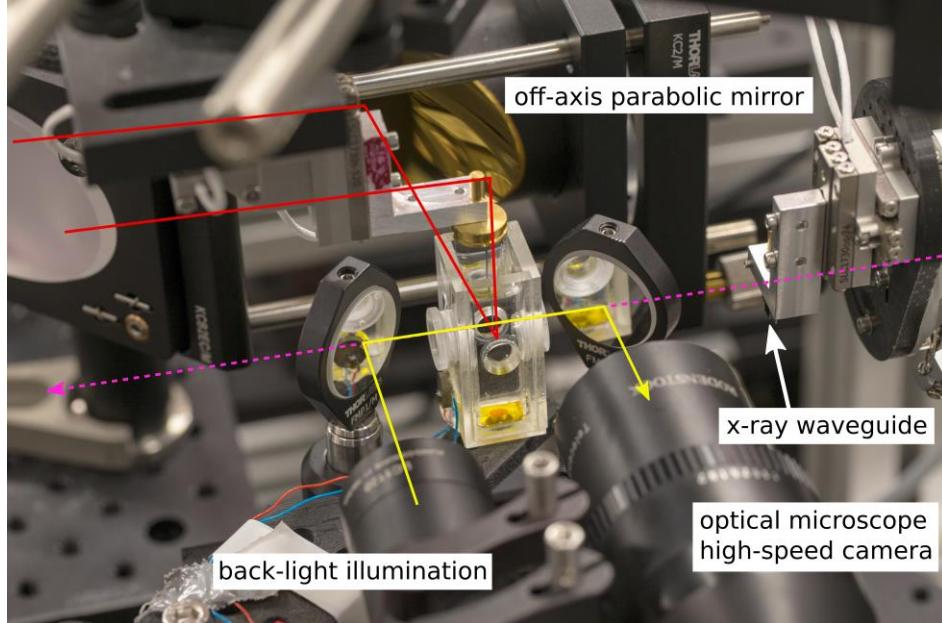
2. Cavitation in a liquid jet & molecular structure of water in the shockwave



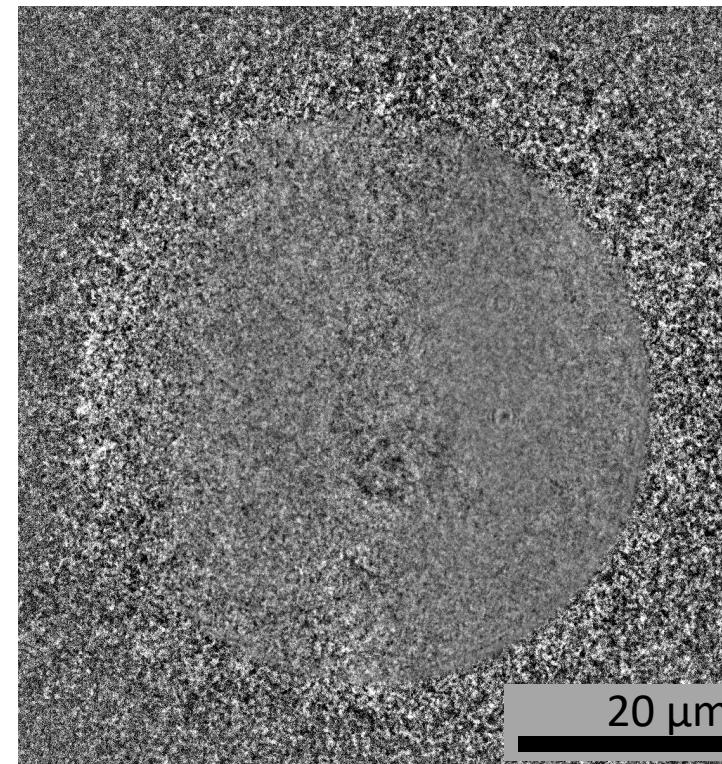
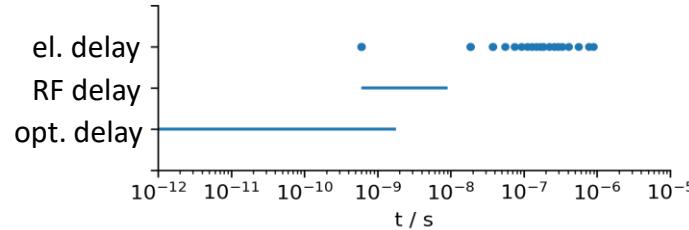
Holography-assisted recording and interpretation of the diffraction signal



3. Femtosecond laser-induced cavitation (p2807 10/2021, MID)



Optical high-speed video:
160 ns shutter, 480.000 fps



Electronic delay: 1200 ns ... 1 ns

MID fs-laser timing options:

- Electronic trigger (18 ns steps)
- RF synchronization system ($\sim 1 \text{ ps}$ steps)
- Optical delay line (fs precision)

X-ray parameters:

$E_{\text{ph}} = 18 \text{ keV}$
 $\tau < 100 \text{ fs}$
 $f_{\text{rep}} = 10 \text{ Hz}$
 $\langle E_p \rangle = 1 \text{ mJ}$

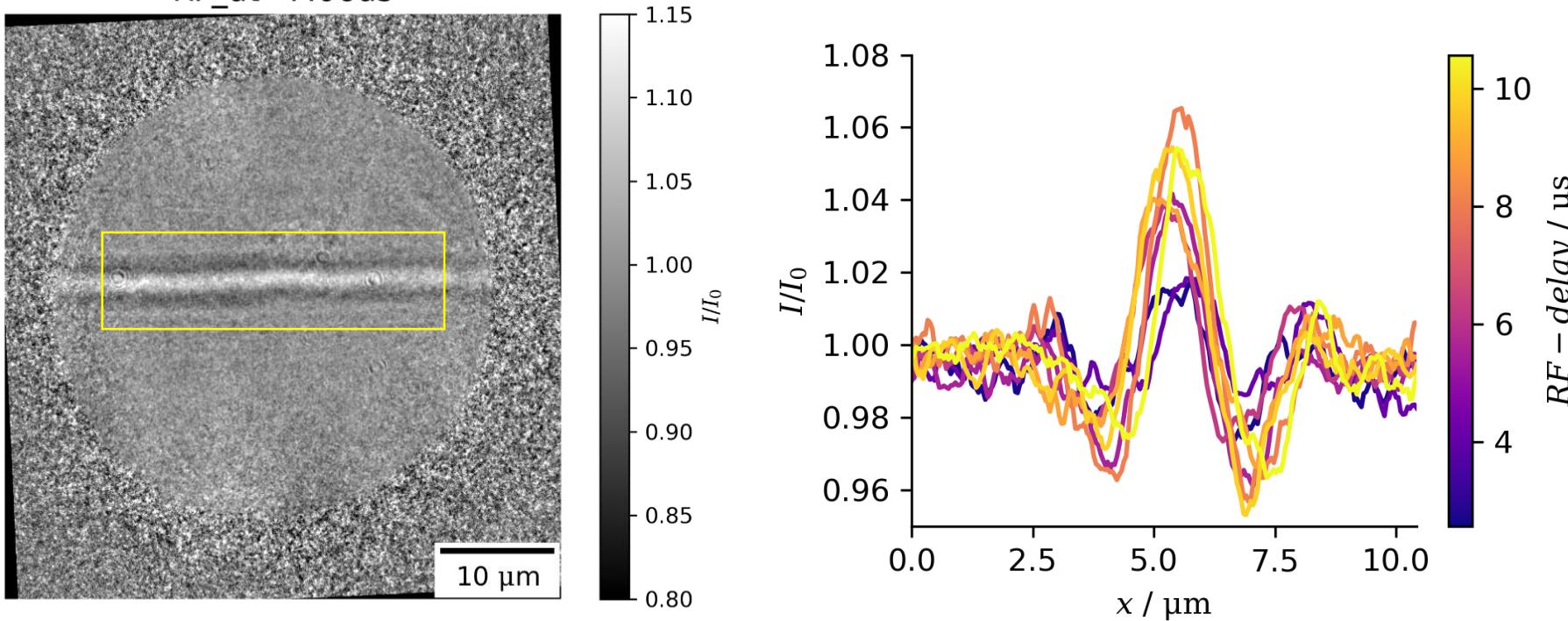
CRL Nanofocus:

$M = 95.2$
 $z_{01} = 102.5 \text{ mm}$
 $z_{12} = 9756 \text{ mm}$
 $p_{\text{eff}} = 68.23 \text{ nm}$

IR Pump laser:

$\lambda = 800 \text{ nm}$
 $\tau \approx 60 \text{ fs}$
 $f_{\text{rep}} = 10 \text{ Hz}$
 $\langle E_p \rangle = 3 \mu\text{J} \dots 120 \mu\text{J}$

Analysis (ongoing)



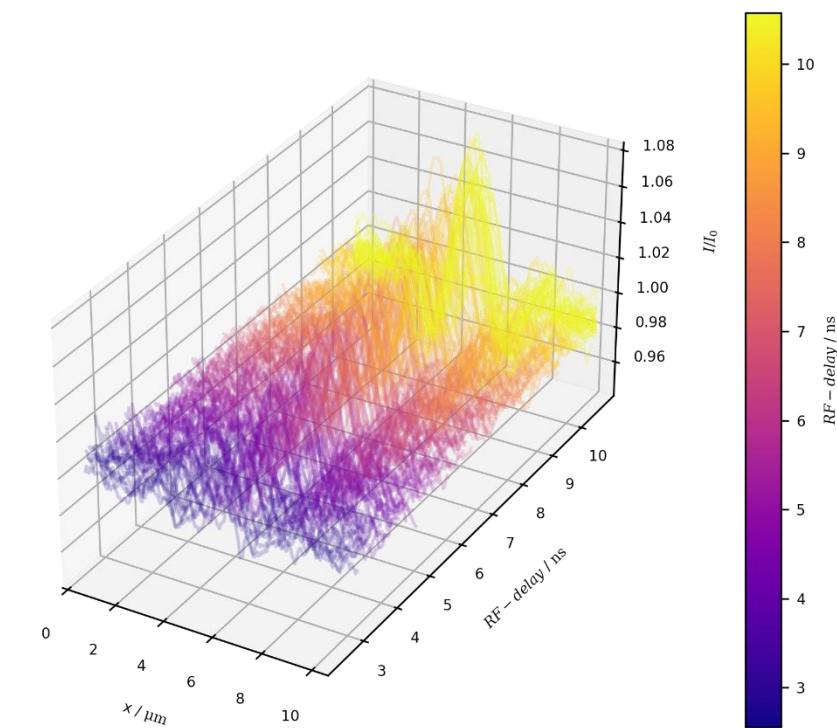
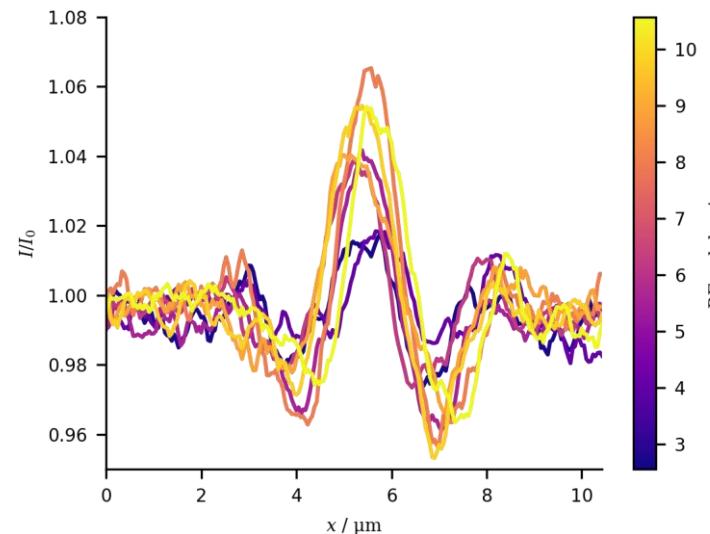
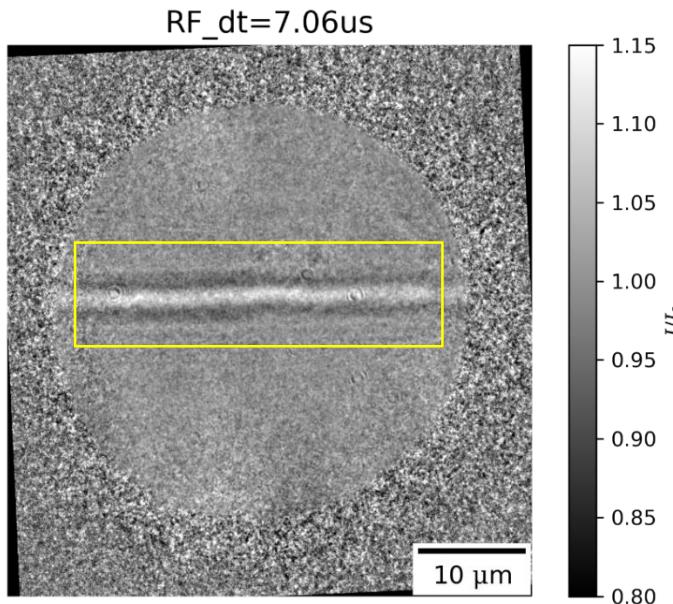
Quantitative phase reconstruction:

- Alternating projections (AP) (2d)
- adapt radially fitted phase (RFP) for cylindrical symmetry

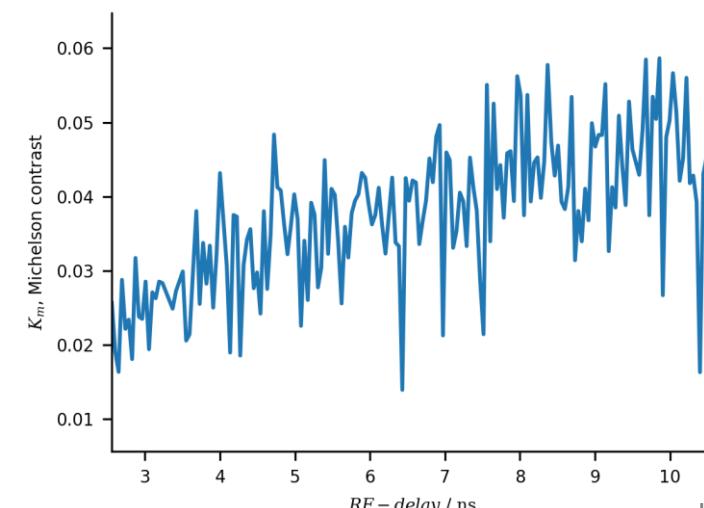
Investigation of:

- optical breakdown
- filamentation
- plasma to vapour transition
- birth of cavitation bubble
- plasma and hydrodynamic simulations...

plasma to bubble transition

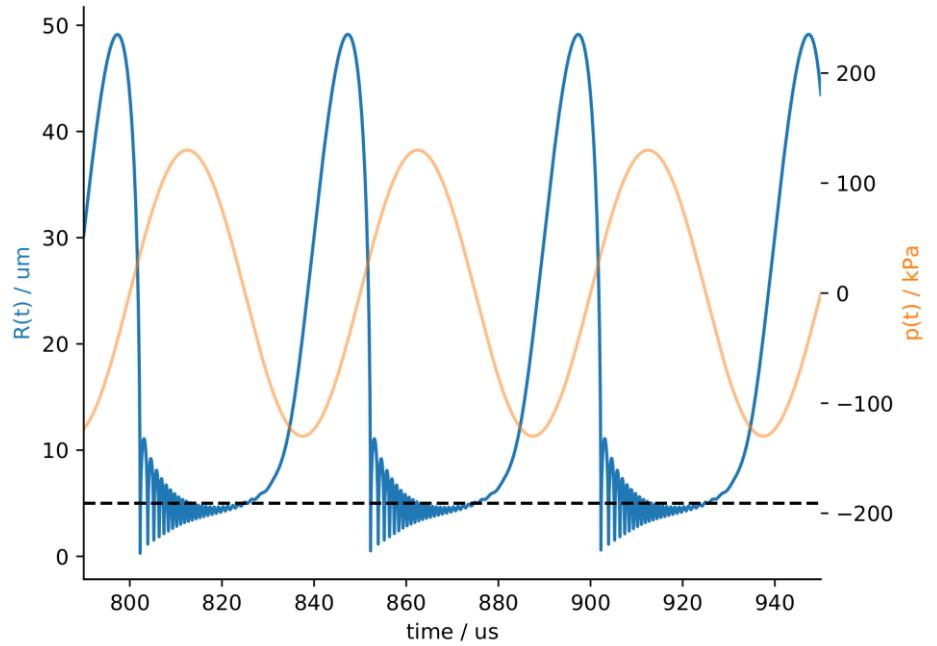


- **μs to ns dynamics:** (slow) bubble growth
- Shock wave emission?
- **$\text{ns} & \text{ps}$ dynamics:** contrast increase (plasma cooling, recombination & transition to vapour cavity)
- -> laser surgery ! study of cavitation in tissues



Hoeppel et al., (in preparation)

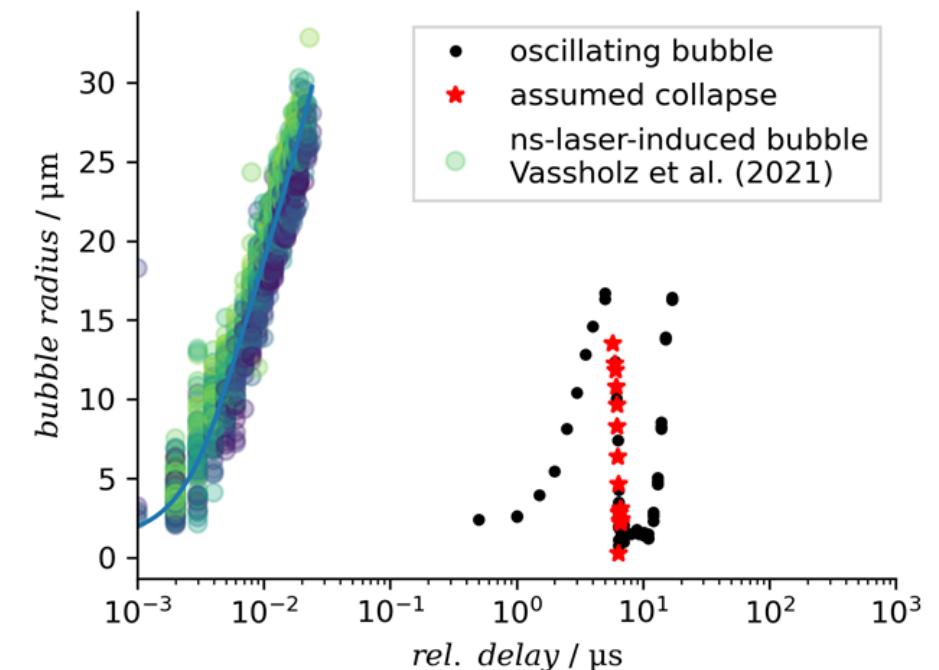
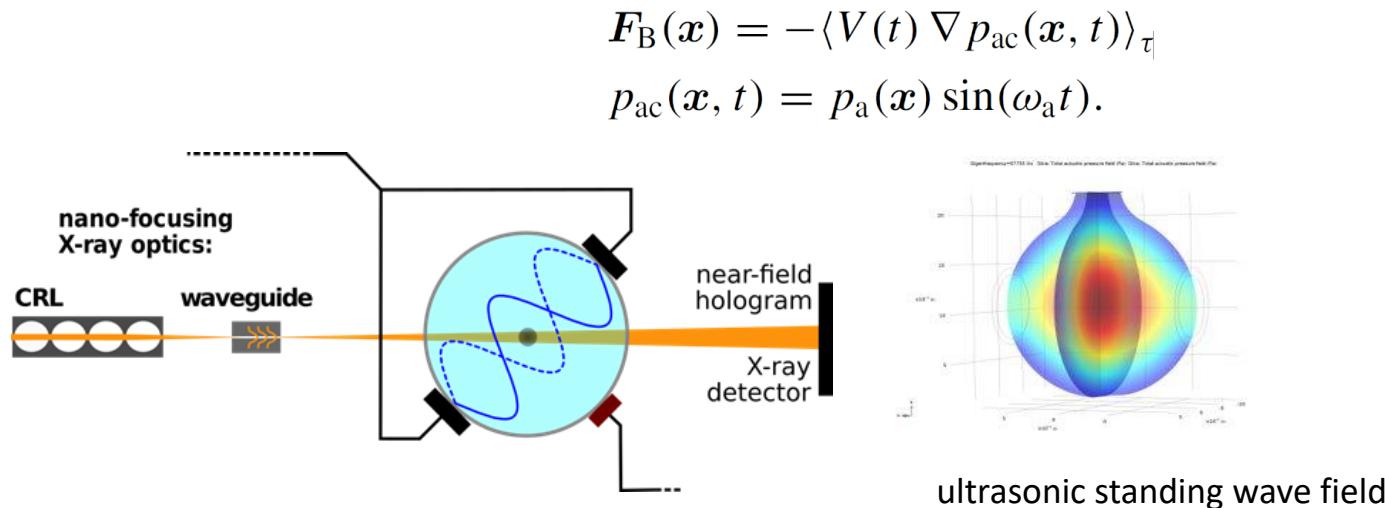
3. Acoustic cavitation and trapping of a bubble



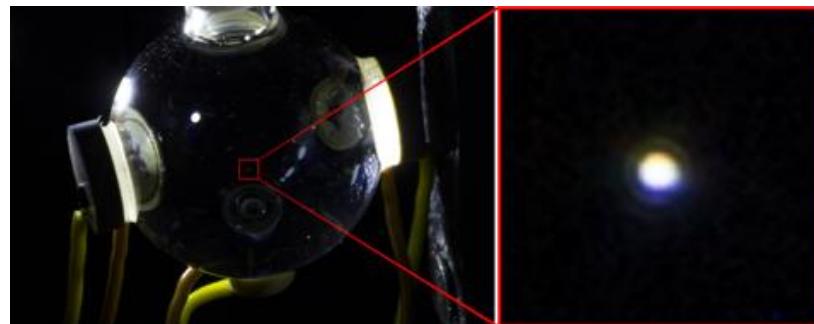
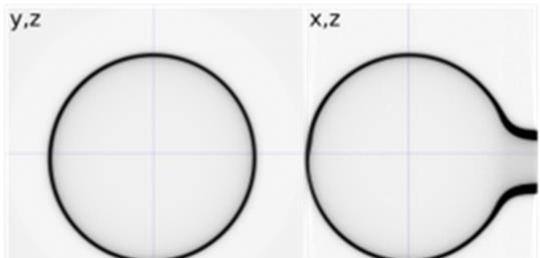
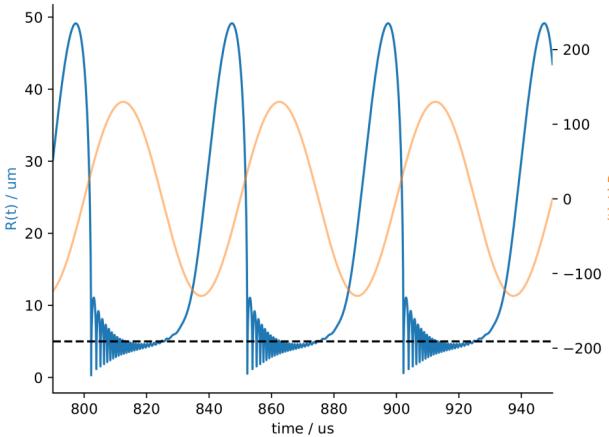
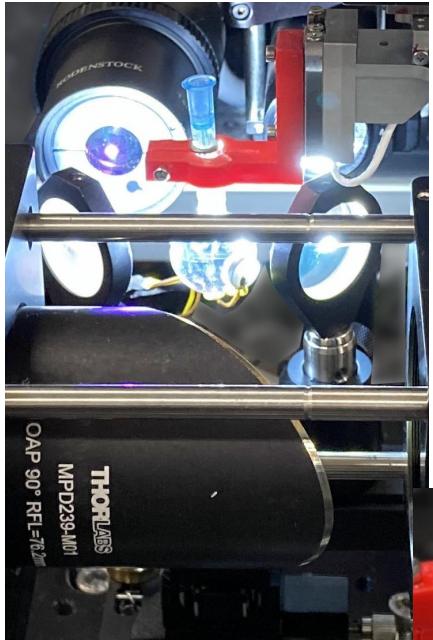
$$R \ddot{R} + \frac{2}{3} \dot{R}^2 = \frac{1}{\rho} \left(p_i - p_{stat} - \frac{2\sigma}{R} - \frac{4\eta}{R} \dot{R} \right)$$

Main requirements for stable trapping:

- positional stability \leftrightarrow Bjernkes force
- Spherical stability \leftrightarrow surface modes
- Diffusional stability \leftrightarrow rectified diffusion



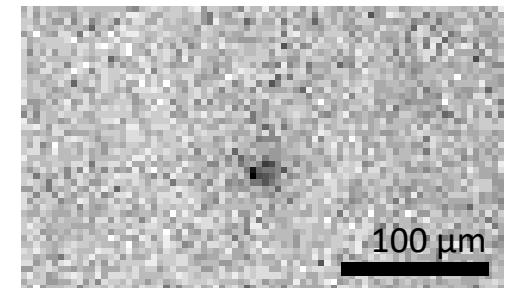
Imaging the bubble collapse – acoustic trapping & SBSL



resonance chamber: d=19 mm cuvette with a trapped bubble at f=88 kHz

The non-linear bubble oscillator:

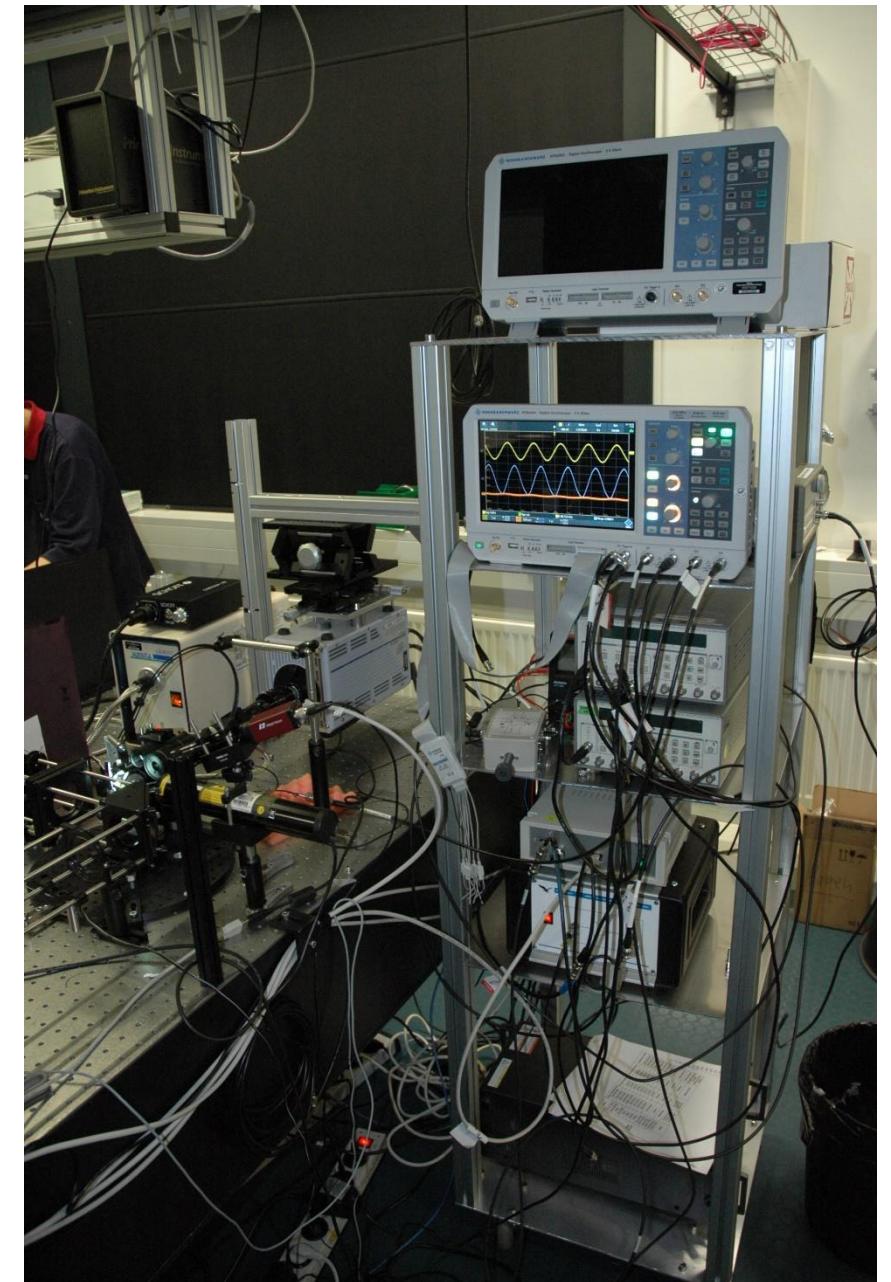
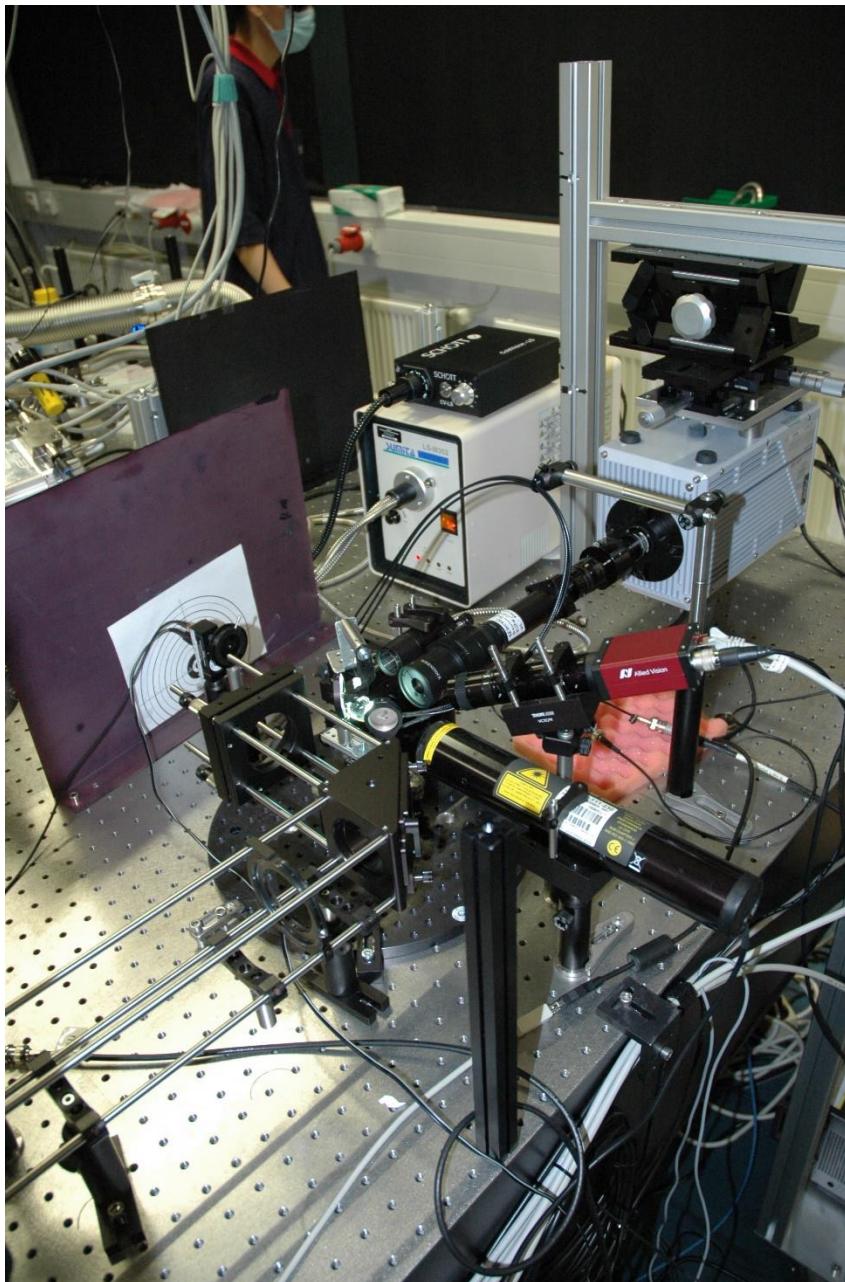
- **Expansion** to maximum radius, e.g. $\sim 50\mu\text{m}$
- **Collapse**: to extremely compressed bubble
 - $T \approx 4.000 - 10.000 \text{ K}$
 - Shock wave emission, $\approx 90\%$ energy loss
 - Compression to almost liquid-like densities
 - Chemical dissociation reactions
 - **sonoluminescence, thermal bremsstrahlung & radiative recombination?**
- after-bounces and repeated expansion



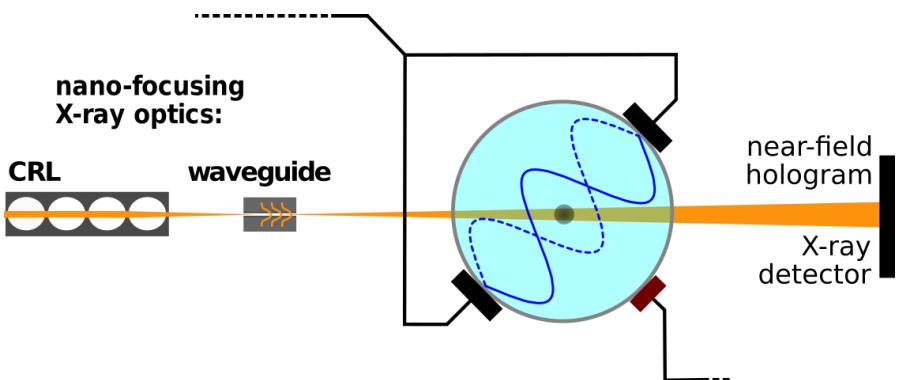
160 ns shutter, 480.000 fps
 $\sim 88 \text{ kHz}$ ultrasonic driving

In-house setup

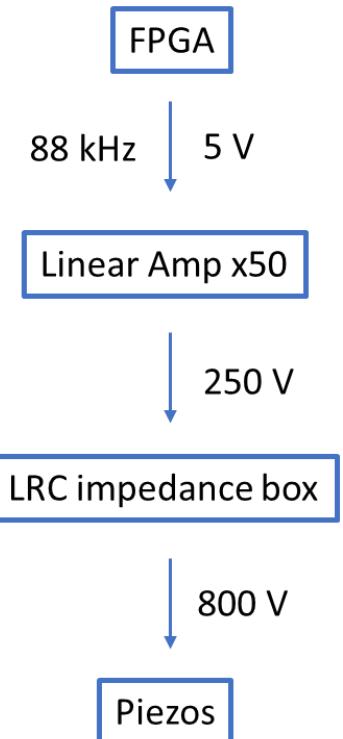
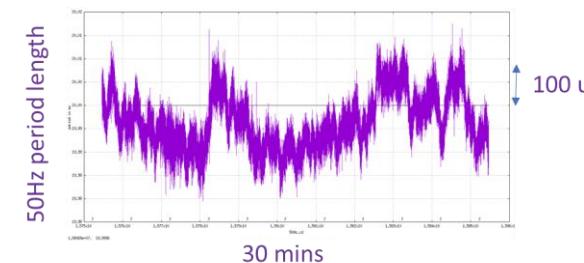
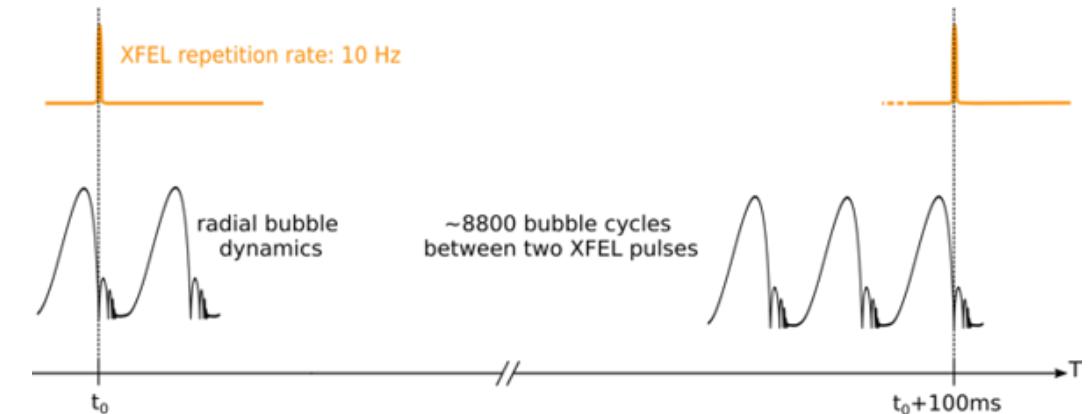
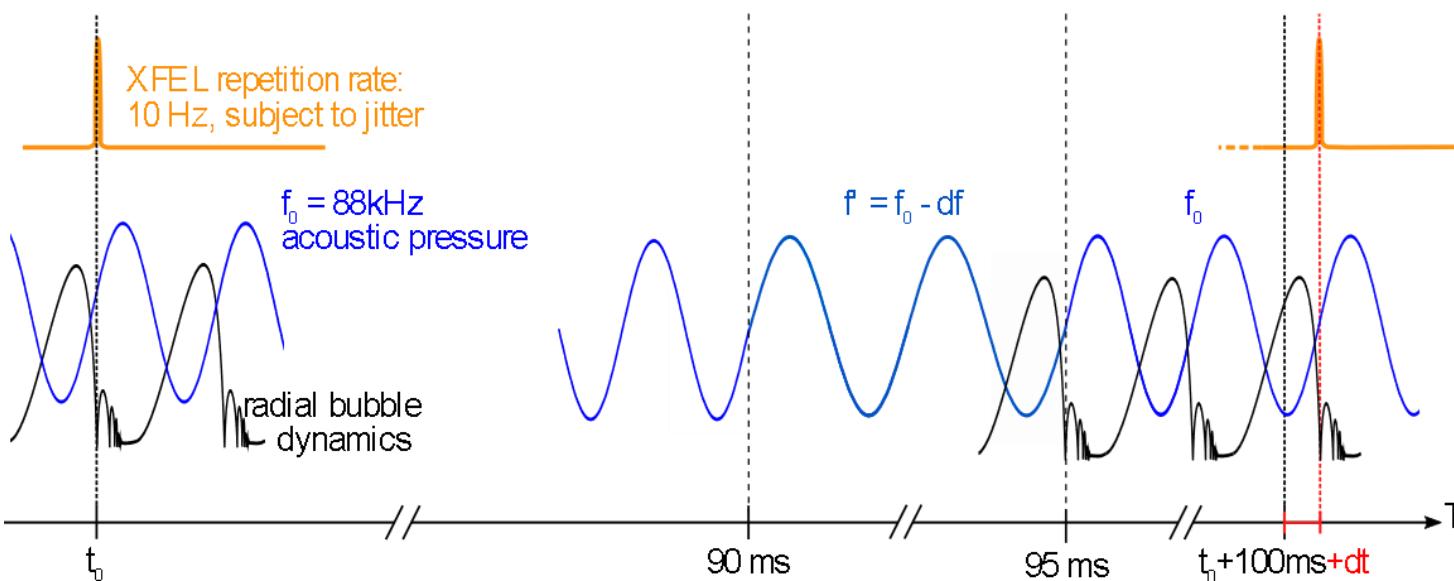
PXS lab:
TiSa fs laser (Coherent)
Acoustics & timing



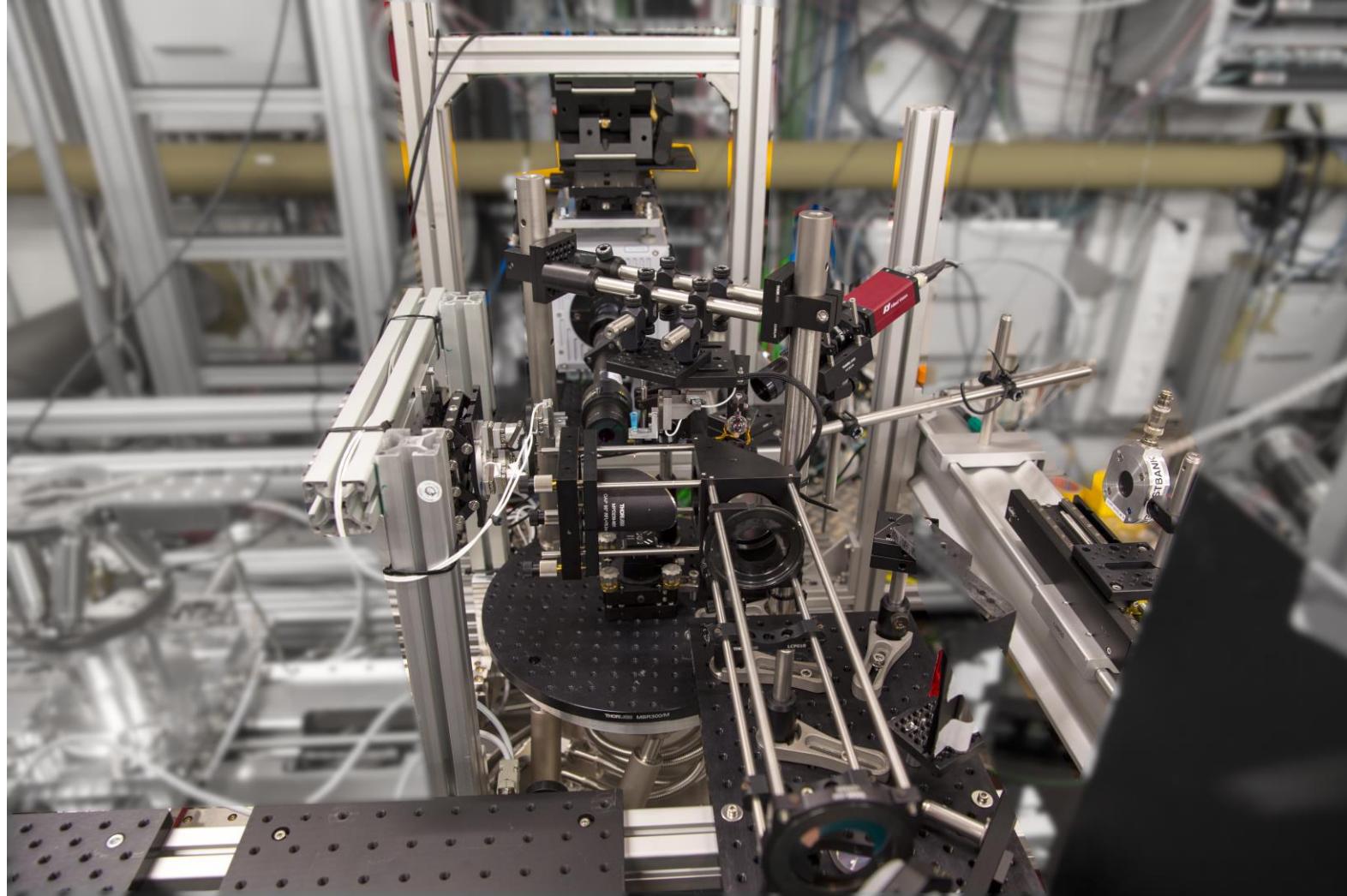
Acoustic synchronization & timing



challenge: bunchclock synchr. to the mains: *jitter!*



XFEL beamtime p2807 10/21: Experimental parameters at MID



X-ray parameters:

$E_{\text{ph}} = 18 \text{ keV}$

$\tau < 100 \text{ fs}$

$f_{\text{rep}} = 10 \text{ Hz}$

$\langle E_p \rangle = 1 \text{ mJ}$

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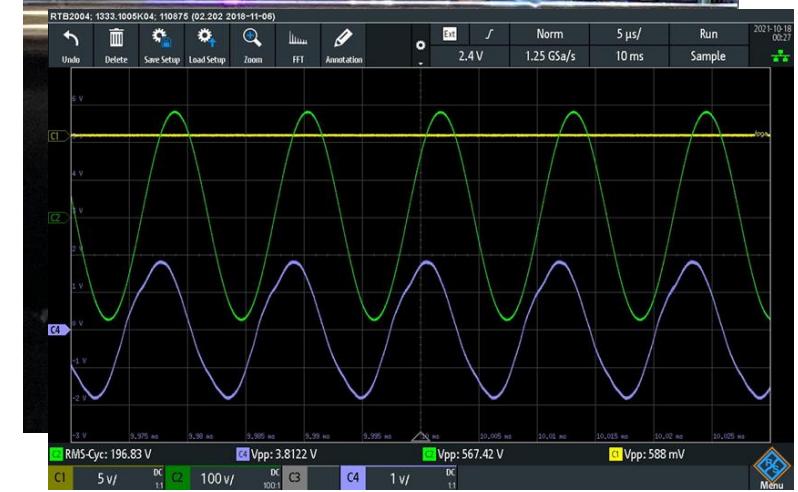
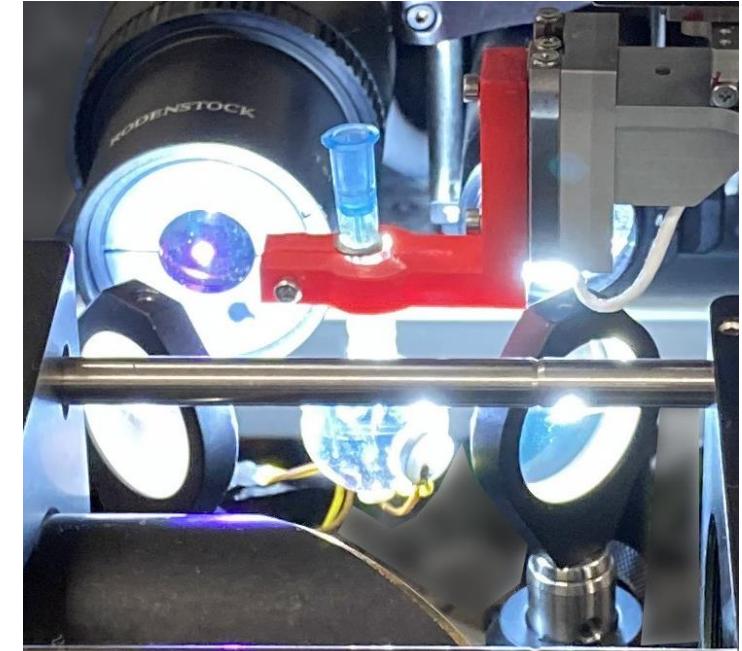
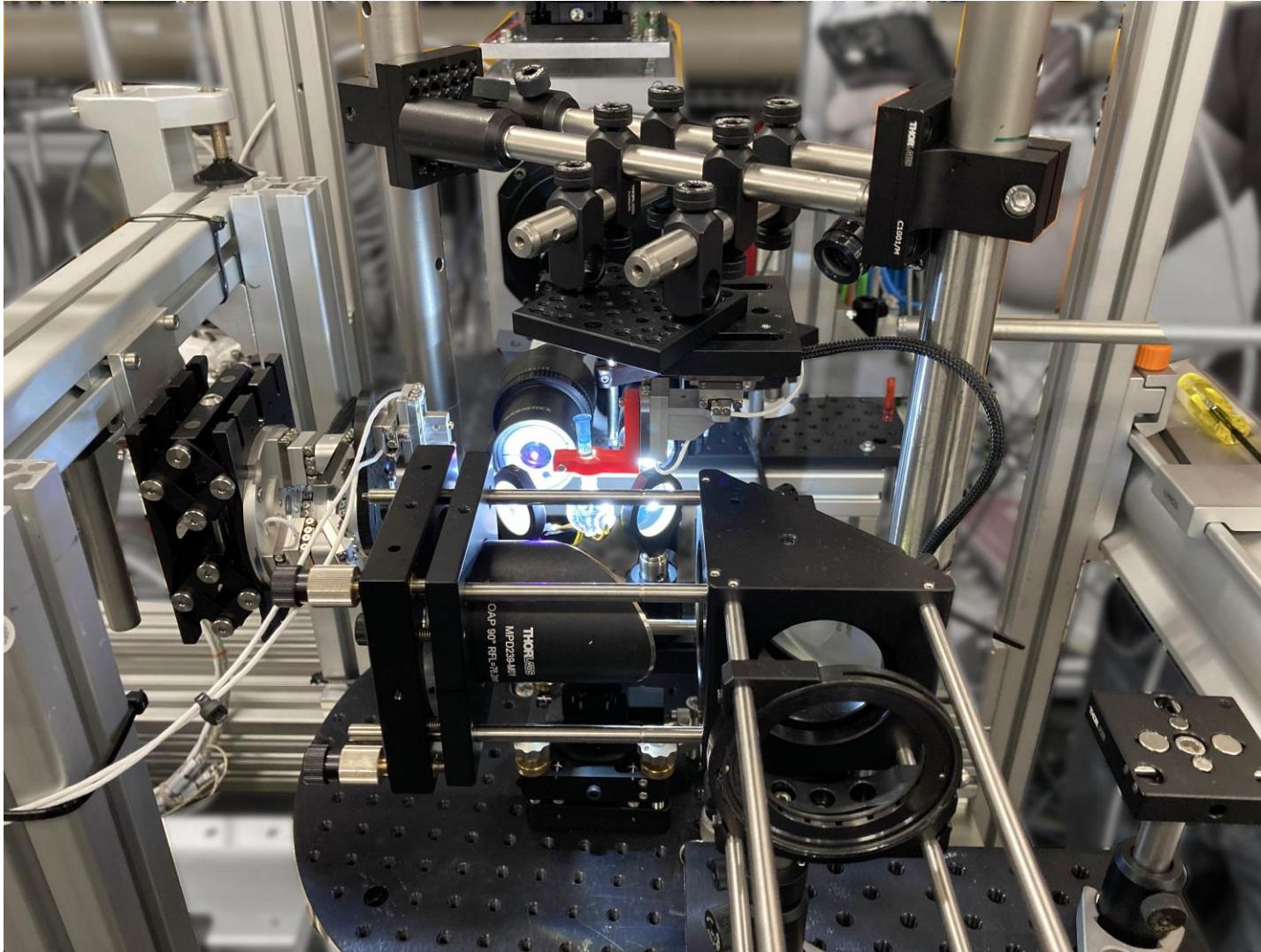
$\lambda = 800 \text{ nm}$

$\tau \approx 60 \text{ fs}$

$f_{\text{rep}} = 10 \text{ Hz}$

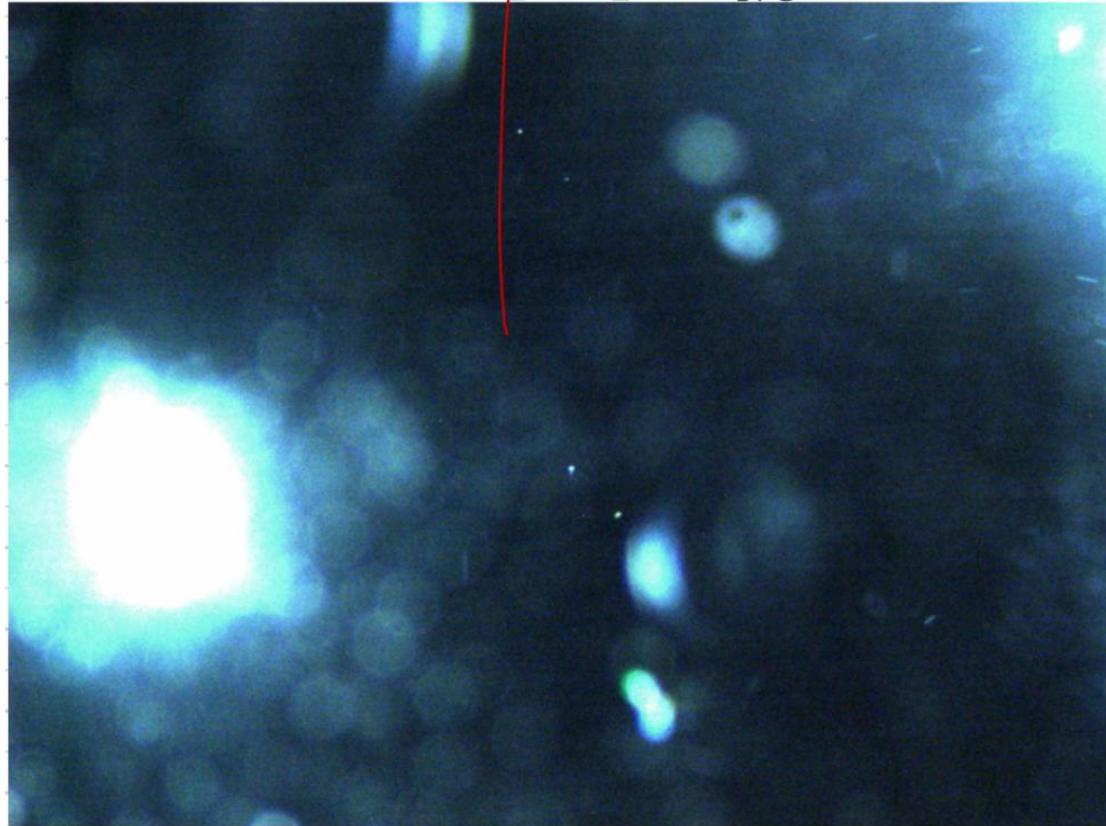
$\langle E_p \rangle = 3 \mu\text{J} \dots 120 \mu\text{J}$

Experimental Setup for acoustic trapping installed at MID



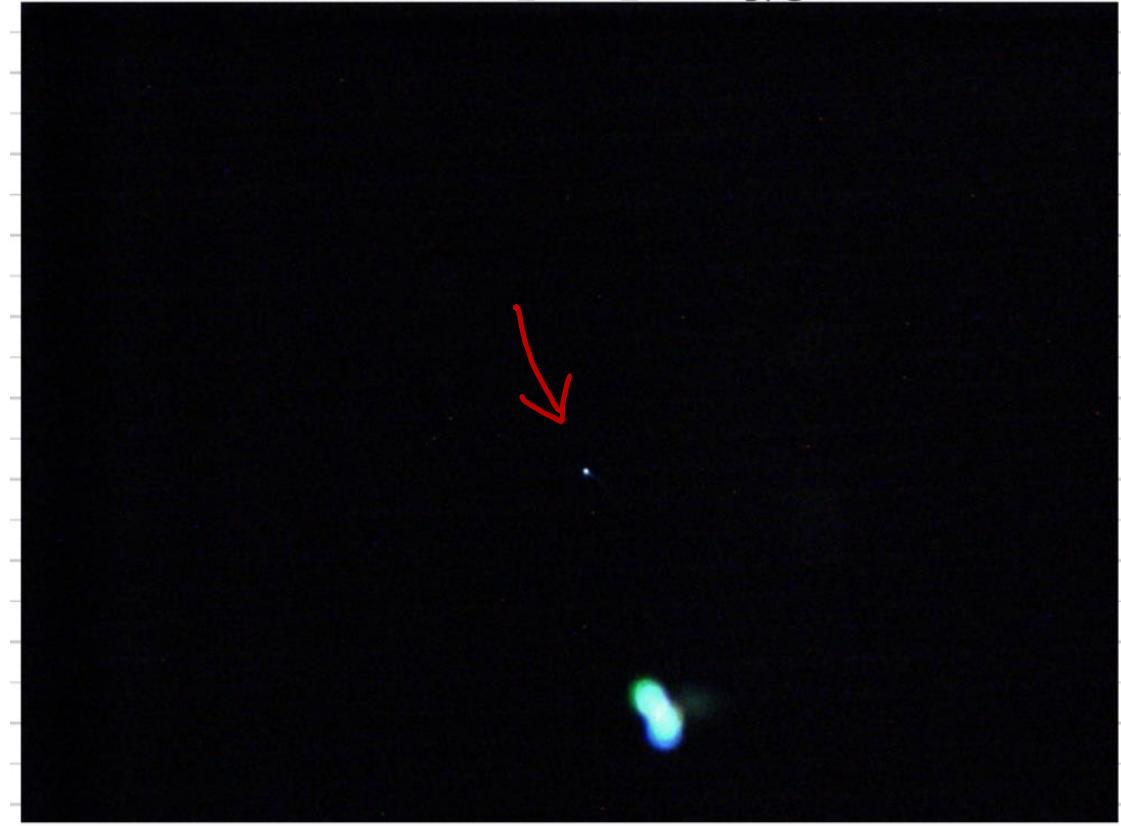
Live observation of luminescence

camera: cavi, file: init_cavi_0000.jpg



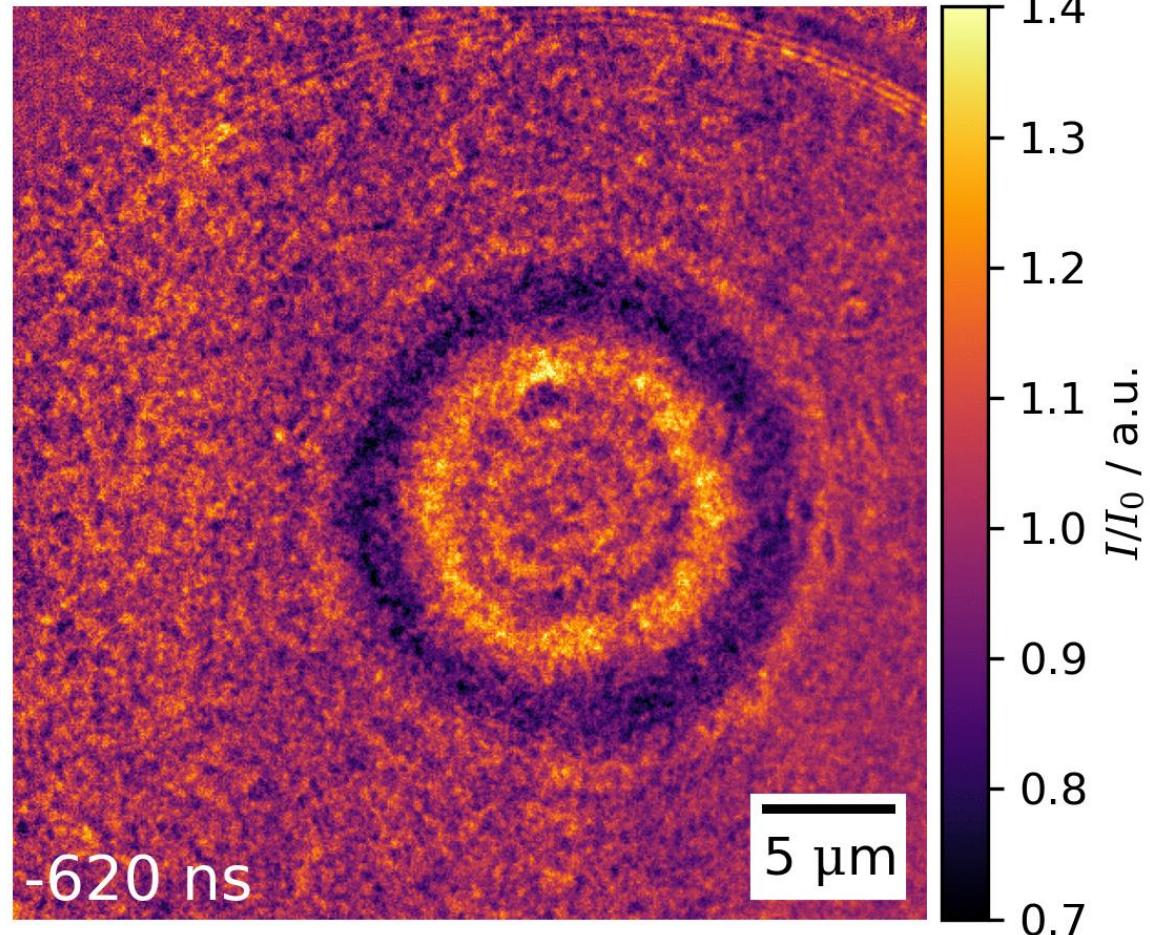
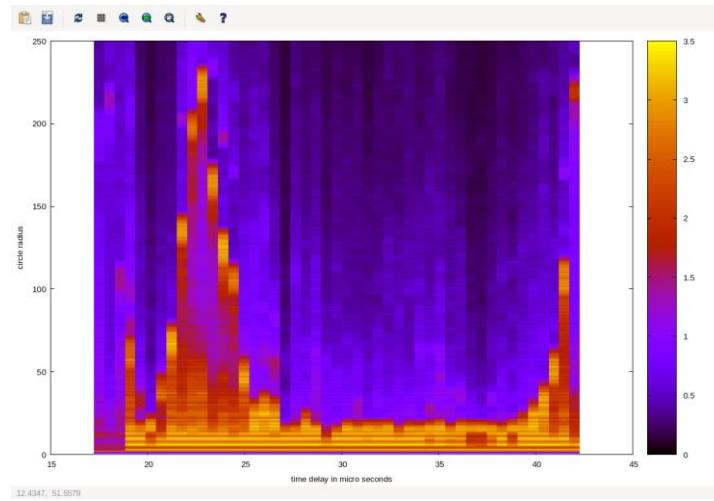
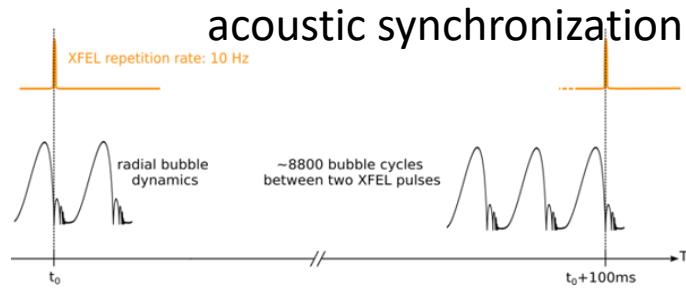
Sun Oct 17 23:14:21 2021

camera: cavi, file: init_cavi_0000.jpg

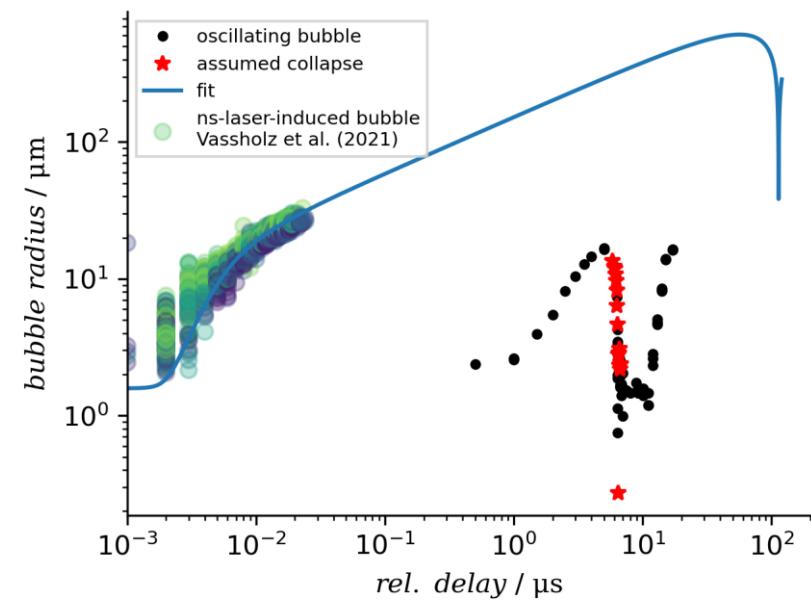
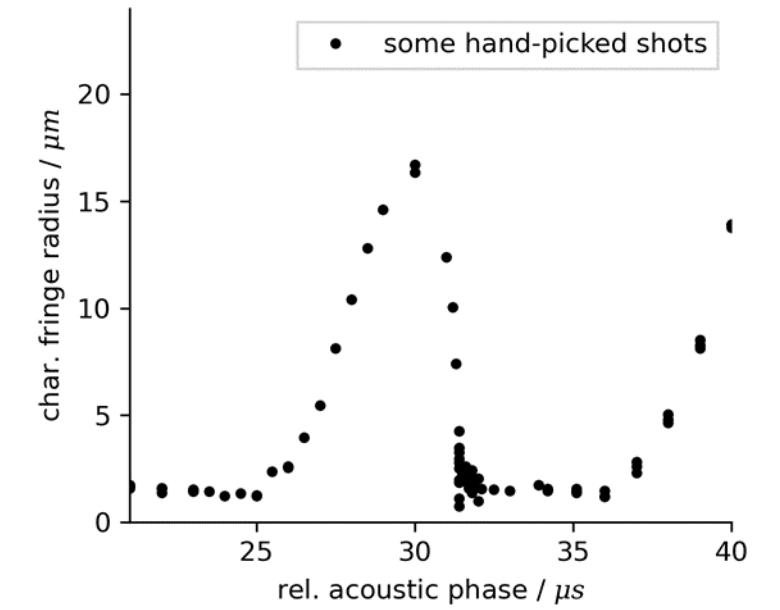
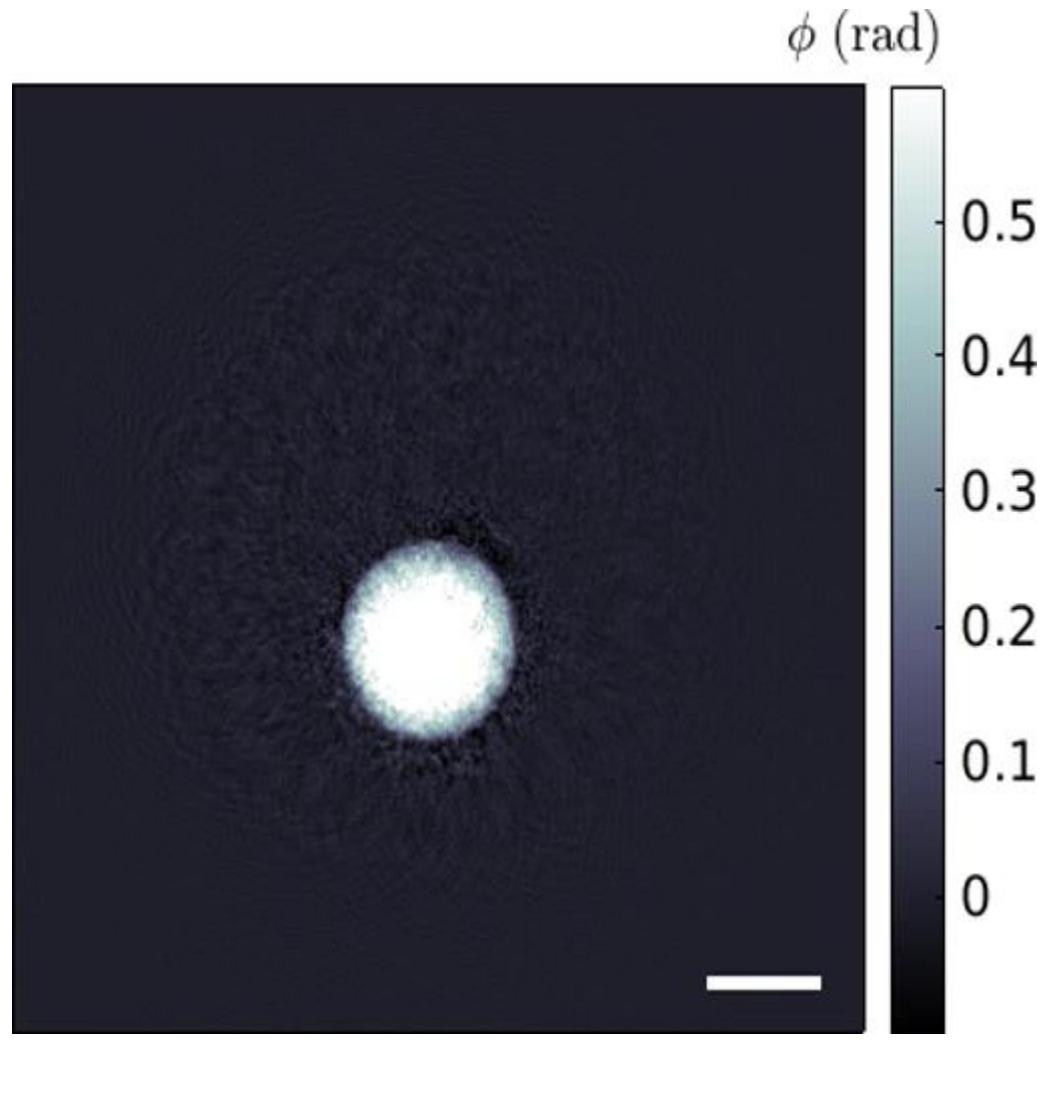


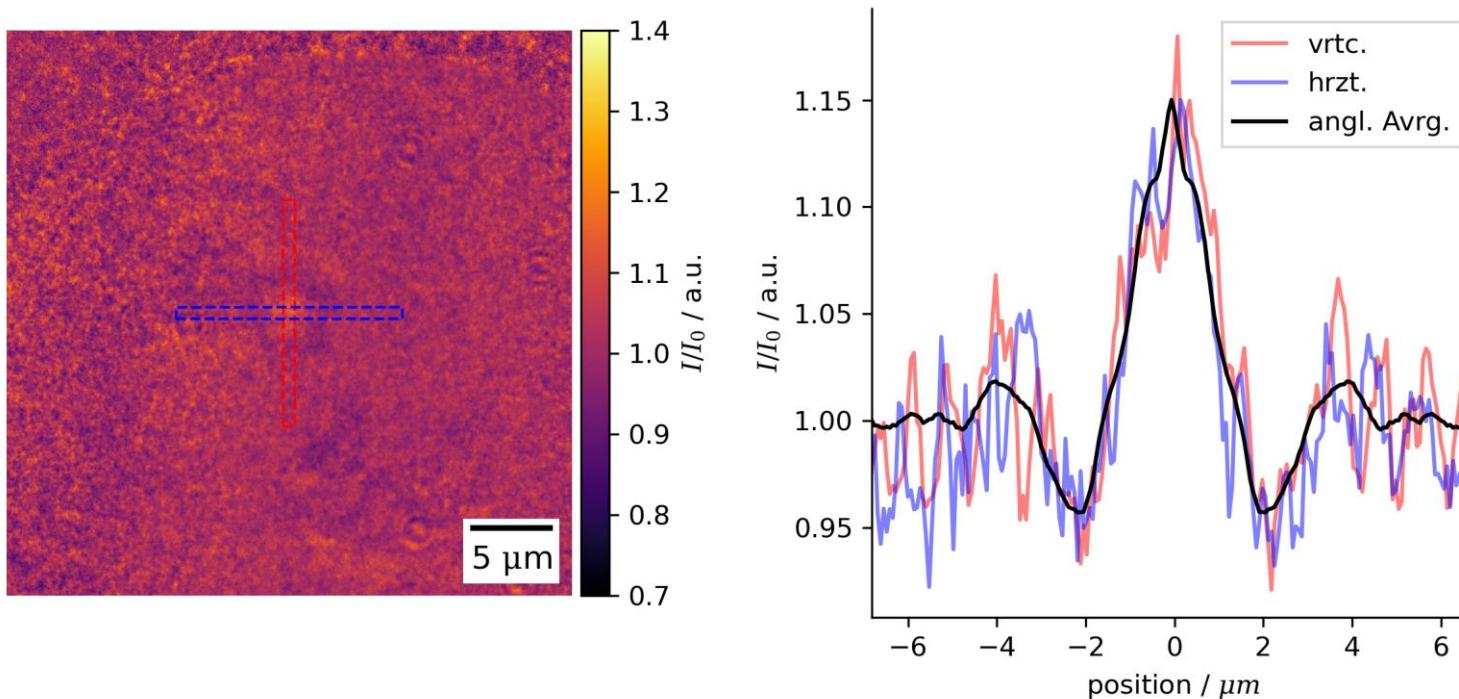
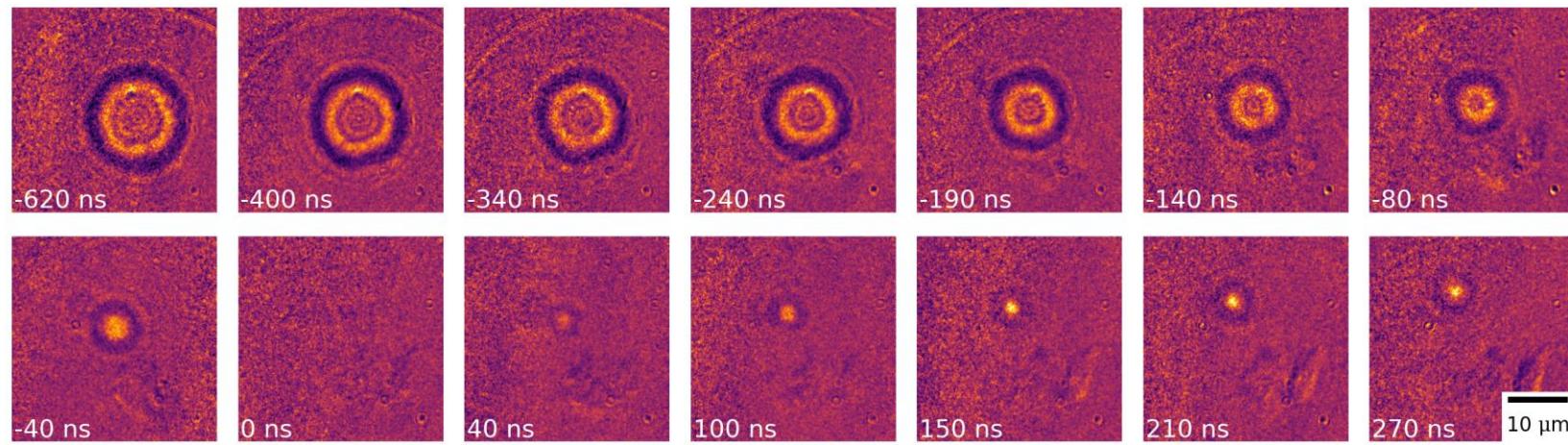
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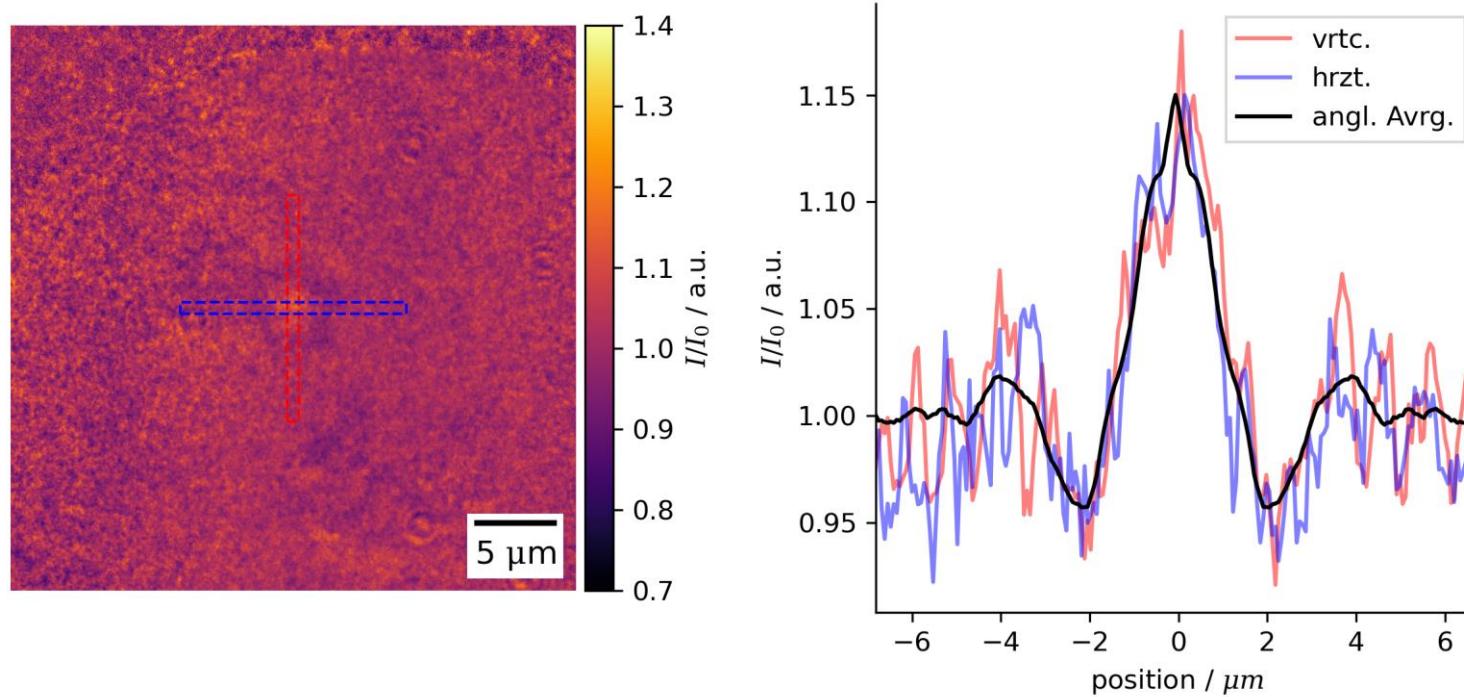
First results: sampling of the collapse



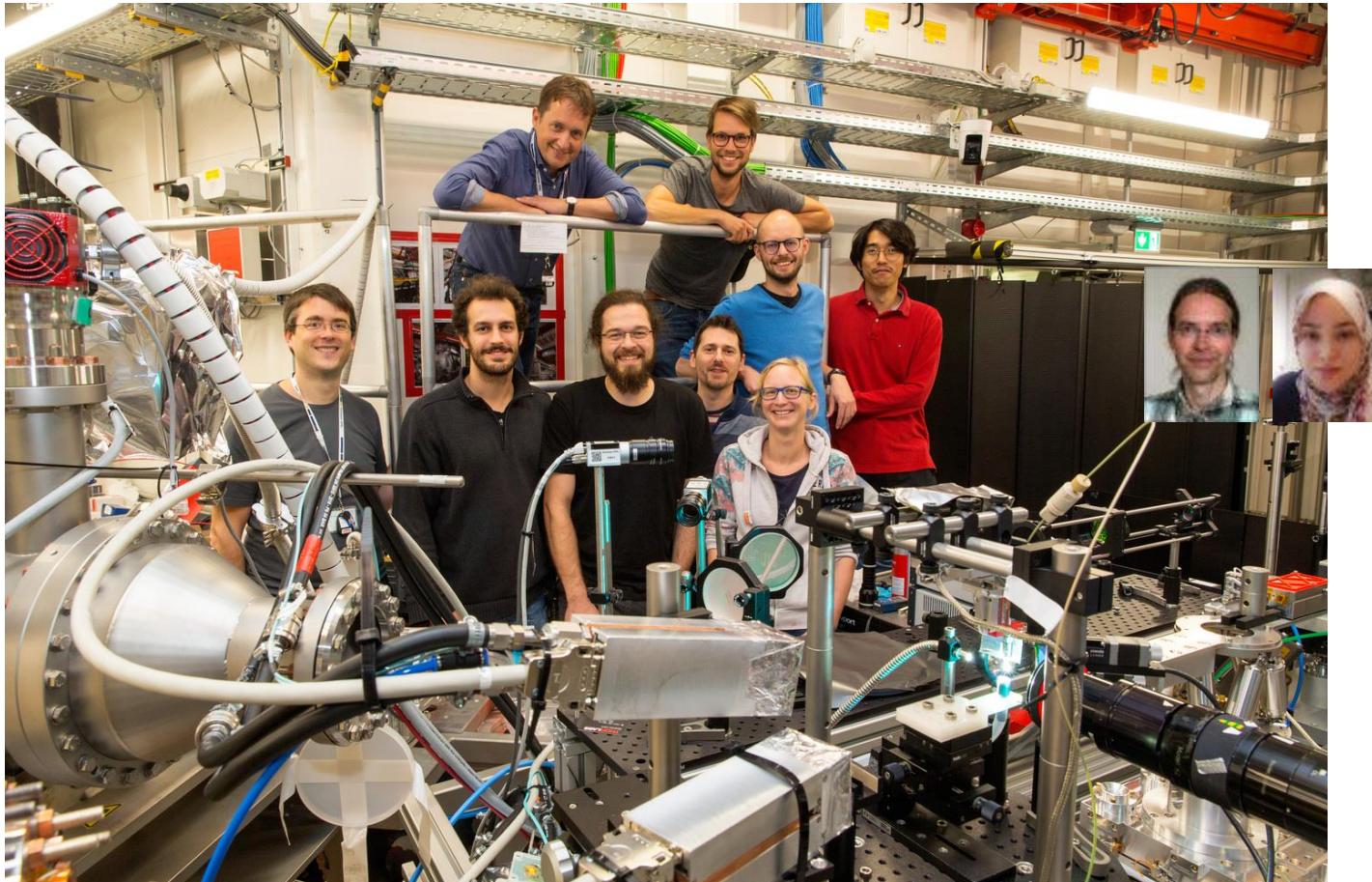
First results: phase reconstruction







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IRP, Uni Göttingen:
Hannes Paus Hoeppe
Malte Vassholz, Markus
Osterhoff, Peter Luley, Jan
Goemann, Tim Salditt



DPI, Uni Göttingen: Juan M.
Rosselló, Atiye Aghelmaleki,
Robert Mettin

DESY: Johannes Hagemann,
Thea Engler, Andreas Schropp,
Christian G. Schroer

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thanks for an amazing instrument
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