

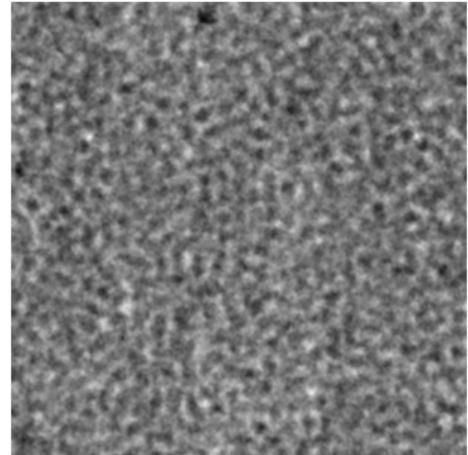
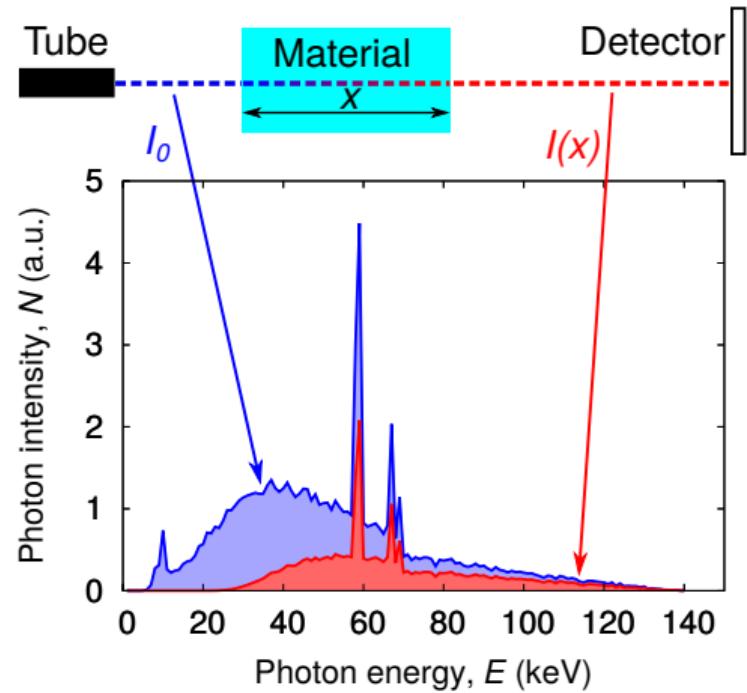


## PhD defense **Manuel Baur**

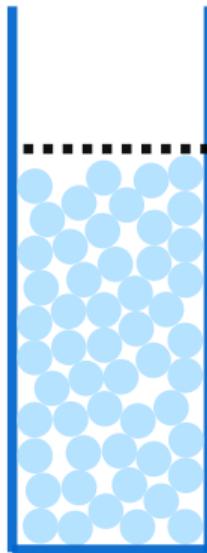
Funded by the German  
Federal Ministry for  
Economic Affairs and  
Energy, grant no. 50WM

1653

# X-ray radiography of granular systems – particle densities and dynamics

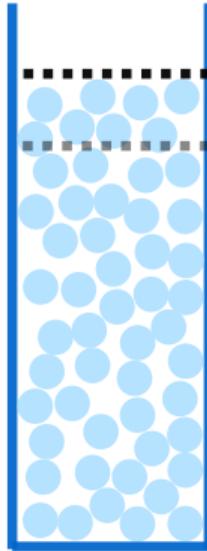


# X-ray radiography of granular systems – particle densities and dynamics



$$\Phi = \text{RLP}$$

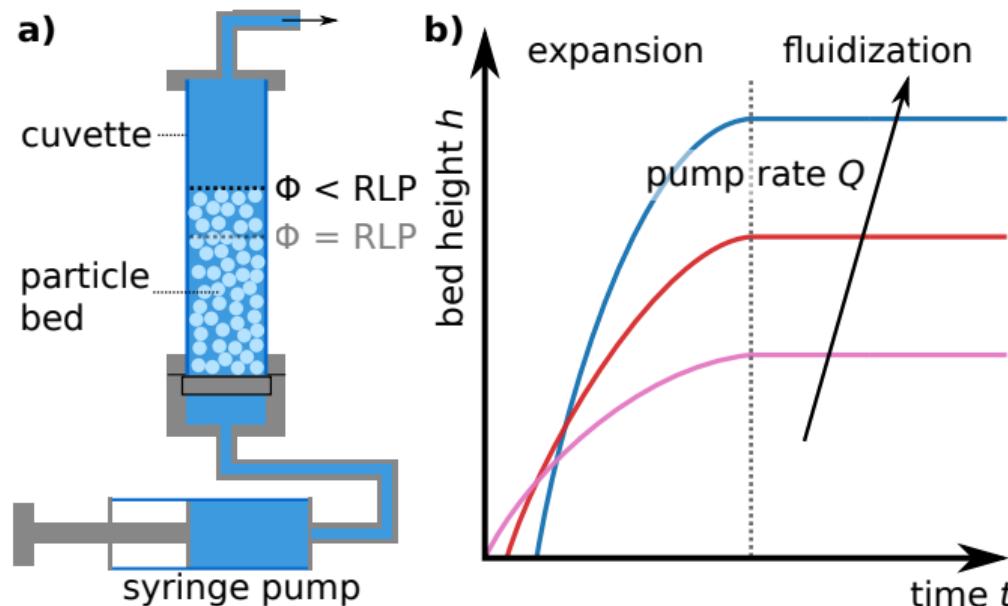
# X-ray radiography of granular systems – particle densities and dynamics



$\Phi < \text{RLP}$   
 $\Phi = \text{RLP}$

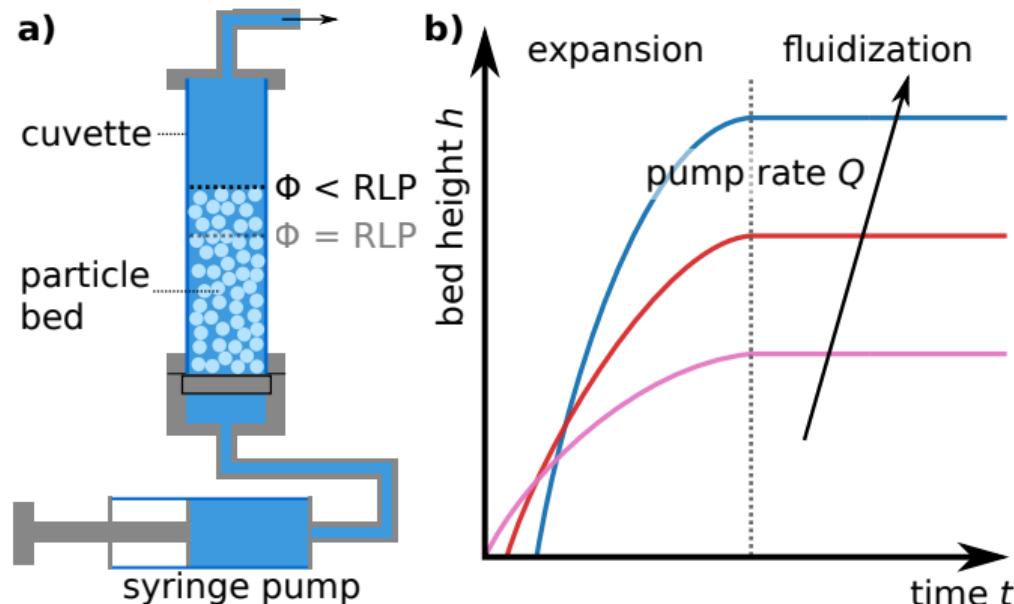
# X-ray radiography of granular systems – particle densities and dynamics

Fluidized bed



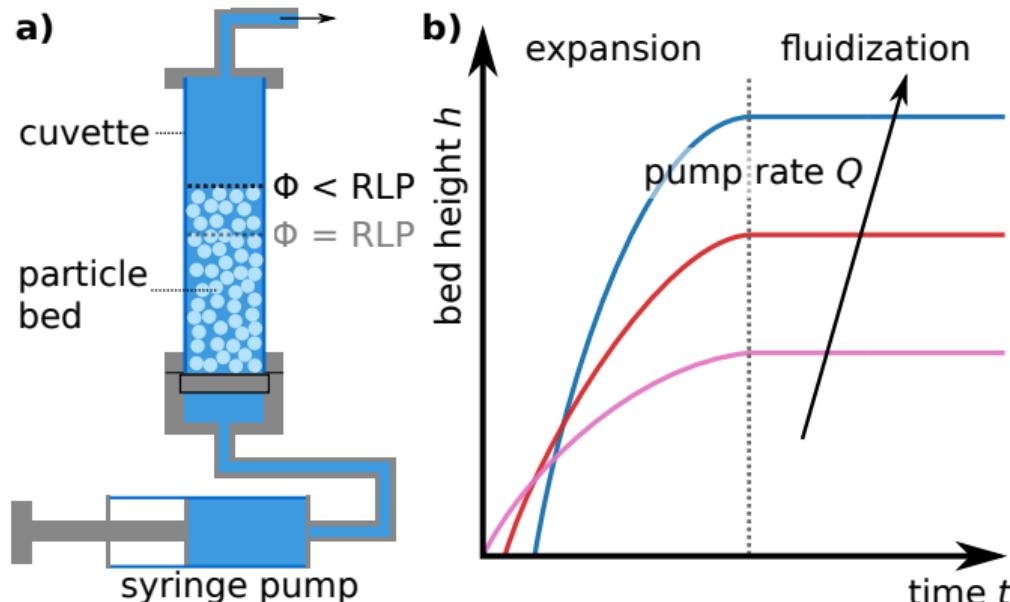
# X-ray radiography of granular systems – particle densities and dynamics

Fluidized bed



# X-ray radiography of granular systems – particle densities and dynamics

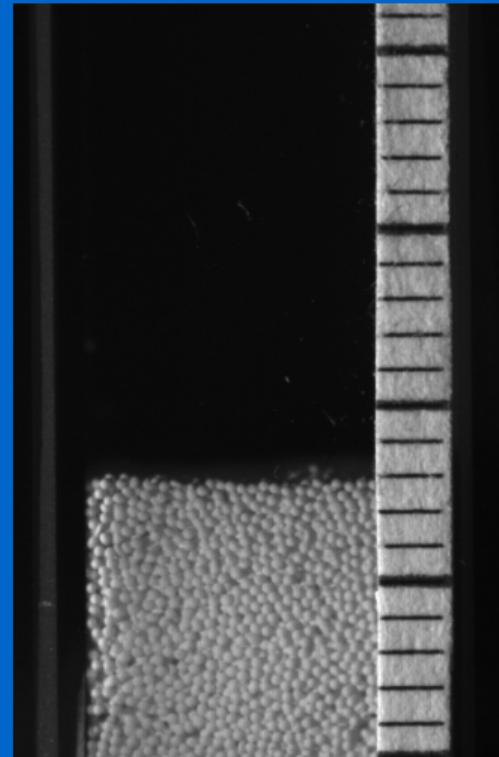
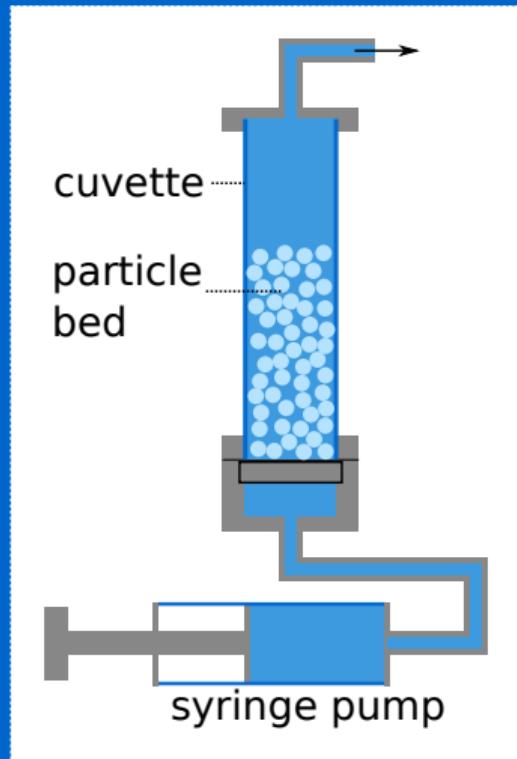
Fluidized bed



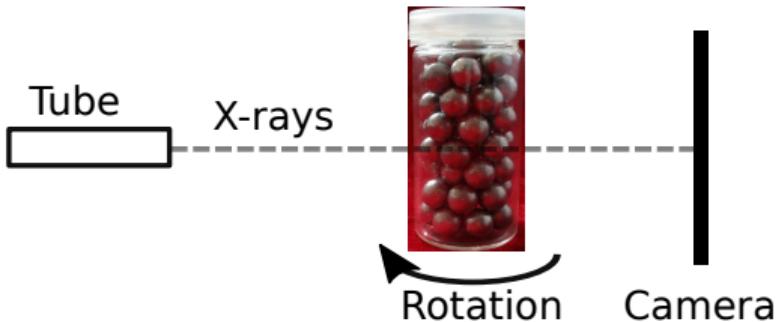
Fluidized bed reactor

**"Lack of understanding:**  
It is very difficult to predict and calculate the **complex mass** [...] **flows** within the bed."

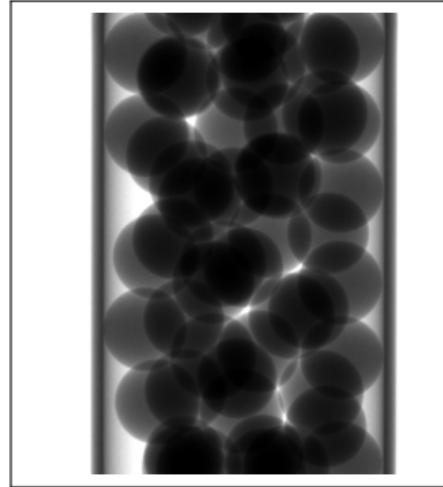
# Particulate flows are **opaque**



# X-ray radiography

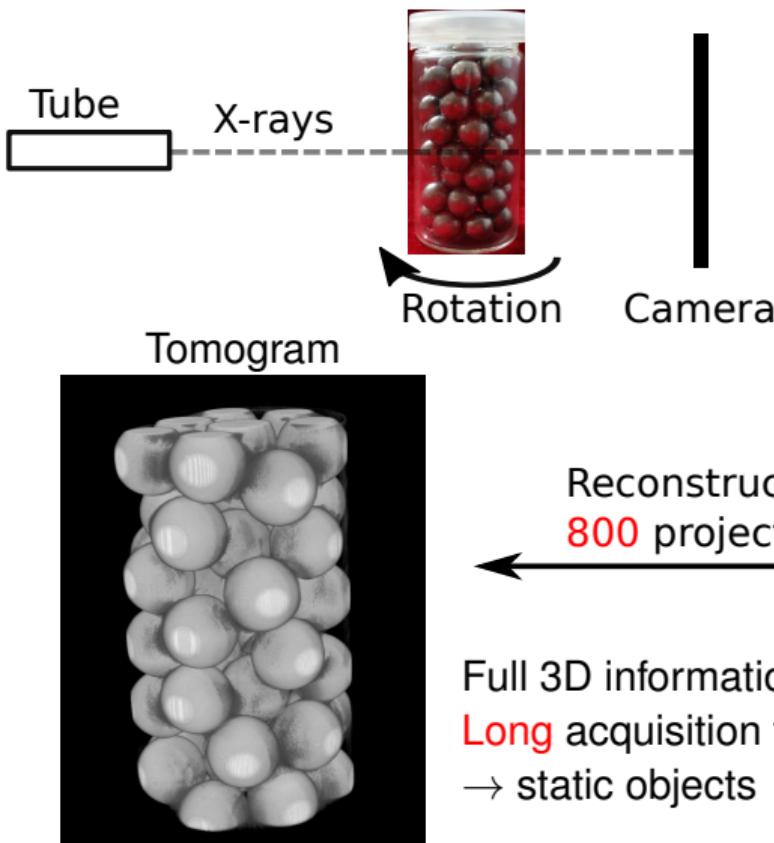


Radiogram

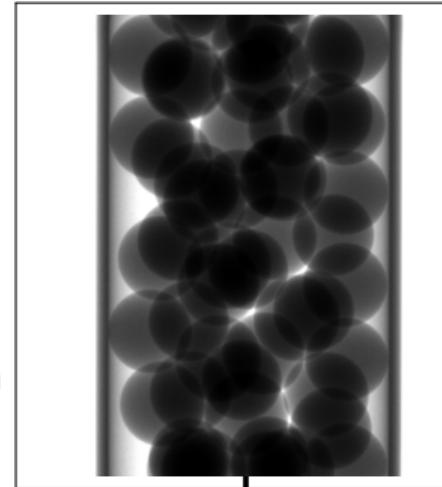


2D projections of 3D object  
Short acquisition time

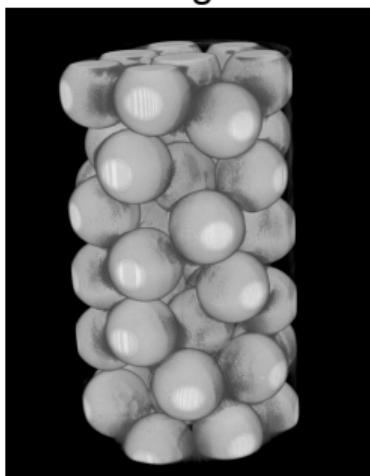
# X-ray radiography



Radiogram



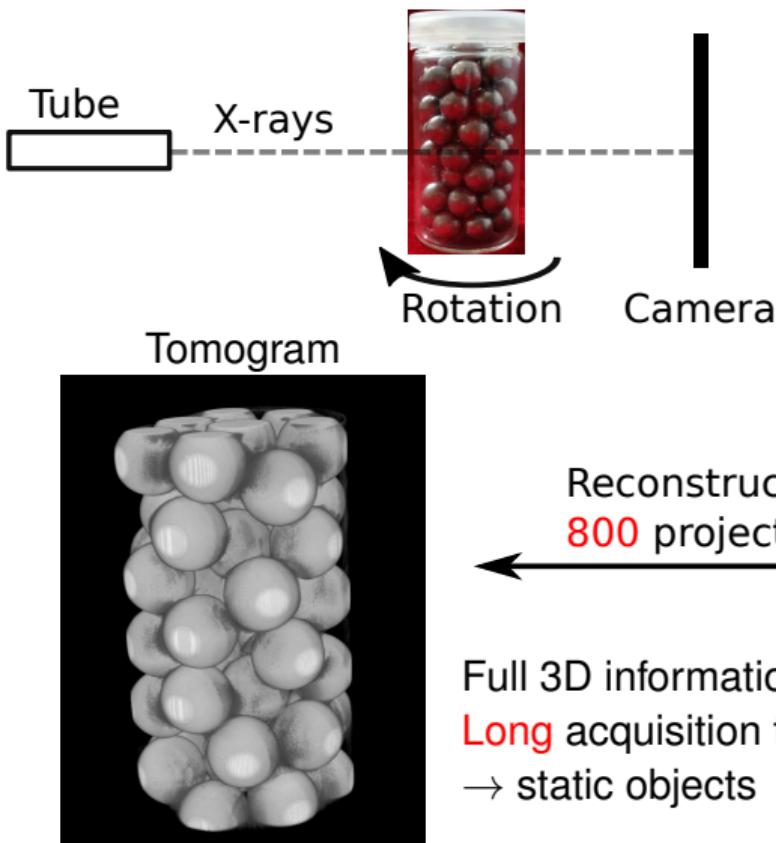
2D projections of 3D object  
Short acquisition time



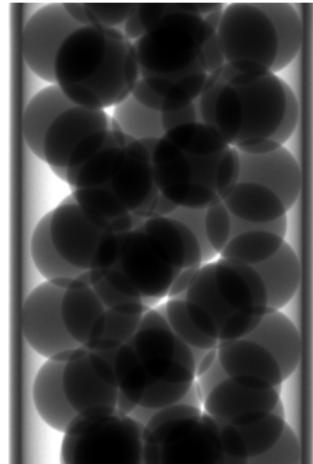
Reconstruction from  
800 projections

Full 3D information  
Long acquisition time  
→ static objects

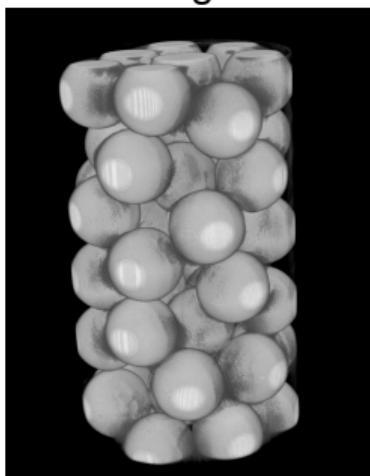
# X-ray radiography



Radiogram

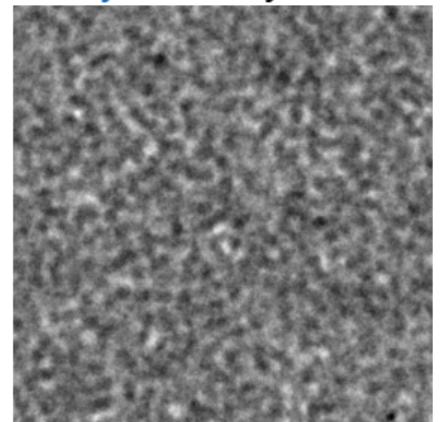


2D projections of 3D object  
Short acquisition time

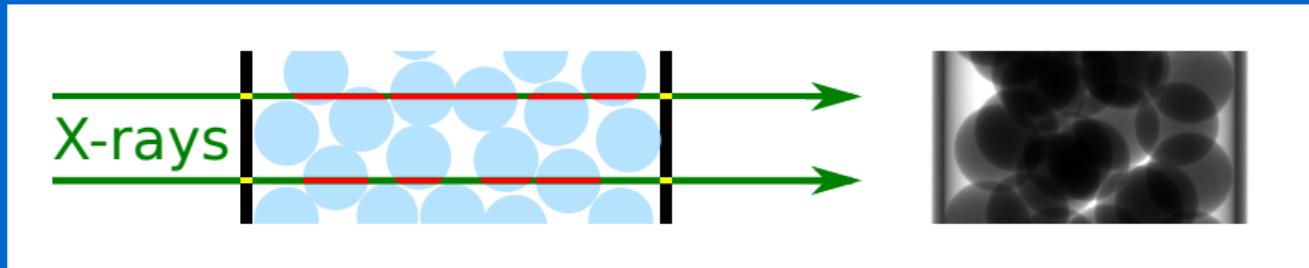


Full 3D information  
Long acquisition time  
→ static objects

Dynamic system



# Measuring the volume fraction of **dynamic** granular systems

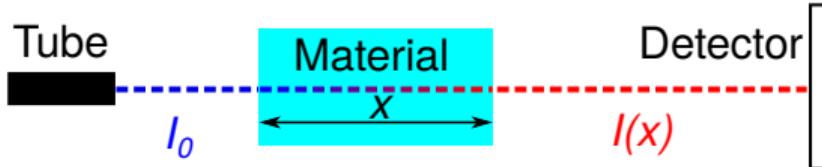


Correction of beam hardening  
in X-ray radiograms

Baur *et al*, *Rev. Sci. Instrum.* (2019)

In collaboration with Norman Uhlmann, Fraunhofer EZRT

# Attenuation of X-rays

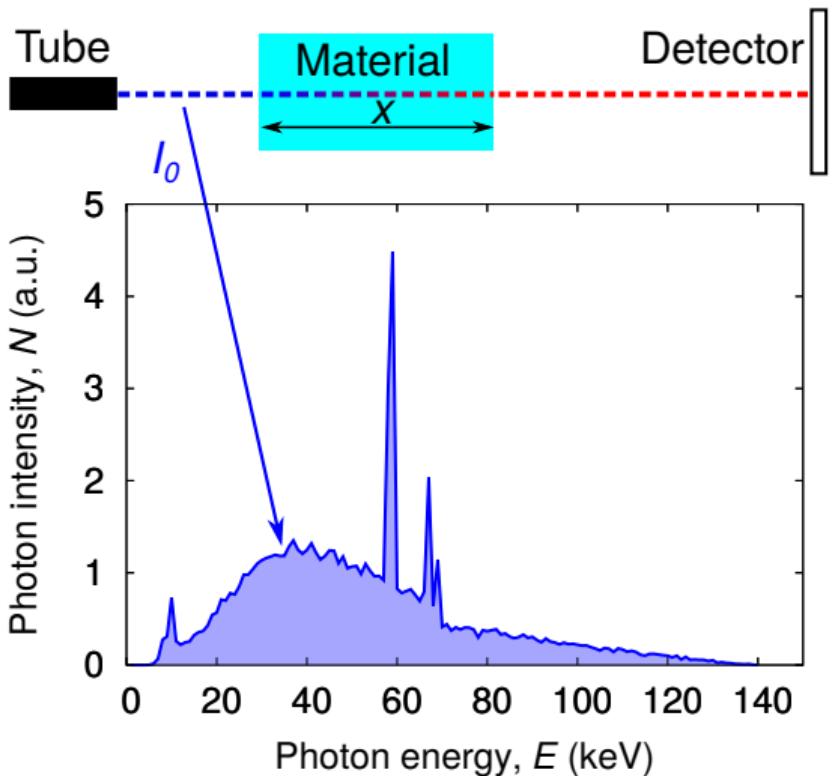


Beer-Lambert's law

$$I(x) = I_0 \exp(-\mu x)$$

$$\text{Thickness: } x = -\frac{1}{\mu} \ln \frac{I(x)}{I_0}$$

# Attenuation of X-rays

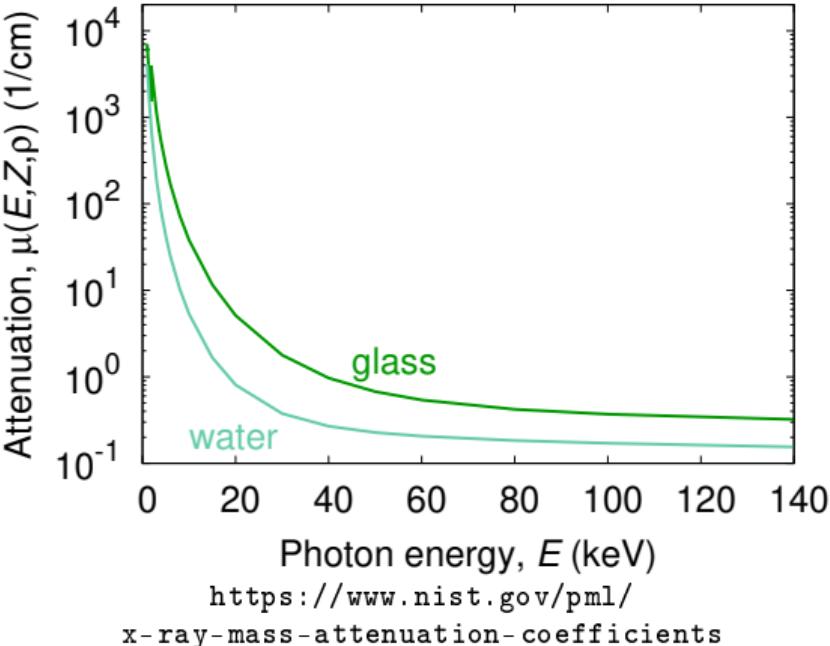


Beer-Lambert's law

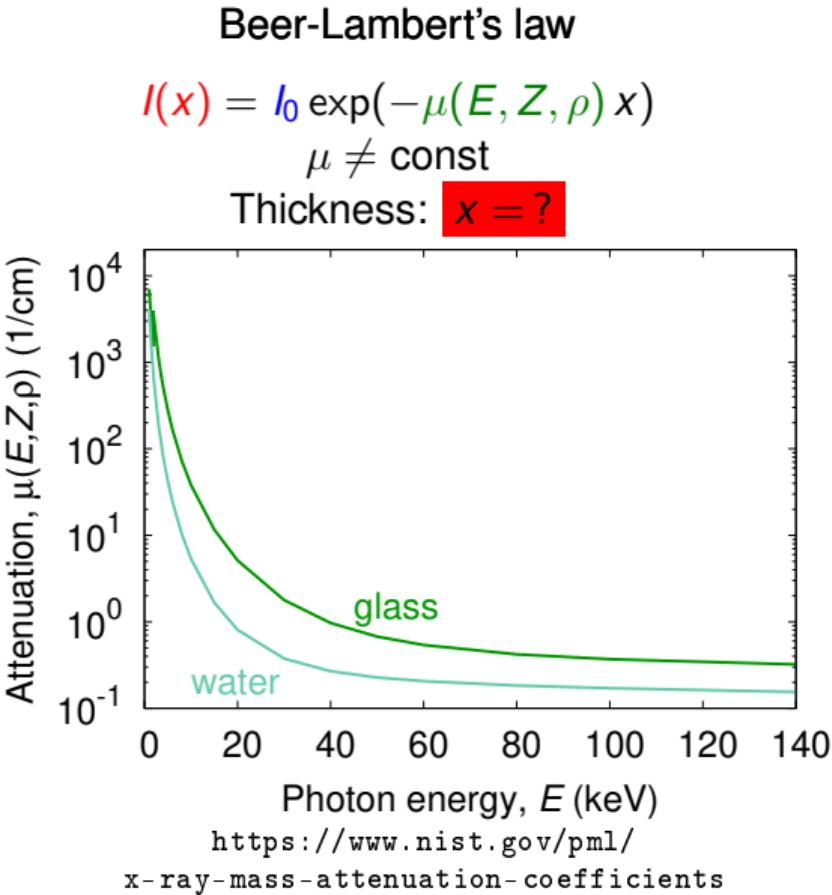
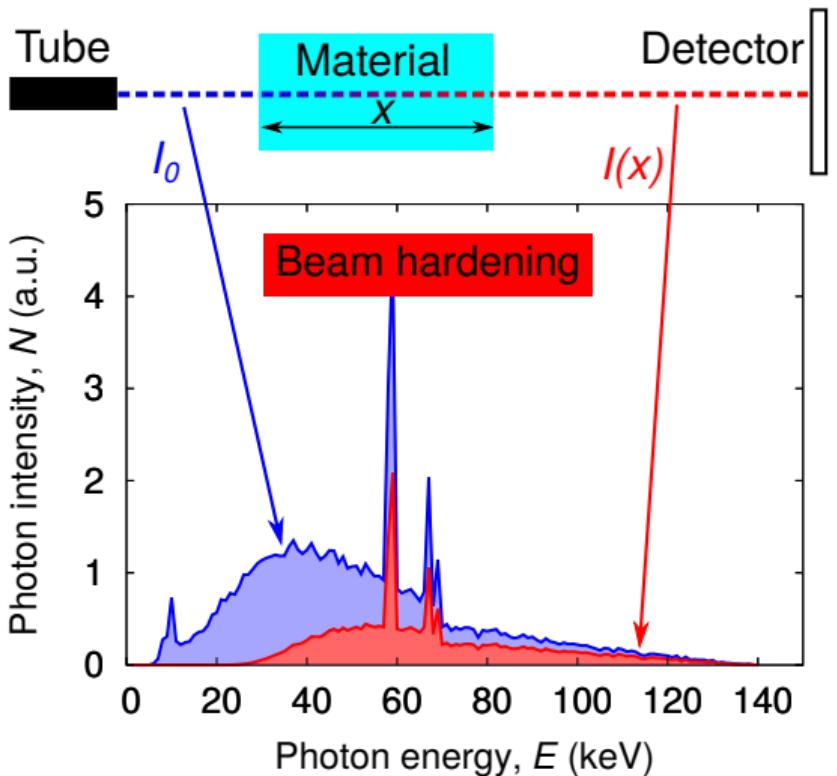
$$I(x) = I_0 \exp(-\mu(E, Z, \rho)x)$$

$\mu \neq \text{const}$

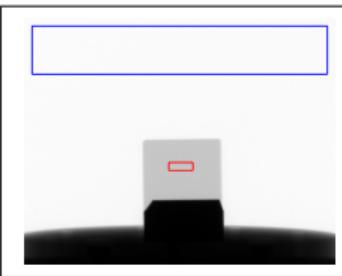
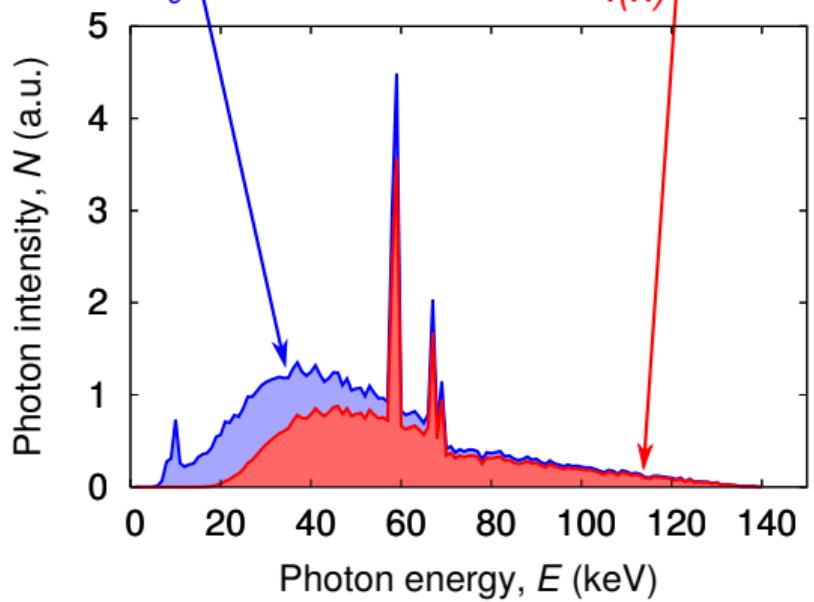
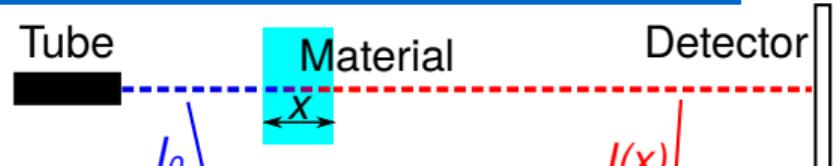
Thickness:  $x = ?$



# Attenuation of X-rays

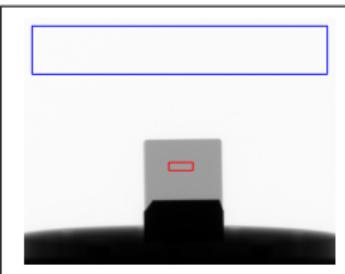
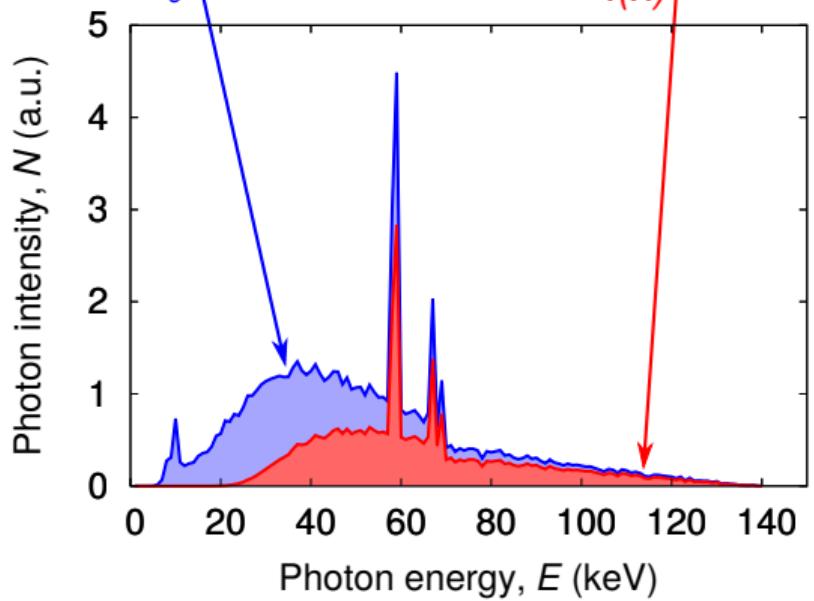
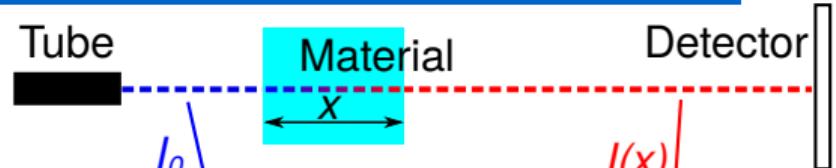


# The effective attenuation, $\mu_{\text{eff}}$



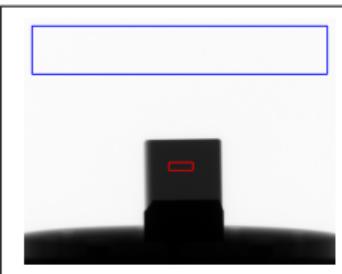
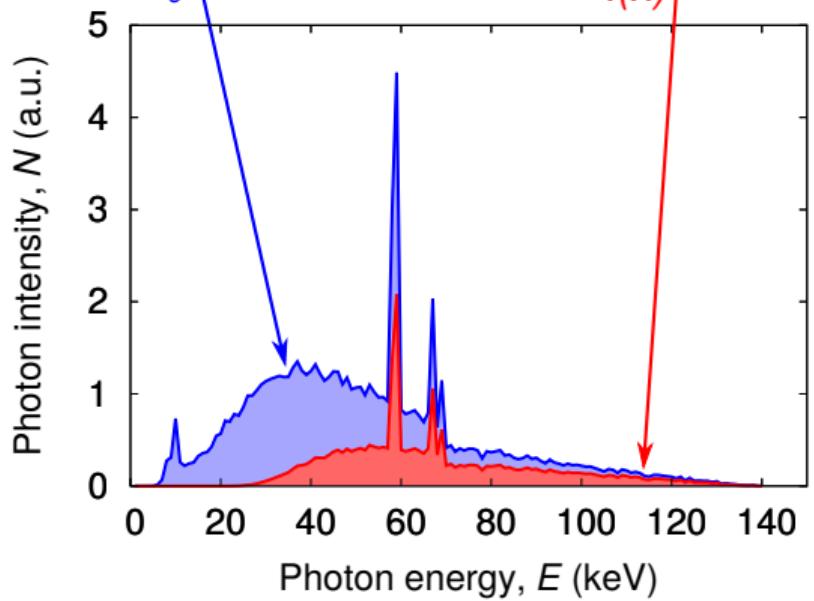
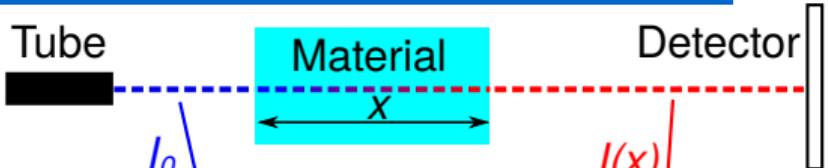
*effective* attenuation:  
 $I(x) = I_0 \exp(-\mu_{\text{eff}}(x) x)$

# The effective attenuation, $\mu_{\text{eff}}$



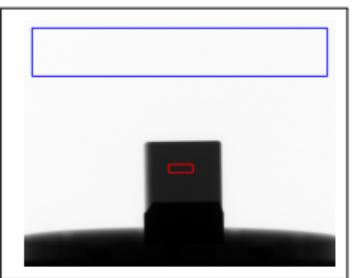
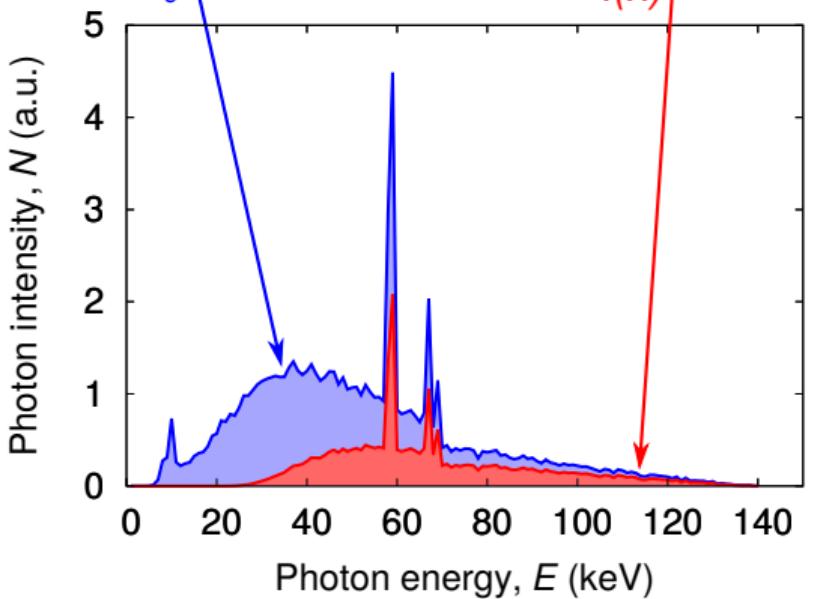
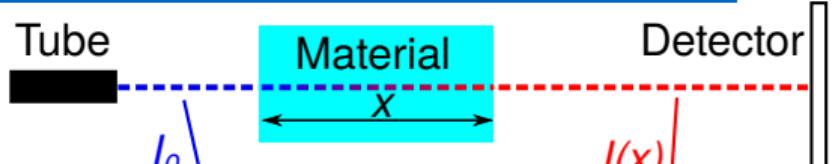
effective attenuation:  
 $I(x) = I_0 \exp(-\mu_{\text{eff}}(x) x)$

# The effective attenuation, $\mu_{\text{eff}}$



effective attenuation:  
 $I(x) = I_0 \exp(-\mu_{\text{eff}}(x) x)$

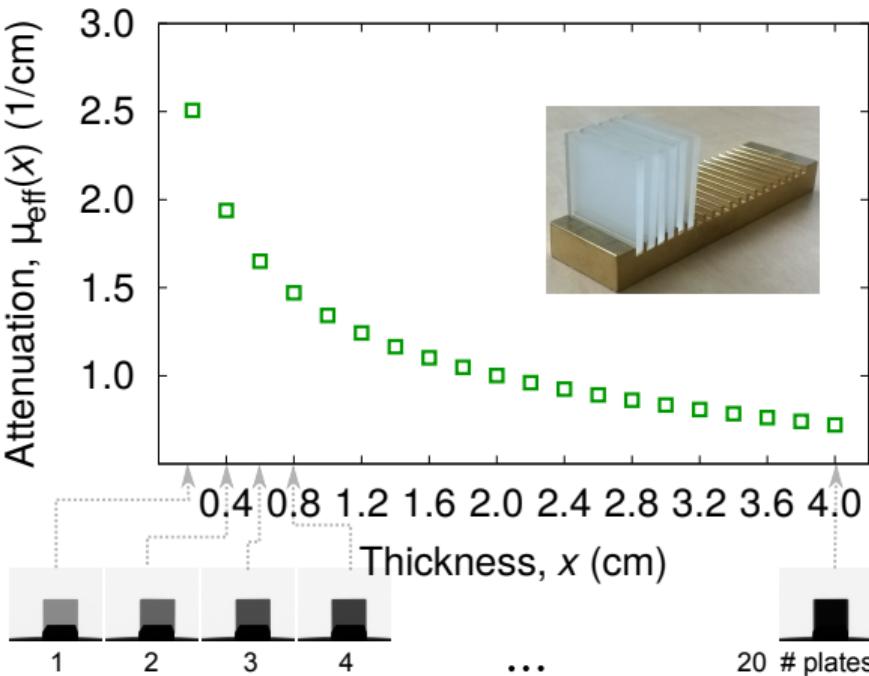
# The effective attenuation, $\mu_{\text{eff}}$



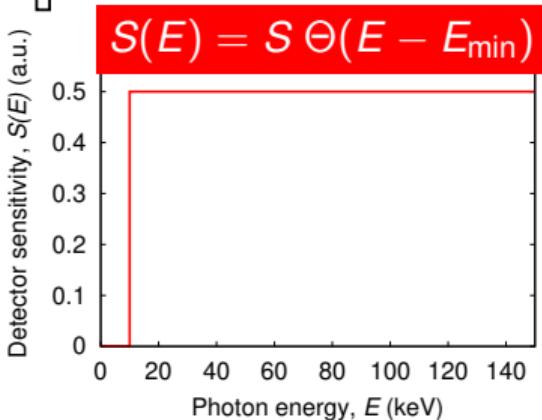
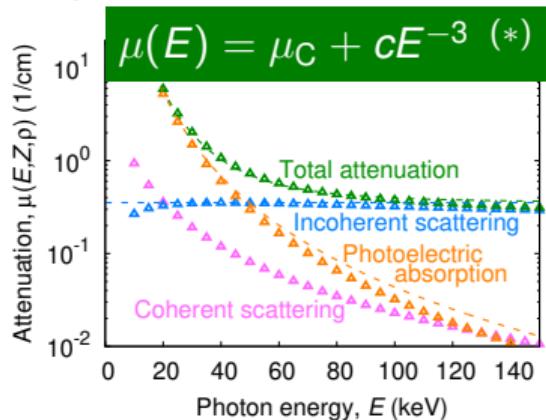
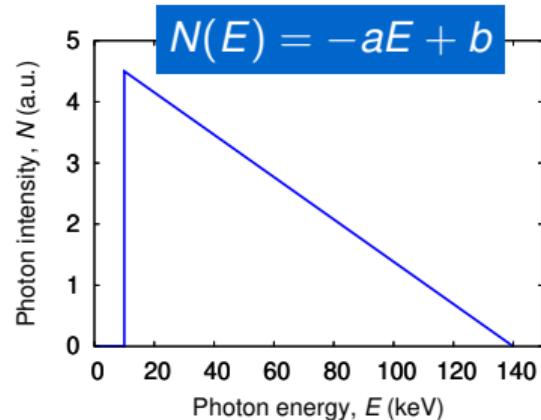
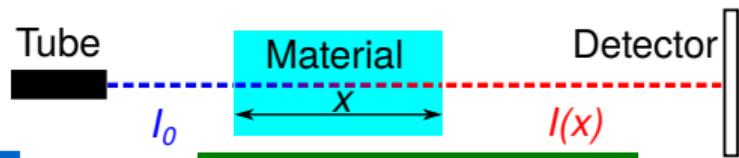
effective attenuation:

$$I(x) = I_0 \exp(-\mu_{\text{eff}}(x)x)$$

$$\mu_{\text{eff}}(x) = -\frac{1}{x} \frac{I(x)}{I_0}$$



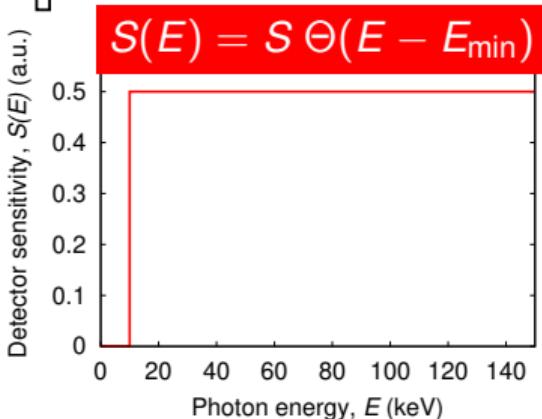
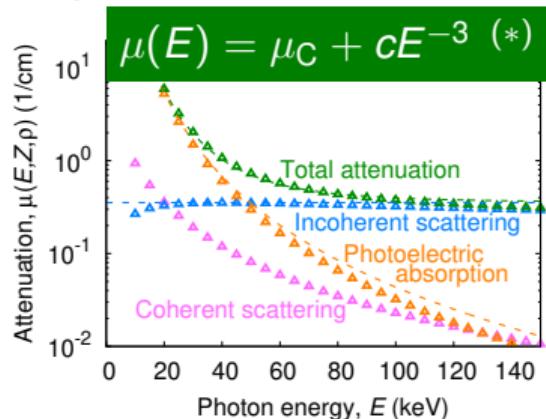
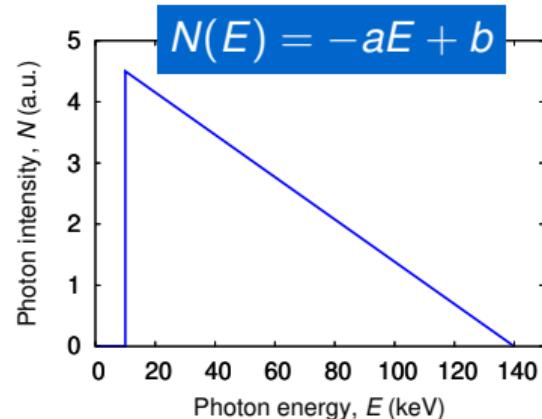
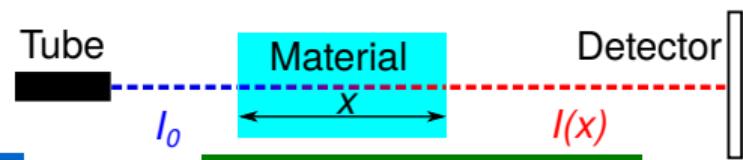
# Modeling of $\mu_{\text{eff}}$



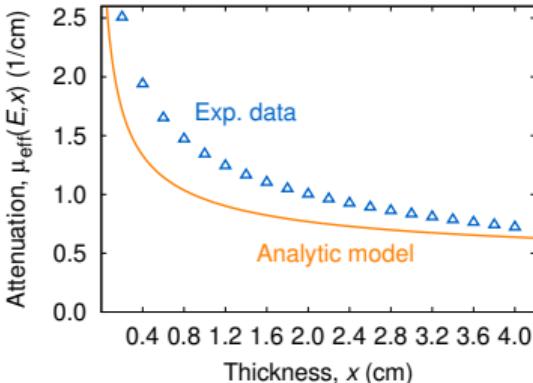
$$I(x) \propto \int N(E) \exp\{-\mu(E, Z, \rho)x\} S(E) dE$$

(\*) XCOM supplied by NIST

# Modeling of $\mu_{\text{eff}}$

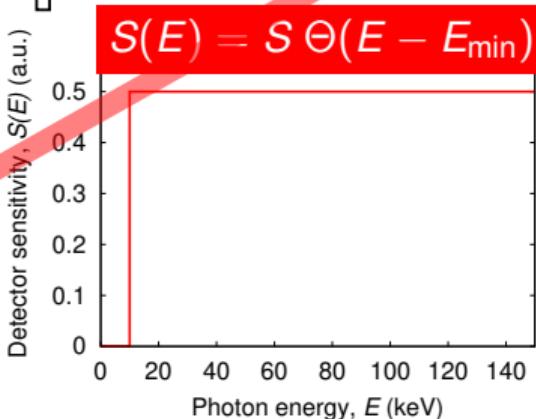
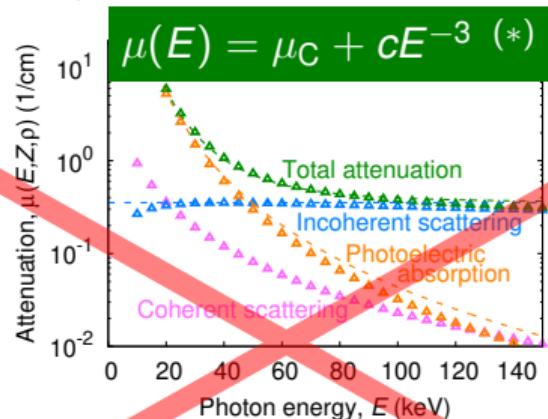
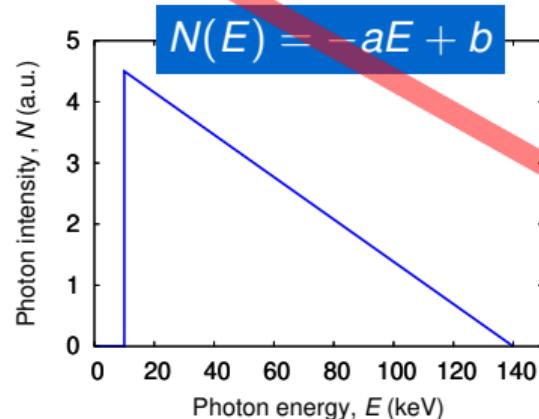
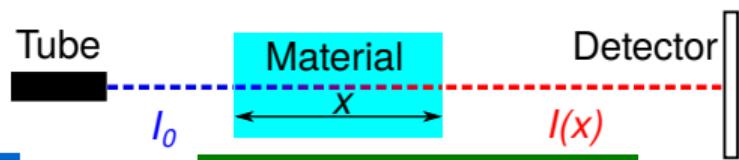


$$\begin{aligned} I(x) &\propto \int N(E) \exp\{-\mu(E, Z, \rho)x\} S(E) dE \\ &\propto S \int_{E_{\min}}^{E_{\max}} (-aE + b) \exp[-(\mu_C + cE^{-3})x] dE \end{aligned}$$



(\*) XCOM supplied by NIST

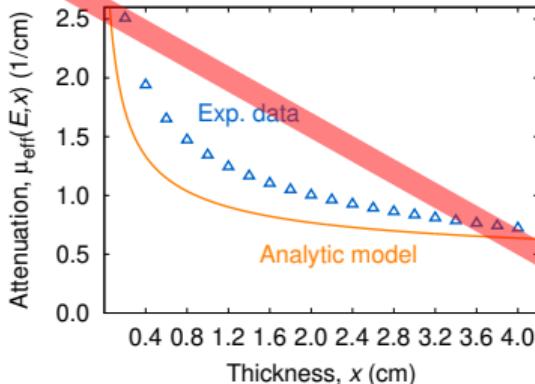
# Modeling of $\mu_{\text{eff}}$



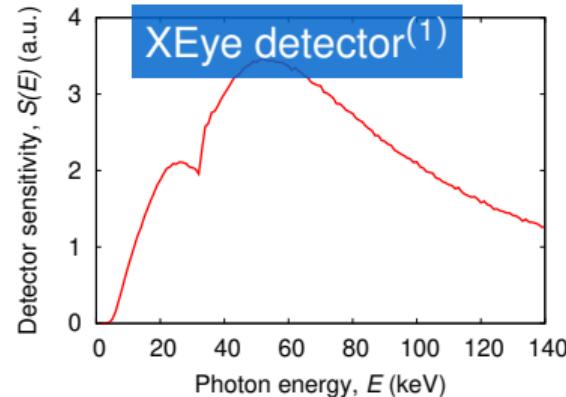
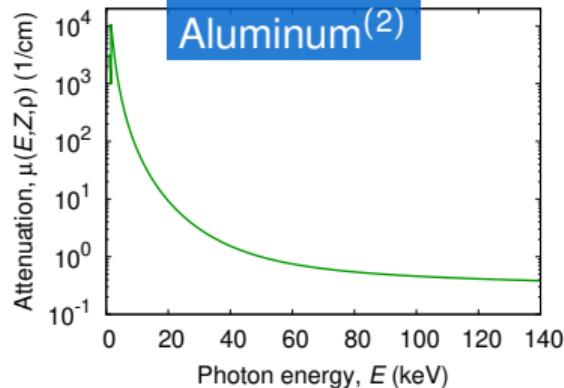
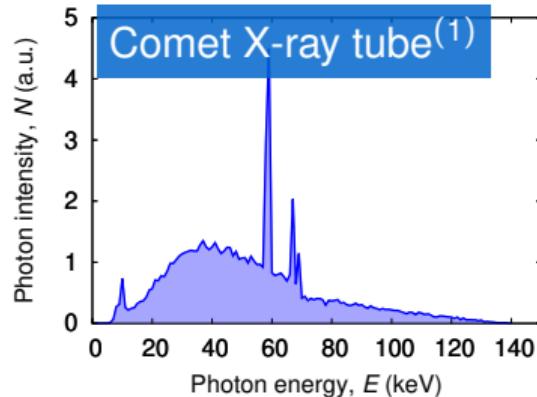
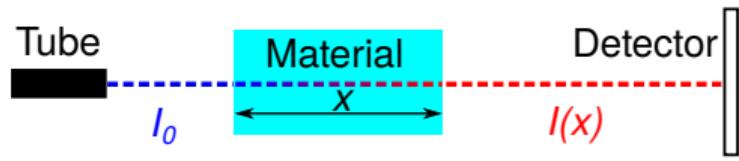
$$I(x) \propto \int N(E) \exp\{-\mu(E, Z, \rho)x\} S(E) dE$$

$$\propto S \int_{E_{\min}}^{E_{\max}} (-aE + b) \exp[-(\mu_C + cE^{-3})x] dE$$

(\*) XCOM supplied by NIST



# Numerical approx. of $\mu_{\text{eff}}$

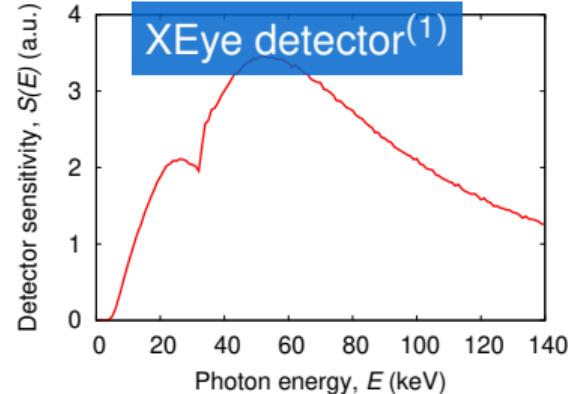
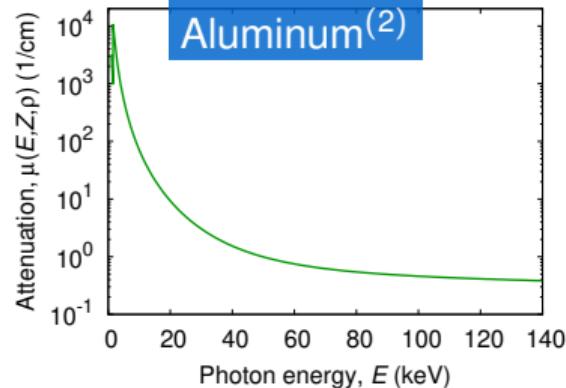
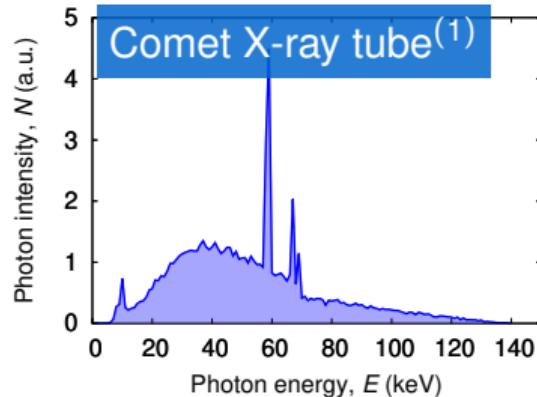
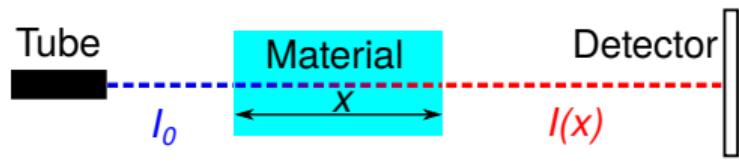


$$I(x) \propto \int N(E) \exp\{-\mu(E, Z, \rho)x\} S(E) dE$$
$$\int \rightarrow \sum$$

(1) Supplied by Norman Uhlmann, Fraunhofer EZRT

(2) XCOM supplied by NIST

# Numerical approx. of $\mu_{\text{eff}}$

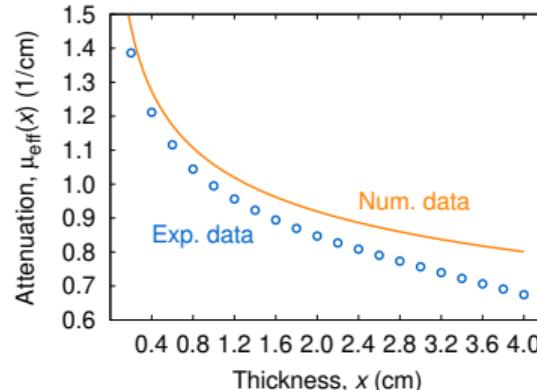


$$I(x) \propto \int N(E) \exp\{-\mu(E, Z, \rho)x\} S(E) dE$$

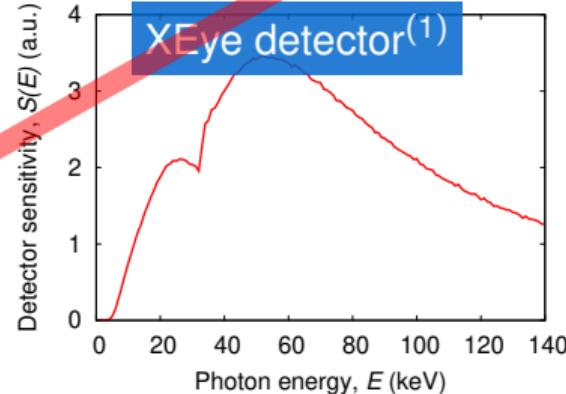
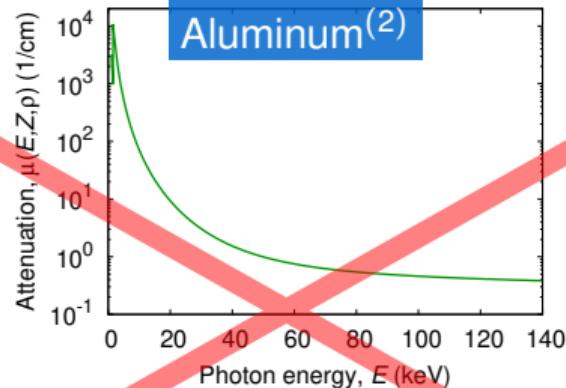
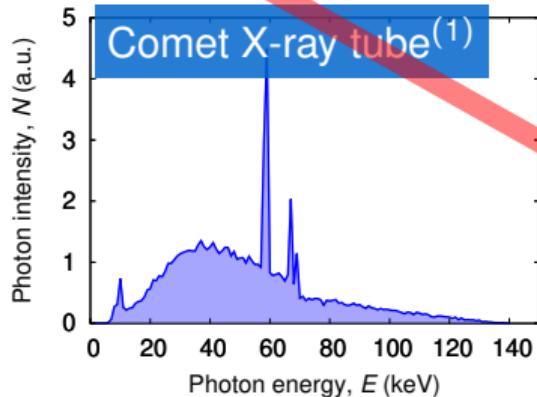
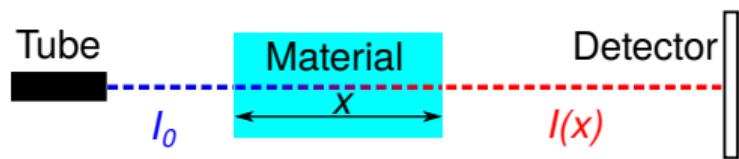
$$\int \rightarrow \sum$$

(1) Supplied by Norman Uhlmann, Fraunhofer EZRT

(2) XCOM supplied by NIST

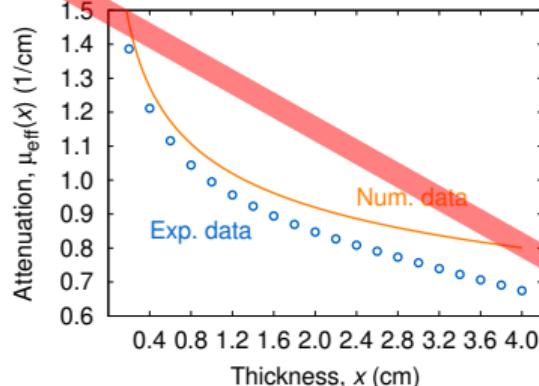


# Numerical approx. of $\mu_{\text{eff}}$



$$I(x) \propto \int N(E) \exp\{-\mu(E, Z, \rho)x\} S(E) dE$$

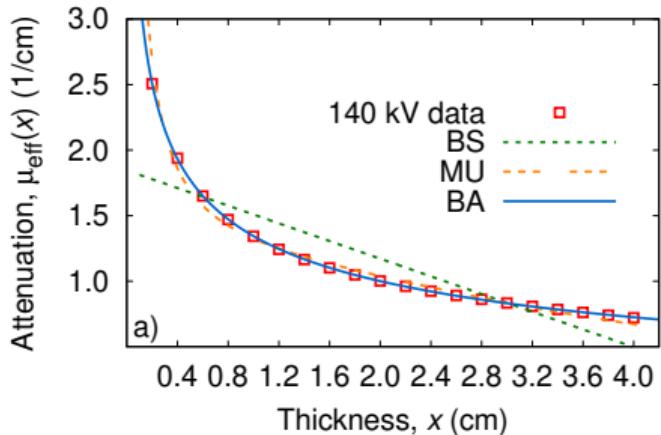
$$\int \rightarrow \sum$$



(1) Supplied by Norman Uhlmann, Fraunhofer EZRT

(2) XCOM supplied by NIST

# Heuristic model functions for $\mu_{\text{eff}}$



$$\mu_{\text{eff}}(x) = \mu_0 - \lambda x$$

Bjärngard & Shackford  
(1994)

$$\mu_{\text{eff}}(x) = \frac{\mu_0}{1 + \lambda x}$$

$$\mu_{\text{eff}}(x) = \frac{\mu_0}{(1 + \lambda x)^\beta}$$

Yu *et al.* (1997)

$$\mu_{\text{eff}}(x) = \mu(E_{\text{max}}) + \frac{2\mu_1}{x\sqrt{-\lambda_1^2 + 4\lambda_2}} \times$$

$$\left[ \arctan \left( \frac{\lambda_1 + 2\lambda_2 x}{\sqrt{-\lambda_1^2 + 4\lambda_2}} \right) - \arctan \left( \frac{\lambda_1}{\sqrt{-\lambda_1^2 + 4\lambda_2}} \right) \right]$$

Kleinschmidt (1999)

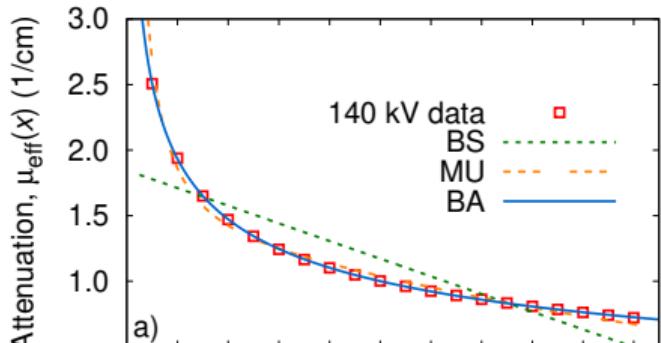
$$\mu_{\text{eff}}(x) = -\frac{1}{x} \ln [A + B \exp(-x/C)]$$

Mudde *et al.* (2008)

$$\mu_{\text{eff}}(x) = a + \frac{b}{x^\alpha}$$

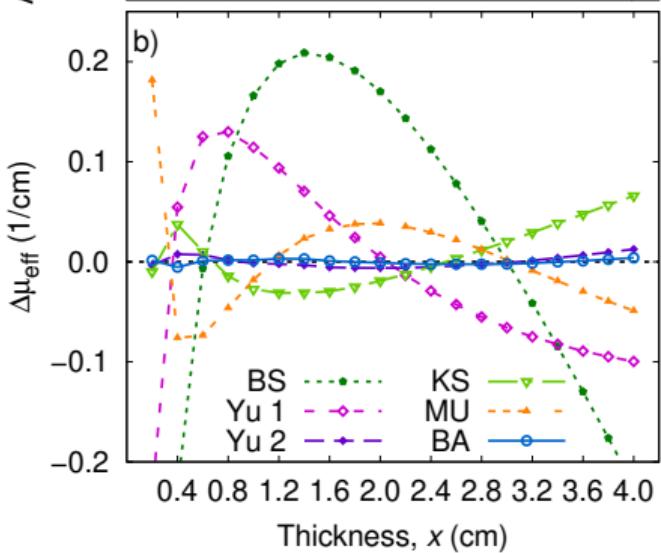
Baur *et al.* (2019)  
(this work)

# Heuristic model functions for $\mu_{\text{eff}}$



$$\mu_{\text{eff}}(x) = \mu_0 - \lambda x$$

Bjärngard & Shackford  
(1994)



$$\mu_{\text{eff}}(x) = \frac{\mu_0}{1+\lambda x}$$

$$\mu_{\text{eff}}(x) = \frac{\mu_0}{(1+\lambda x)^\beta}$$

Yu *et al.* (1997)

$$\mu_{\text{eff}}(x) = \mu(E_{\text{max}}) + \frac{2\mu_1}{x\sqrt{-\lambda_1^2+4\lambda_2}} \times \left[ \arctan\left(\frac{\lambda_1+2\lambda_2 x}{\sqrt{-\lambda_1^2+4\lambda_2}}\right) - \arctan\left(\frac{\lambda_1}{\sqrt{-\lambda_1^2+4\lambda_2}}\right) \right]$$

Kleinschmidt (1999)

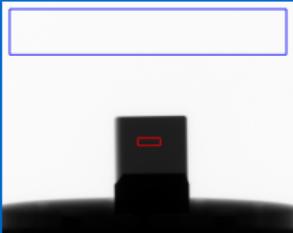
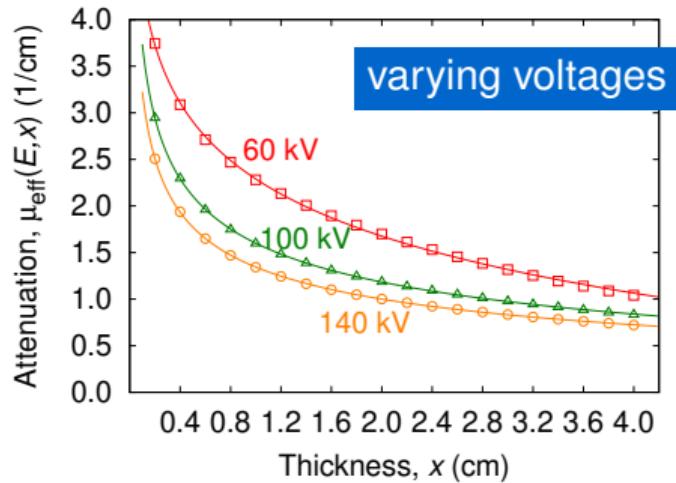
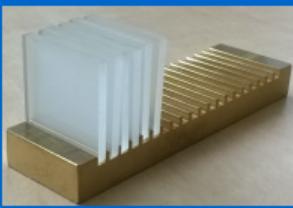
$$\mu_{\text{eff}}(x) = -\frac{1}{x} \ln [A + B \exp(-x/C)]$$

Mudde *et al.* (2008)

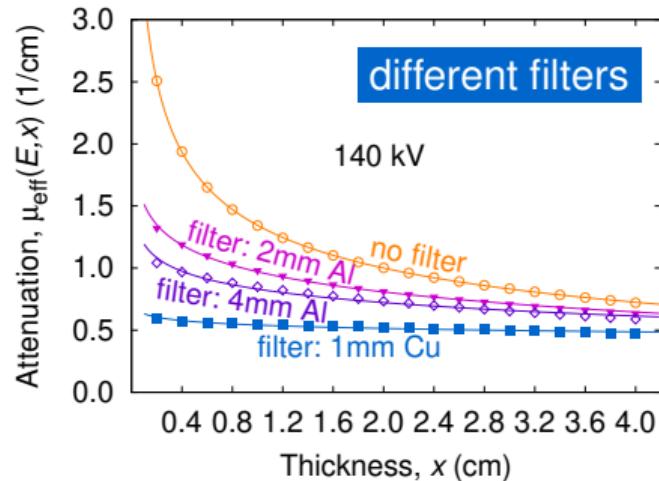
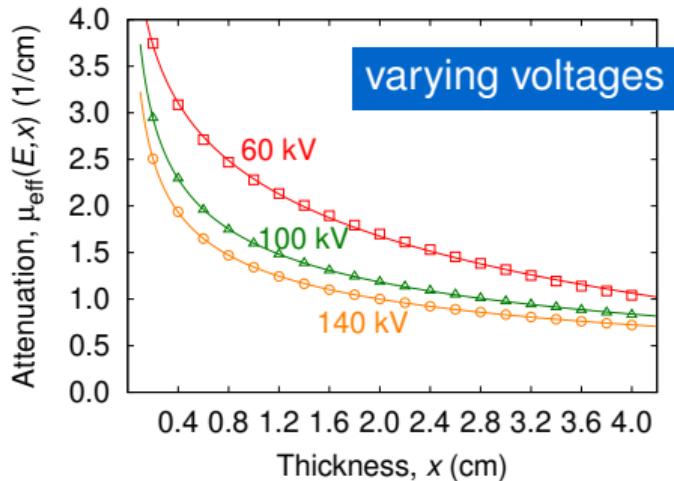
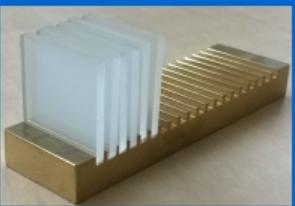
$$\mu_{\text{eff}}(x) = a + \frac{b}{x^\alpha}$$

Baur *et al.* (2019)  
(this work)

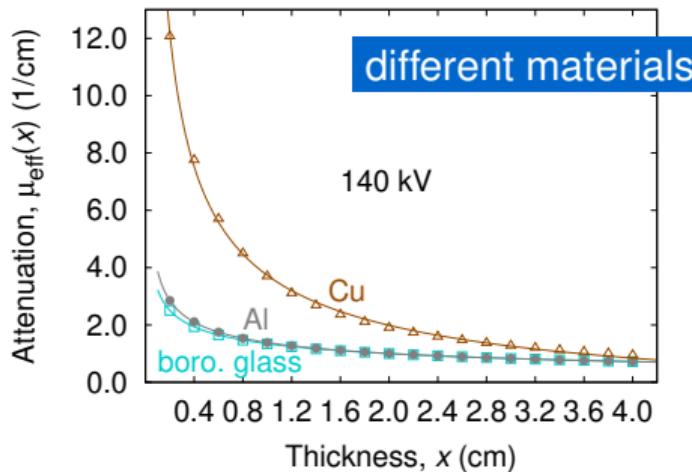
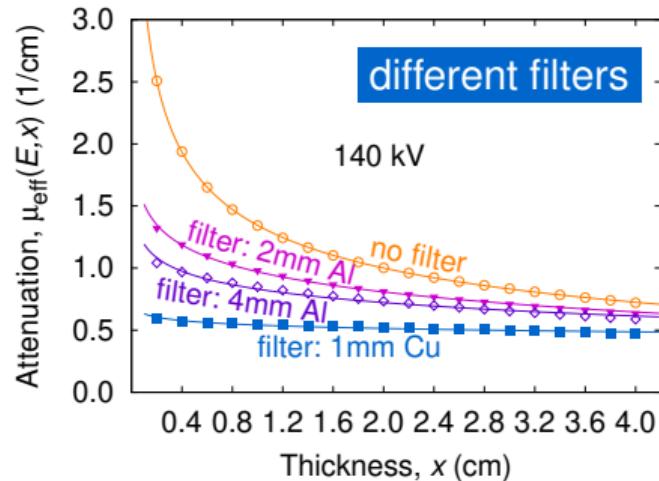
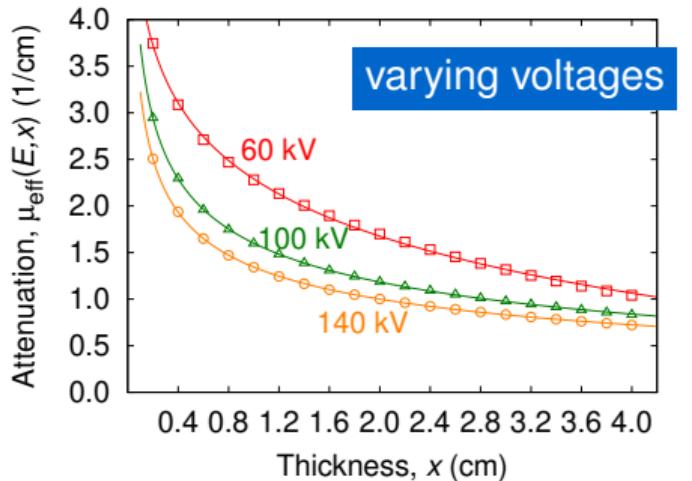
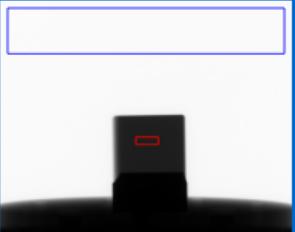
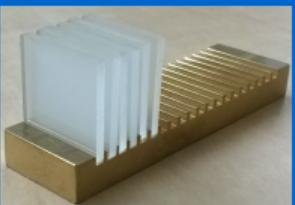
Universality of  
 $\mu_{\text{eff}}(x) = a + \frac{b}{x^\alpha}$



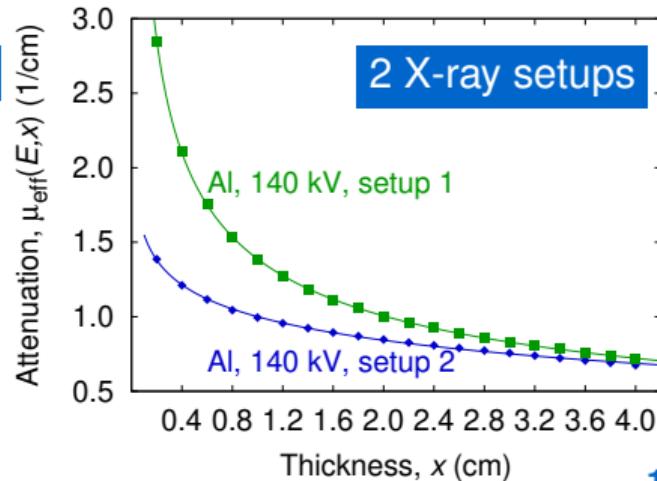
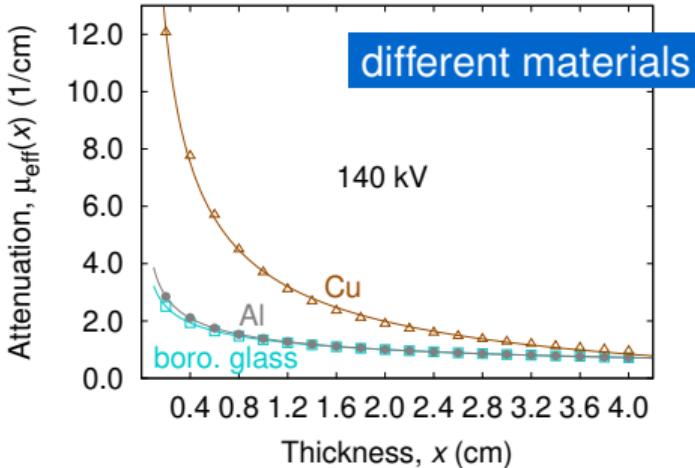
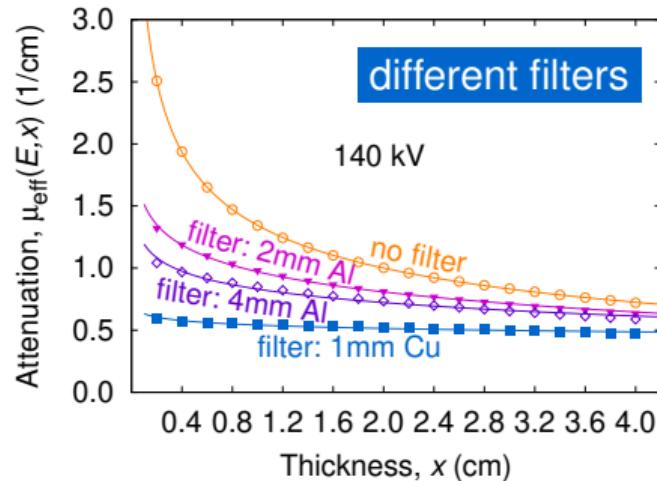
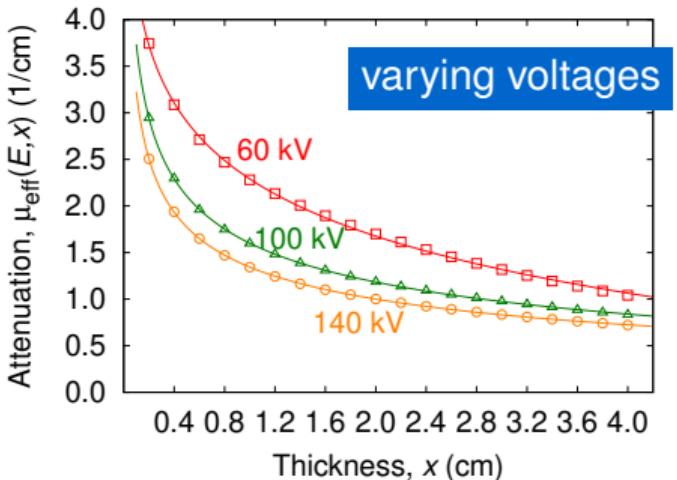
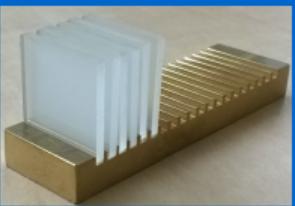
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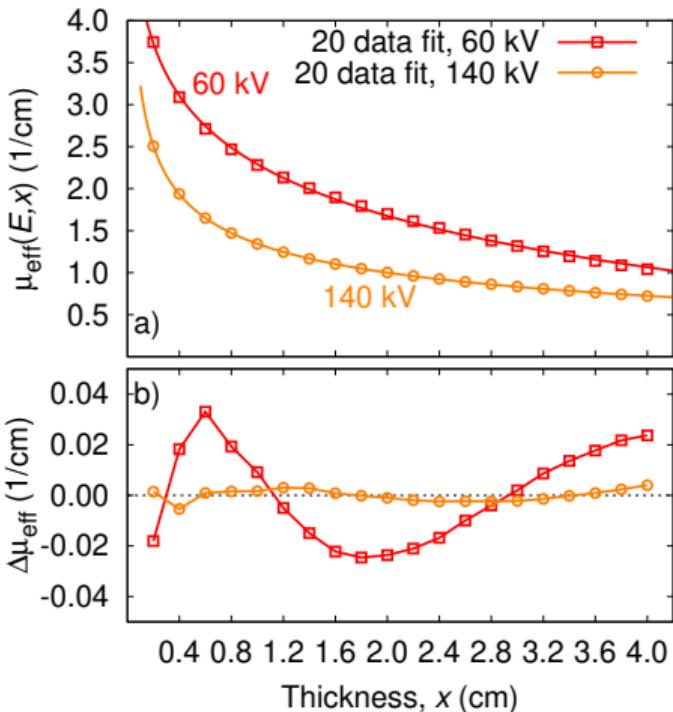
# Determining the material thickness $x$

Generalized Beer-Lambert

$$I(x) = I_0 \exp(-\mu_{\text{eff}}(x) x)$$

Model function

$$\mu_{\text{eff}}(x) = a + \frac{b}{x^\alpha}$$



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Generalized Beer-Lambert

$$I(x) = I_0 \exp(-\mu_{\text{eff}}(x) x)$$

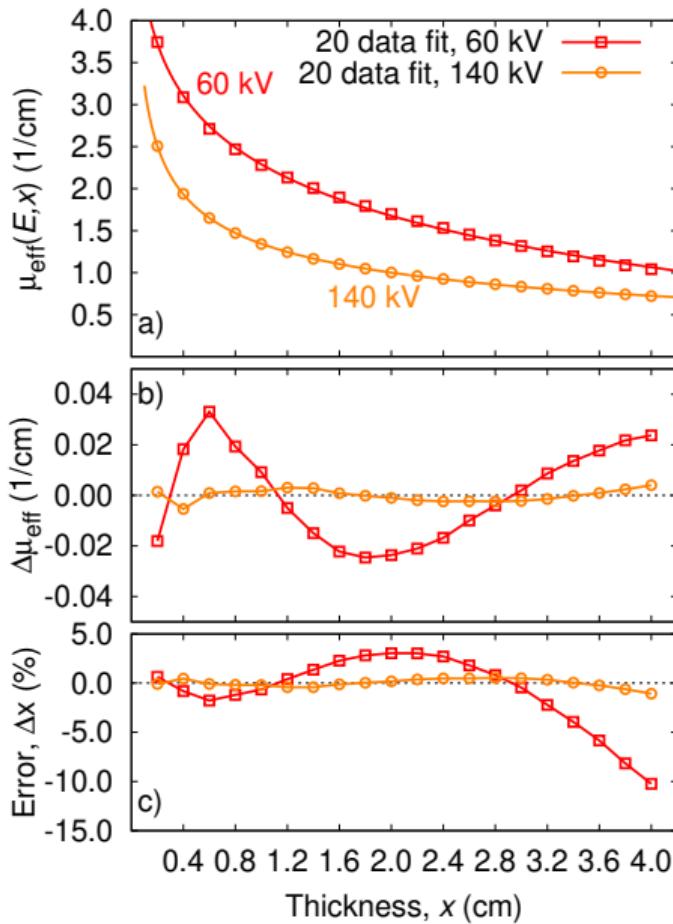
Model function

$$\mu_{\text{eff}}(x) = a + \frac{b}{x^\alpha}$$

Solve

$$ax + bx^{1-\alpha} + \ln\left(\frac{I(x)}{I_0}\right) = 0$$

e.g. Newton's method or look-up table



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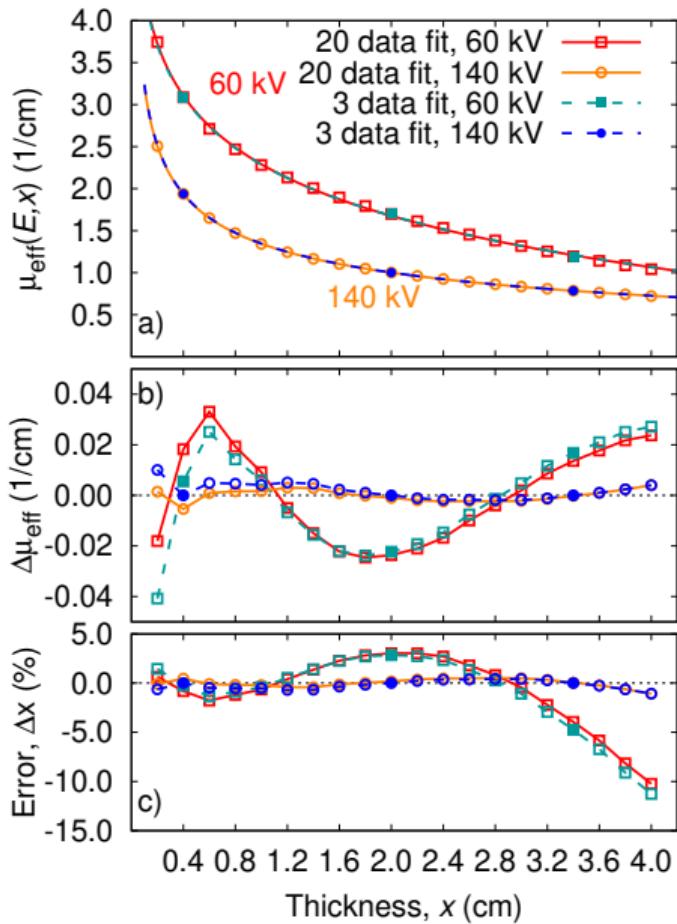
Model function

$$\mu_{\text{eff}}(x) = a + \frac{b}{x^\alpha}$$

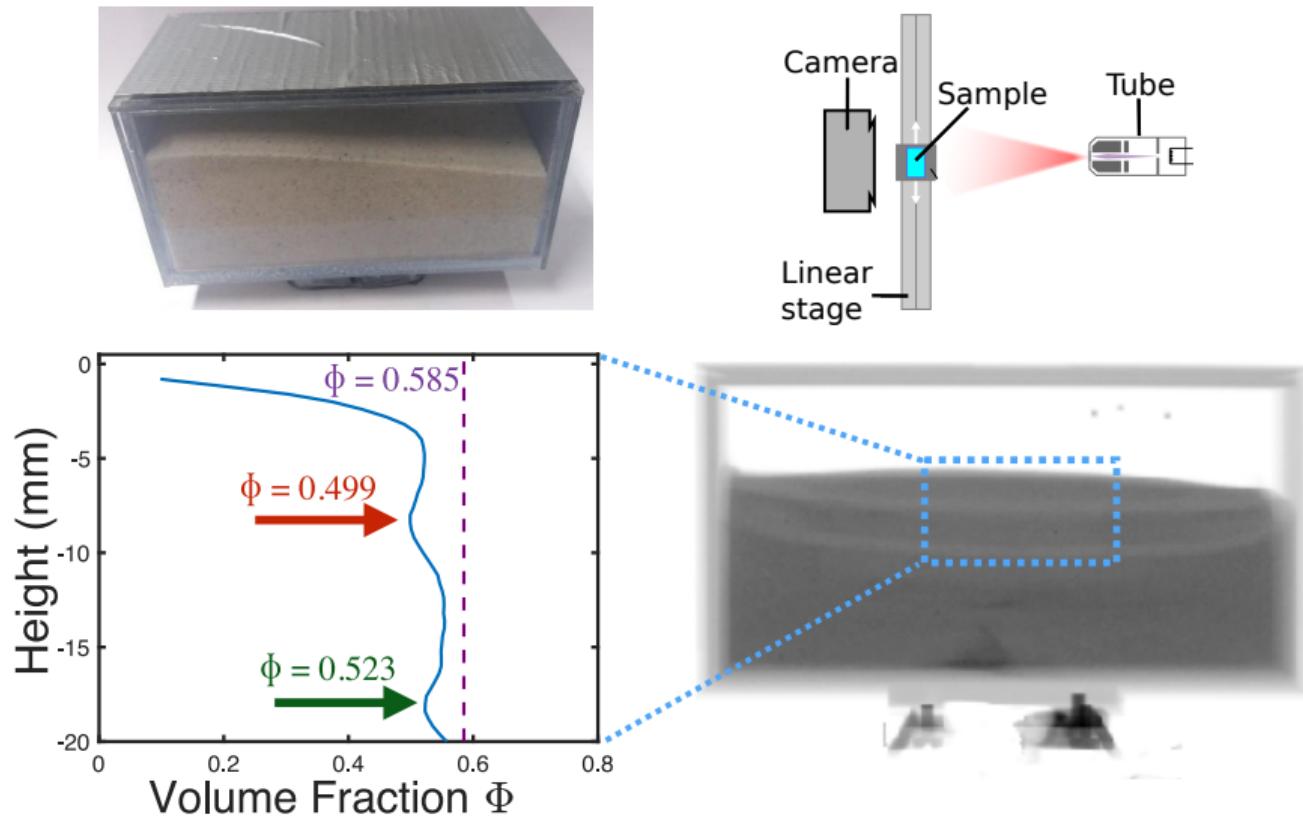
Solve

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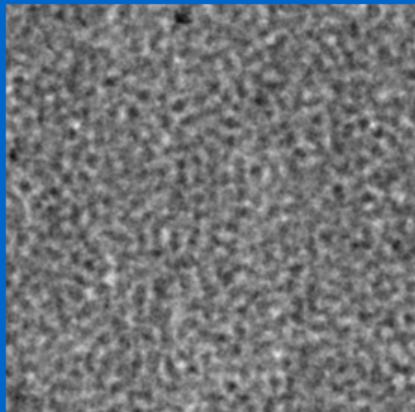


# Migrating shear bands in shaken granular matter, Kollmer *et al* (2020)

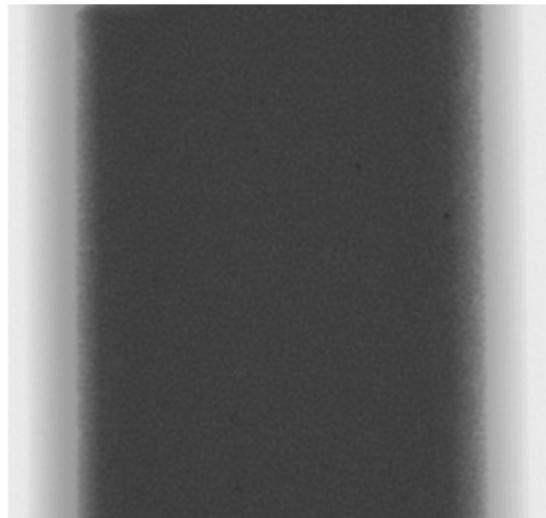
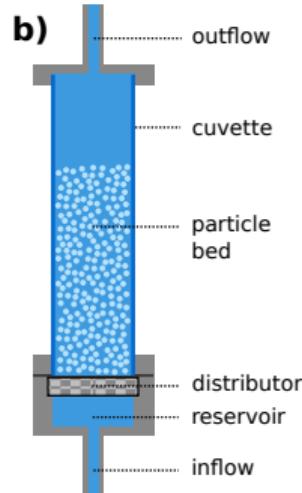
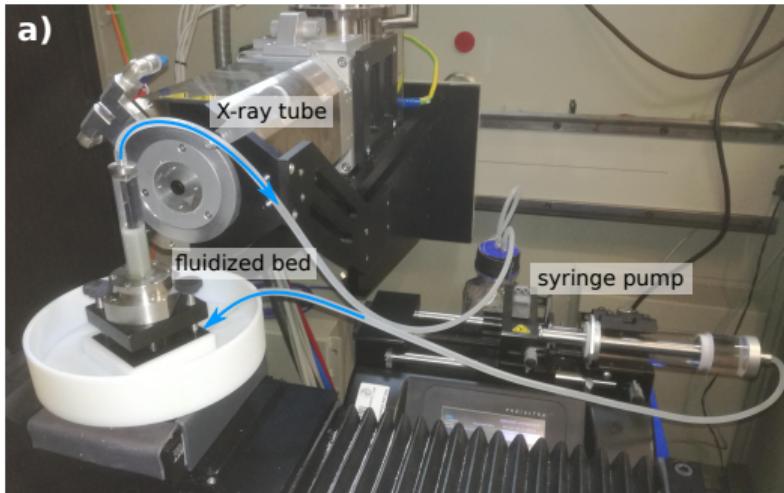


# X-ray Digital Fourier Analysis (X-DFA)

## A technique to measure granular dynamics

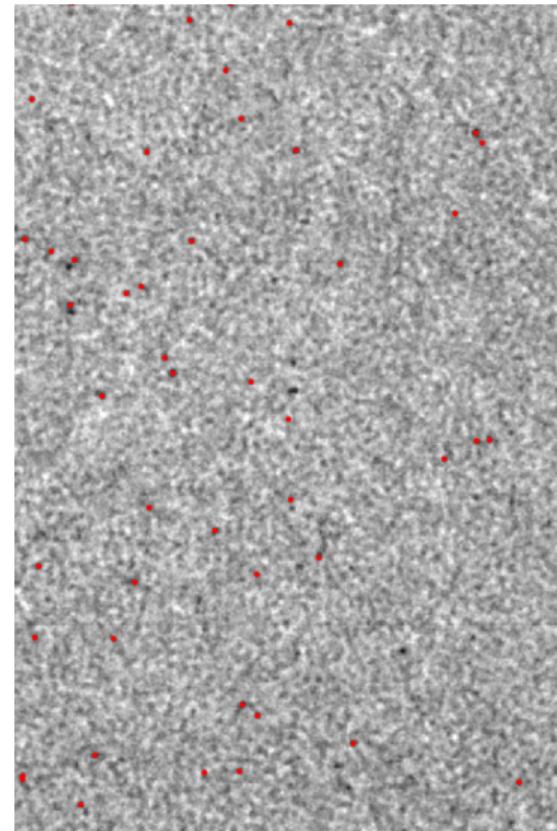
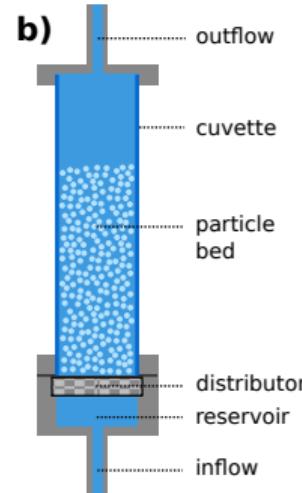
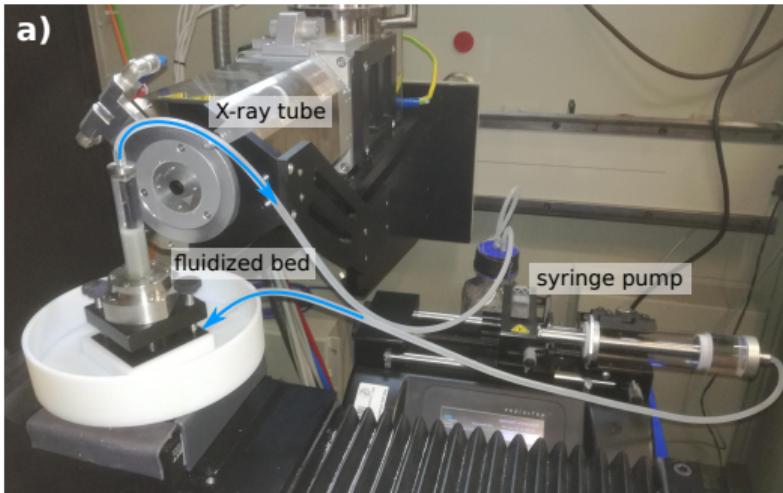


# Experiments



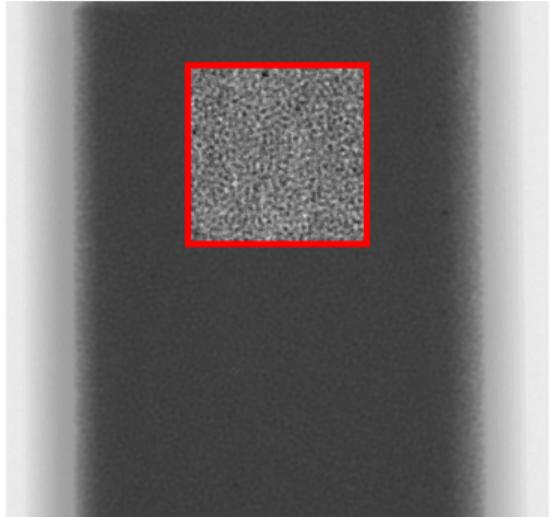
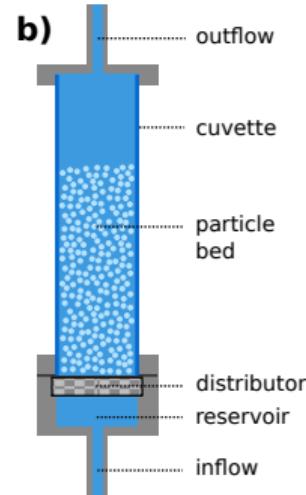
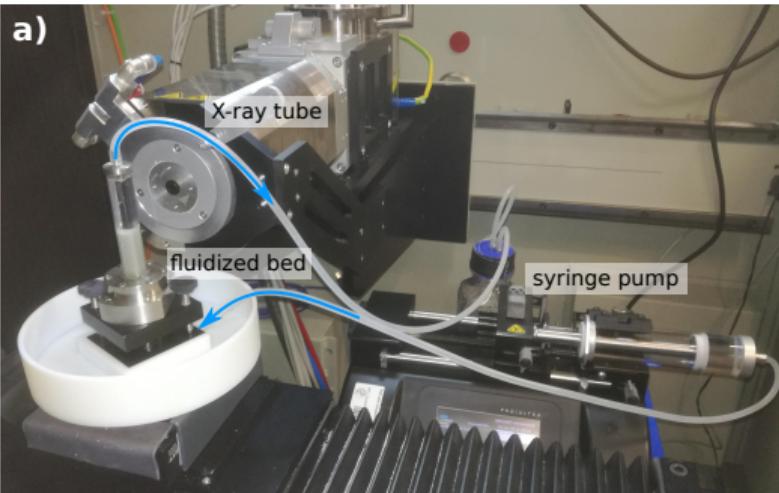
- volume fraction:  $0.45 < \Phi < 0.56$
- control dynamics and  $\Phi$  via pump rate

# Experiments

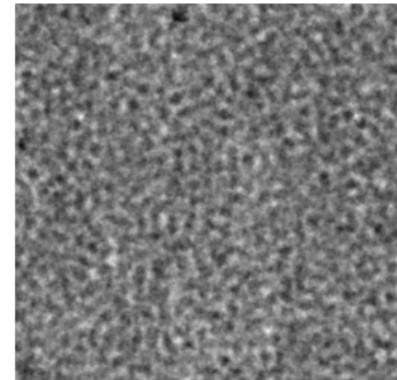


- volume fraction:  $0.45 < \Phi < 0.56$
- control dynamics and  $\Phi$  via pump rate

# Experiments



- volume fraction:  $0.45 < \Phi < 0.56$
- control dynamics and  $\Phi$  via pump rate



## Differential Dynamic Microscopy (DDM)

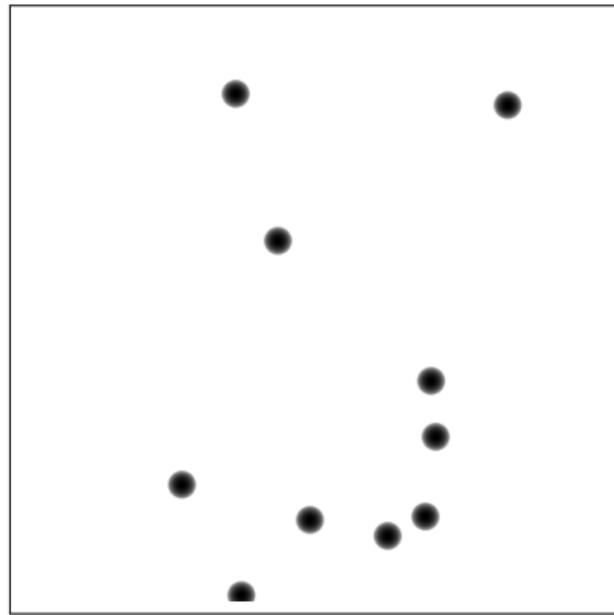
	<b>up to now</b>	<b>this work</b>
<b>system</b>	dispersion, gels	fluidized bed
<b>particles</b>	colloids	granulate
<b>part. diameter</b>	$< 1 \mu\text{m}$	$\approx 200 \mu\text{m}$
<b>volume fraction</b>	$\Phi \leq 0.33$	$0.45 < \Phi < 0.56$
<b>imaging</b>	light microscope	x-ray radiography
<b>dynamics</b>	Brownian motion, caging, glassy, collective motion	

# Extending Differential Dynamic Microscopy (DDM) to X-ray imaging

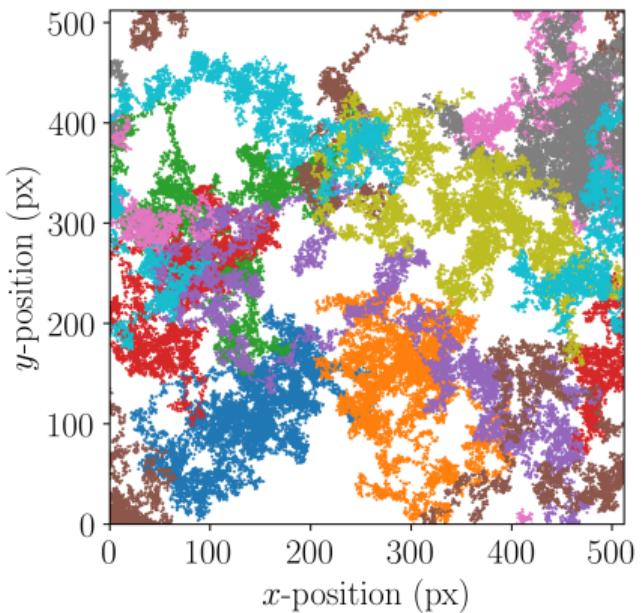
	<b>up to now</b>	<b>this work</b>
<b>system</b>	dispersion, gels	fluidized bed
<b>particles</b>	colloids	granulate
<b>part. diameter</b>	$< 1 \mu\text{m}$	$\approx 200 \mu\text{m}$
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<b>imaging</b>	light microscope	x-ray radiography
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## Digital Fourier Analysis of X-Ray Radiograms (X-DFA)

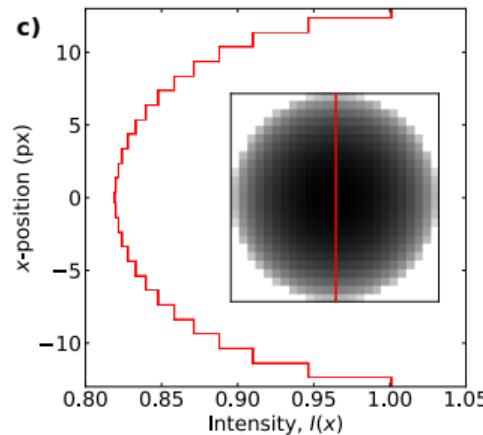
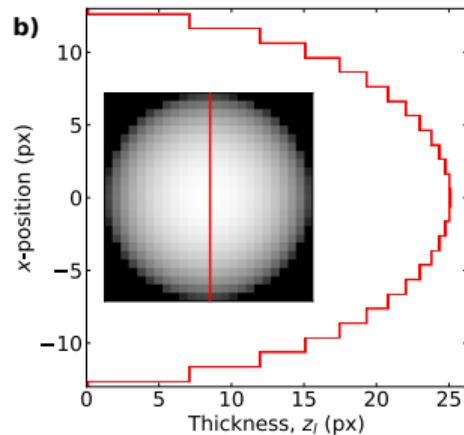
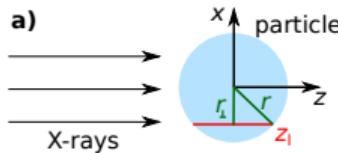
## Synthetic radiograms



Video 10 Particles

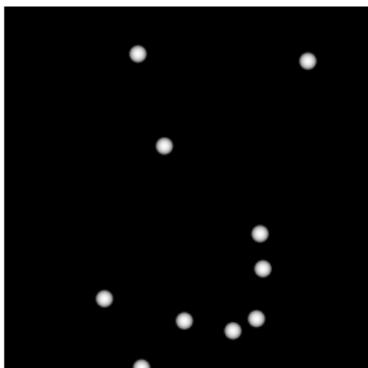
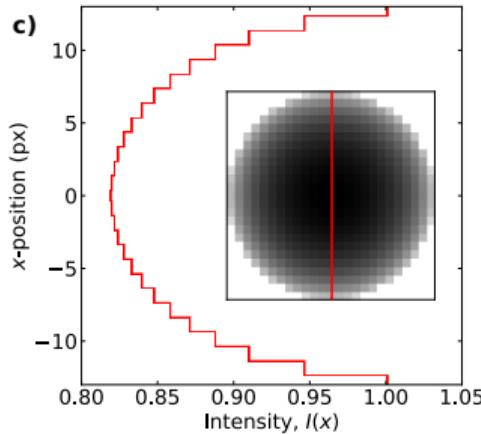
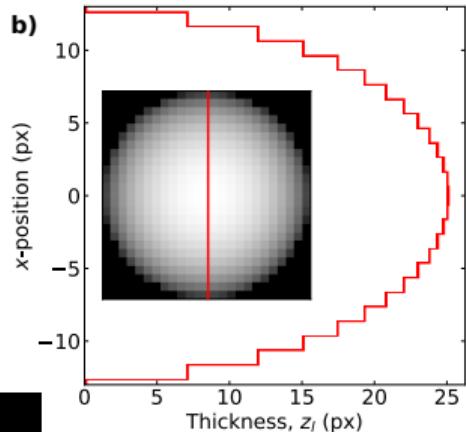
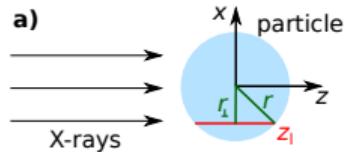


# Synthetic radiograms

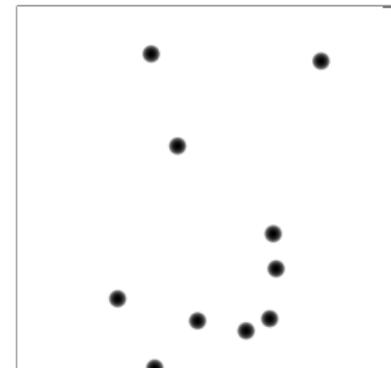


Beer-Lambert  
 $I(z_I) = I_0 \exp(-\mu z)$

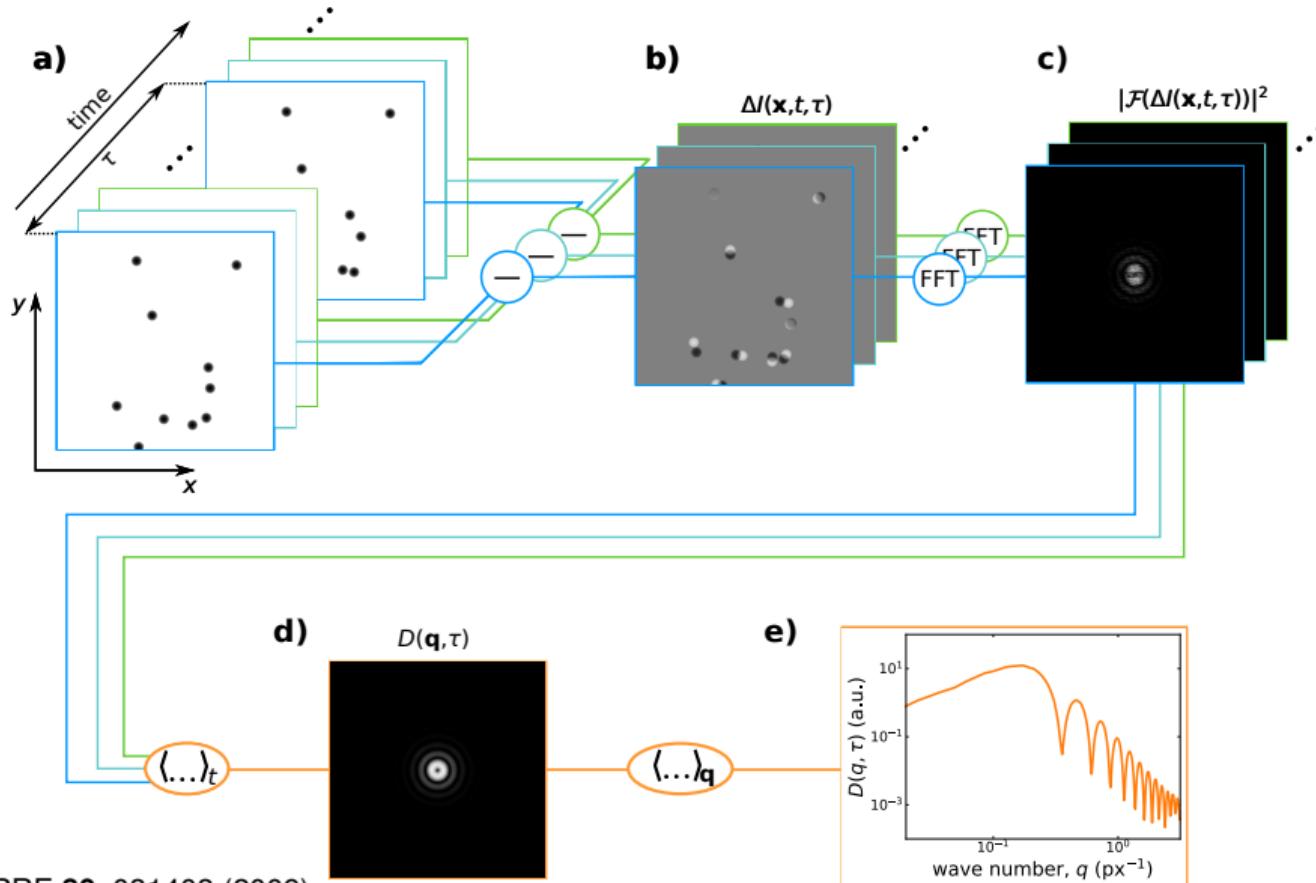
# Synthetic radiograms



Beer-Lambert  
 $I(z_l) = I_0 \exp(-\mu z)$

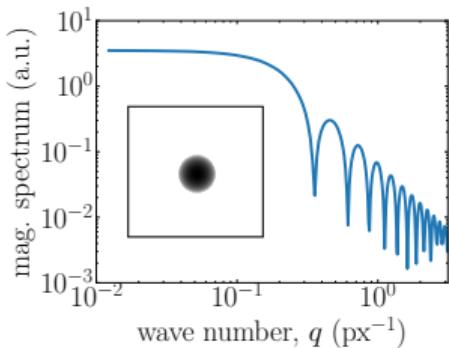
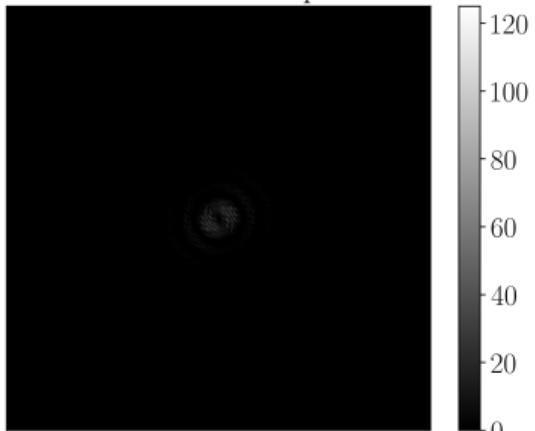


# The image structure function $D(\mathbf{q}, \tau)$



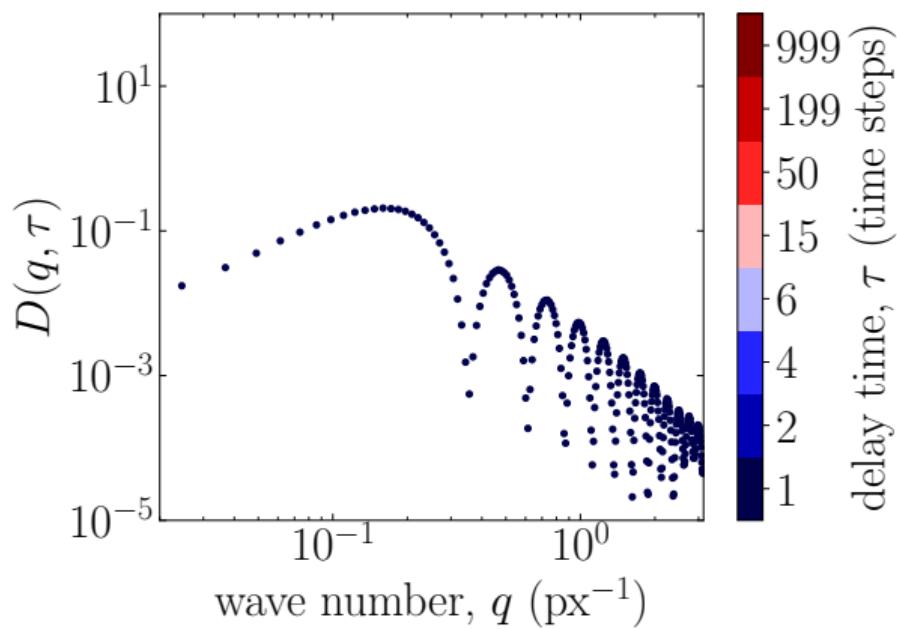
# The image structure function $D(q, \tau)$

$\tau = 1$  time steps



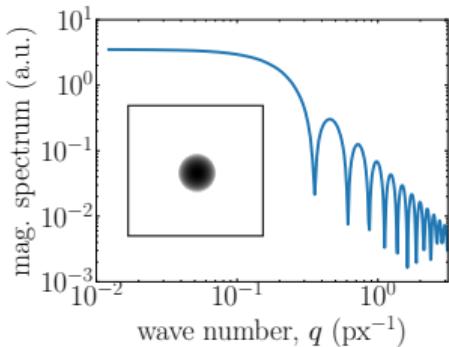
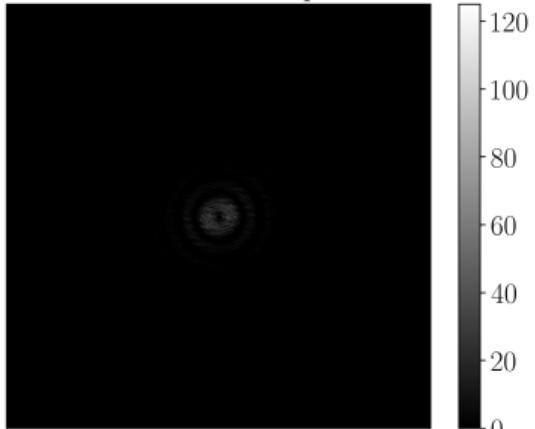
Time averaging:  $D(\mathbf{q}, \tau) = \langle |\mathcal{F}(\Delta I)|^2 \rangle_t$

Azimuthal averaging:  $D(\mathbf{q}, \tau) \rightarrow D(\mathbf{q}, \tau) \dots$



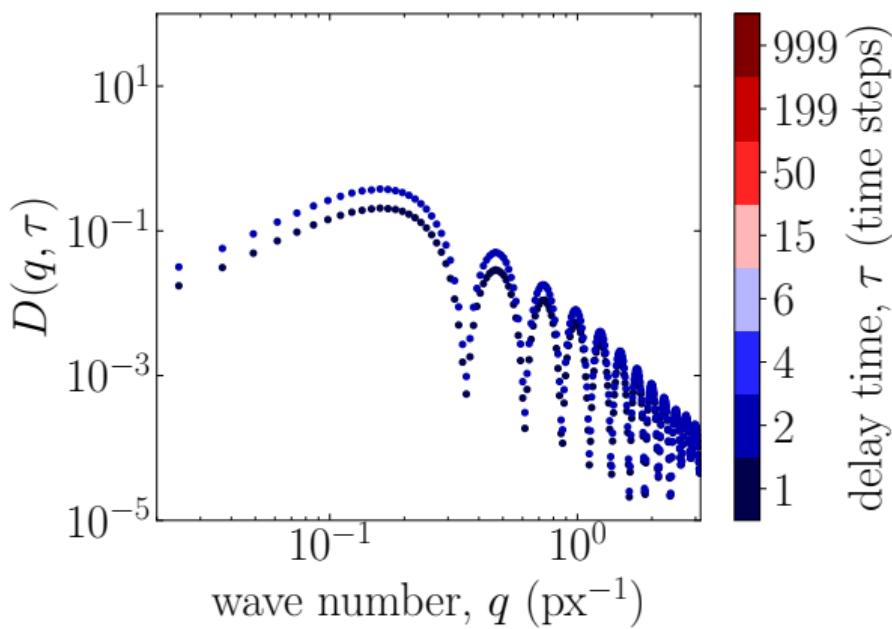
# The image structure function $D(q, \tau)$

$\tau = 2$  time steps



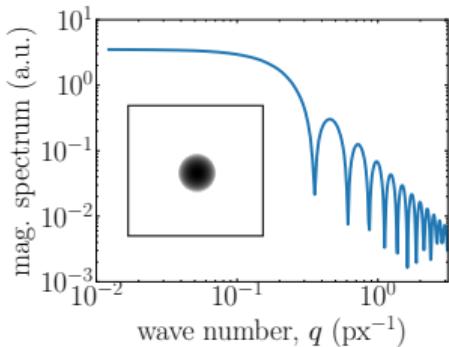
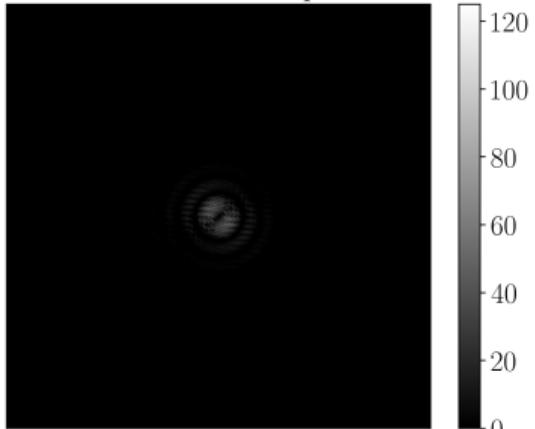
Time averaging:  $D(\mathbf{q}, \tau) = \langle |\mathcal{F}(\Delta I)|^2 \rangle_t$

Azimuthal averaging:  $D(\mathbf{q}, \tau) \rightarrow D(\mathbf{q}, \tau) \dots$



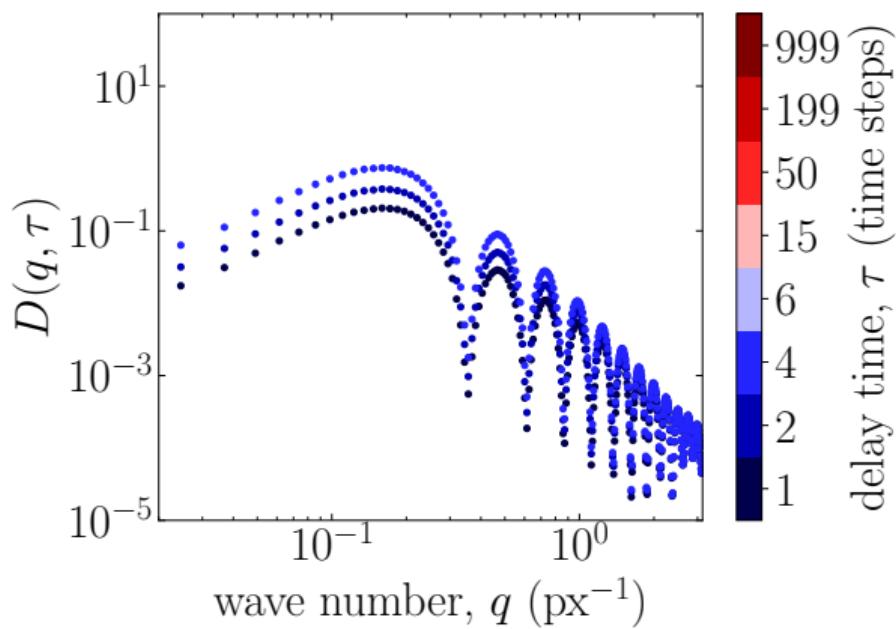
# The image structure function $D(q, \tau)$

$\tau = 4$  time steps



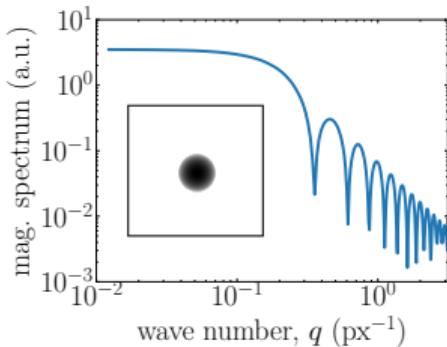
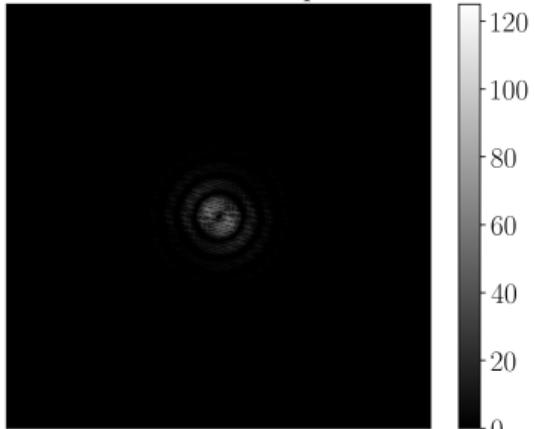
Time averaging:  $D(\mathbf{q}, \tau) = \langle |\mathcal{F}(\Delta I)|^2 \rangle_t$

Azimuthal averaging:  $D(\mathbf{q}, \tau) \rightarrow D(\mathbf{q}, \tau) \dots$



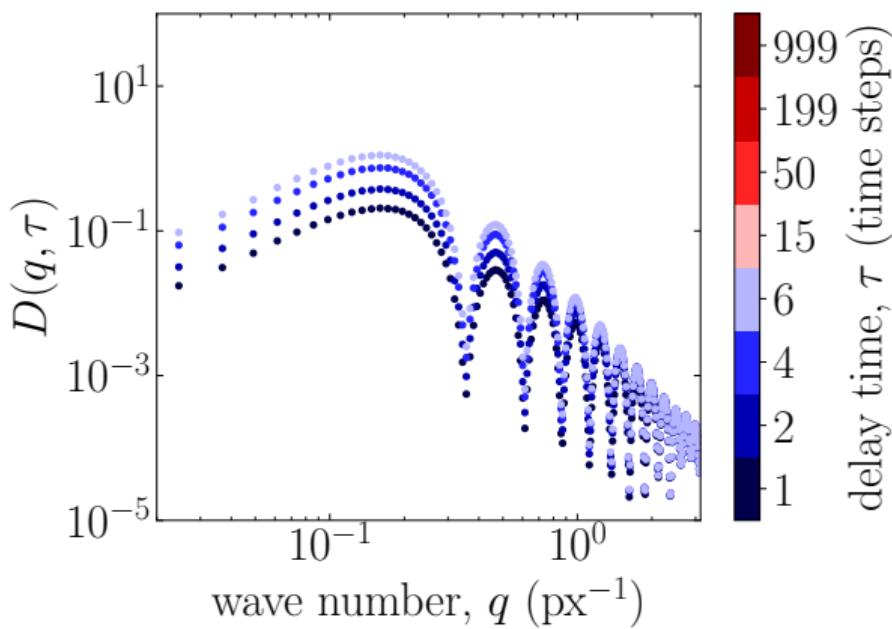
# The image structure function $D(q, \tau)$

$\tau = 6$  time steps

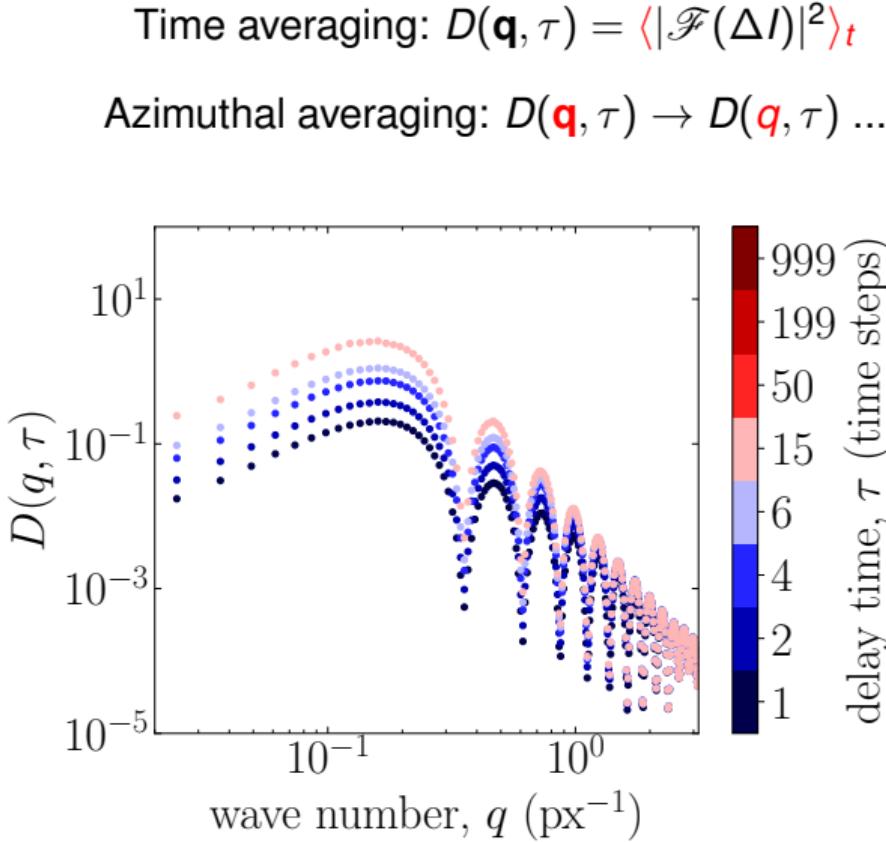
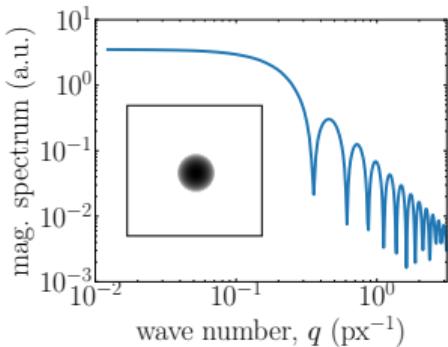
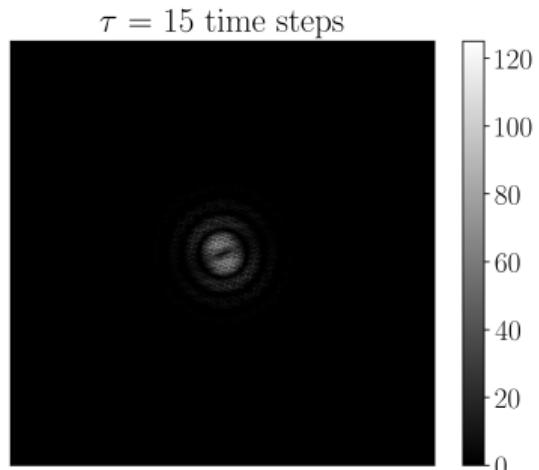


Time averaging:  $D(\mathbf{q}, \tau) = \langle |\mathcal{F}(\Delta I)|^2 \rangle_t$

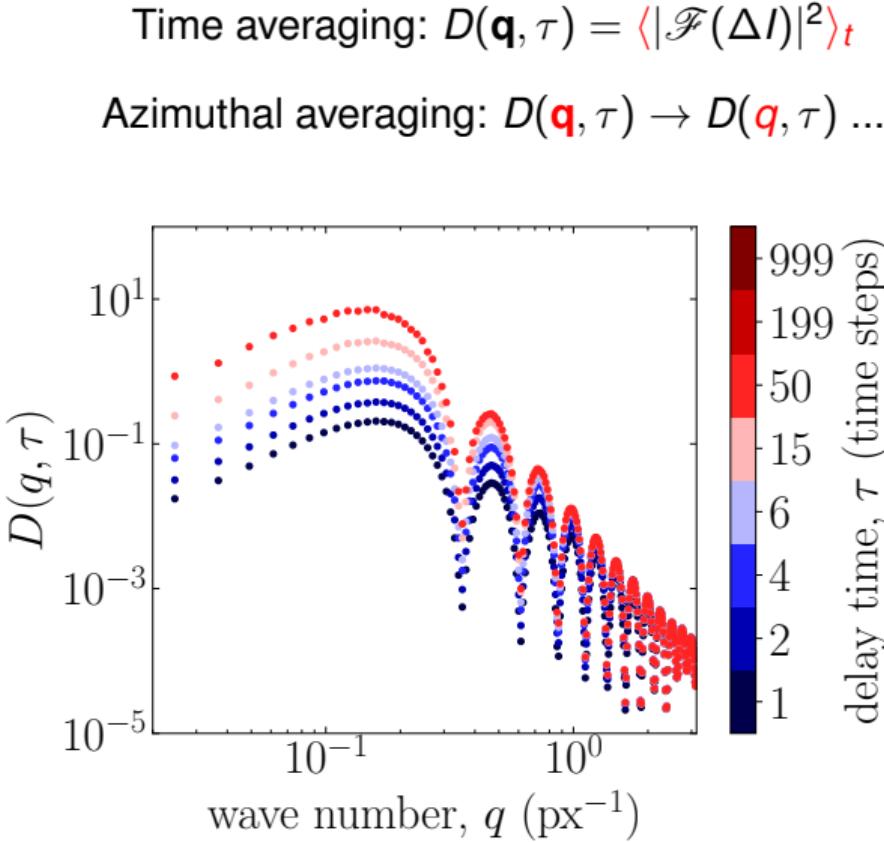
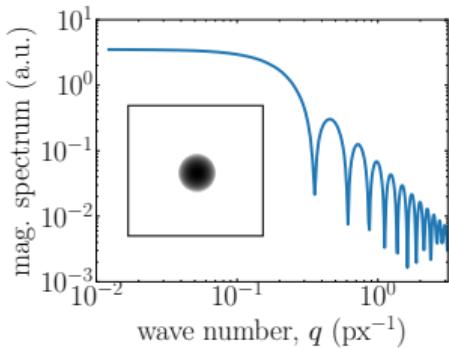
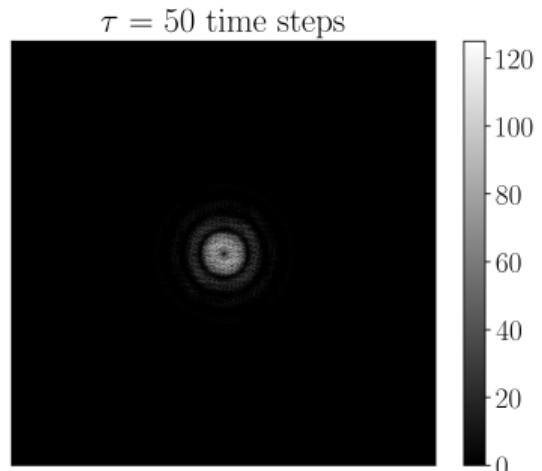
Azimuthal averaging:  $D(\mathbf{q}, \tau) \rightarrow D(\mathbf{q}, \tau) \dots$



# The image structure function $D(q, \tau)$

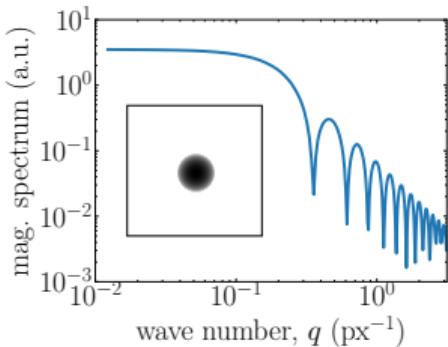
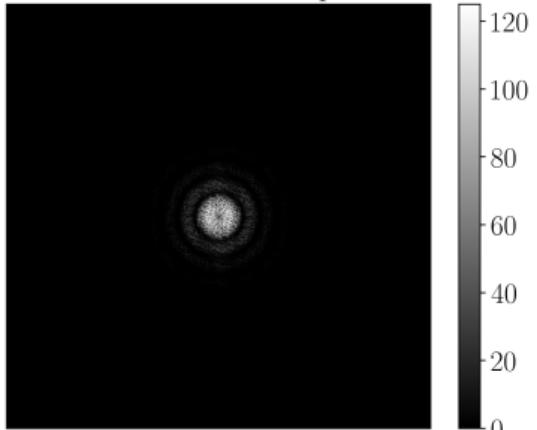


# The image structure function $D(q, \tau)$



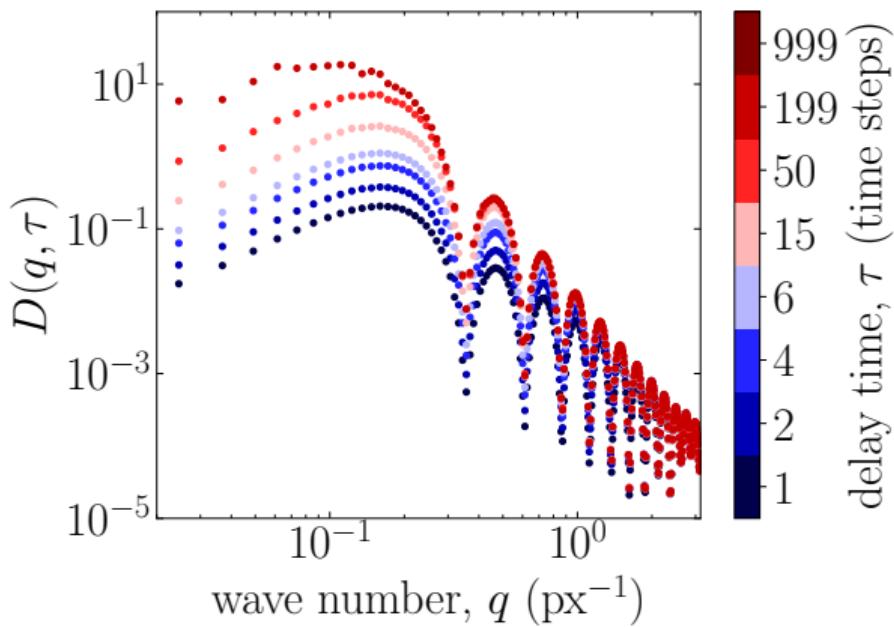
# The image structure function $D(q, \tau)$

$\tau = 199$  time steps



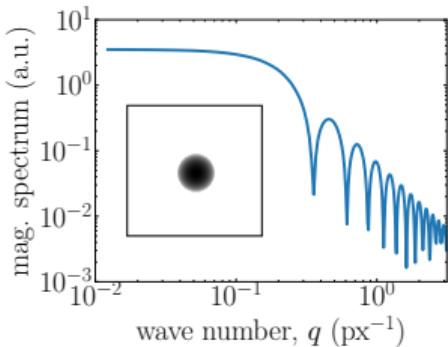
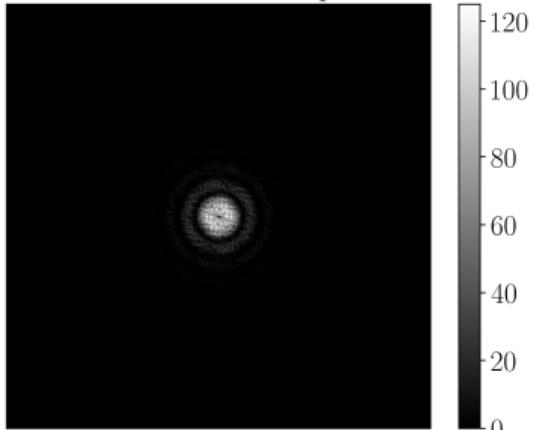
Time averaging:  $D(\mathbf{q}, \tau) = \langle |\mathcal{F}(\Delta I)|^2 \rangle_t$

Azimuthal averaging:  $D(\mathbf{q}, \tau) \rightarrow D(\mathbf{q}, \tau) \dots$



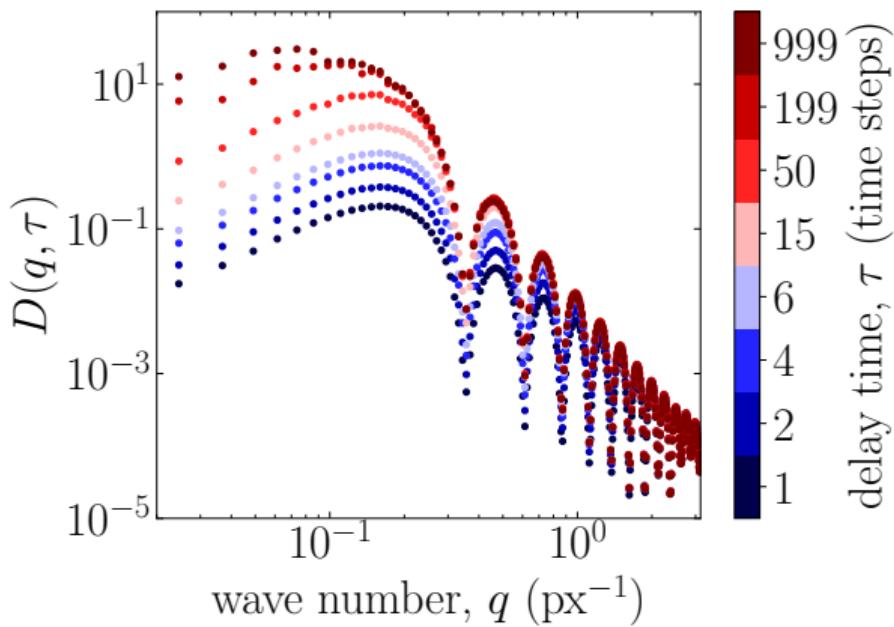
# The image structure function $D(q, \tau)$

$\tau = 999$  time steps

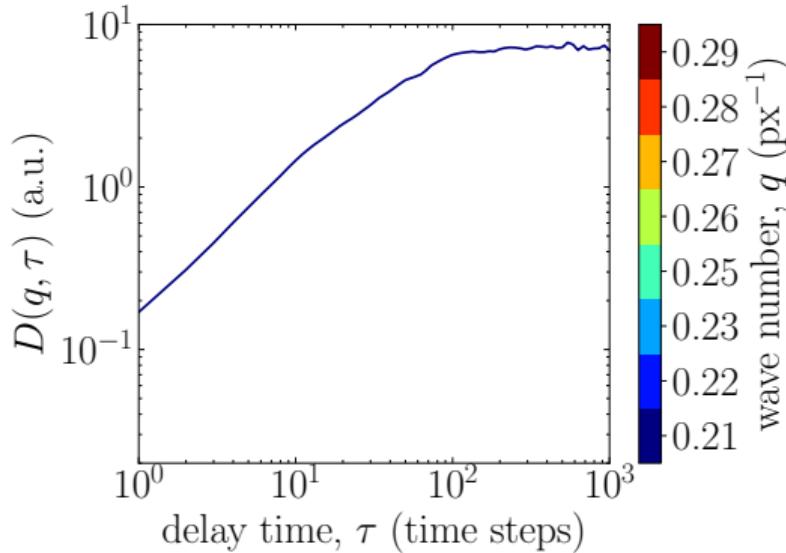
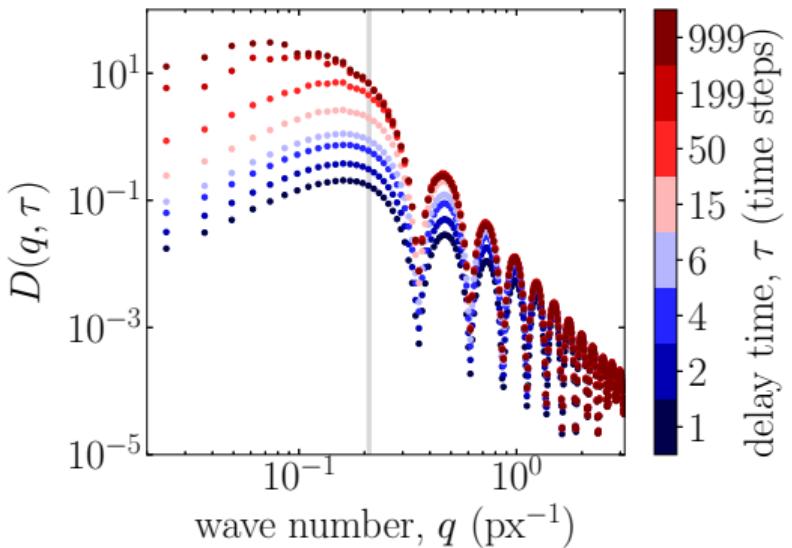


Time averaging:  $D(\mathbf{q}, \tau) = \langle |\mathcal{F}(\Delta I)|^2 \rangle_t$

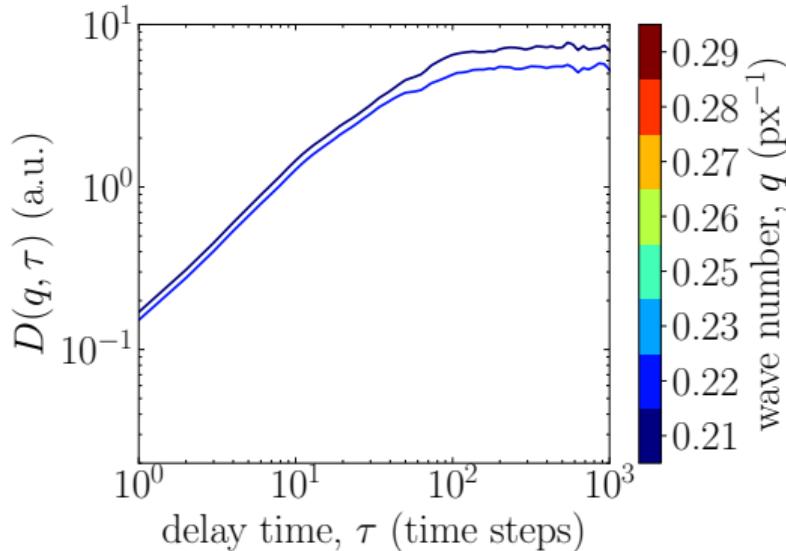
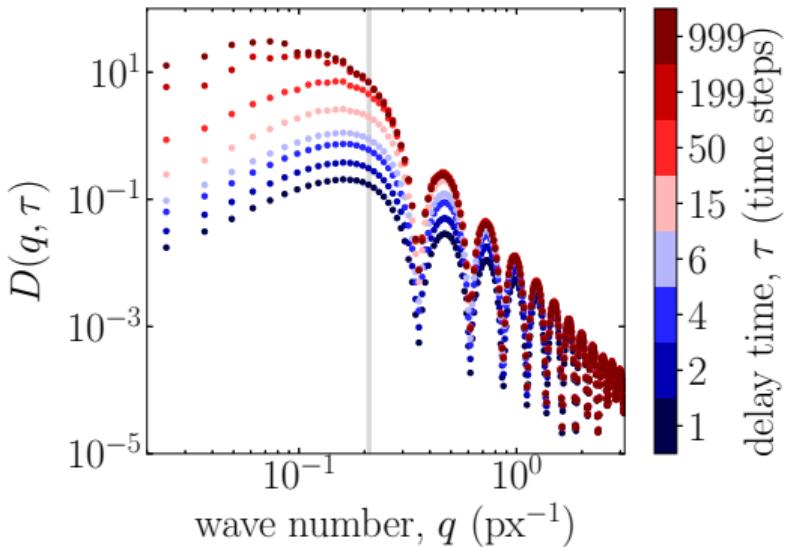
Azimuthal averaging:  $D(\mathbf{q}, \tau) \rightarrow D(\mathbf{q}, \tau) \dots$



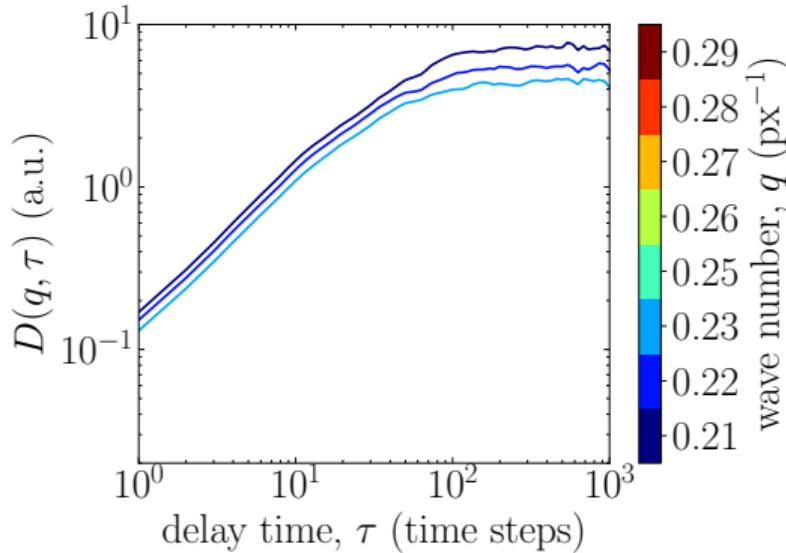
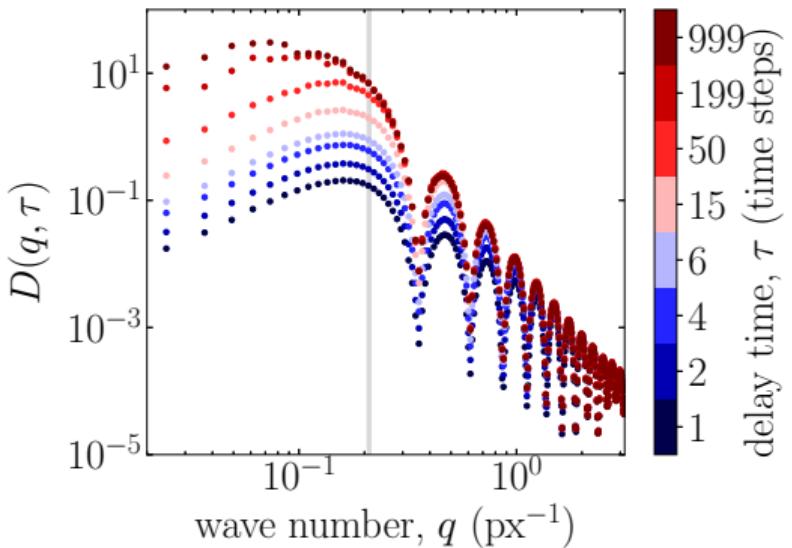
## The image structure function $D(q, \tau)$



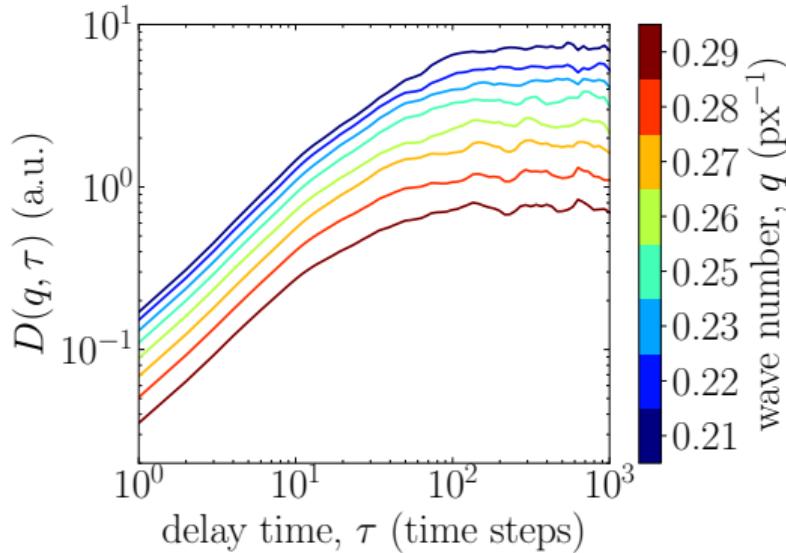
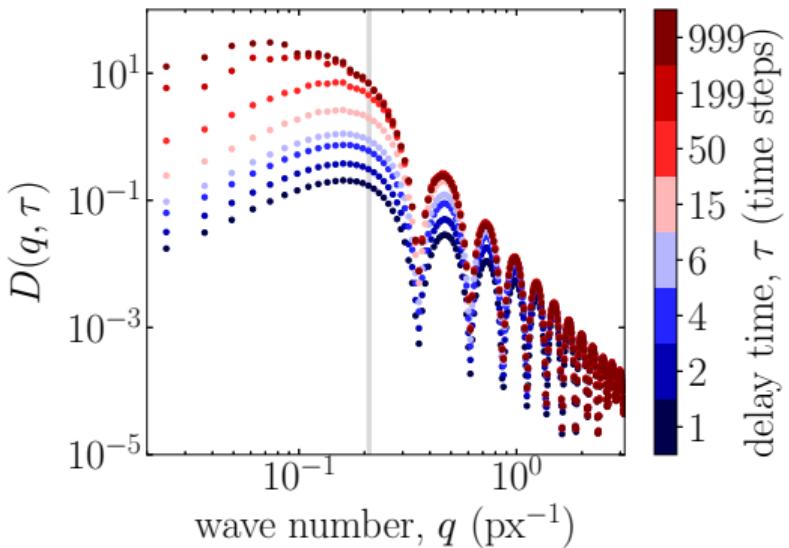
## The image structure function $D(q, \tau)$



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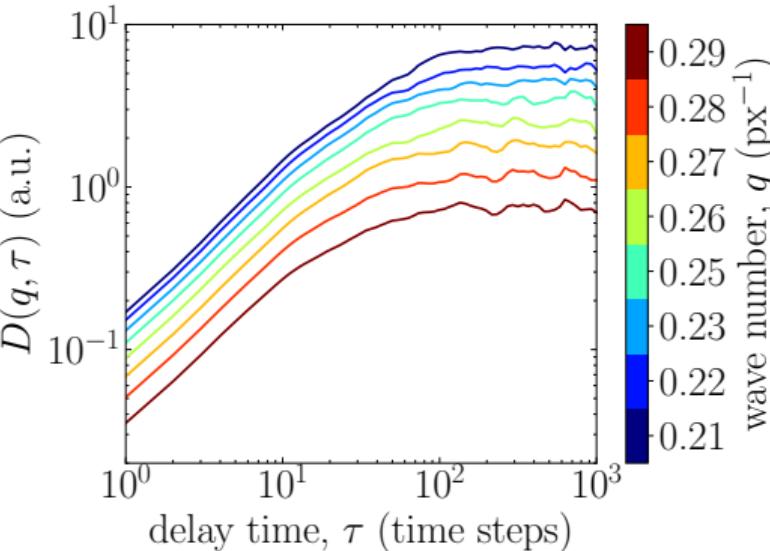
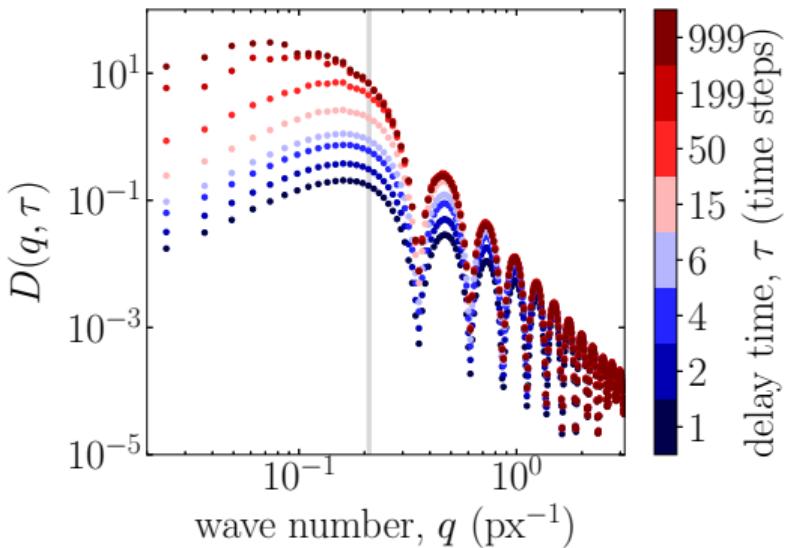


## The image structure function $D(q, \tau)$



$$\begin{aligned}
 D(q, \tau) &= \left\langle |I(q, t + \tau) - I(q, t)|^2 \right\rangle_t \\
 &= A(q) \left[ 1 - \frac{\left\langle I^*(q, t) I(q, t + \tau) \right\rangle_t}{\left\langle |I(q, t)|^2 \right\rangle_t} \right] + B(q)
 \end{aligned}$$

## The image structure function $D(q, \tau)$



$$\begin{aligned}
 D(q, \tau) &= \left\langle |I(q, t + \tau) - I(q, t)|^2 \right\rangle_t \\
 &= A(q) \left[ 1 - \underbrace{\frac{\langle I^*(q, t) I(q, t + \tau) \rangle_t}{\langle |I(q, t)|^2 \rangle_t}}_{\text{image correlation function}} \right] + B(q)
 \end{aligned}$$

## Linear space invariant imaging

image correlation function

$$g(\mathbf{q}, \tau) = \frac{\langle I^*(\mathbf{q}, t) I(\mathbf{q}, t + \tau) \rangle_t}{\langle |I(\mathbf{q}, t)|^2 \rangle_t}$$

# Linear space invariant imaging

image correlation function

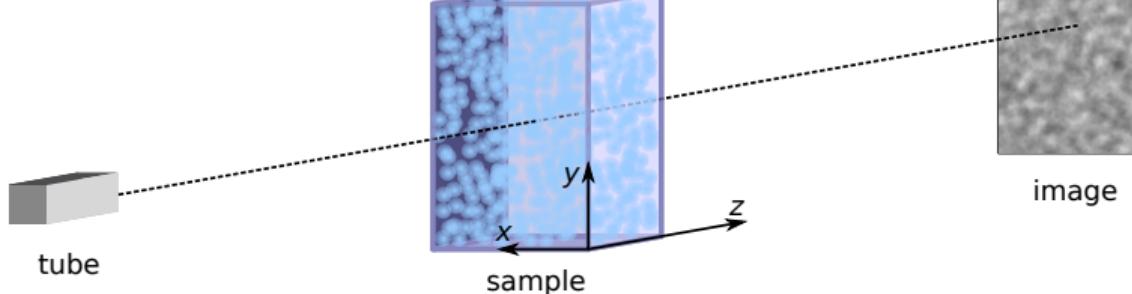
$$g(\mathbf{q}, \tau) = \frac{\langle I^*(\mathbf{q}, t)I(\mathbf{q}, t + \tau) \rangle_t}{\langle |I(\mathbf{q}, t)|^2 \rangle_t}$$

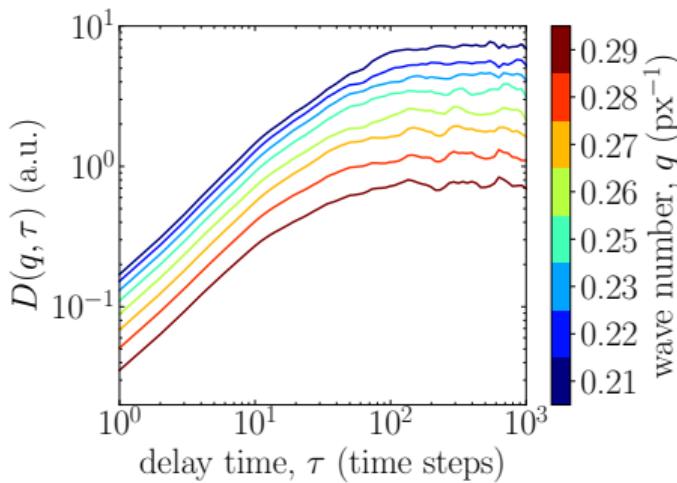
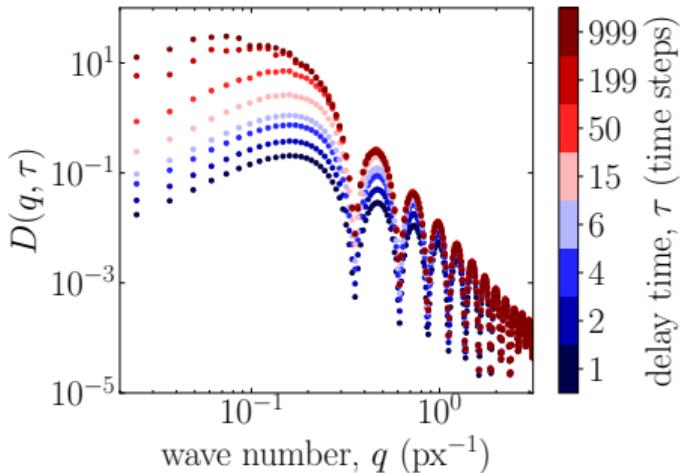
intermediate scattering function

$$f(\mathbf{q}, \tau) = \frac{\langle \rho^*(\mathbf{q}, t)\rho(\mathbf{q}, t + \tau) \rangle_t}{\langle |\rho(\mathbf{q}, t)|^2 \rangle_t}$$

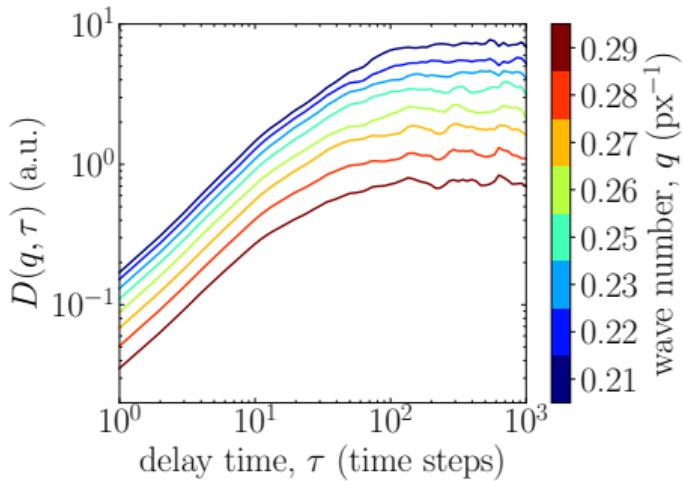
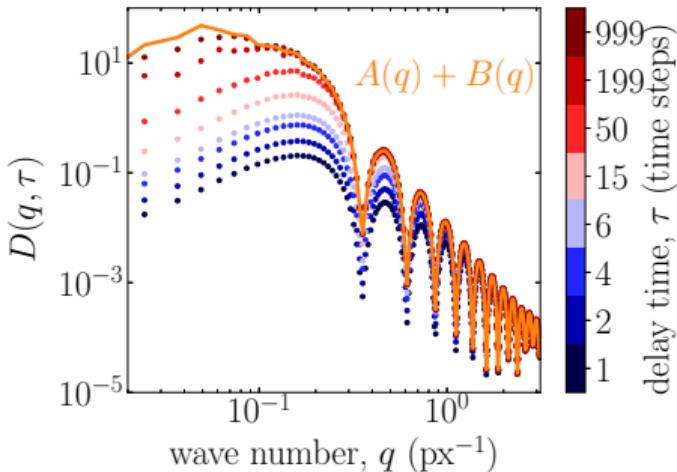
Linear space-invariant imaging:

$$I(\mathbf{r}, t) = I_0 + \int d\mathbf{r}' dz' T(\mathbf{r} - \mathbf{r}', -z') c(\mathbf{r}', z', t)$$



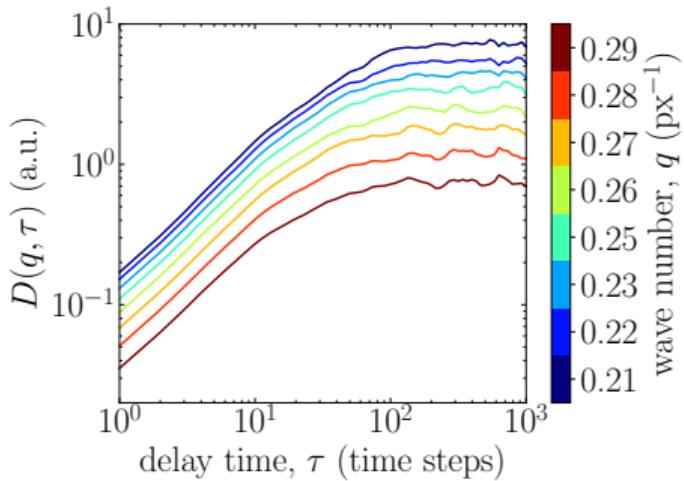
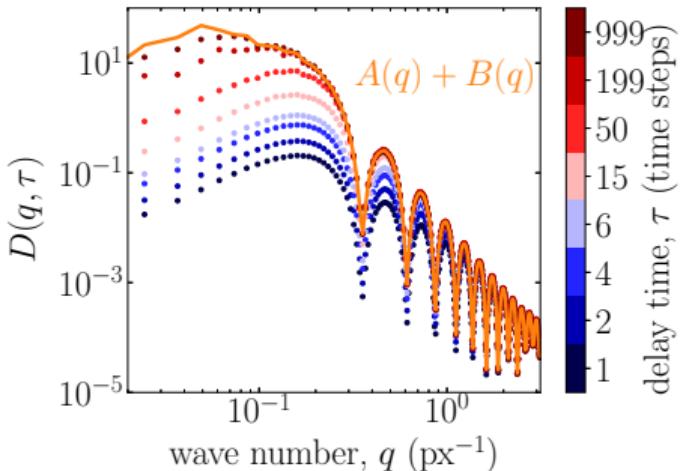


$$\begin{aligned}
 D(q, \tau) &= \left\langle |I(q, t + \tau) - I(q, t)|^2 \right\rangle_t \\
 &= A(q) \underbrace{\left[ 1 - \frac{\langle I^*(q, t) I(q, t + \tau) \rangle_t}{\langle |I(q, t)|^2 \rangle_t} \right]}_{\text{image correlation function}} + B(q)
 \end{aligned}$$



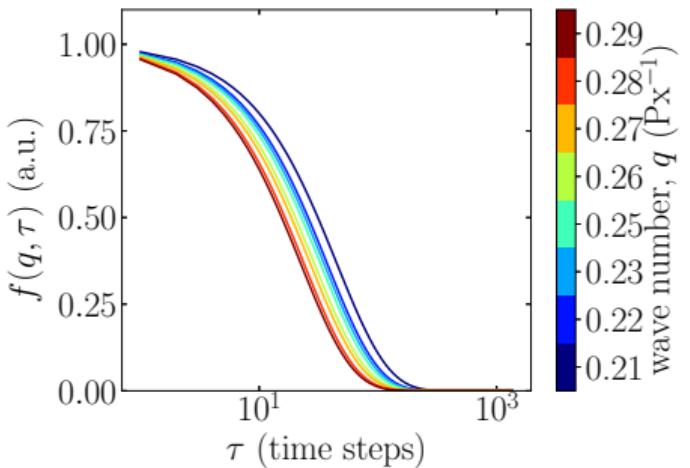
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- $d(q, \tau \rightarrow 0) = B(q) = 0$
- $d(q, \tau \rightarrow 0) = A(q) + B(q)$

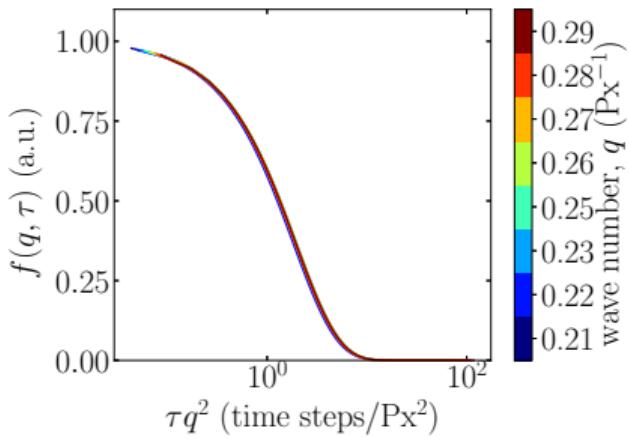
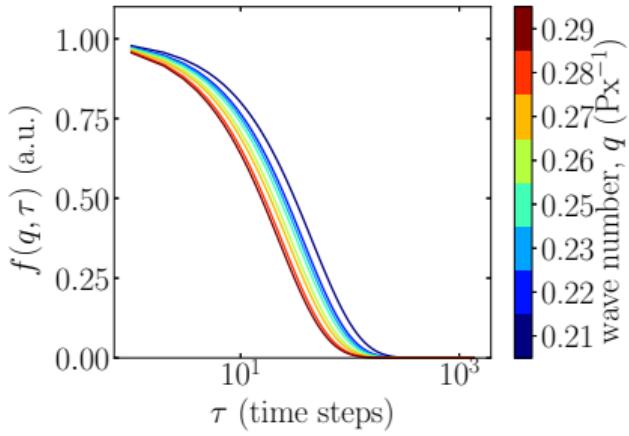


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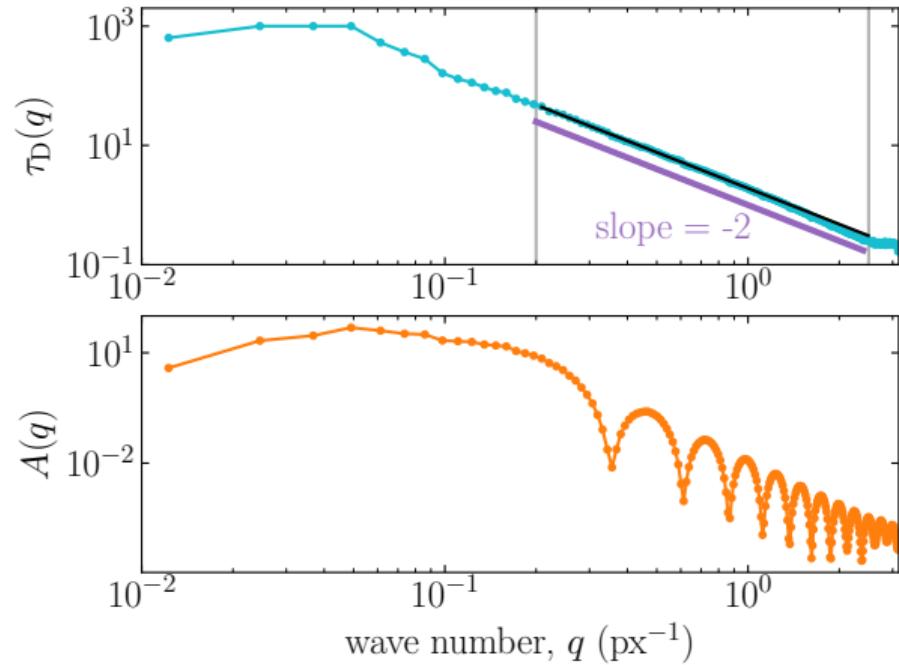
- $d(q, \tau \rightarrow 0) = B(q) = 0$
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# Intermediate scattering function $f(q, \tau)$



Brownian motion:  
 $f(q, \tau) = \exp(-q^2\tau/\tau_D)$   
Accuracy: 2% - 6%

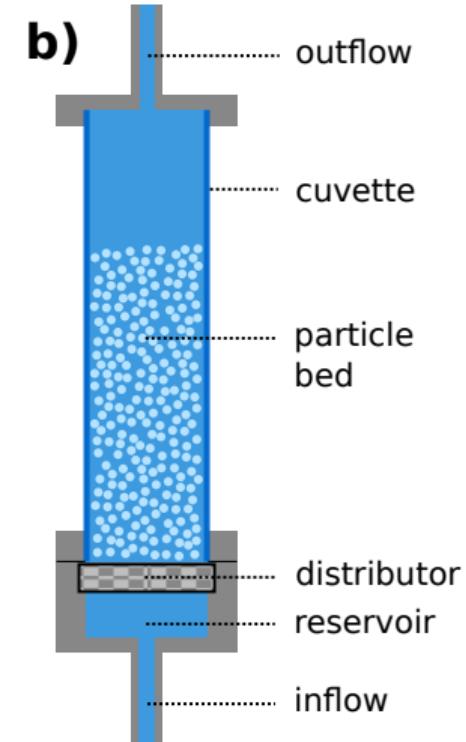
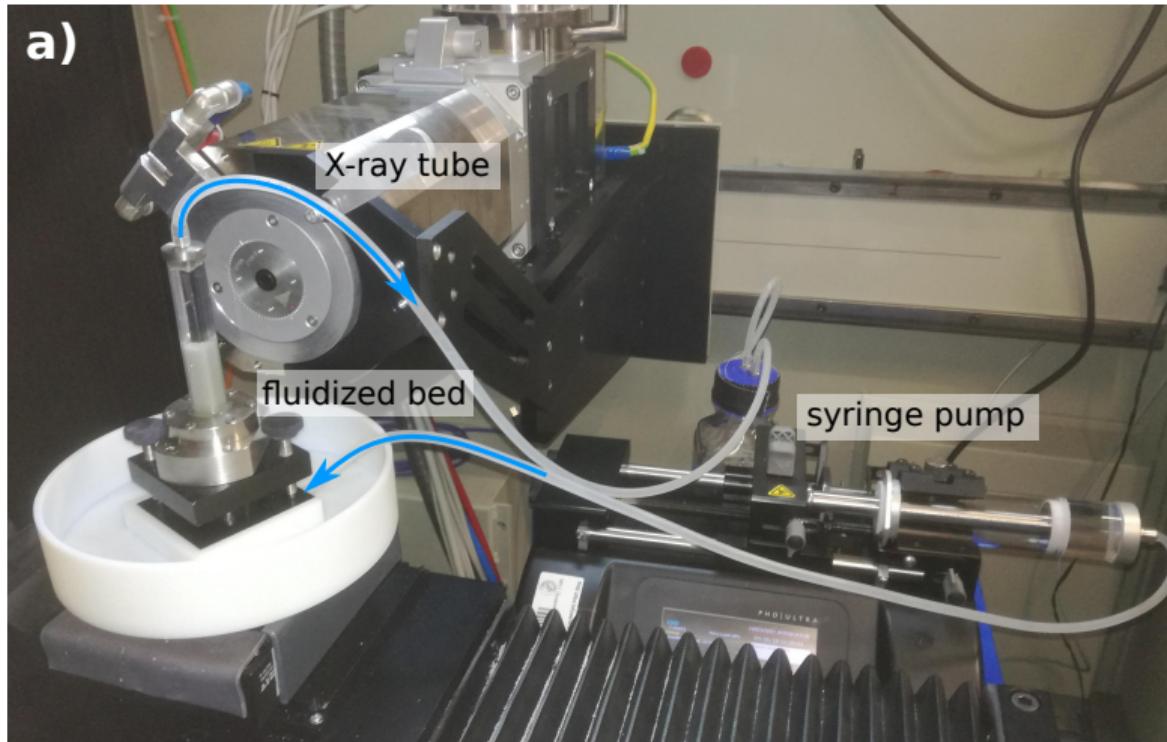


Validating X-DFA on a sedimenting  
suspension

Measurements on a fluidized bed

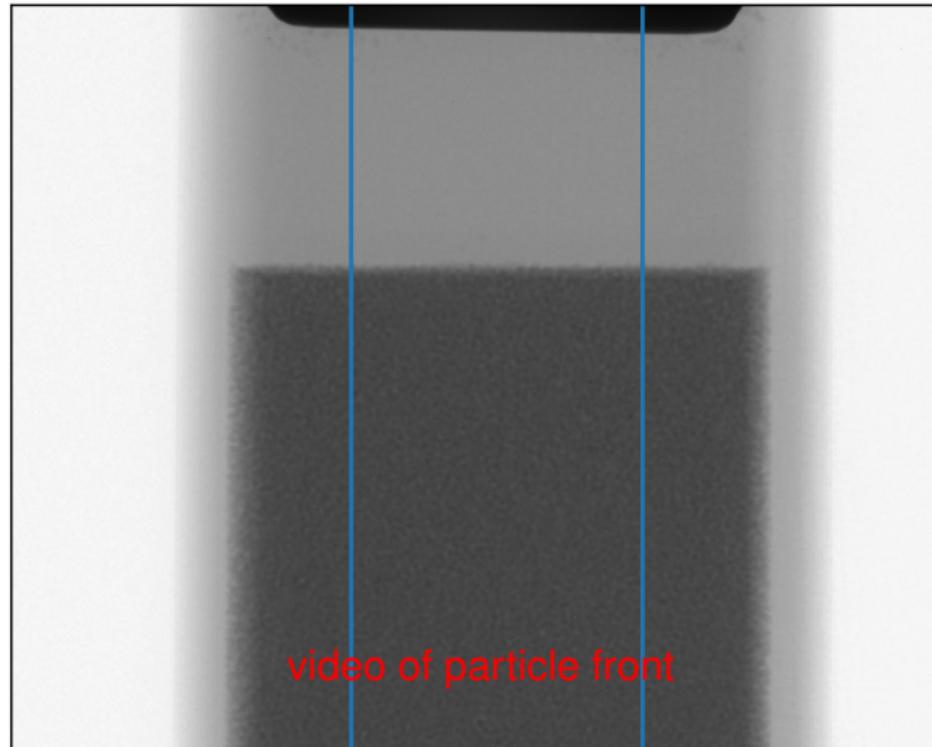
# Experimental validation: Sedimenting suspension

track sedimentation front ↔ DFA

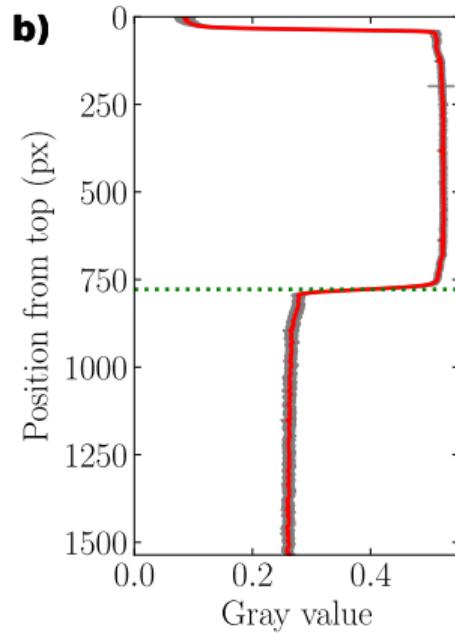
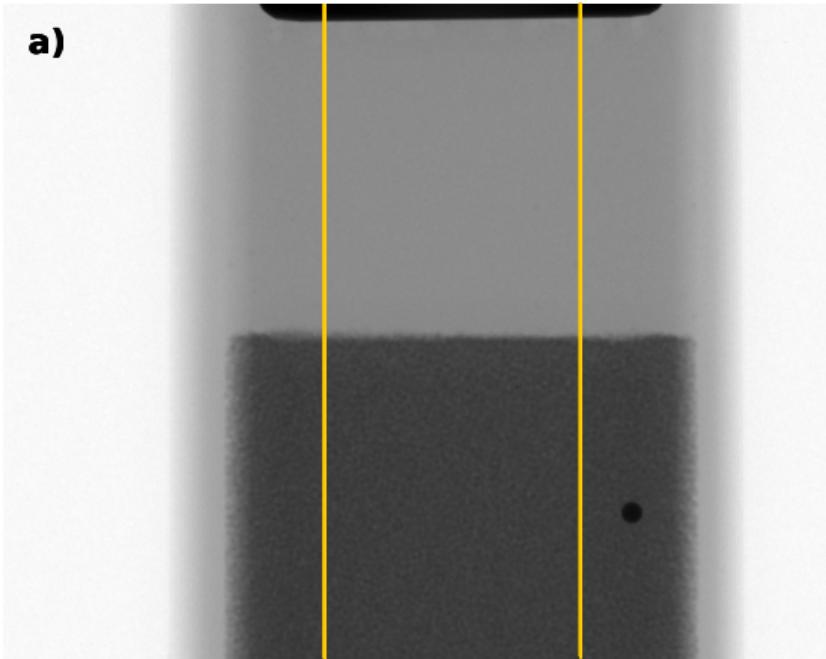


## Experimental validation: Sedimenting suspension

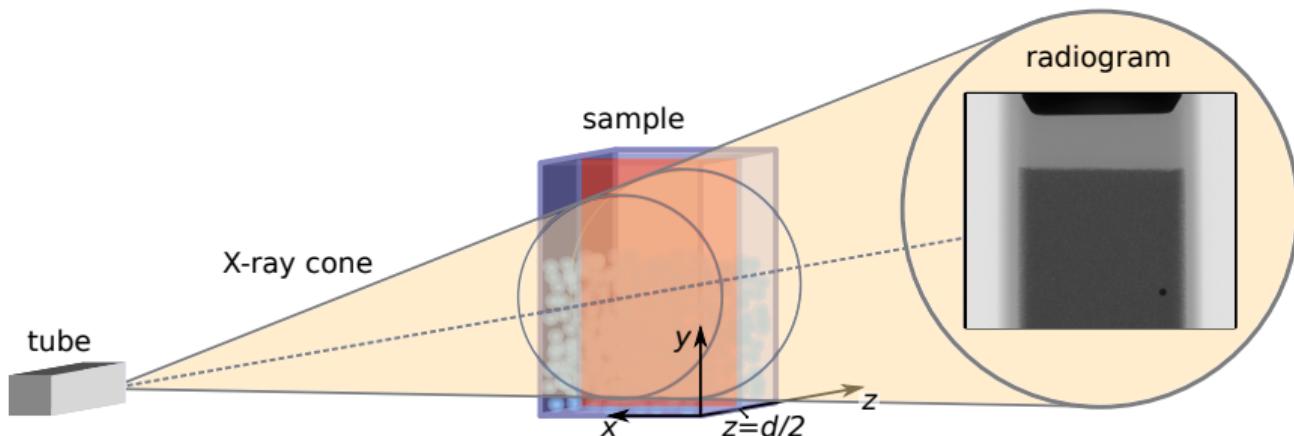
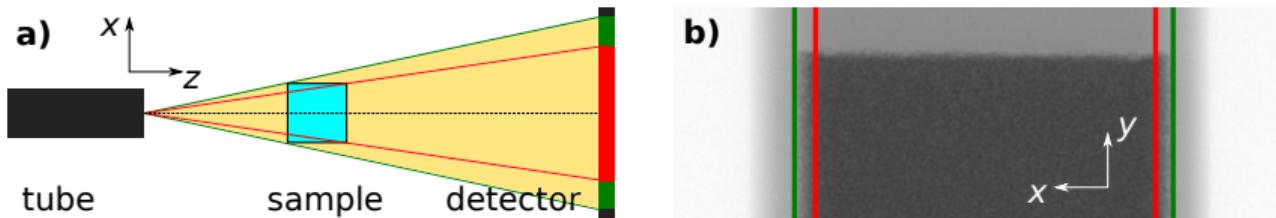
track sedimentation front ↔ DFA



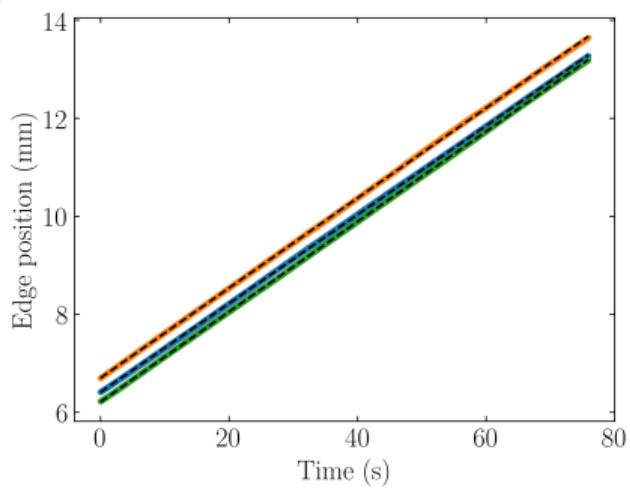
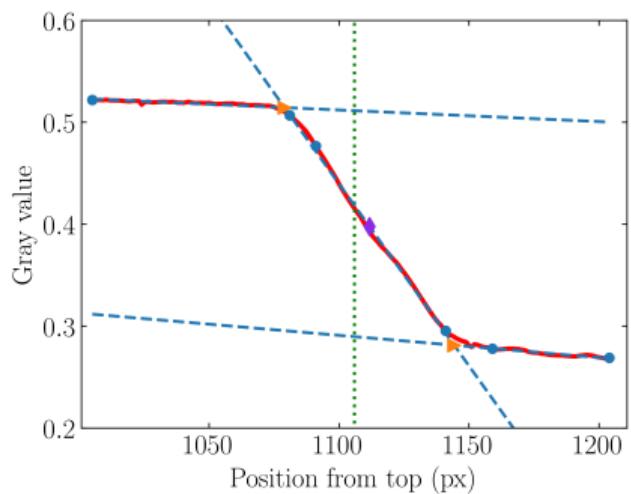
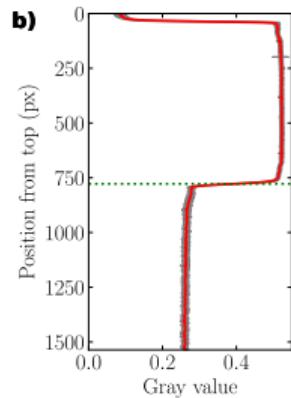
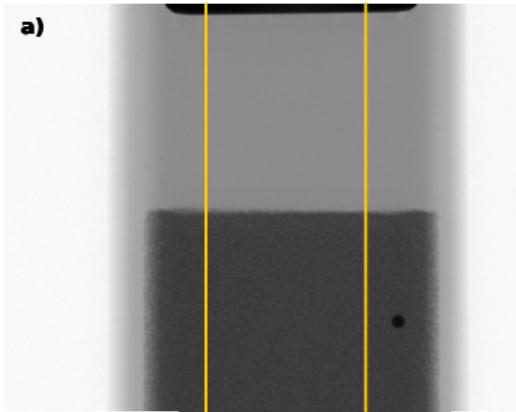
## Tracking of particle front



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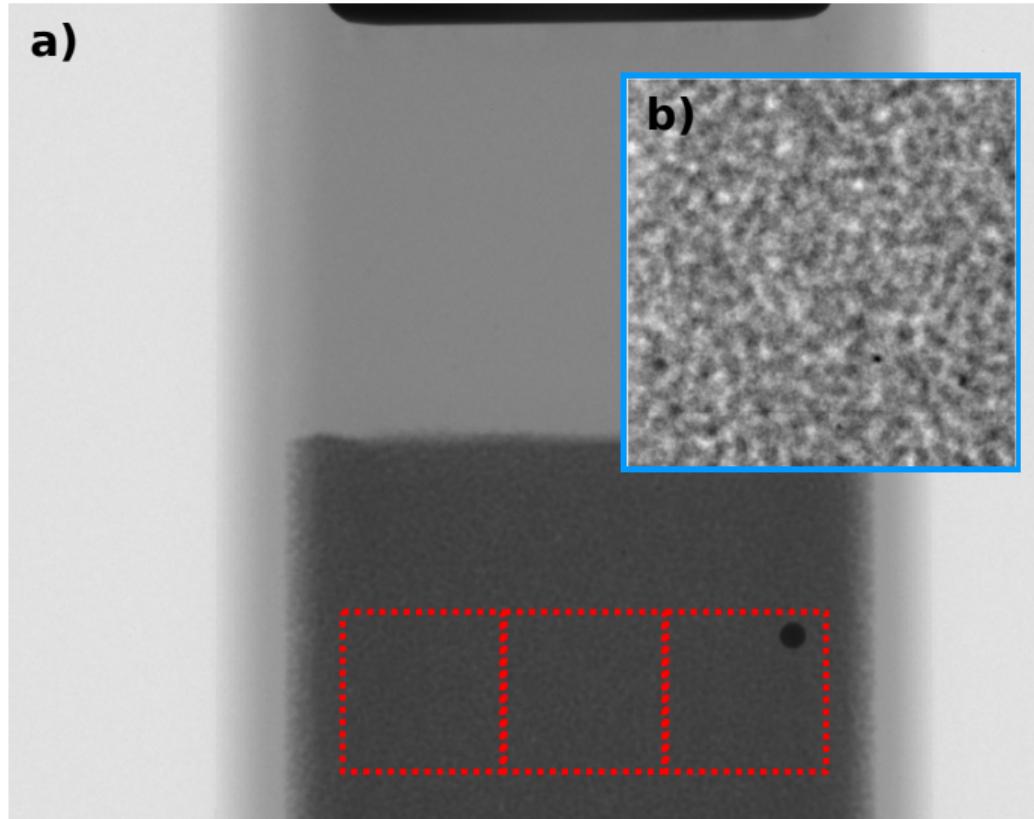


# Tracking of particle front

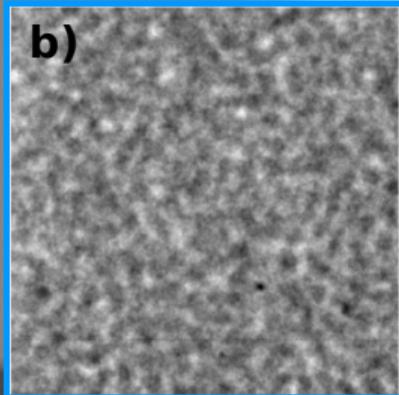


# X-DFA for sedimenting particles

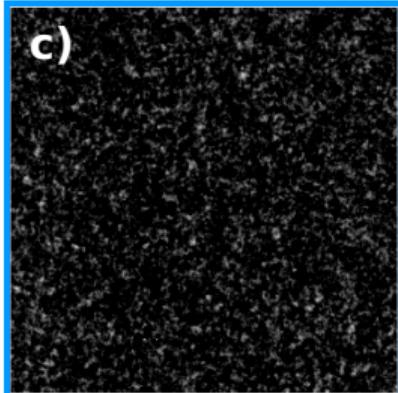
a)



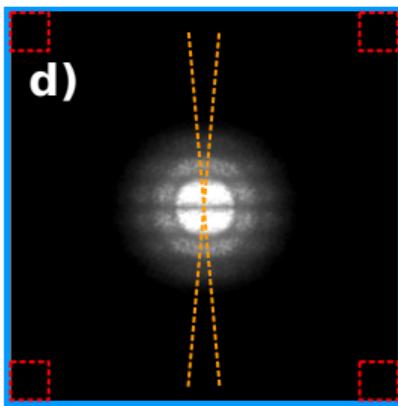
b)



c)

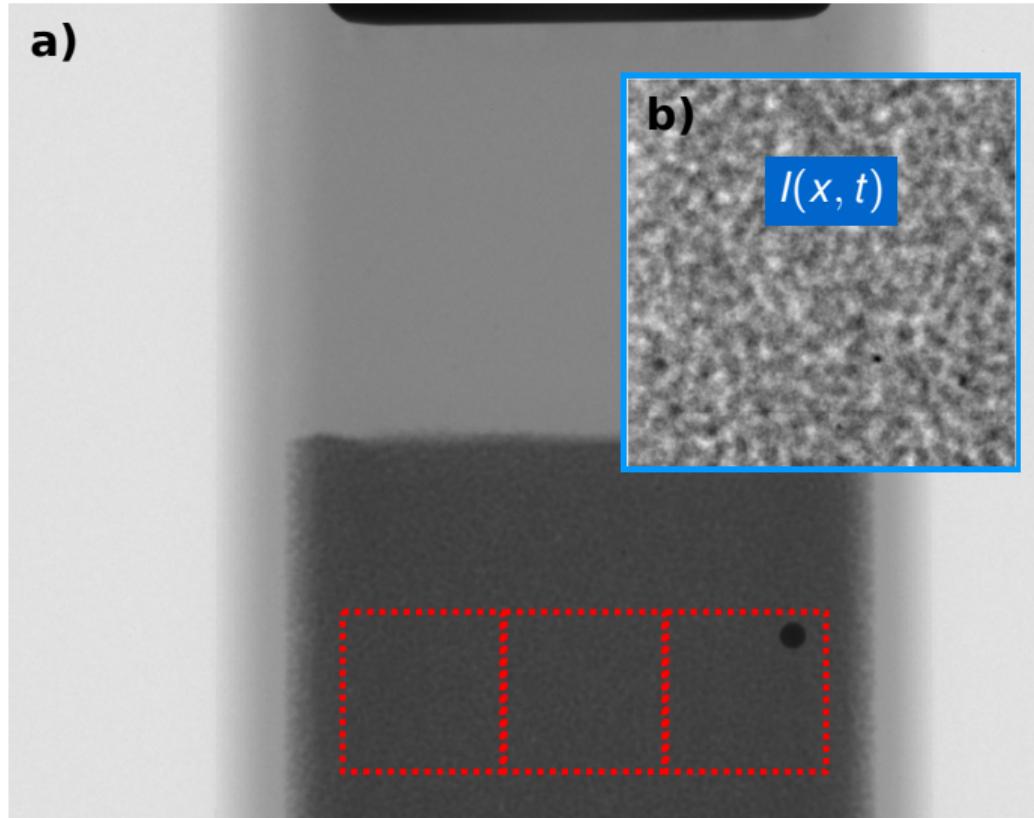


d)

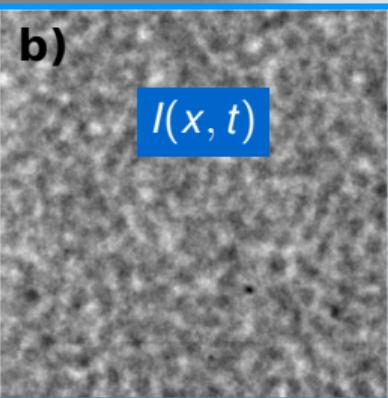


# X-DFA for sedimenting particles

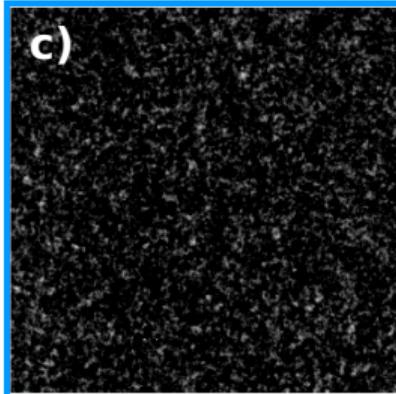
a)



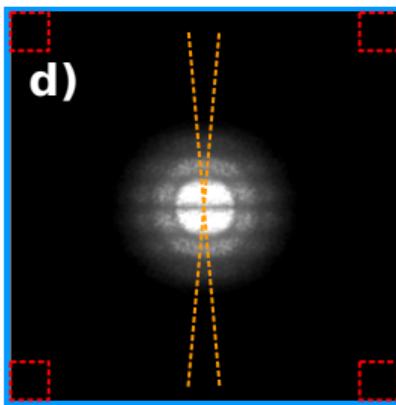
b)



c)

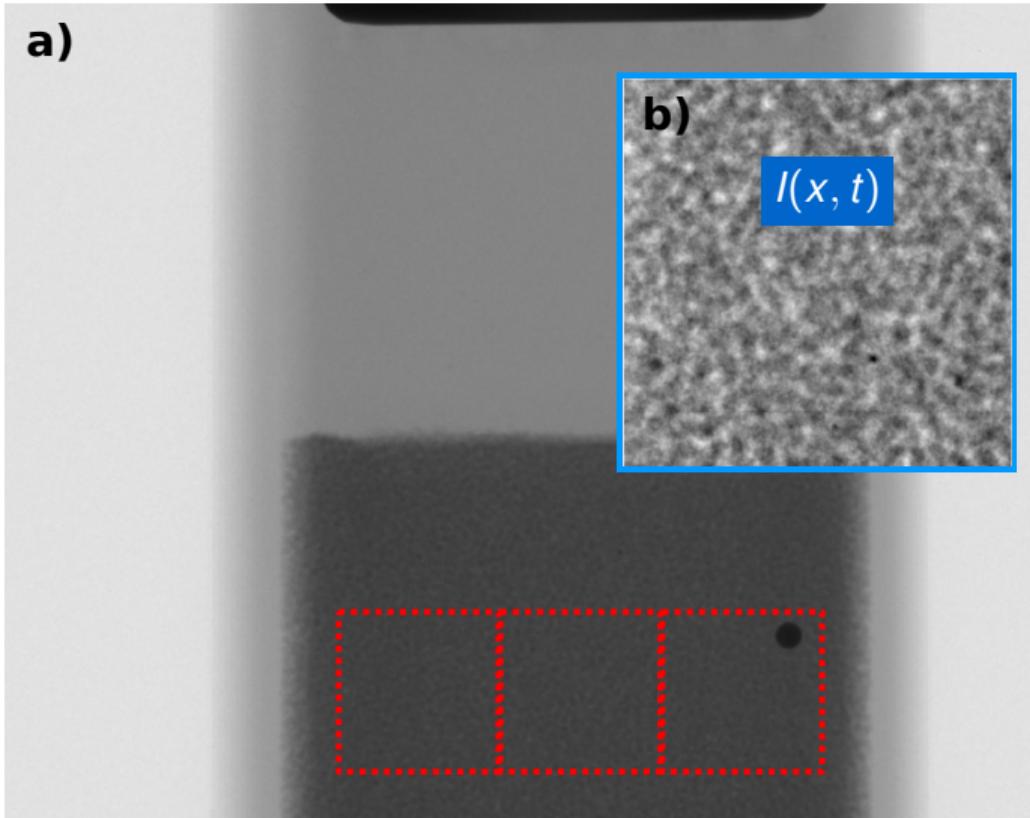


d)

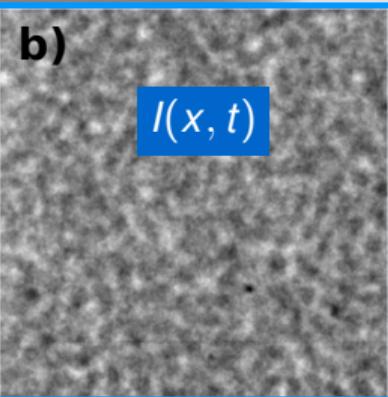


# X-DFA for sedimenting particles

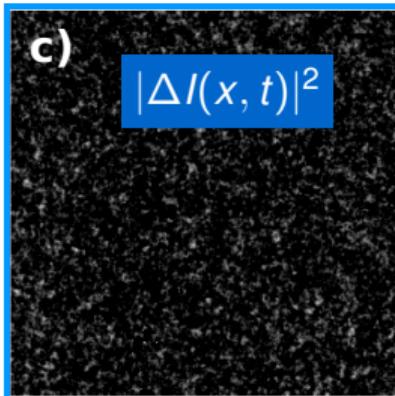
a)



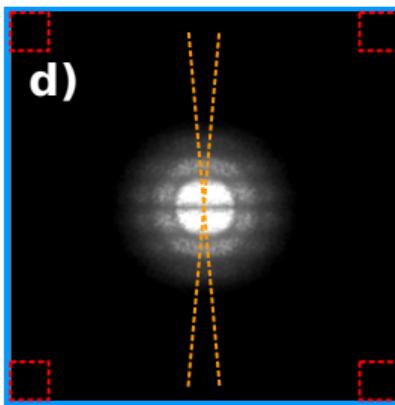
b)



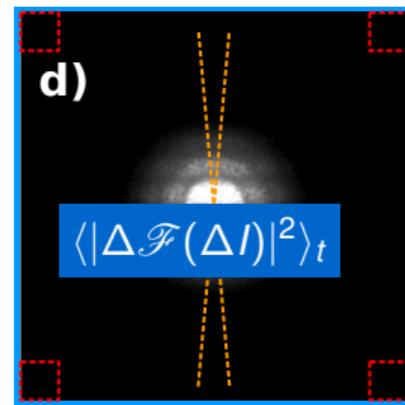
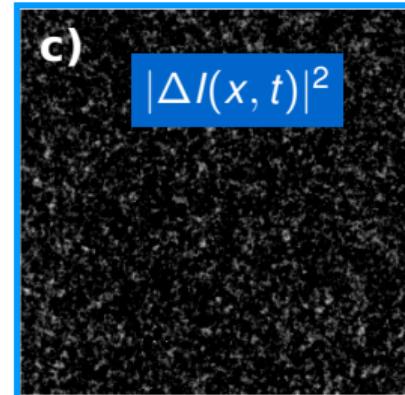
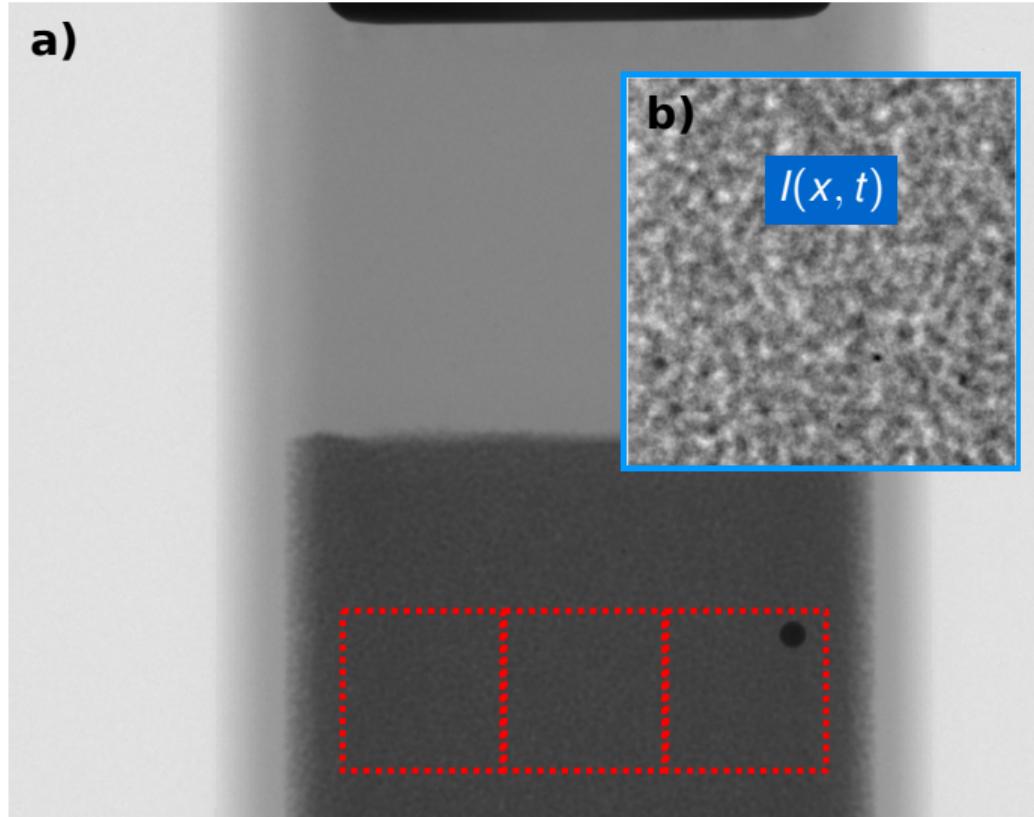
c)



d)



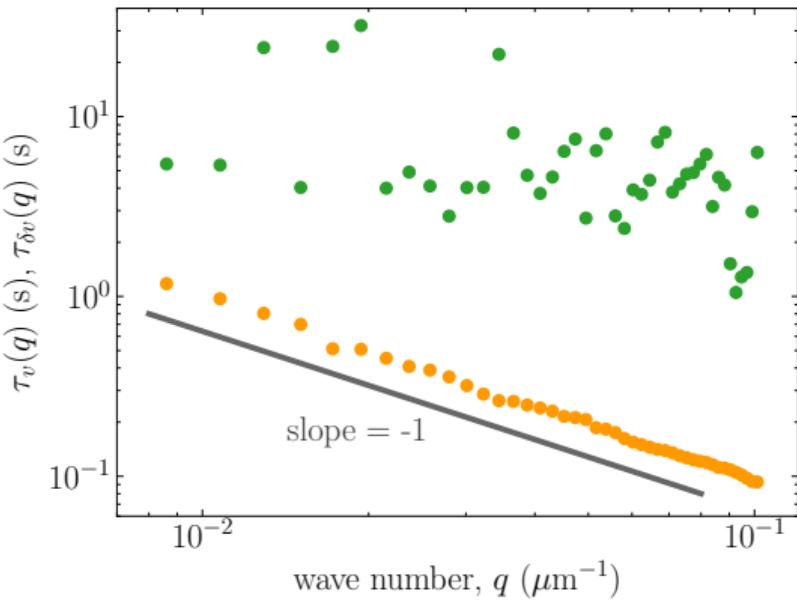
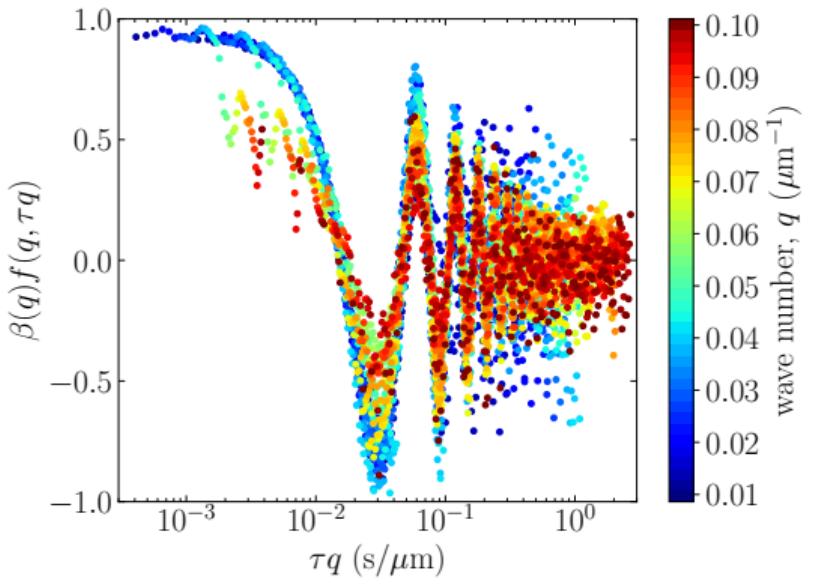
# X-DFA for sedimenting particles



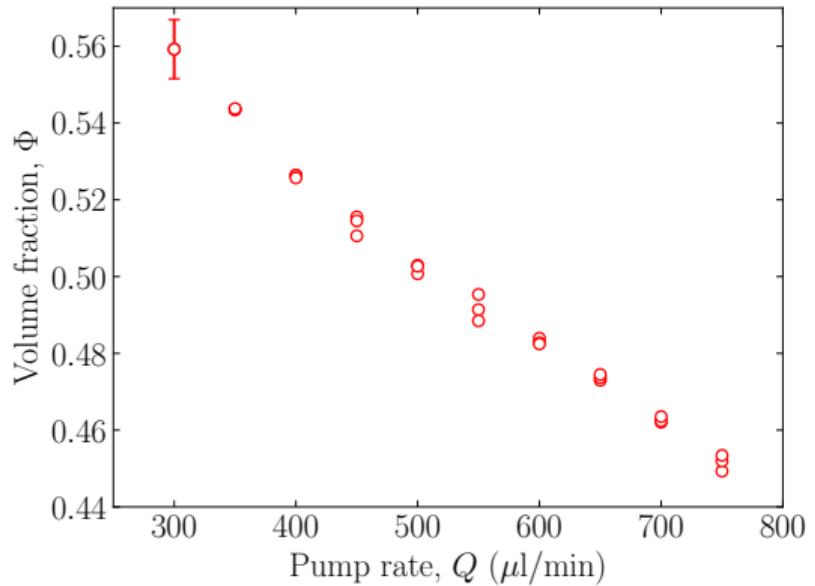
## X-DFA for sedimenting particles

$$f(q, \tau) = \cos(q\langle v_s \rangle \tau) \exp\left(-\frac{1}{2}q^2 \delta v^2 \tau^2\right)$$

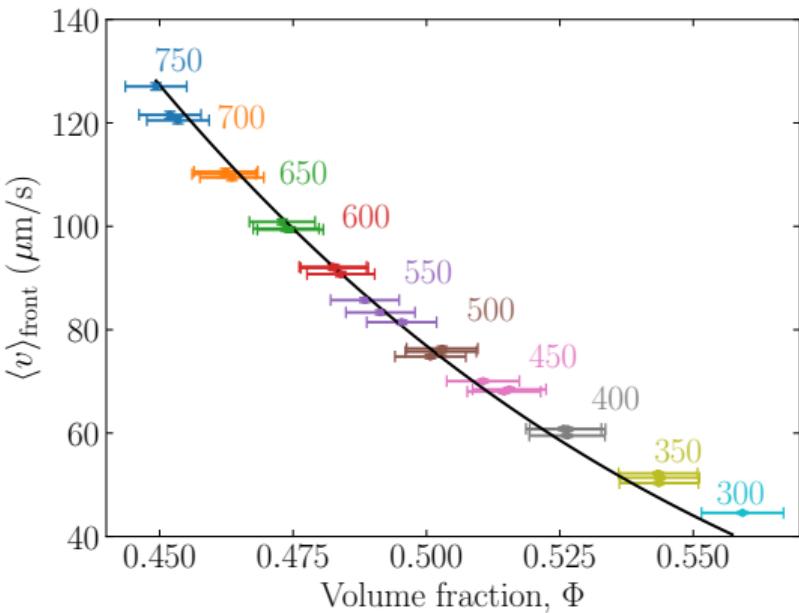
$$\langle v_s \rangle = \langle \Delta r \rangle / \tau_\nu, \langle \delta v \rangle = \langle \delta r \rangle / \tau_{\delta\nu}$$



## Richardson-Zaki law

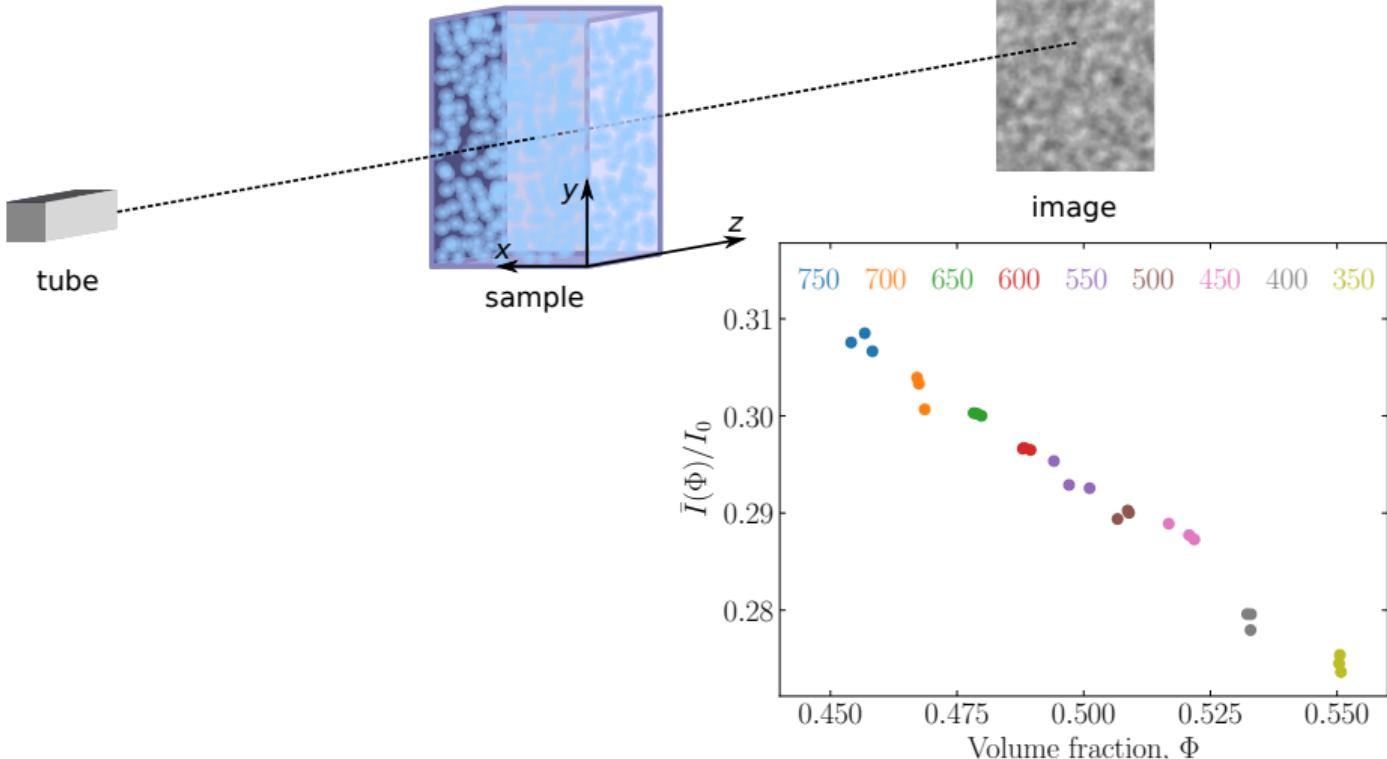


$$\frac{\langle v \rangle_{\text{front}}}{v_{\text{Stokes}}} = (1 - \Phi)^n$$

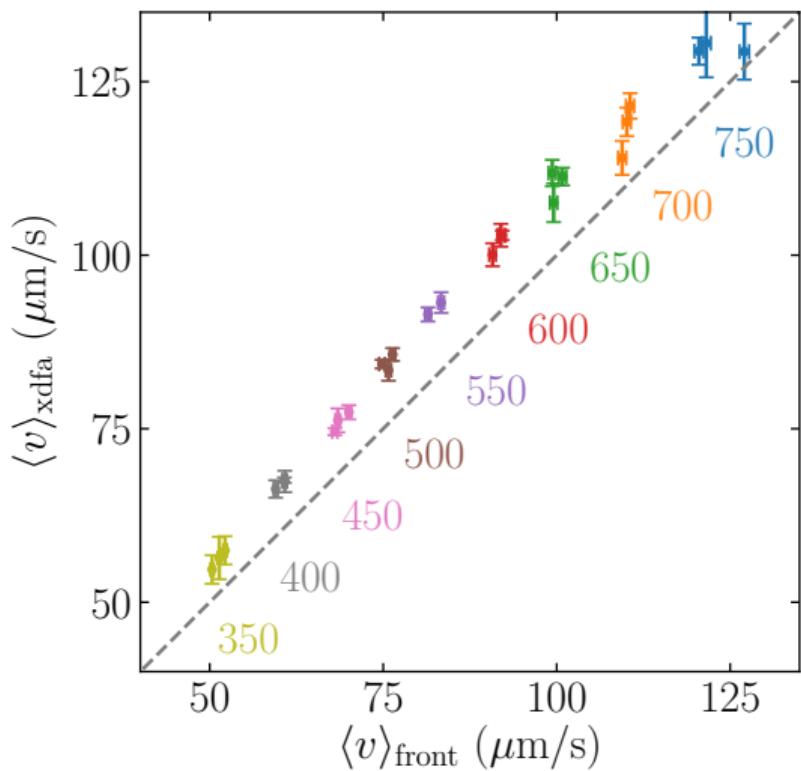


# X-DFA: Requirement of linear space invariant imaging

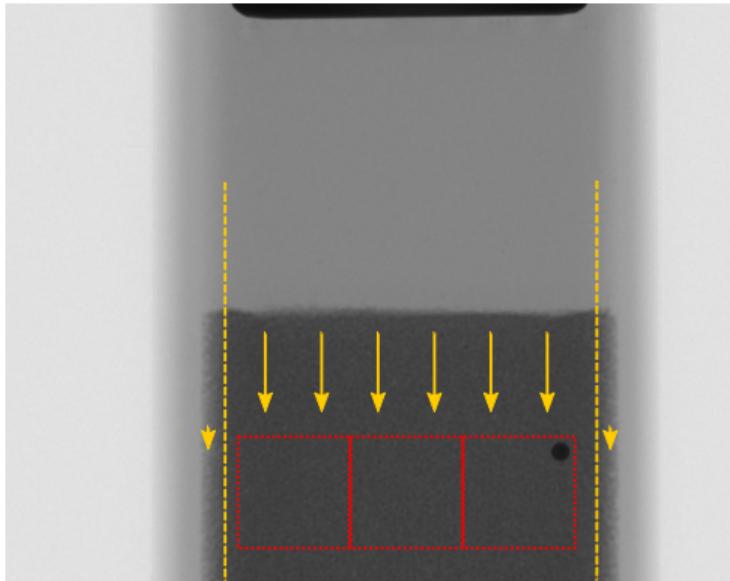
$$I(\mathbf{r}, t) = I_0 + \int d\mathbf{r}' dz' T(\mathbf{r} - \mathbf{r}', -z') c(\mathbf{r}', z', t)$$



## Front tracking vs. X-DFA

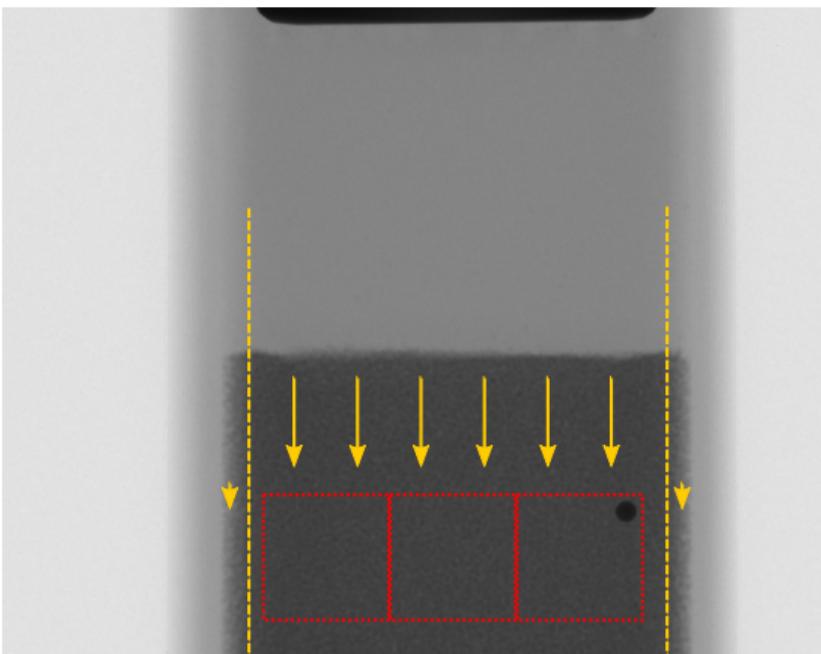


$\langle v \rangle_{\text{xdfa}} > \langle v \rangle_{\text{front}}$  by 9.4%



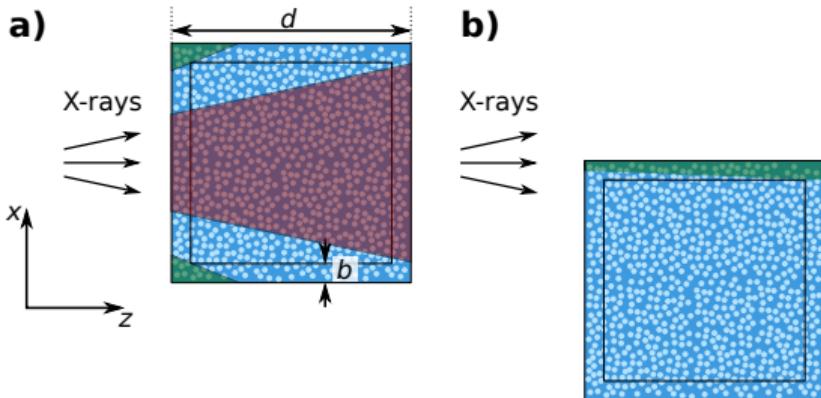
sample video

## Estimate width of boundary layer



$\langle v \rangle_{\text{xdfa}} > \langle v \rangle_{\text{front}}$  by 9.4%

$\langle v \rangle_{\text{xdfa}}$  takes two layers into account  
 $\langle v \rangle_{\text{front}}$  takes four layers into account



### Estimation:

Boundary velocity = 0

Else = const.

$\rightarrow b \approx 3$  particle diameters

Thank you for your attention!

