

# 中性子散乱実験装置についてもう一度考える

## —積み木型の中性子散乱実験装置—

北大院工 古坂道弘、佐藤他加志、藤田文行、本間彰、  
奥沢康裕、石川直樹、杉田宰、武田晋、宮田環、中西誠一  
京大原子炉実験所 杉山正明、日野正裕

**In preparation...**



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## —積み木型の中性子散乱実験装置—

2

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# Conventional SANS instrument

# Neutron SAS instruments

4

- Typical SANS instruments
- about 10 mmø sample size



SANS-U@JRR-3



New D11 @ ILL

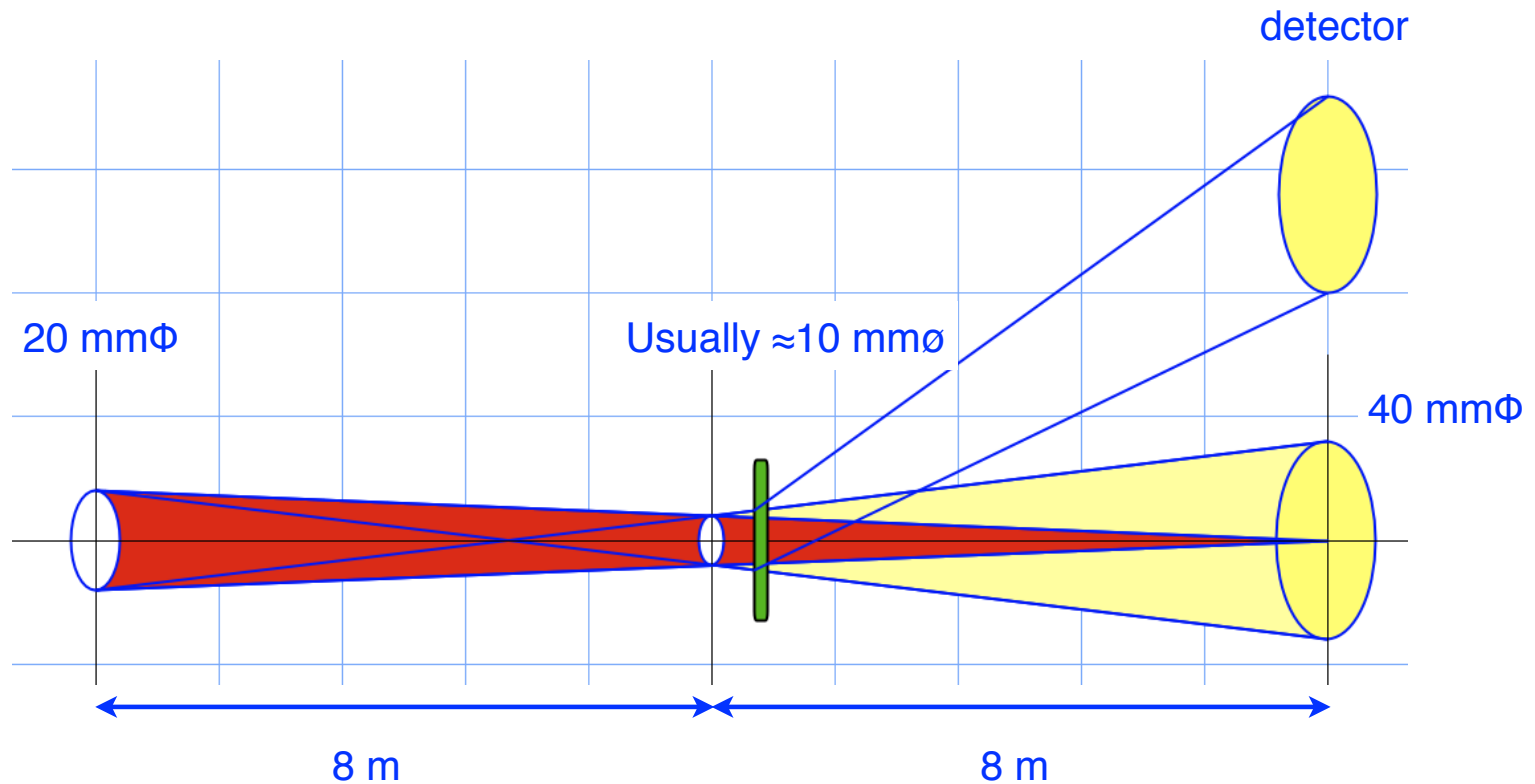
<http://www.ill.eu/instruments-support/instruments-groups/instruments/d11/news-from-d11/>



# Small Pin-hole Time-of-flight SANS

5

- Usually, sample diameter  $\approx 10 \text{ mm } \varnothing$  at SANS-U
- 16 m+16m at maximum
- $Q_{\min} \approx 5 \times 10^{-3} \text{ \AA}^{-1}$



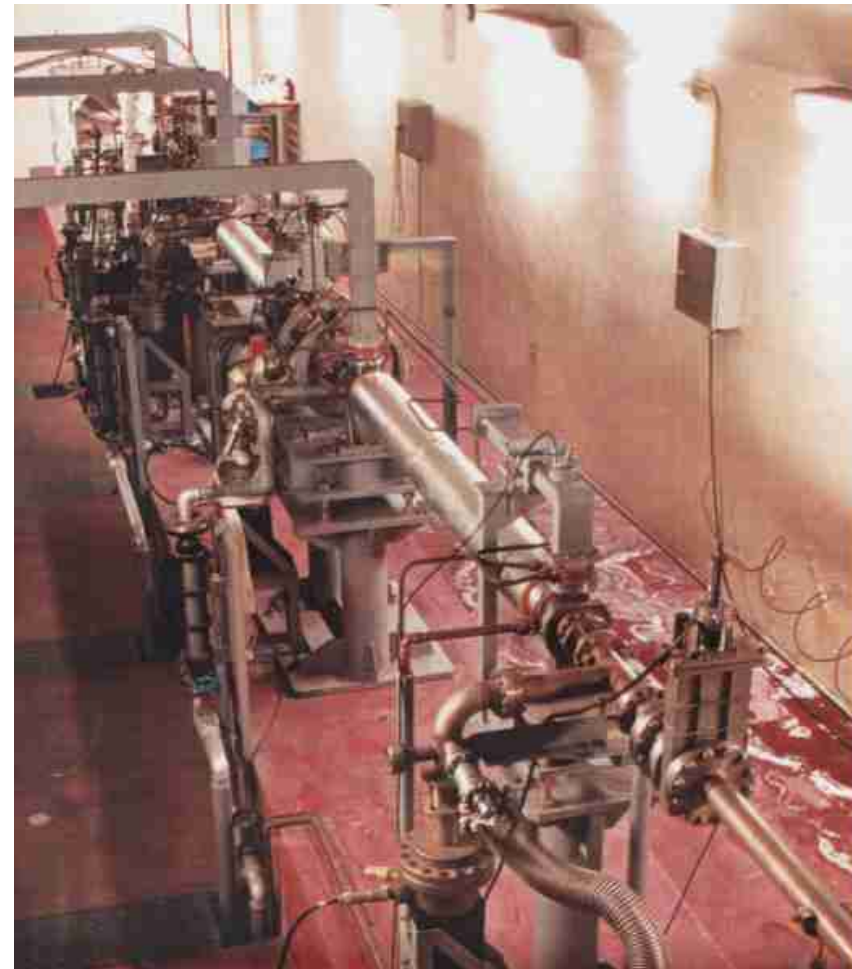
# Electron linac based pulsed neutron source at Hokkaido University

# 45MeV Electron Linac @Hokkaido University

7

- The first generation **compact pulsed neutron source**
  - First beam  $\approx 1975$
  - still running...
- 35 MeV, 30  $\mu\text{A}$ , 50 pps

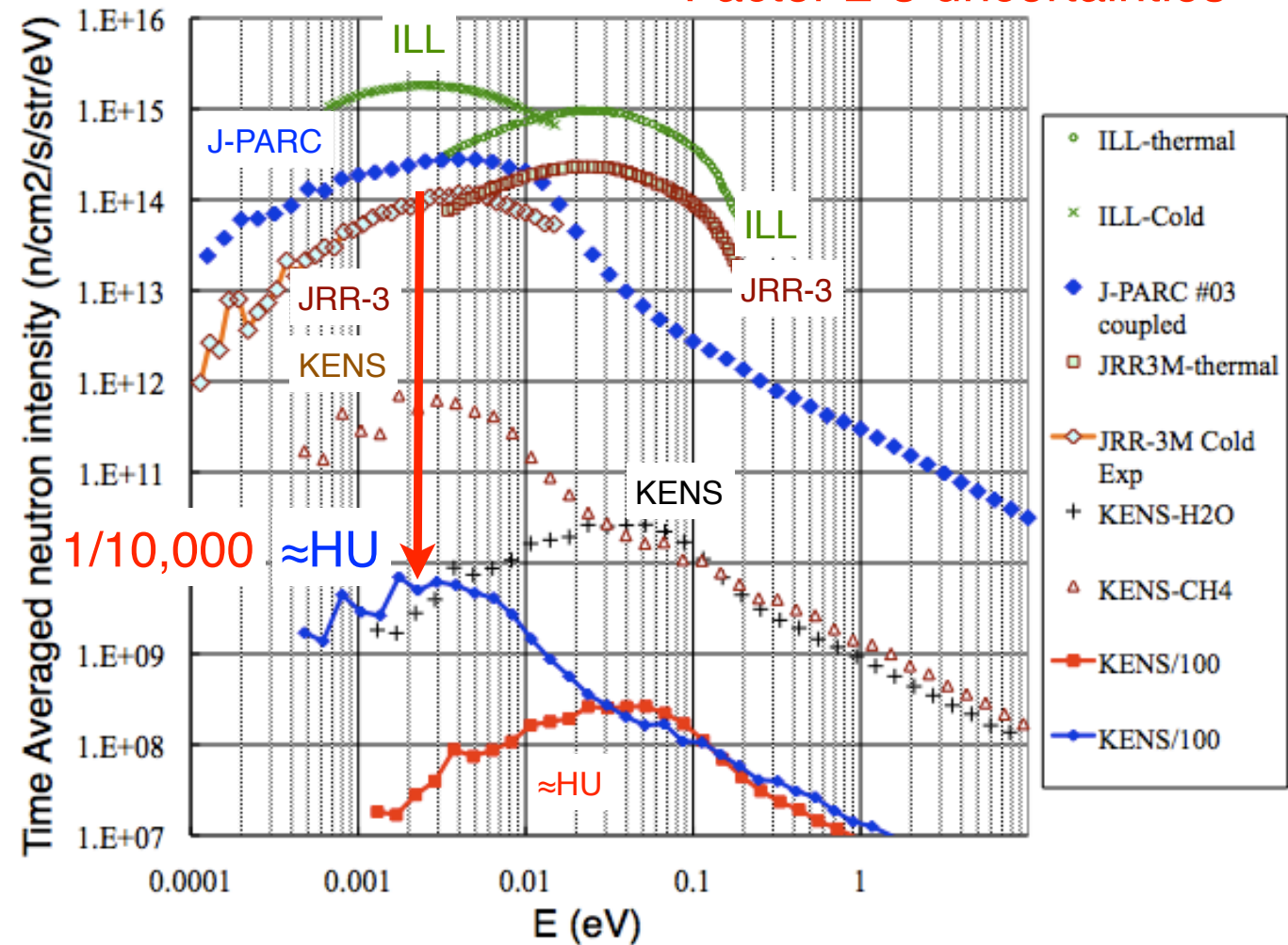
Hokkaido University Electron Linac



# Time averaged intensity

8

Factor 2-3 uncertainties



Tailor made experiment.  
example 1.

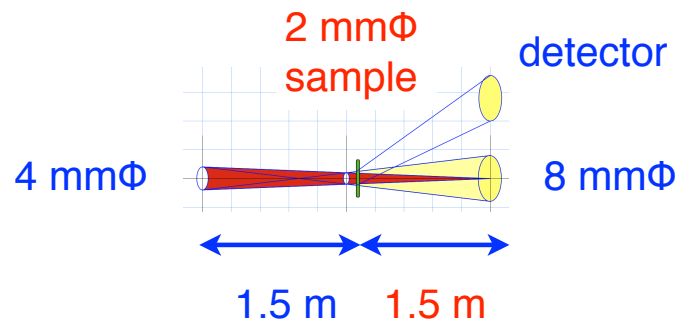
# Small Pin-hole Time-of-flight SANS

# Small Pin-hole Time-of-flight SANS

10

- What do you think of this  
SANS instrument at Hokkaido Univ.
- Sample size  $\approx 2 \text{ mm } \phi$
- flight path only 1.5m
- no vacuum tube
- Poor intensity  $\approx 1/25???$

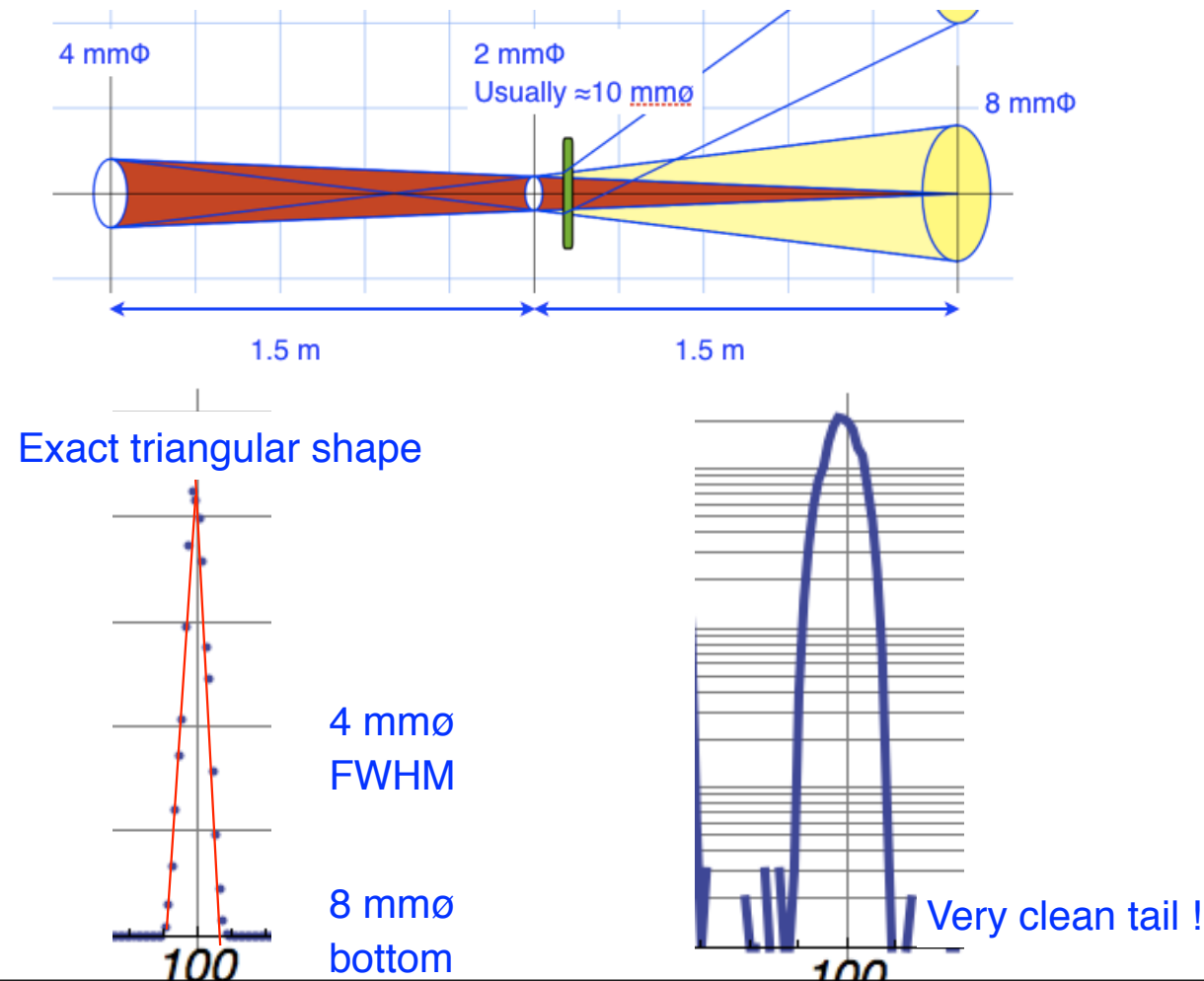
$$I(Q) \propto \phi \cdot d\Omega_i \cdot \frac{d\Sigma}{d\Omega} \cdot V_{\text{sample}} \cdot \eta \cdot d\Omega_f$$



# Very clean direct beam!

11

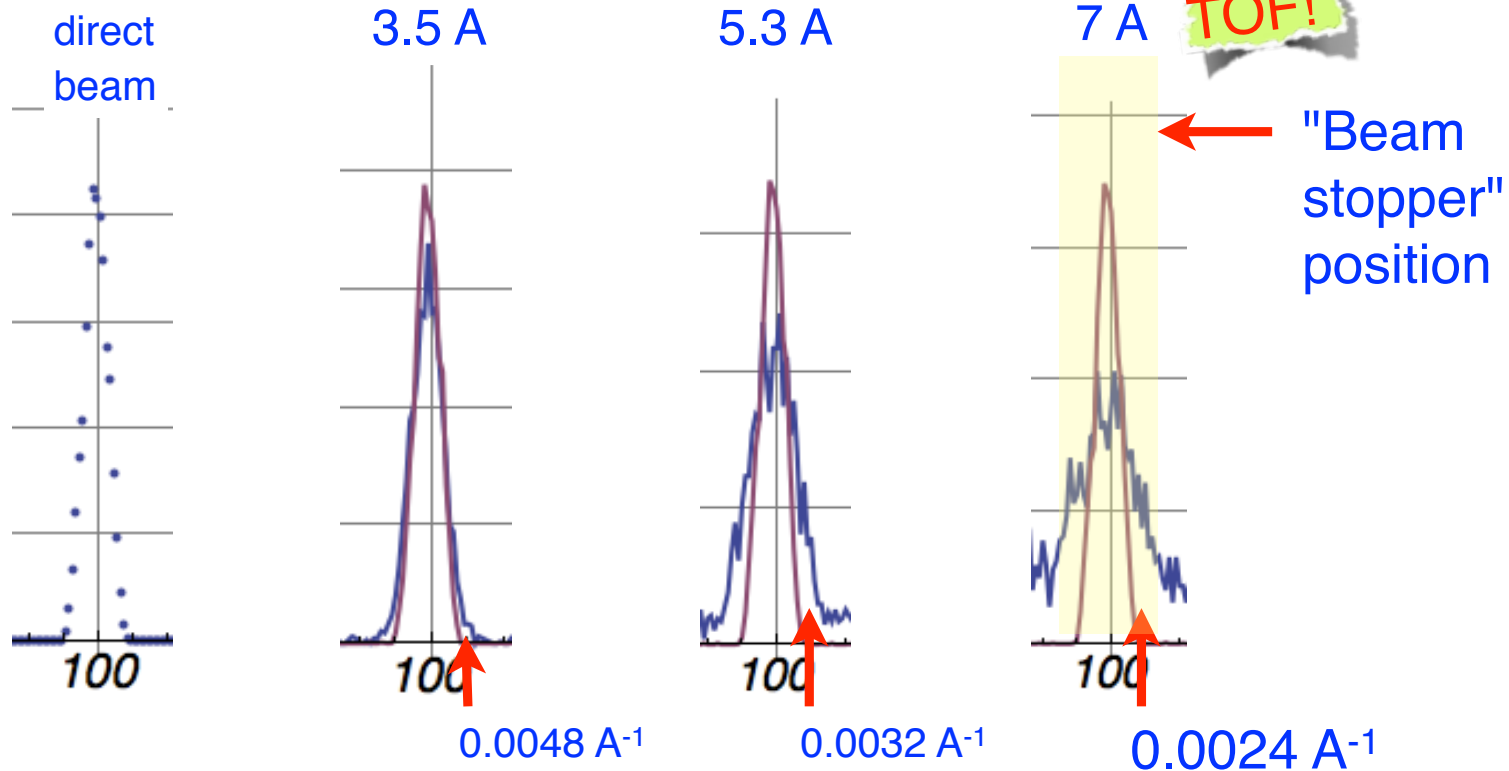
- Hokkaido Univ. small pin-hole SANS, 2 mm $\phi$
- Cd plate drilled with Boric acid



# SANS near the direct beam

Preliminary

12



- Cf. Conventional SANS machine:
  - Direct beam stopper covers low angle part!
  - SANS B.G. due to collimation

Cf.  $\approx 0.004 \text{ Å}^{-1}$  with 8 m

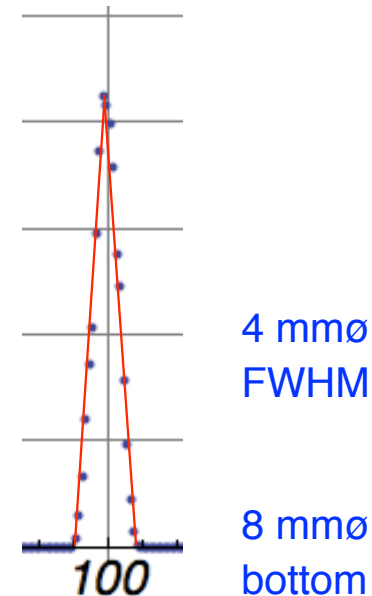


# Data analysis

13

- 2mmø sample SANS is possible even at Hokkaido University.
- TOF necessary
- Direct beam profile is precisely defined
  - Triangular shape direct beam
  - With fine resolution area detector
- How to de-convolute the result?
  - Fit with resolution function convoluted with  $S(Q)$ ?

Exact triangular shape



# Ex. 2. Diffractometer

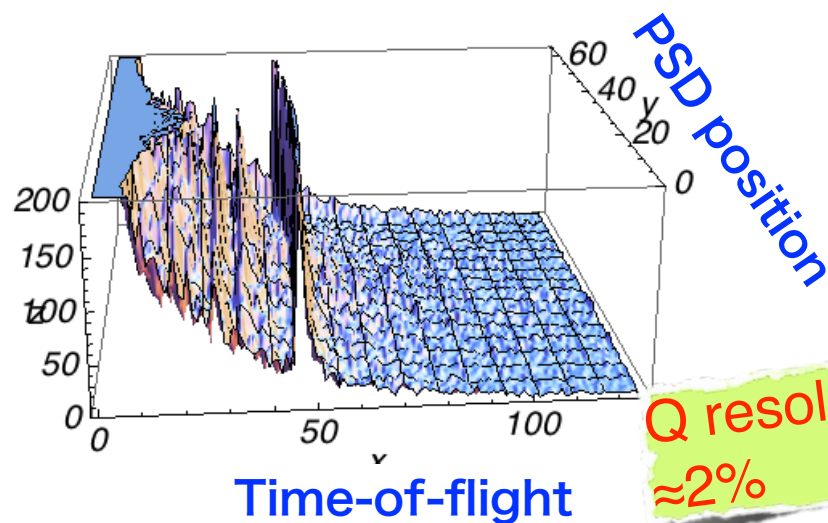
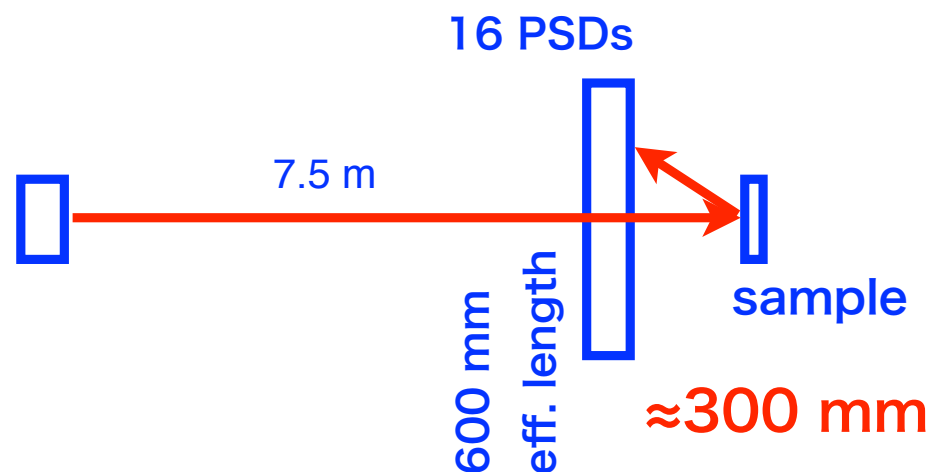
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Very low resolution

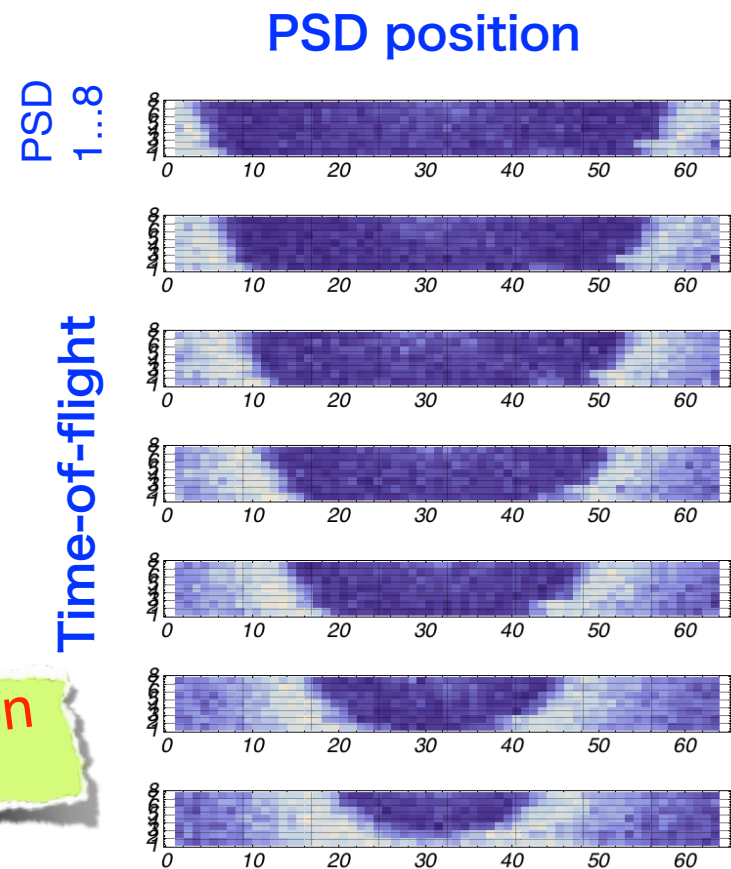
# Diffraction in a steel plate

Preliminary

15



Q resolution  
 $\approx 2\%$



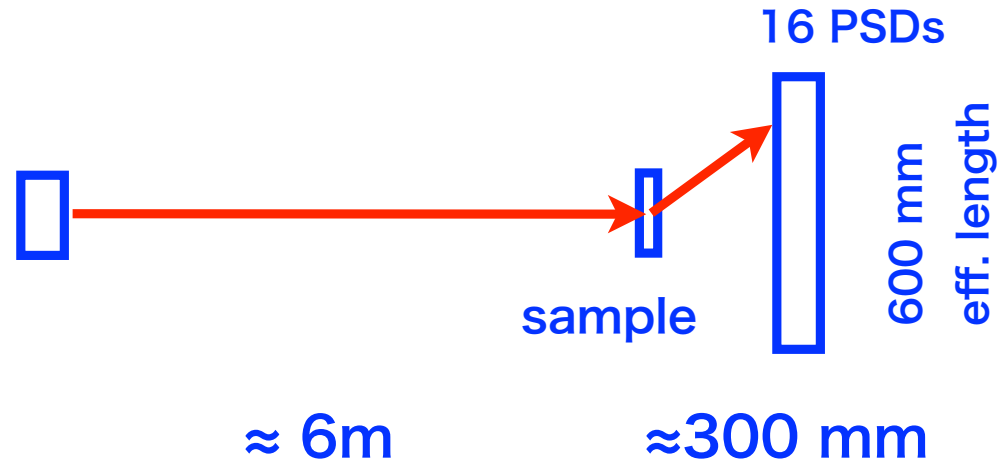
# Ex. 3. Intermediate-angle scattering

16

In between small-angle and wide angle.

# Intermediate-angle scattering

17



To be  
performed

- $0.05 \text{ \AA}^{-1} < Q < 4 \text{ \AA}^{-1}$ 
  - with  $\lambda=1, 12 \text{ \AA}$
- Ex. Precipitations in steel

# What is the tailor-made experiment (TME) beamline

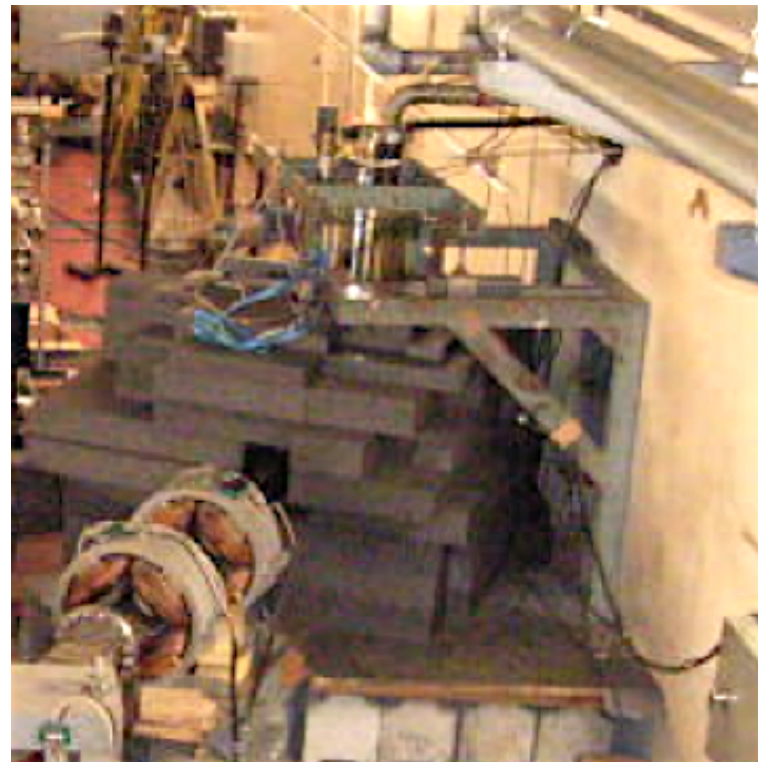
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Compact pulsed neutron source is a  
best place for TME beamline.

# Moderator development

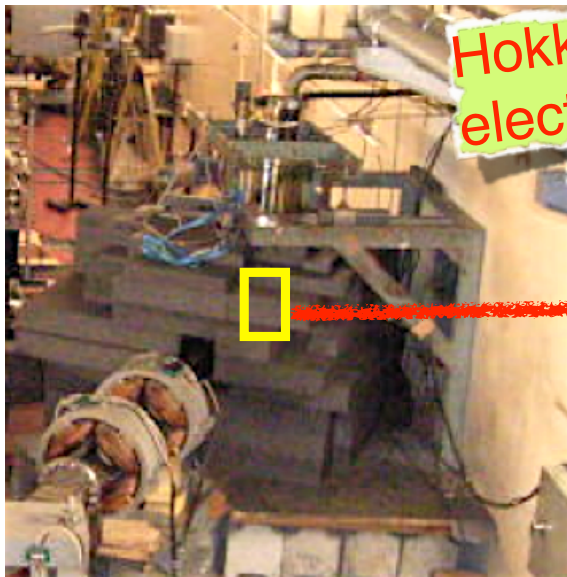
19

- Best place for **developing** high-performance **moderator**
  - Coupled moderator concept
  - Solid methane moderator
- Hands-on modification!
  - Low flux means low activation
- J-PARC moderators are developed here.
  - SNS composite moderator tested.



# Just imagine some components

20



Hokkaido University  
electron linac



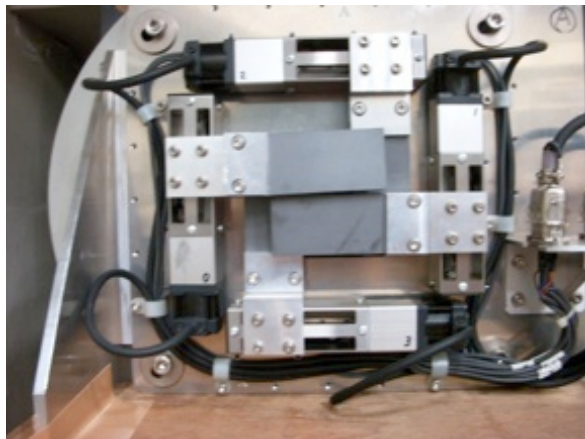
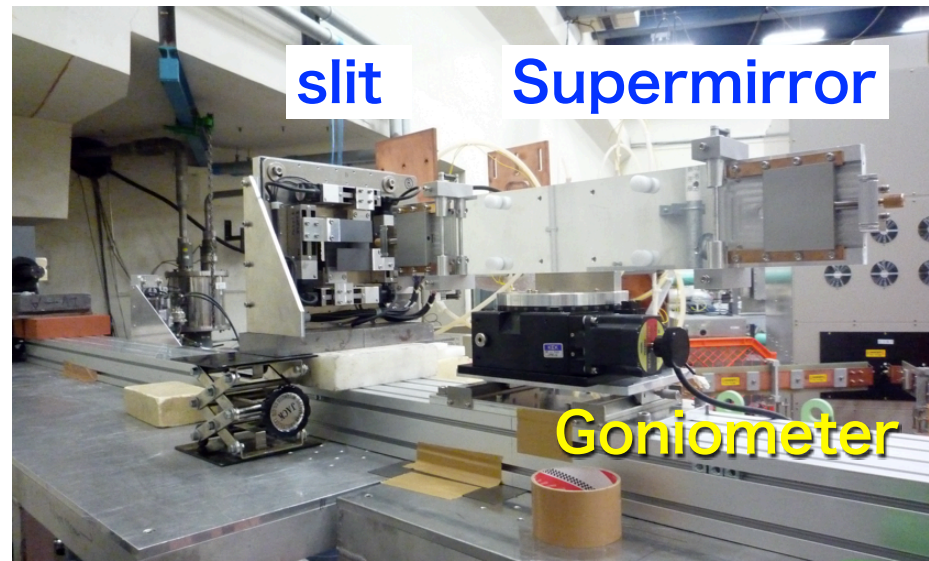
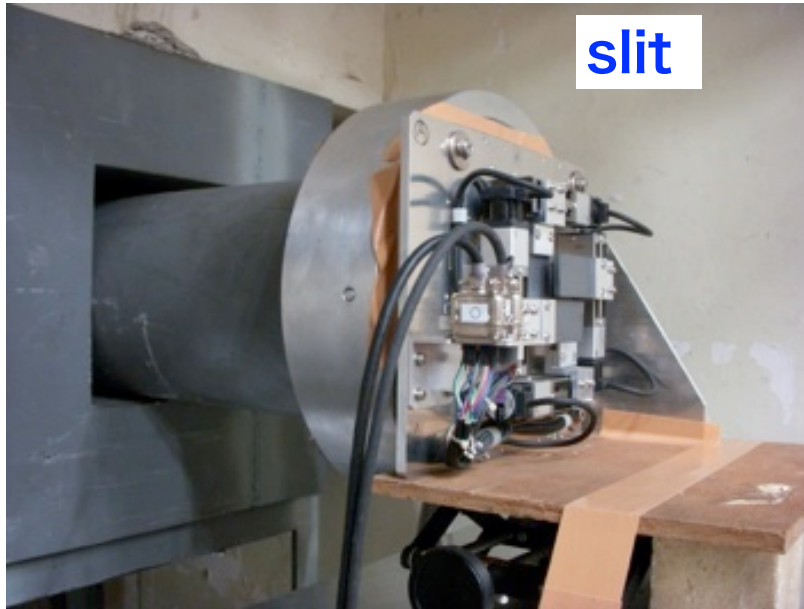
Pulsed cold neutron source  
solid methane@ $\approx 20\text{K}$

Neutron beam exit



# Just imagine some components

21

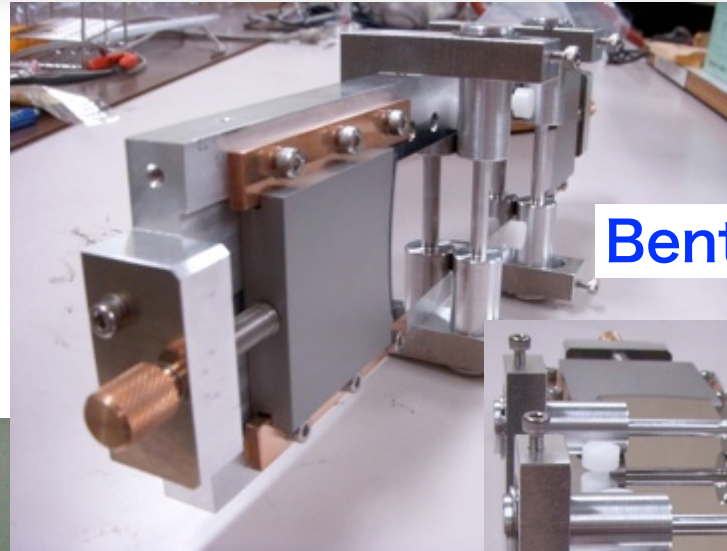
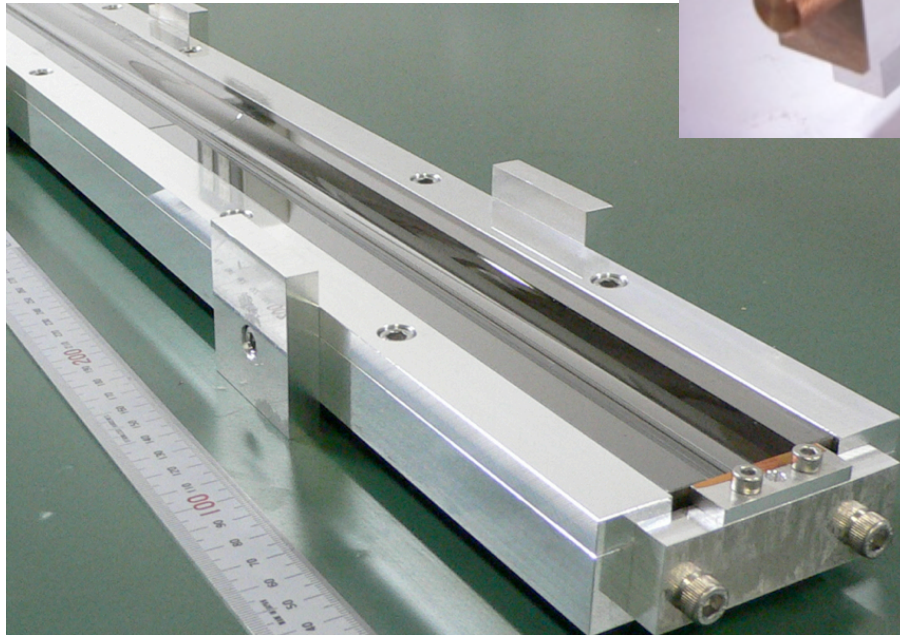


Tables "optical bench"

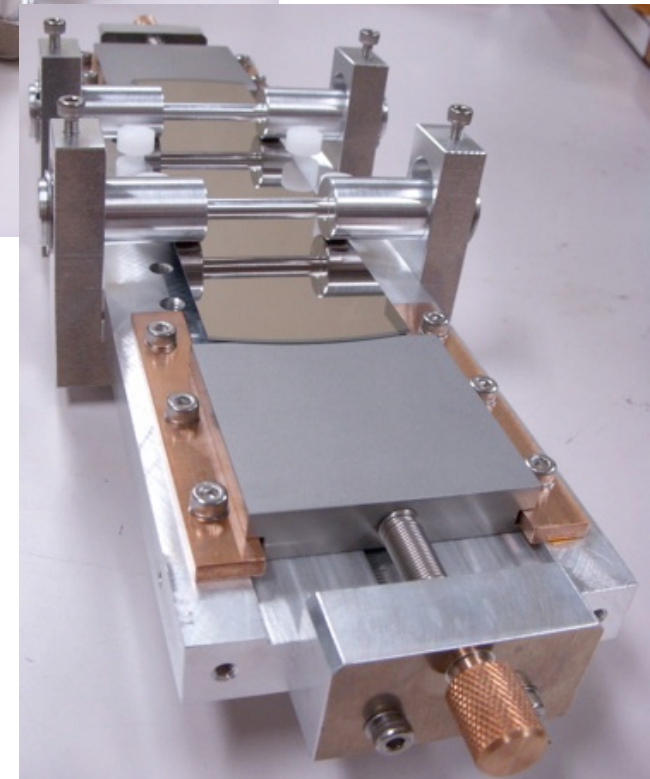
# Supermirror components

22

ellipsoidal mirror



Bent supermirror

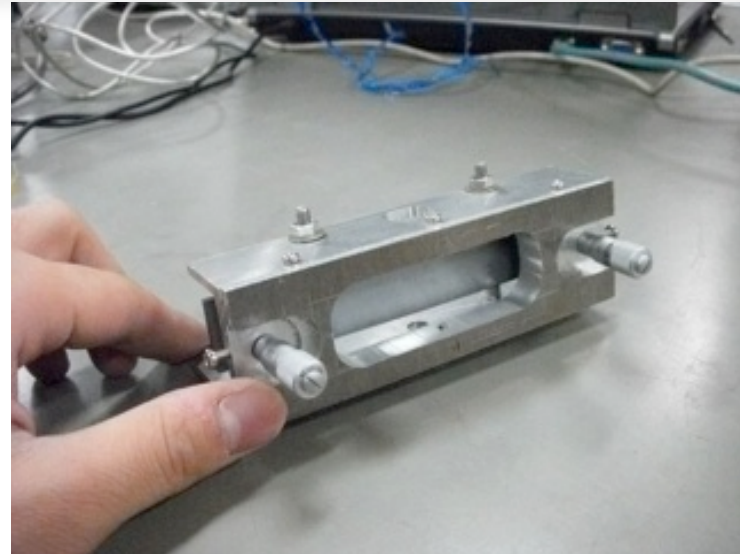
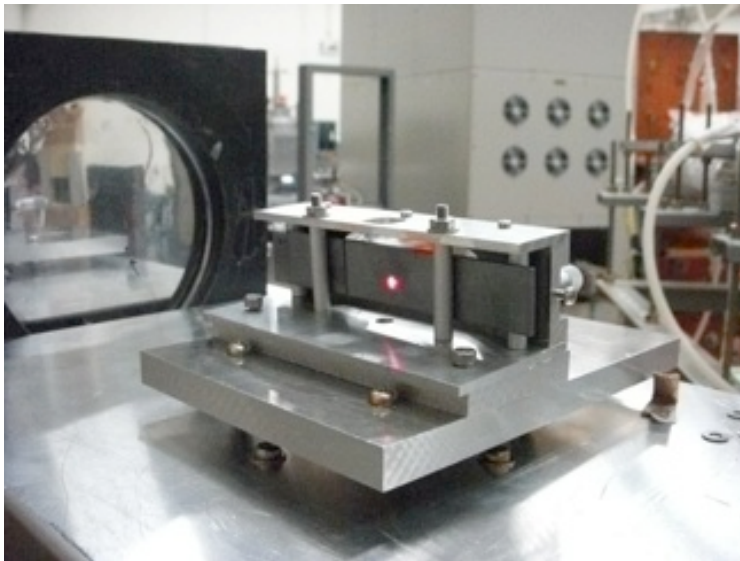




# Si perfect crystal bender

23

- High resolution area detector (RPMT)  
Resistive wire type  
Photomultiplier



# Shielding blocks!

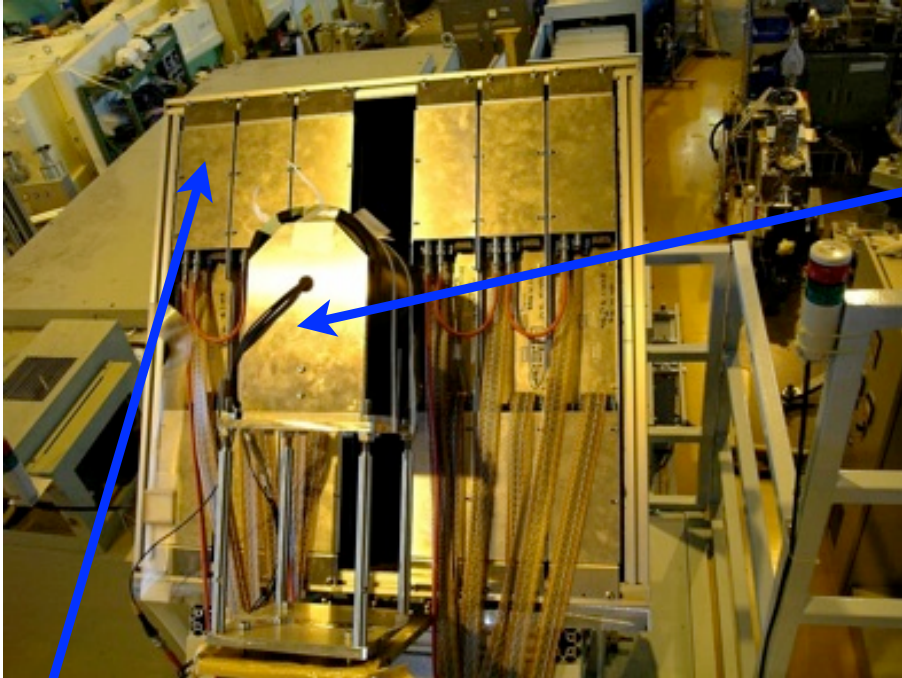
24

Tape-engineering!



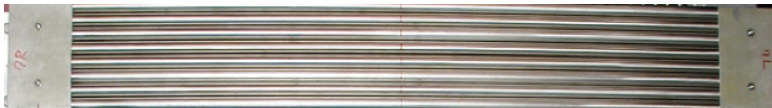
# Detector components

25



- High resolution area detector  
(RPMT)  
Resistive wire type  
Photomultiplier

- Linear Position Sensitive Detectors  
(LPSDs)  
8 LPSDs/module x 6 units



- He-gas:  
difficult to purchase?

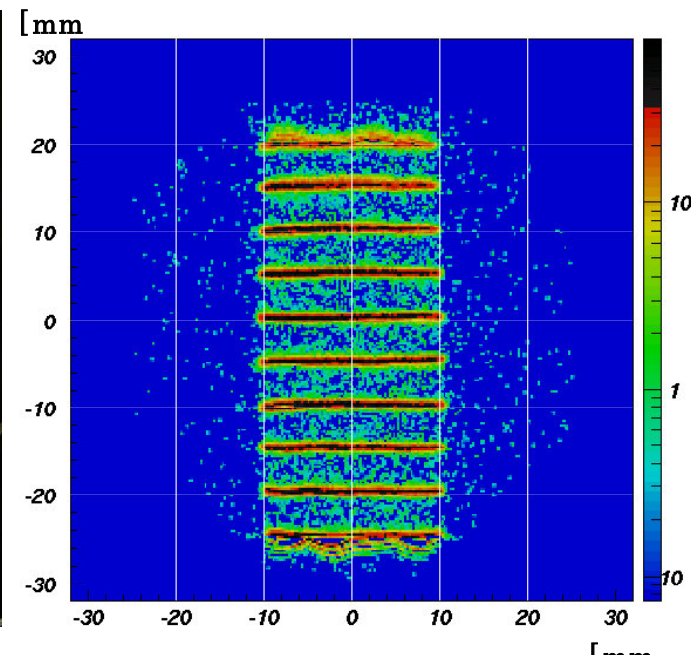
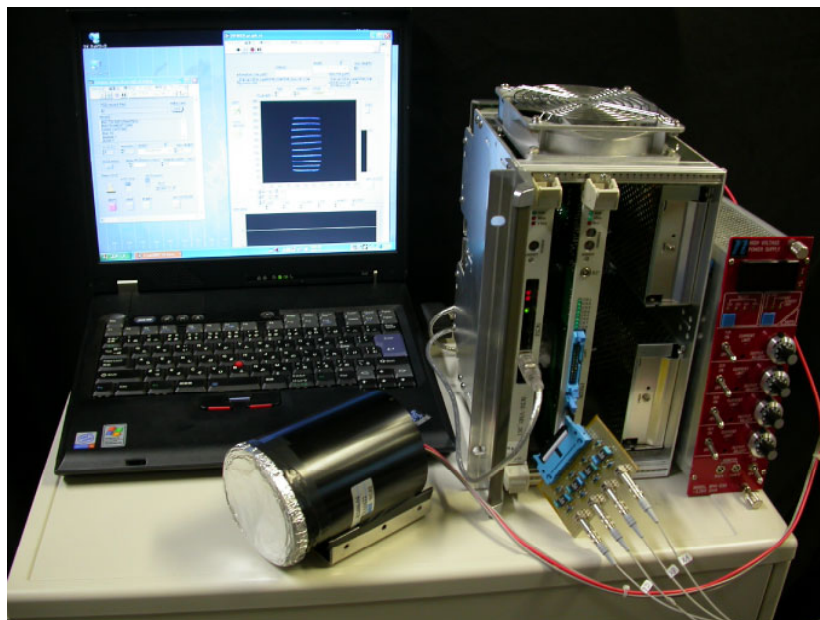


# Resistive wire type PMT +ZnS scintillator

26

- $\text{Li (n, } \alpha \text{)}$ ; ZnS(Ag) scintillation
- 3inch, 5inch PMT
  - R2486-04
- Good resolution
  - $<1\text{mm}$
- convenient system for optical device test.

Hirota, Satoh et al.  
(RIKEN, KEK, NOP)



# mfSANS at Hokkaido Univ.

27

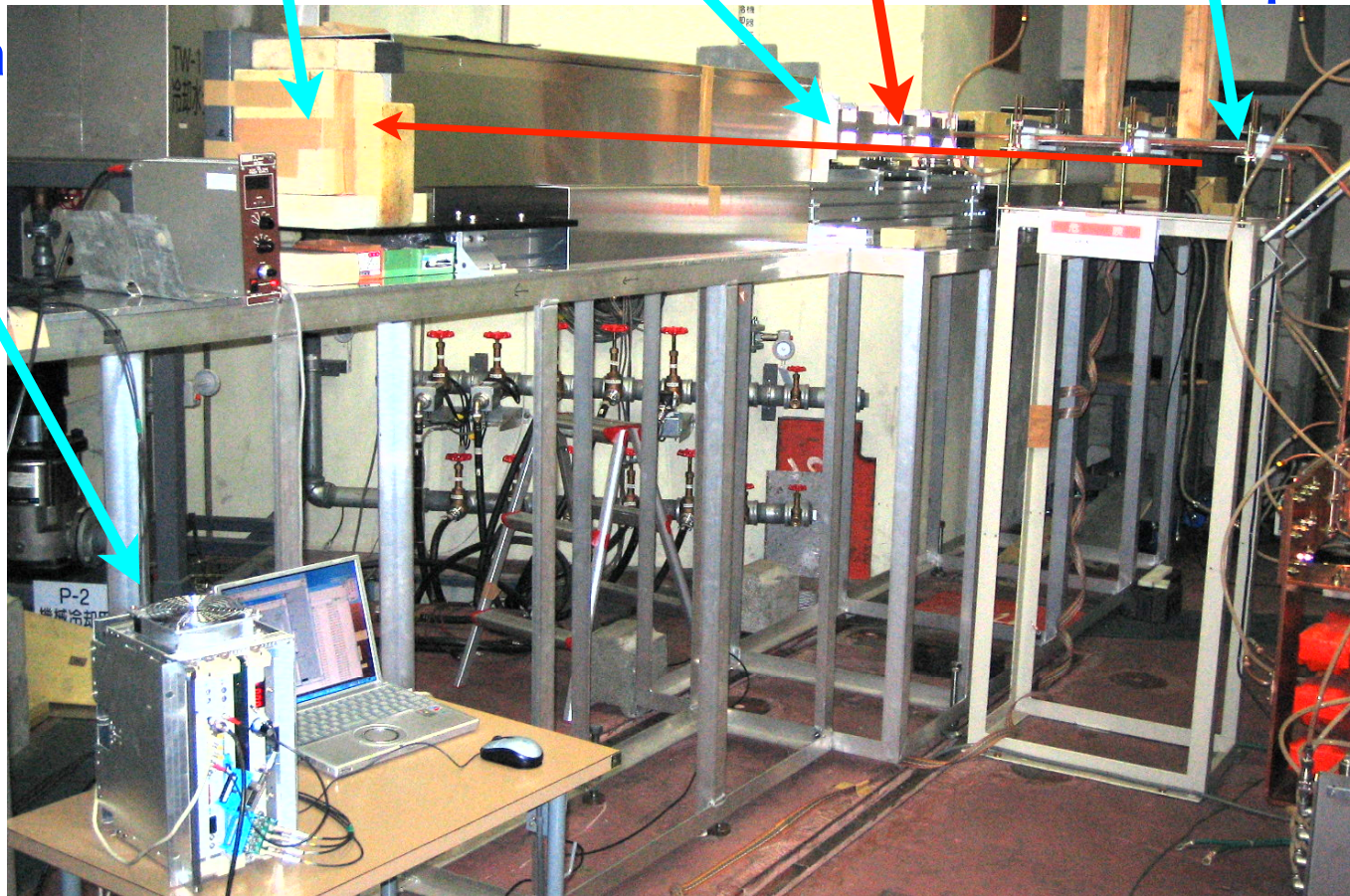
Data  
acquisition  
system

Detector

Sample

Ellipsoidal  
mirror

Beam  
port



# Ex. 4. High intensity monochromator

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for mfSANS@JRR-3



# Commonly used monochromator

29

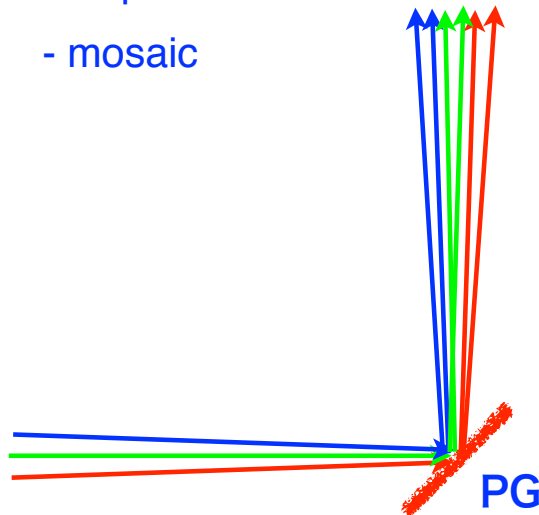
- Pyrolytic Graphite
  - Mosaic crystal
  - Reflectivity  $\approx 80\%$
  - $\Delta\lambda/\lambda \leq 1\%$ 
    - depending on beam divergence

Not suitable to  
SANS, Reflectometer

## Large beam divergence

- $\lambda$  spread
- mosaic

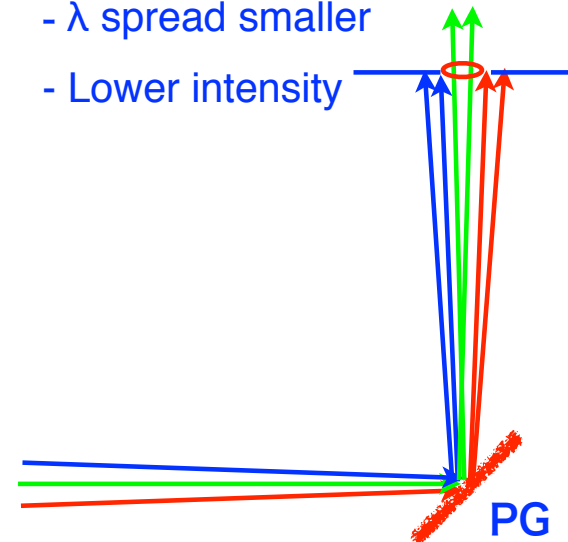
$\lambda > \lambda_0$   
 $\lambda = \lambda_0$   
 $\lambda < \lambda_0$



## Collimation effects

- $\lambda$  spread smaller
- Lower intensity

$\lambda > \lambda_0$   
 $\lambda = \lambda_0$   
 $\lambda < \lambda_0$



# Neutron SAS instruments

30

- 1 instrument at the end of beamline.



SANS-U@JRR-3



New D11 @ ILL

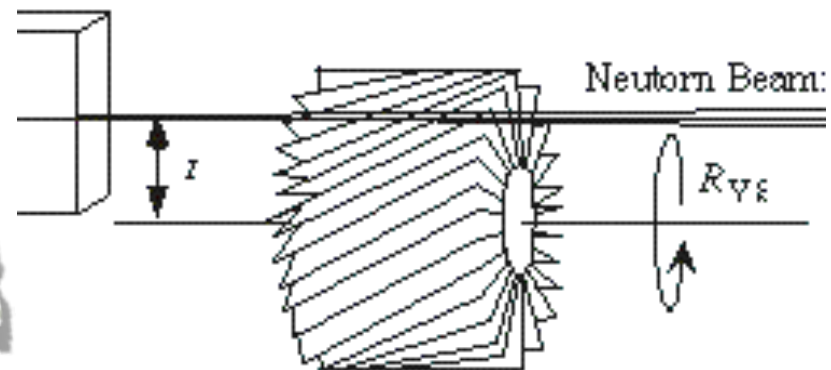
<http://www.ill.eu/instruments-support/instruments-groups/instruments/d11/news-from-d11/>

# SANS monochromator

31

- **Velocity selector** is the choice
  - High intensity quasi-monochromator
  - $\Delta\lambda/\lambda \approx 5 \sim 20 \%$

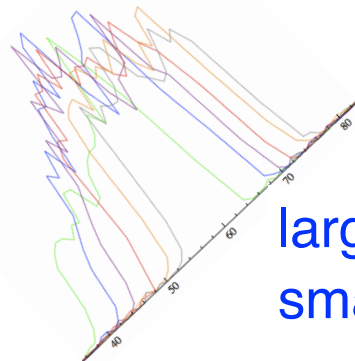
Extremely  
good  
performance



- 1 selector/Guide-end
- **Supermirror/monochromator** would be other possibility
  - NOP beamline

# Test experiment at H.U. **pulsed** neutron source

32



large divergence  
small  $\Delta\lambda/\lambda$

$\lambda < \lambda_0$

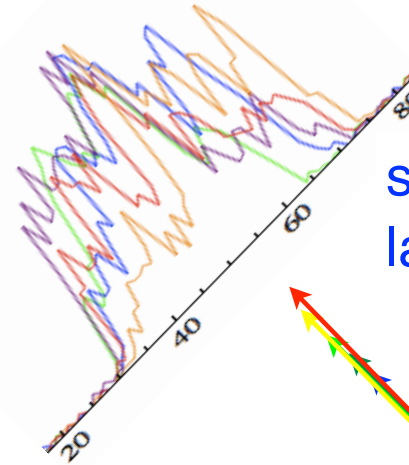
$\lambda > \lambda_0$

Bent Si

$\lambda < \lambda_0$

$\lambda = \lambda_0$

$\lambda > \lambda_0$



small divergence  
large  $\Delta\lambda/\lambda \approx 4\%$

Bent Si

$\lambda < \lambda_0$

$\lambda = \lambda_0$

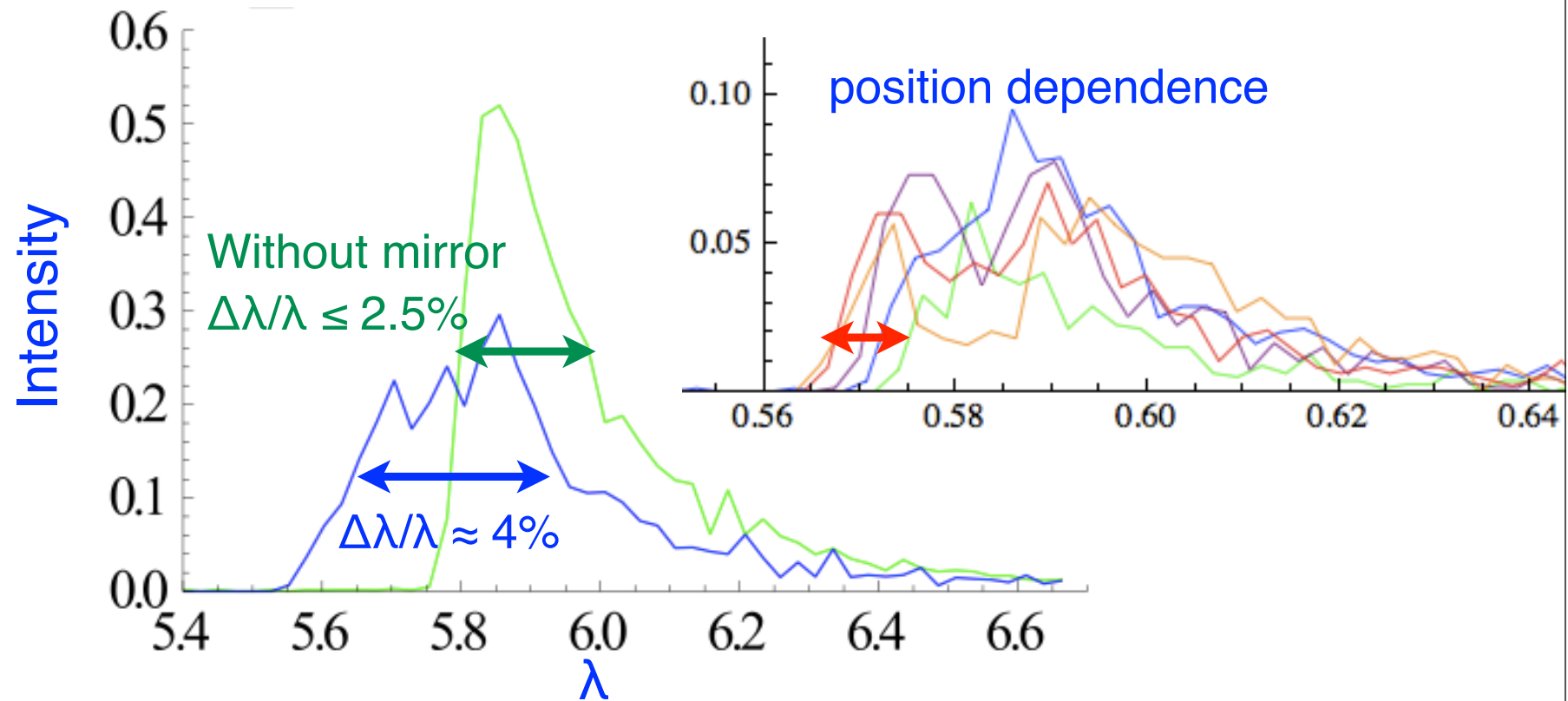
$\lambda > \lambda_0$

Large divergence

# Wide $\lambda$ band realized

33

- $\lambda$  dependence by time-of-flight measurement
  - Not easy at a reactor.

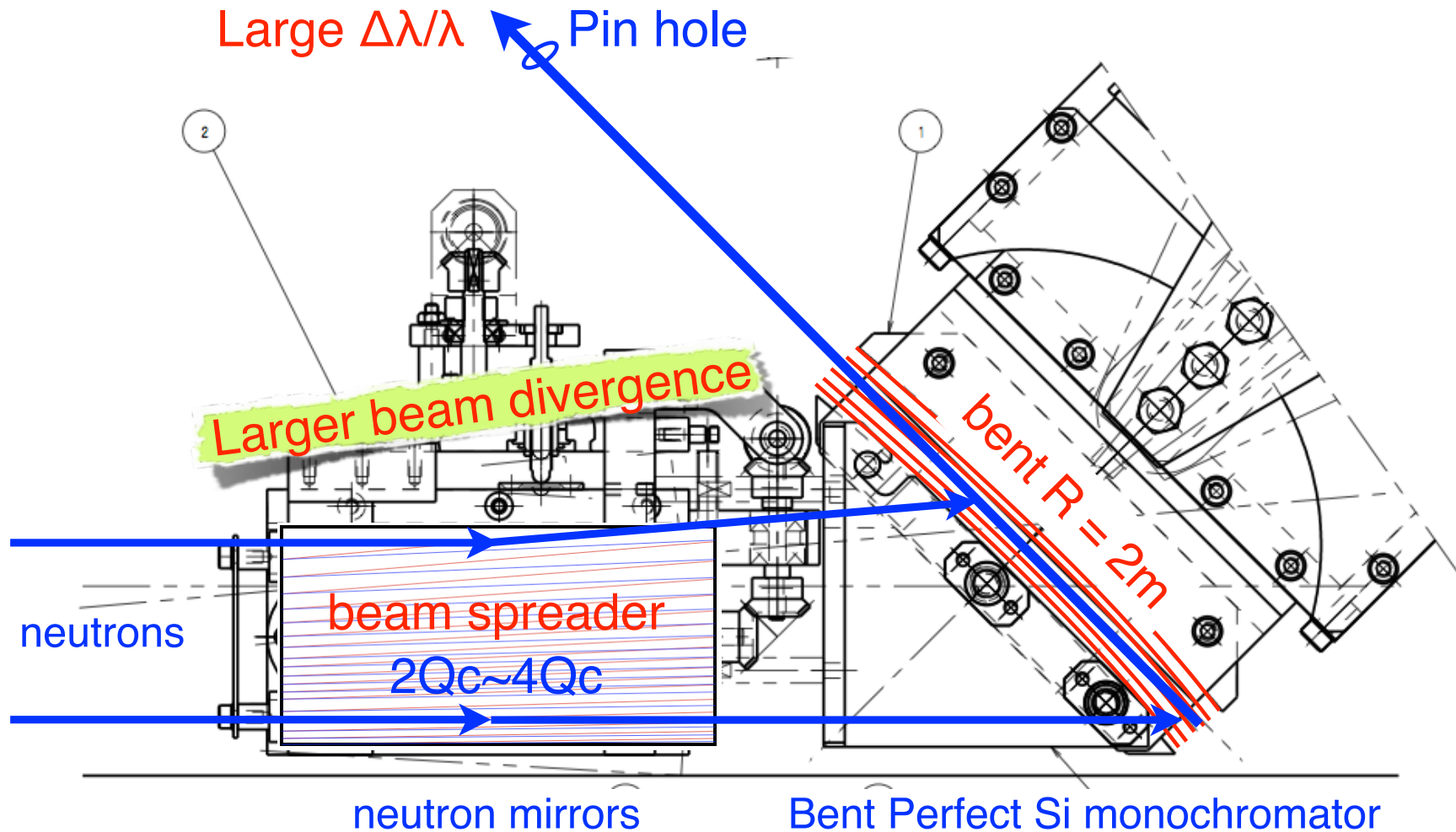


# High intensity monochromator with a beam deflector

New concept!

34

- Improved monochromator being tested



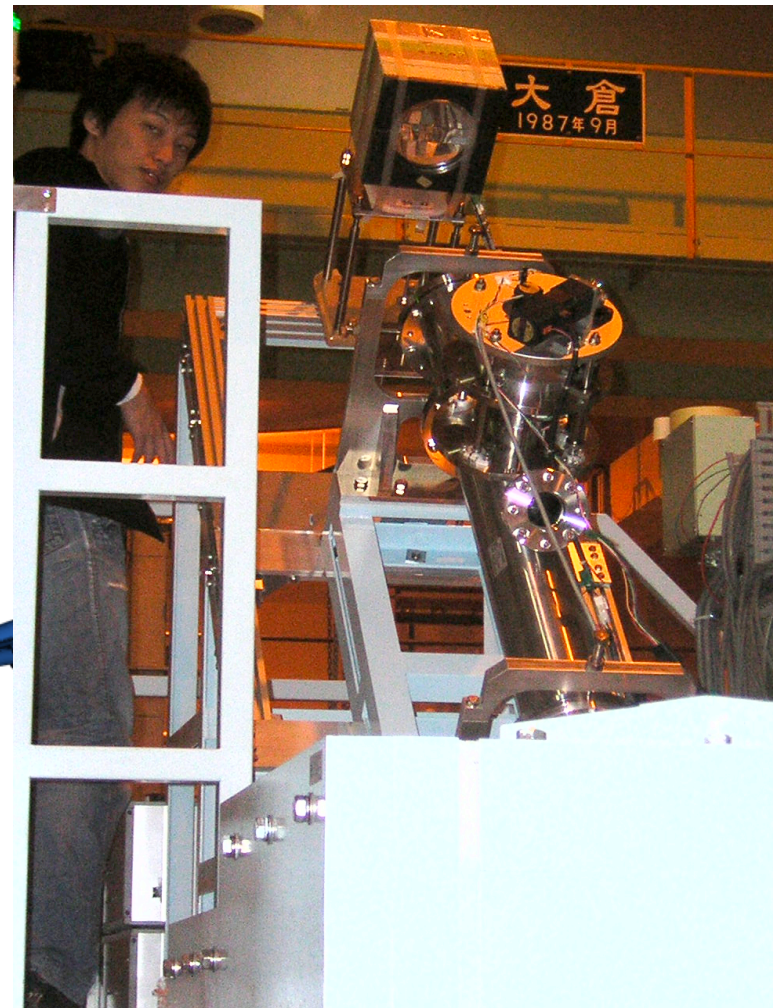
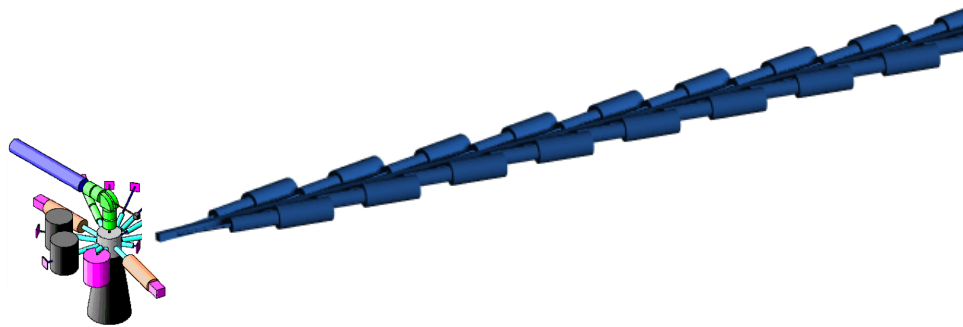


# Idea of compact SANS instruments



35

- Many SANS modules
  - along a beamline
- To have long enough machine time anytime,
  - Ex. For protein solution research
- Educate young scientists
- Check your new ideas
- Your own SANS instrument



# Tailor made experiment beamline

36

- Test **new ideas**
  - Very easy to tailor an instrument.
- Best way of **learning** neutron scattering and instrumentation.
  - To educate young people
  - Teach yourself
- Low cost
- Relaxed resolution to gain intensity
- Tailor made experiment:
  - Ex. just to look at...
  - Intermediate  $Q \approx 0.1 \sim 2 \text{ \AA}^{-1}$  region
  - a magnetic diffraction peak

# Just to review focusing SANS instrument

# Neutron SAS instruments

38

- Need a cold neutron source.
- Only 1 instrument at a guide-end.



SANS-U@JRR-3



New D11 @ ILL

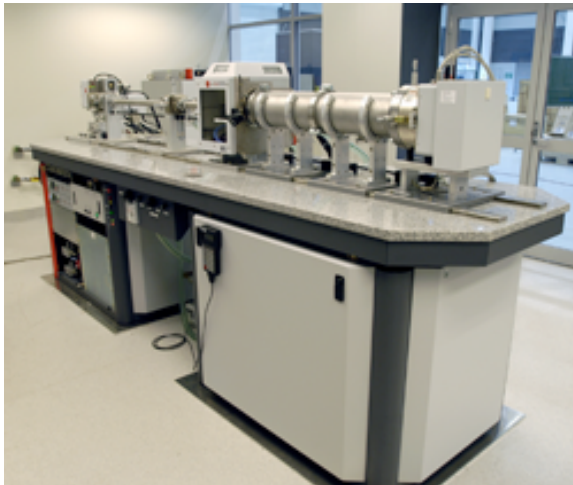
<http://www.ill.eu/instruments-support/instruments-groups/instruments/d11/news-from-d11/>

# X-ray SAS instruments are Small...

# X-ray SAS instrument

40

- Laboratory sources are available anywhere
- Proper training methods are established
  - Variety of books available
  - You can ask experienced people around.
- If laboratory source is not appropriate, you can always use synchrotron radiation



SAXS

[http://matrix-dev.ansto.gov.au/research/bragg\\_institute/facilities/instruments/saxs/mimi\\_saxs](http://matrix-dev.ansto.gov.au/research/bragg_institute/facilities/instruments/saxs/mimi_saxs)



ESRF

<http://www.lightsources.org/cms/?pid=1000103>



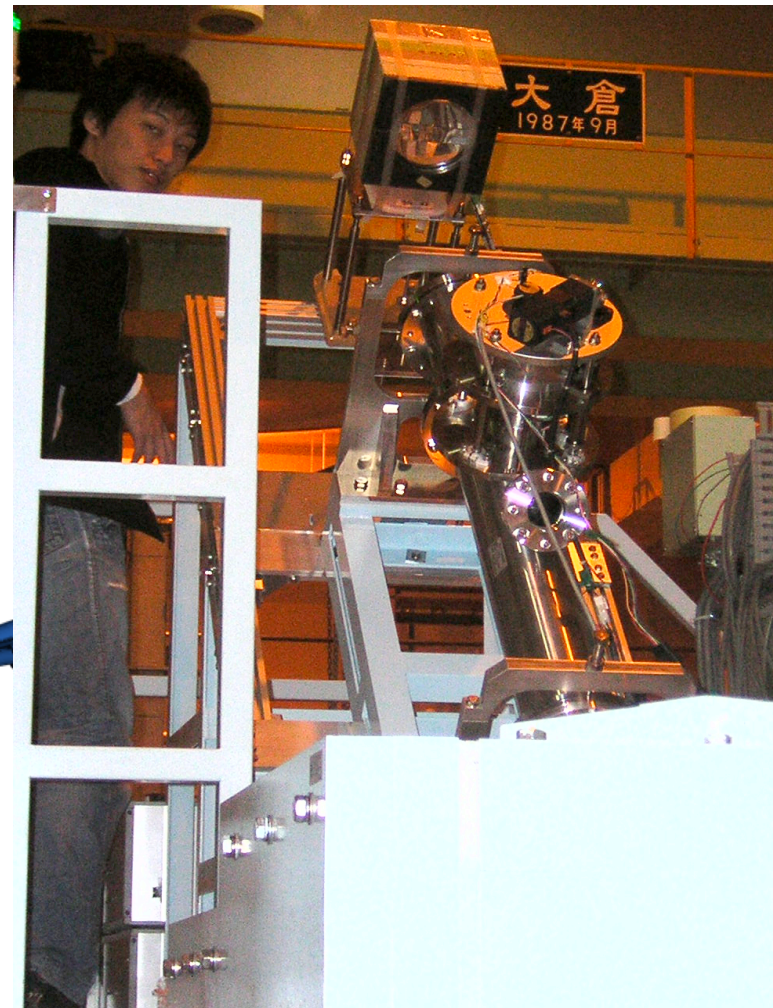
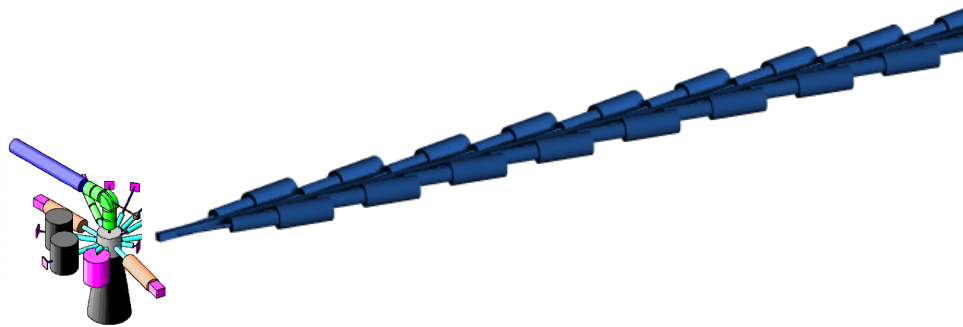
# Why not have compact neutron SAS instrument

# Idea of compact SANS instruments



42

- To have long enough machine time anytime,
  - Ex. For protein solution research
- Educate young scientists
- Check your new ideas
- Many SANS modules
- Your own SANS instrument



# to match with large neutron facilities

43

- Big facilities like
  - ILL, Oak Ridge, Munich, NIST, JRR-3, KAERI, ANSTO, ...
  - ISIS, Luhan center, PSI, SNS, J-PARC



J-PARC

<http://j-parc.jp/ja/layout-j.html>

# How to make compact instruments: **Focusing** is the key.

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44

Focusing makes instrument **compact!**  
compact  $\approx$  low cost

Cf. Focusing gives us **no-gain in intensity!**

# Focusing SANS

45

- Toroidal mirror focusing to extend low-Q limit.
  - Moved from Jülich to München
  - $Q=4 \times 10^{-4} \text{Å}^{-1}$

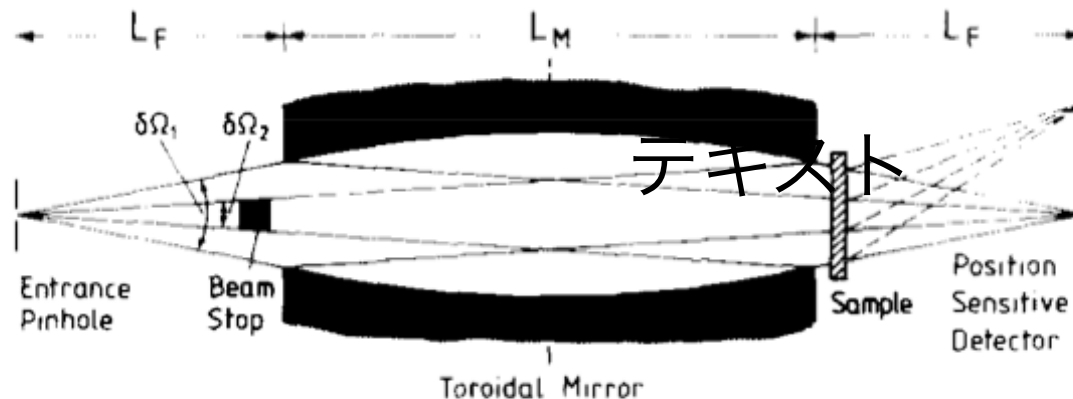


Fig. 1. Toroidal mirror with the image in the detector plane.

B. Alefeld et al./ Physica B 234-236 (1997) 1052-1054

- $\text{MgO}_2$  lens, sextupole lens are available.



# Ellipsoidal mirror focusing SANS

Kamada et al. (Hokkaido Univ.)

46

- Ellipsoidal mirror
- 1~10 mm $\phi$  aperture

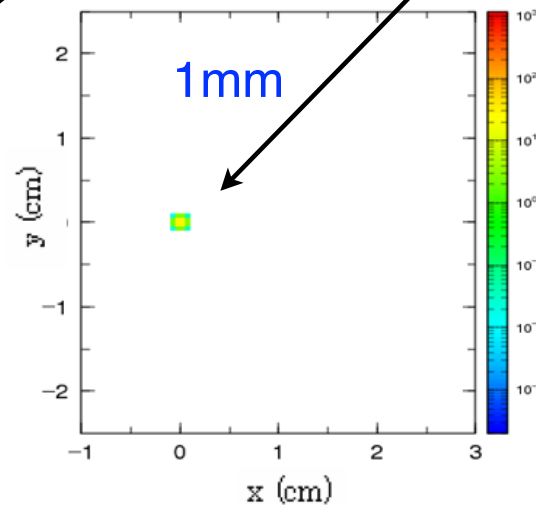
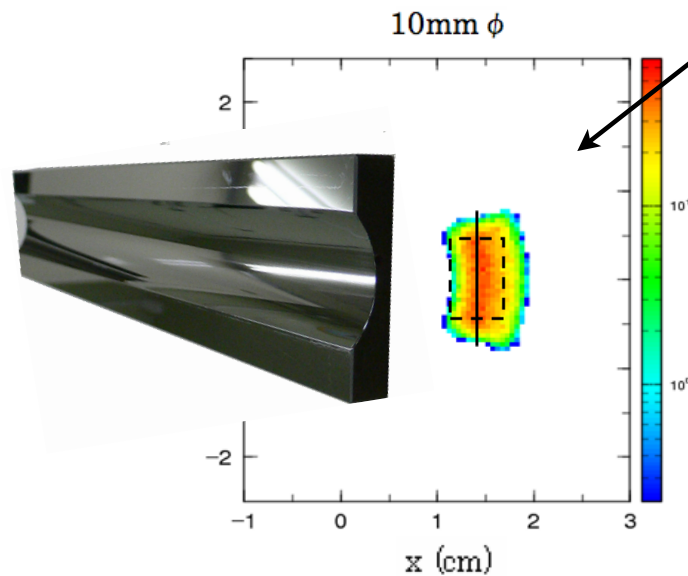
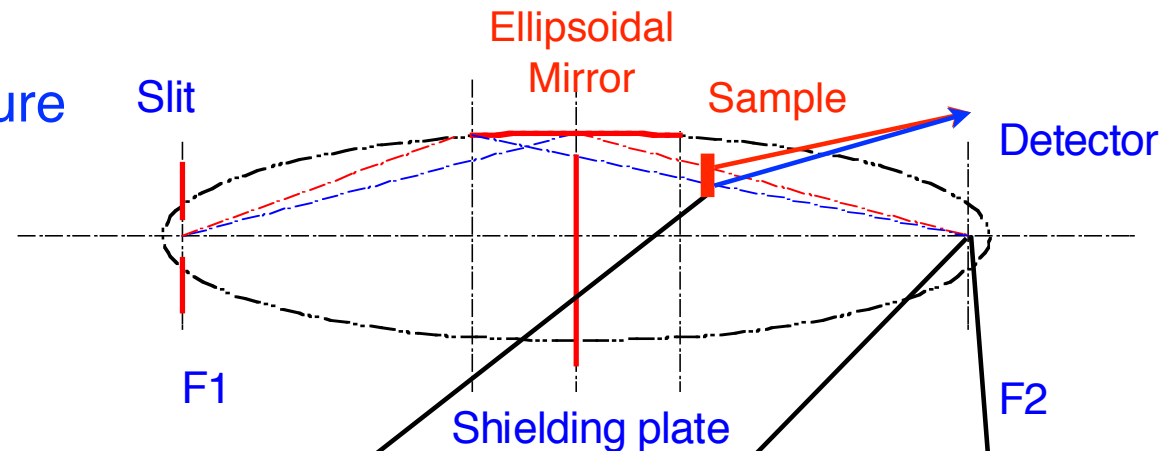


図 4.12 Aperture : 1mm  $\phi$

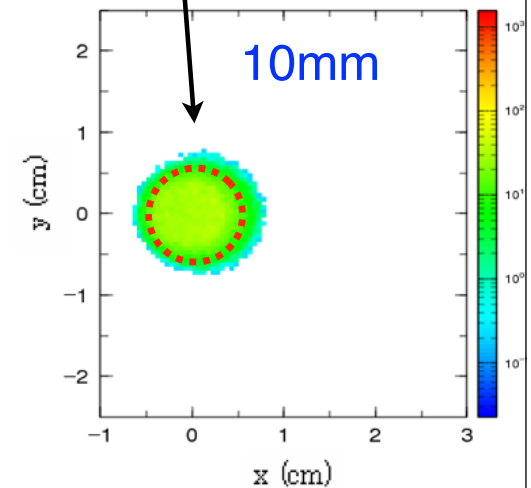


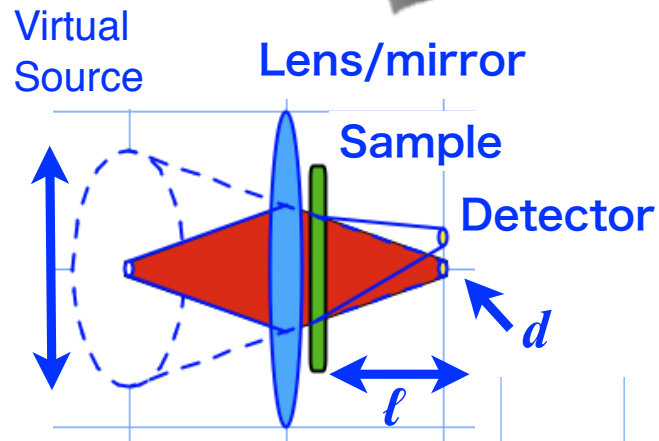
図 4.17 Aperture : 10mm  $\phi$



# Focusing SANS instrument is Compact!

47

## ■ Focusing *≈ compact*



## ■ Same resolution/intensity

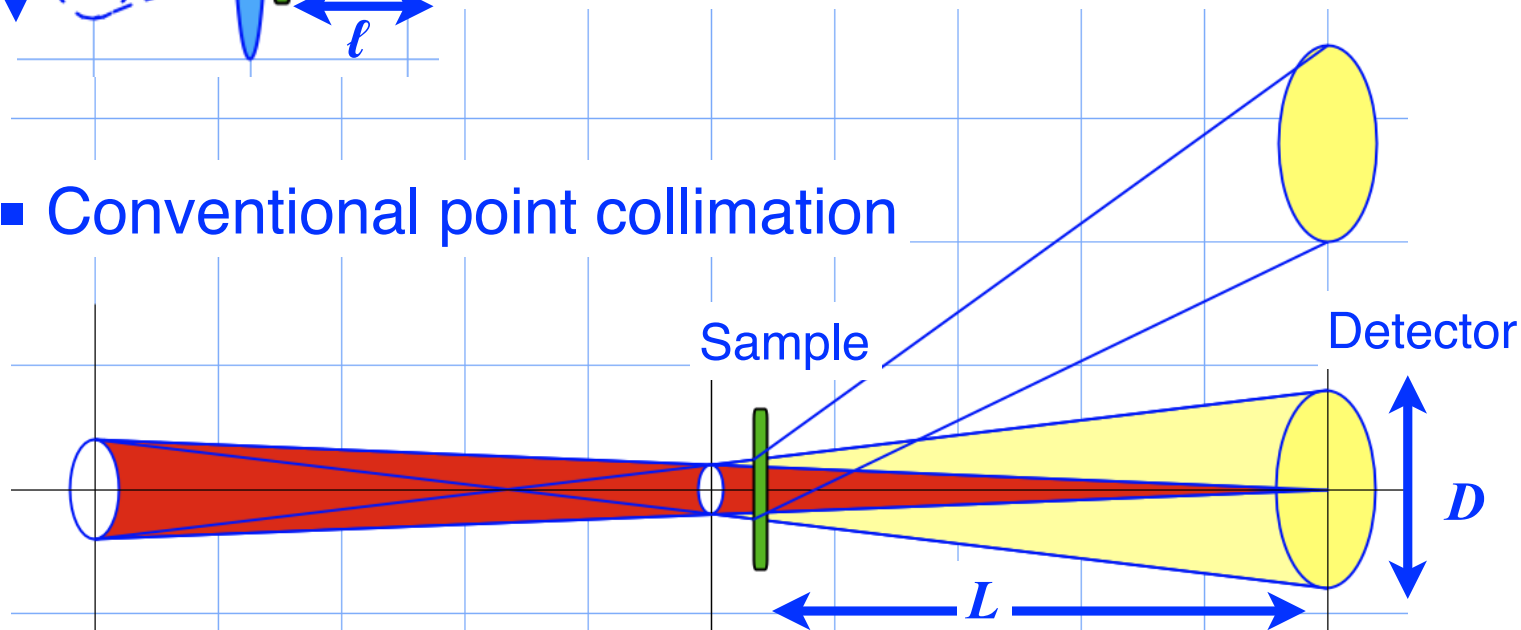
- Angular resolution

$$\approx D/L \approx d/\ell$$

- Intensity:

$$I \propto \phi \cdot d\Omega_i \cdot \frac{d\Sigma}{d\Omega} \cdot V_{sample} \cdot \eta \cdot d\Omega_f$$

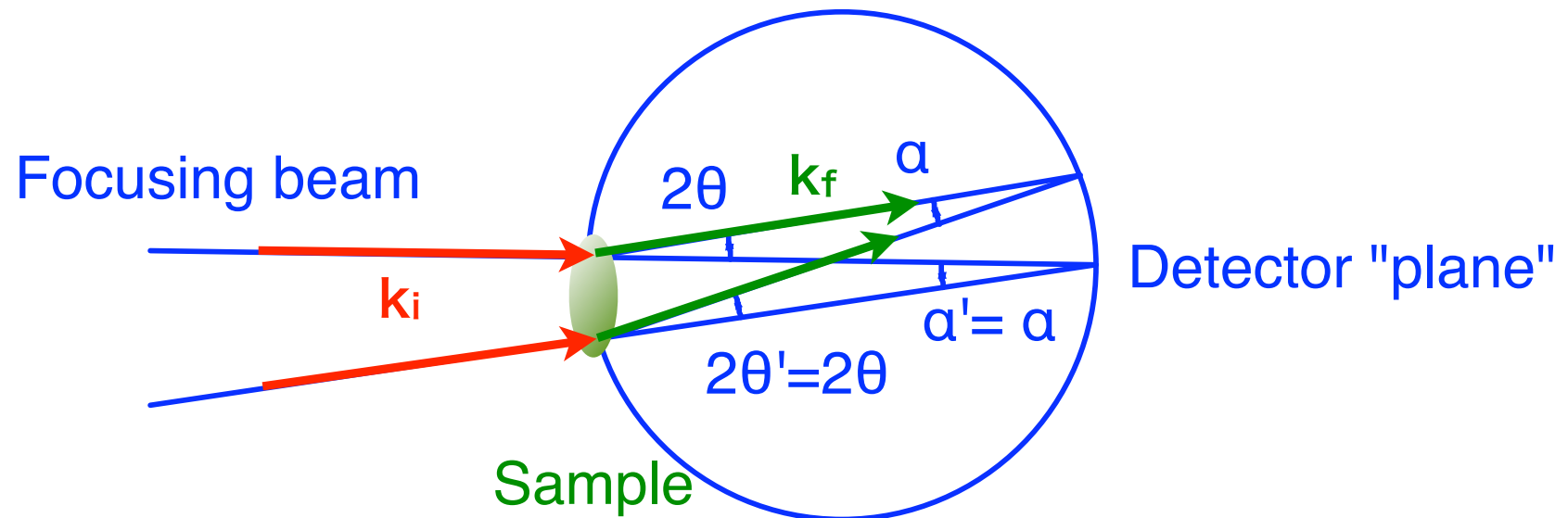
## ■ Conventional point collimation



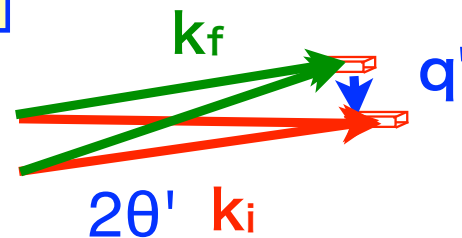
# Focusing in k space

48

Real space



Reciprocal space



Mini-Focusing Small-Angle Neutron  
Scattering Instrument (mfSANS)  
@Hokkaido University

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49

Time-of-flight focusing SANS

# mfSANS at Hokkaido Univ.

50

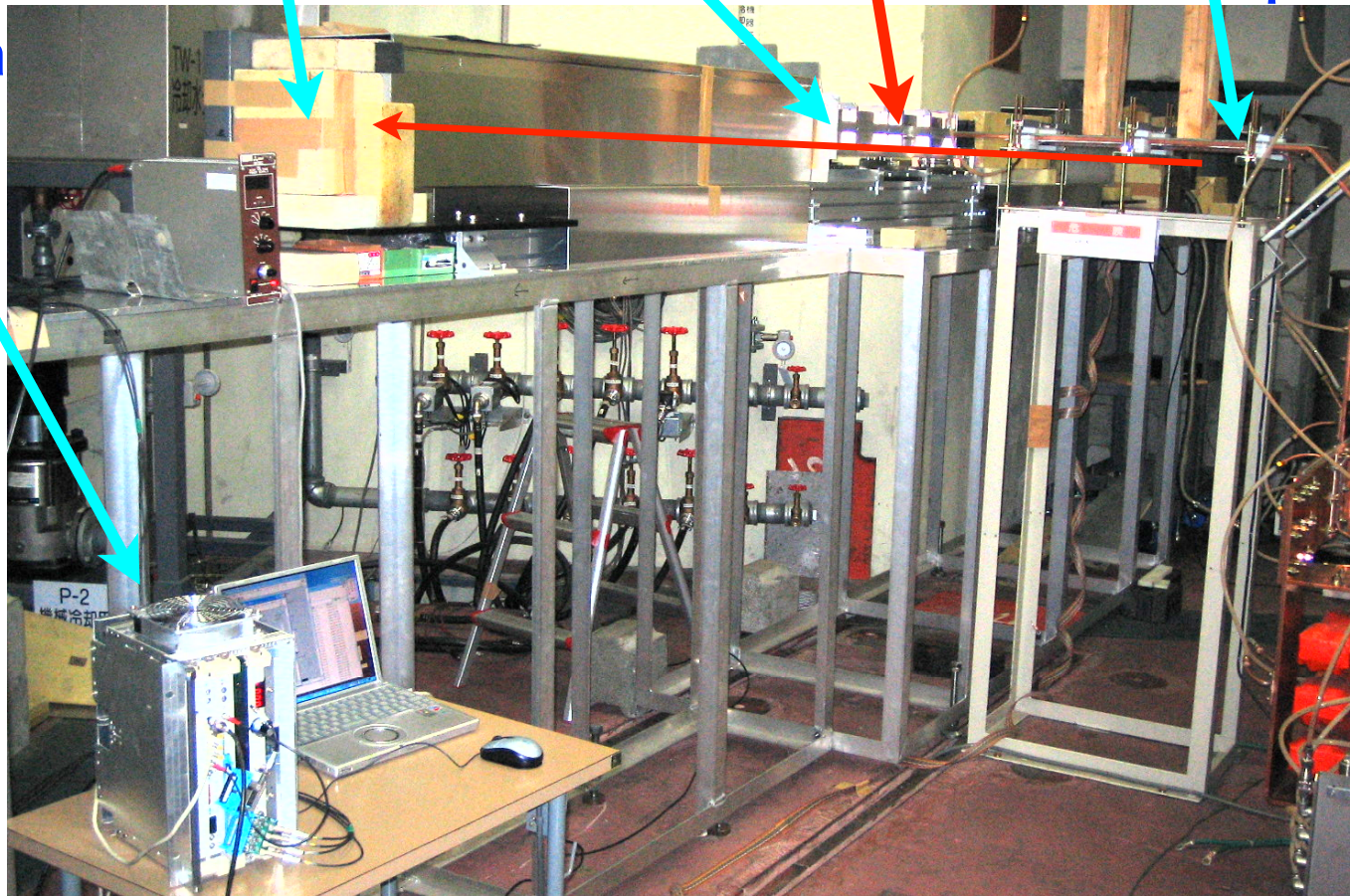
Data  
acquisition  
system

Detector

Sample

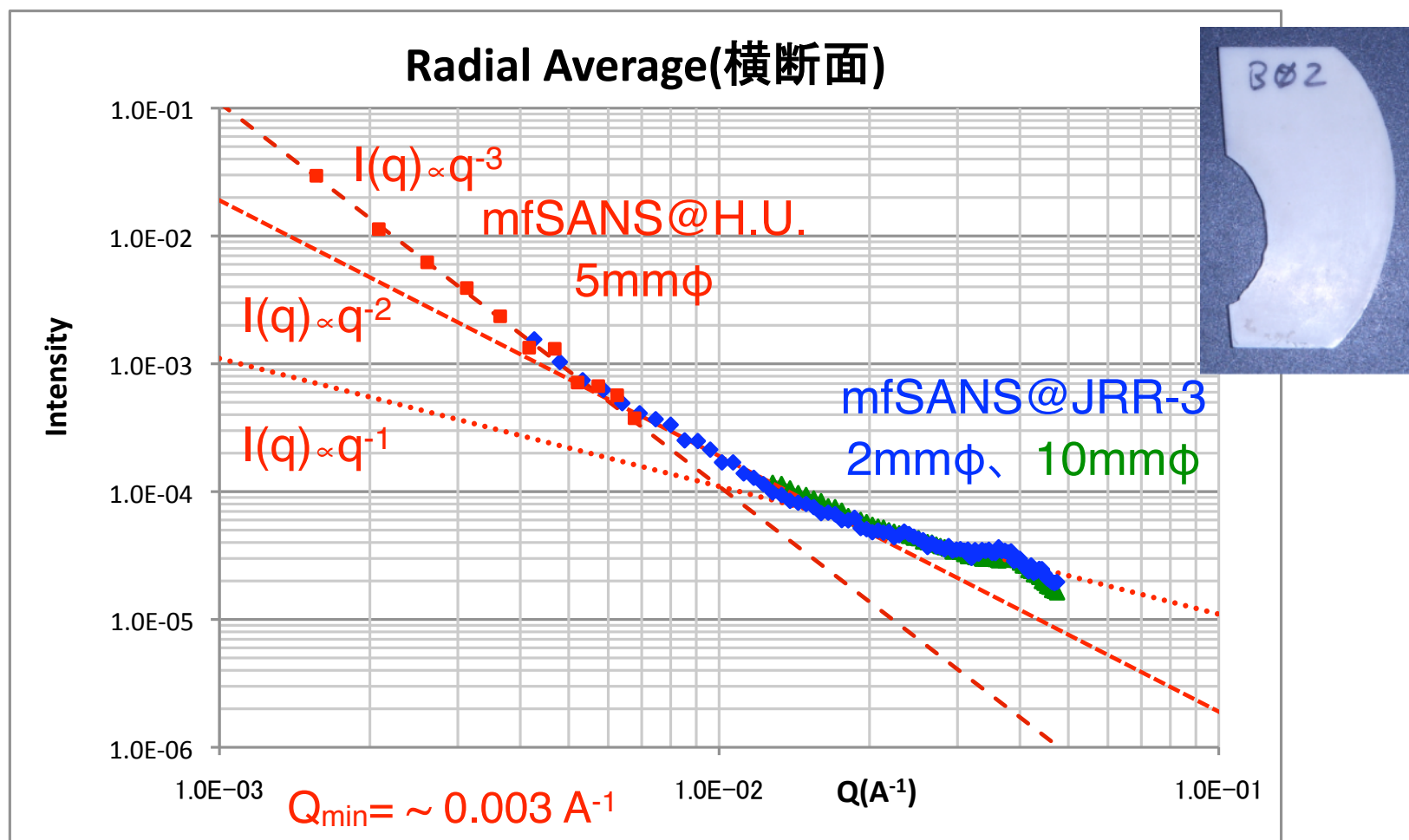
Ellipsoidal  
mirror

Beam  
port



# Bovine thighbone, cross section SANS preliminary analysis

51



Okusawa et al

# mfSANS@JRRR-3/JAEA

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52

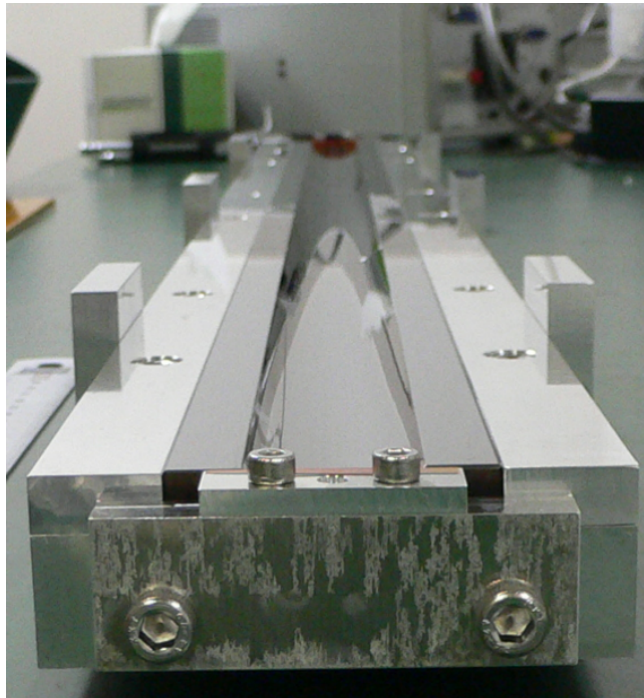
Monochromatic neutron  
focusing SANS



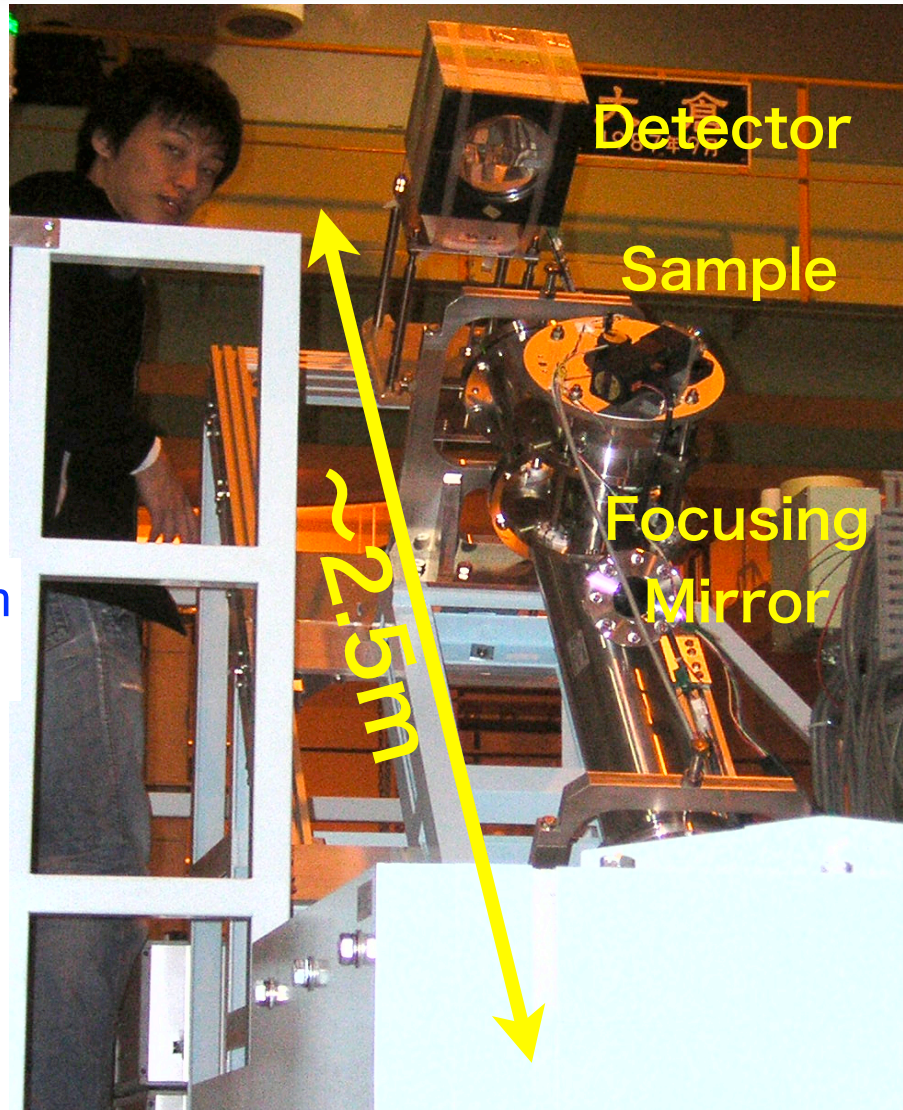
# Prototype focusing SANS@JRR-3

53

- Ellipsoidal mirror
  - 2.5  $Q_c$  supermirror
  - 2.5 m between focal points
  - short radius 20 mm

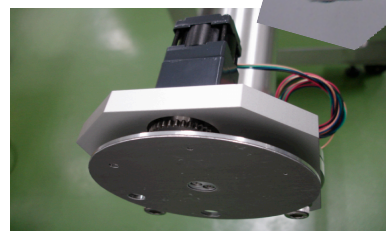
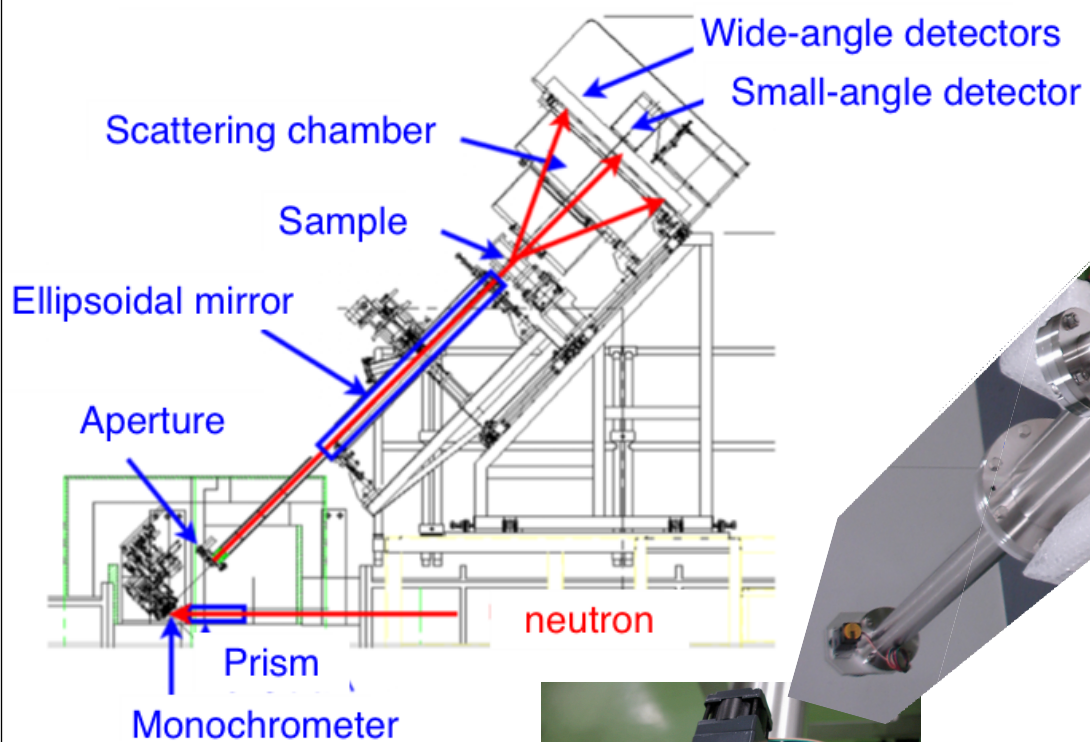


L: 900mm  
W: 20mm



# mfSANS@JRR-3

54

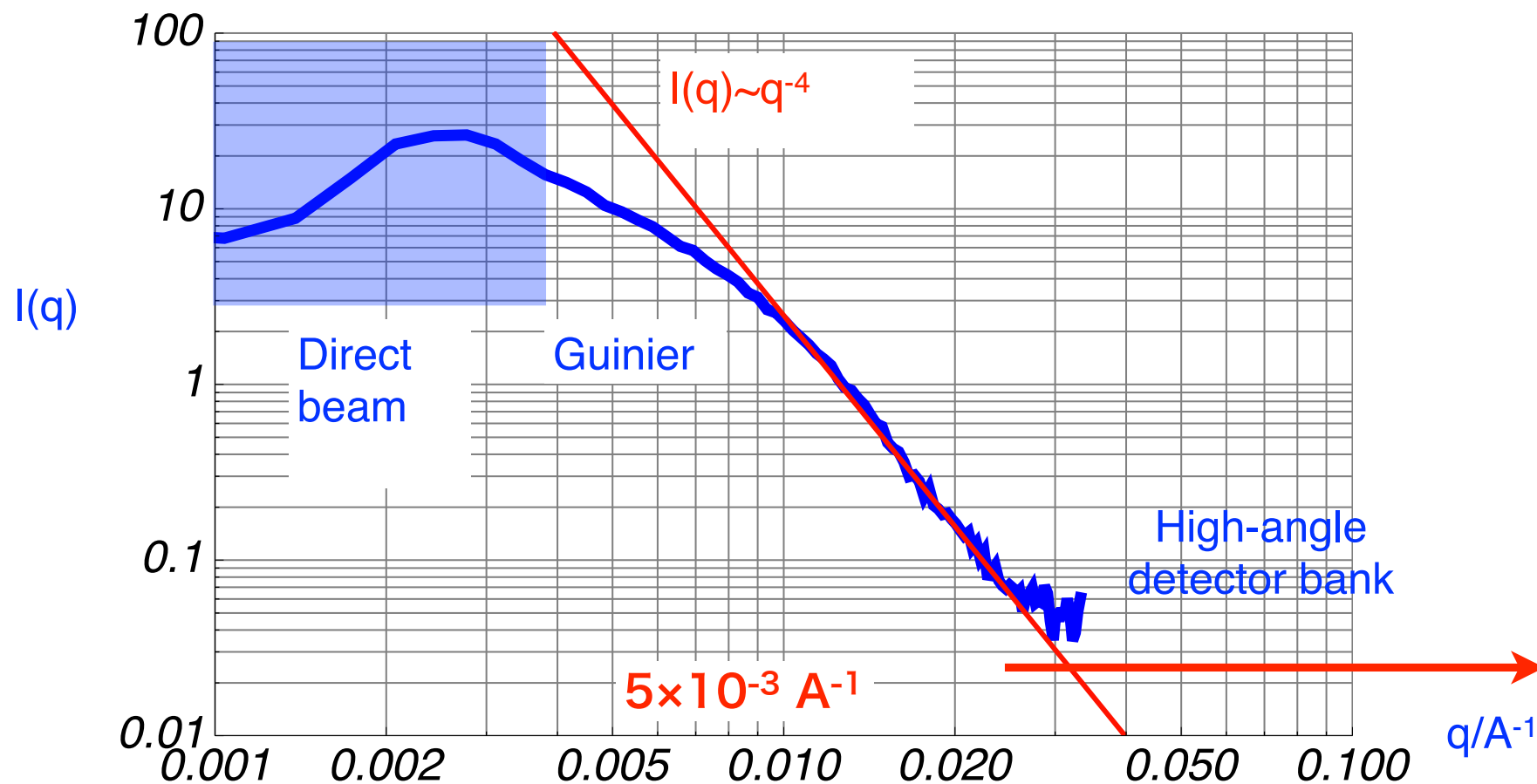


# Ni powder 20nm

Preliminary data

55

- $Q_{\min} = 5 \times 10^{-3} \text{ \AA}^{-1}$  using 2mm $\varnothing$  aperture.

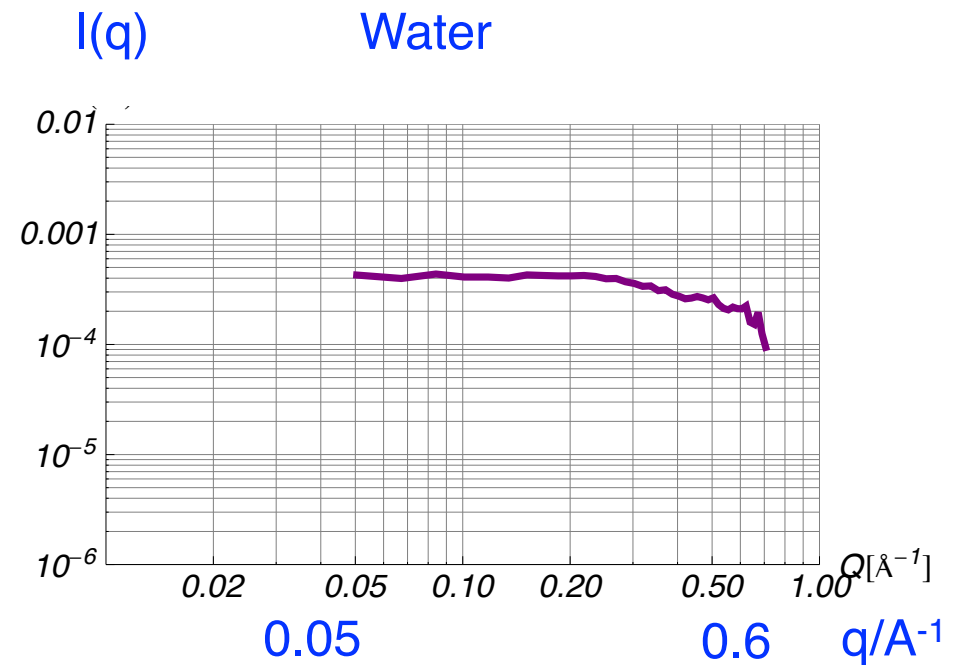
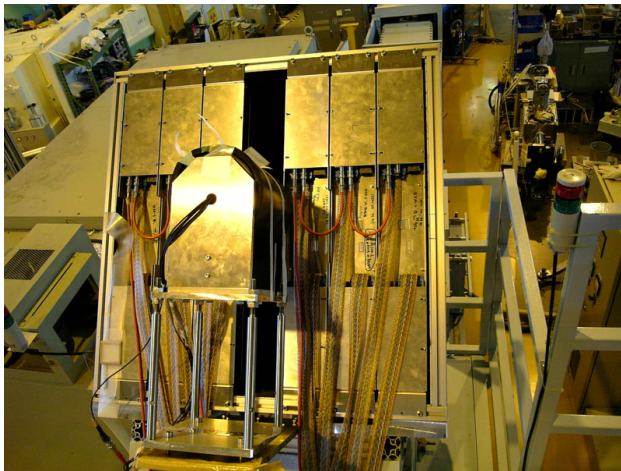




# wider-angle scattering Preliminary data

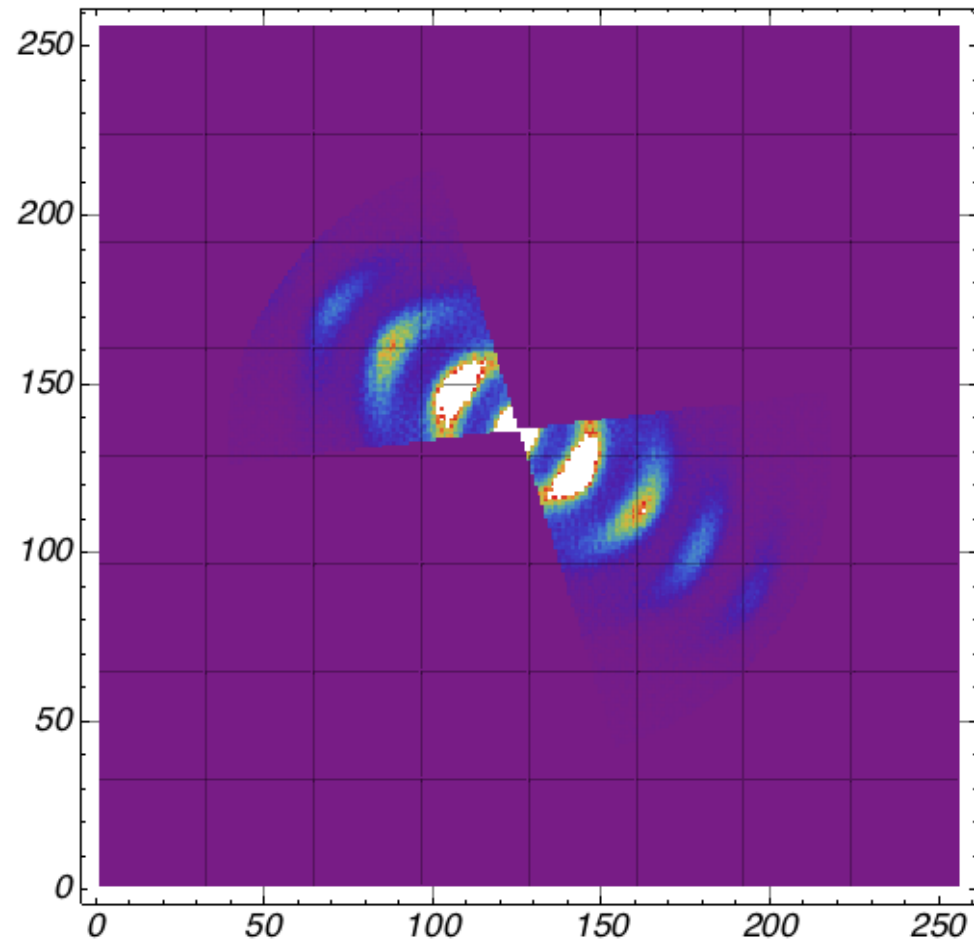
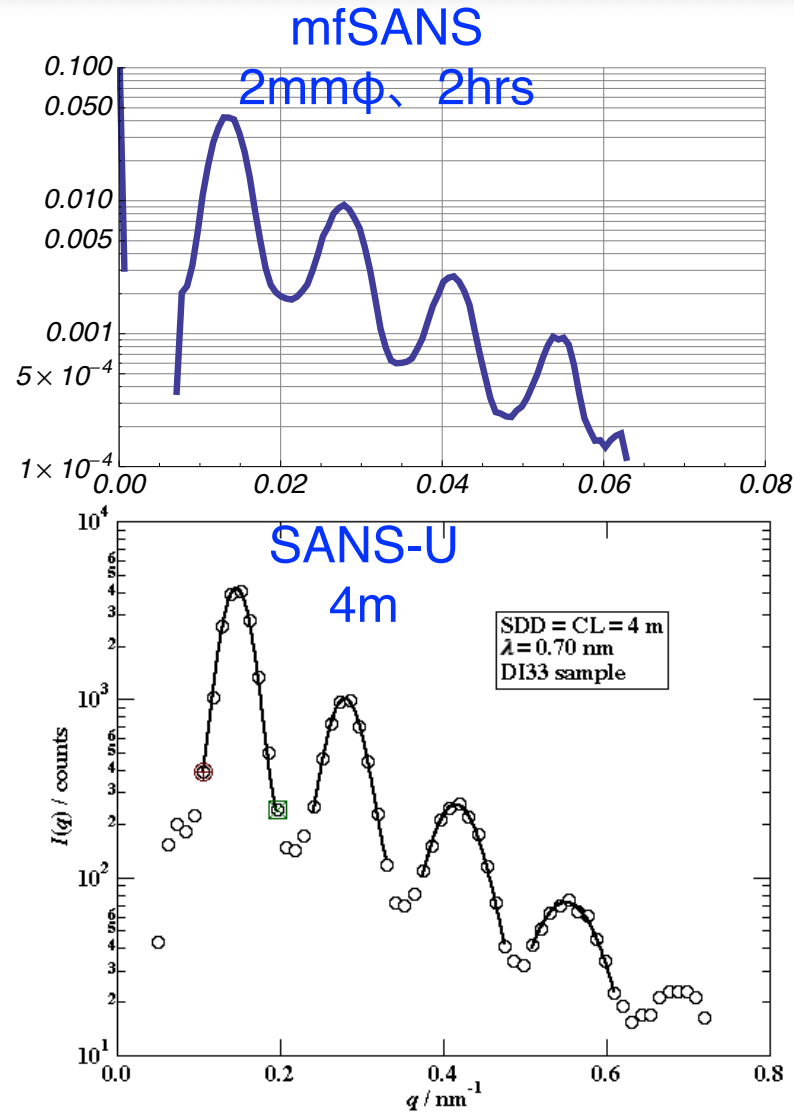
56

- 48 Linear position sensitive detectors at higher angle
  - 1/2 inch dia, 600 mm in length
  - GE made



# Micro-phase separated block copolymer

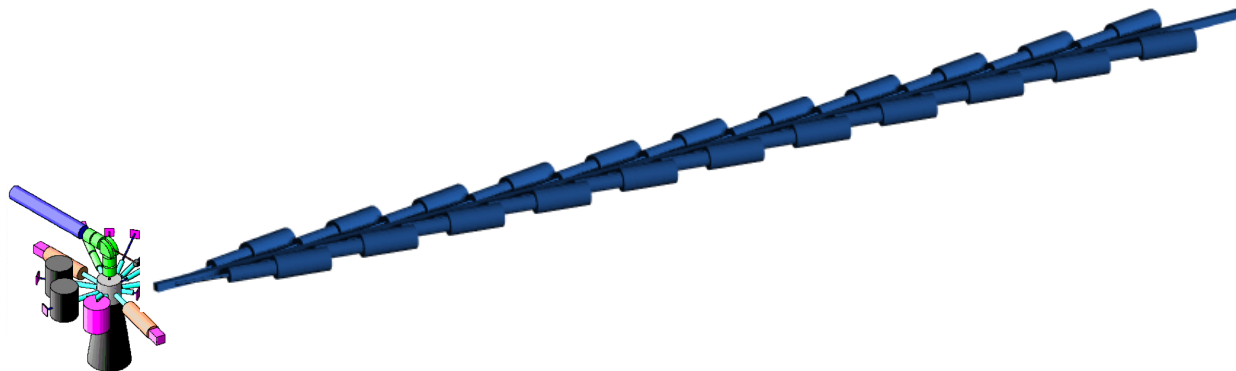
57



# Accelerator-driven Laboratory-size neutron-source

58

- RFQ accelerator + DTL  $\approx 3\text{-}11\text{ MeV}$ 
  - Li or Be target
- Combined with:
  - many mfSANS modules,
  - mini-reflectometers,
  - mini-powder machines??





# Summary

59

- Tailor-made neutron scattering instruments beamline
  - To check **new ideas**
  - Good for self-education
- Small-pinhole SANS demonstrated
- Various new devices under development
  - High performance monochromator
- Very compact SANS instruments under development
  - at Hokkaido university (pulsed neutron source)
  - at JRR-3 reactor of JAEA
  - High-performance SANS modules of your own at large facilities
- With focusing technique  
we can build very compact SANS instrument

