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Recent progress of pulsed neutron imaging in Japan

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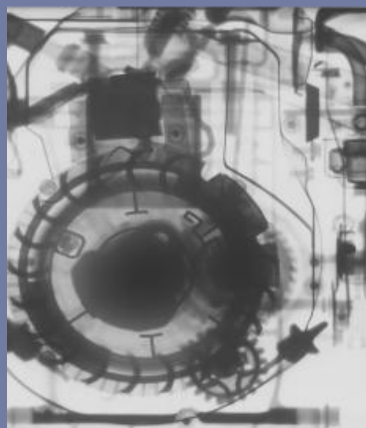
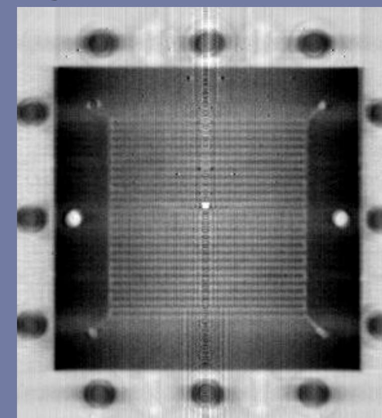
Characteristics of spectroscopic imaging using a pulsed neutron source

Traditional neutron radiography: Contrast



Engine

Lily

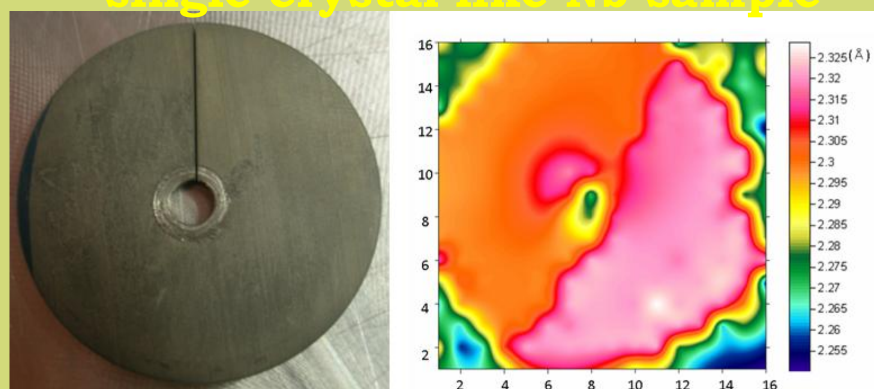


Fuel cell

Spectroscopic imaging (diffraction like)

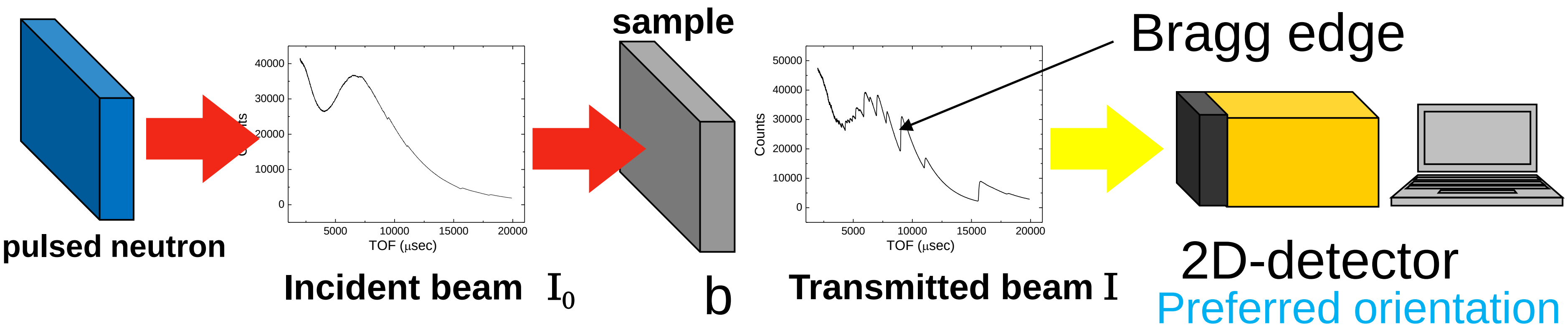
We can get image showing the crystallographic characteristics of a sample.

Lattice spacing image of a single crystal like Nb sample



Principle of pulsed neutron imaging and analysis of the pulsed neutron transmission data

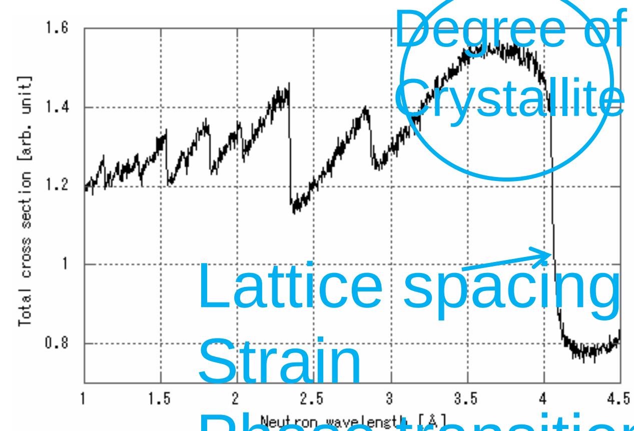
Experimental setup for spectroscopic imaging at a pulsed source and obtained information



$$\Sigma_{tot}(\lambda) = -\frac{\ln\left(I(\lambda) / I_0(\lambda) \right)}{b}$$

b: Sample thickness

λ: Neutron wavelength



(Bragg edge analysis) + (2D-detector)

Position dependent crystal and texture information

Rietveld-type Transmission-Spectrum analysis code: RITS (by H. Sato)

Transmission spectra $Tra(\lambda) = \exp\left[-\sum_p \sigma_{tot,p}(\lambda)\rho_p t_p \right]$

$$\sigma_{tot}(\lambda) = \sigma_{coh}^{el}(\lambda) + \sigma_{incoh}^{el}(\lambda) + \sigma_{coh}^{inel}(\lambda) + \sigma_{incoh}^{inel}(\lambda) + \sigma_{abs}(\lambda)$$

$$\sigma_{coh}^{ela}(\lambda) = \frac{\lambda^2}{2V_0} \sum_{d_{hkl}=0}^{2d_{hkl}<\lambda} \left[|F_{hkl}|^2 \times d_{hkl} \times P_{hkl}(\lambda, d_{hkl}) \times O_{hkl}(\lambda, d_{hkl}) \times E_{hkl}(\lambda, F_{hkl}) \right]$$

Profile function near the edge (Lattice space)

March-Dollase function (Preferred orientation)

Extinction function (Crystallite size)

V_0: Volume of a unit cell

d_{hkl}: Lattice plane distance of (*hkl*) plane

F_{hkl}: Crystal structure factor

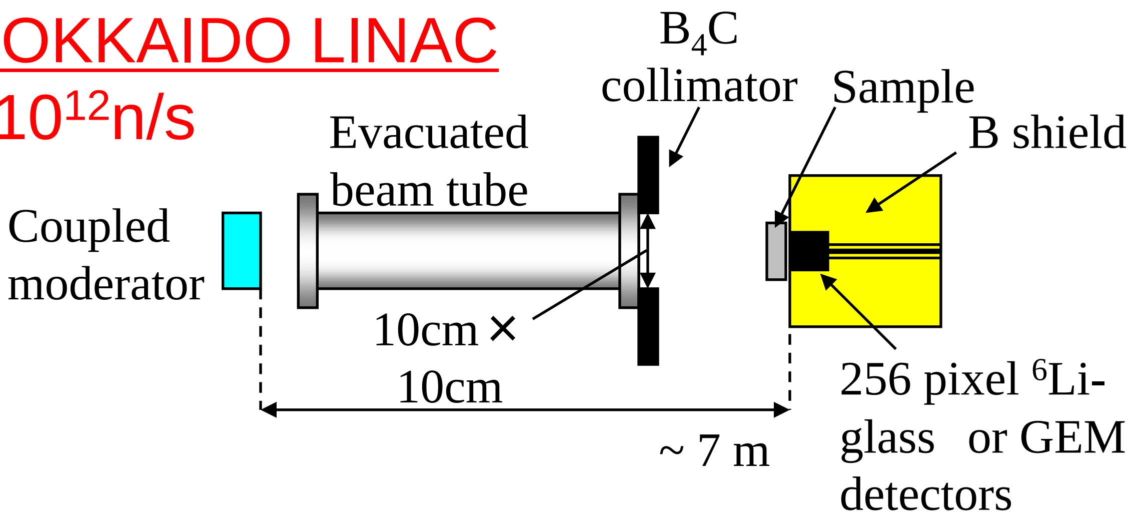
Examples of evaluation of crystallographic characteristics of materials by pulsed neutron transmission

Experimental setup at Hokkaido linac and BL10 at J-PARC

14

HOKKAIDO LINAC

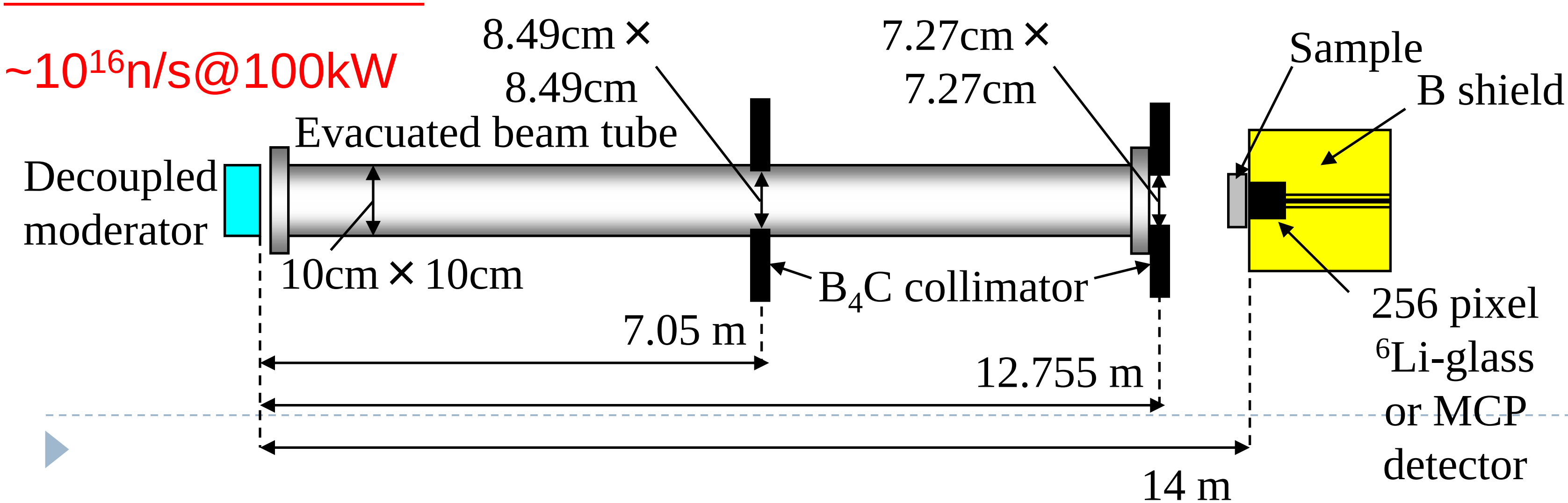
~10^{12}n/s



J-PARC

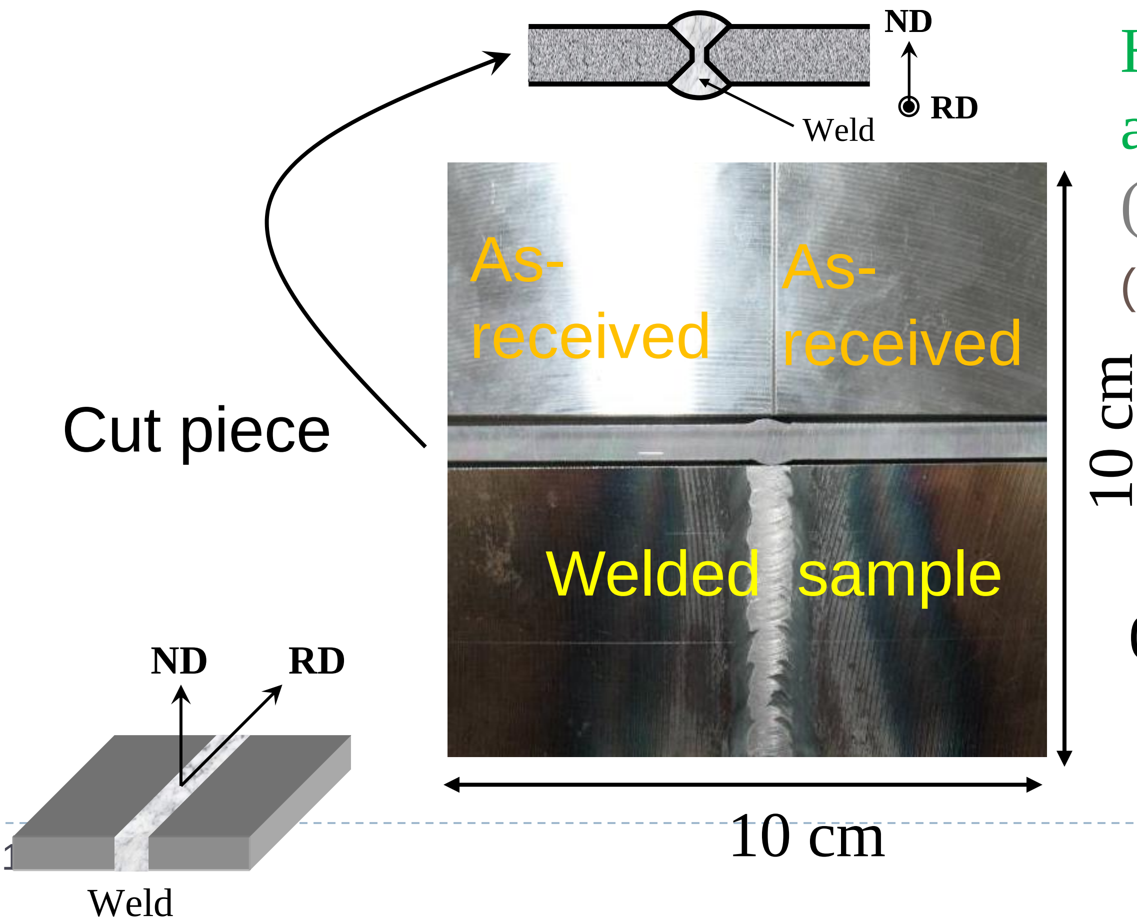
J-PARC MLF BL10

~10^{16}n/s@100kW



a. Welded iron plate

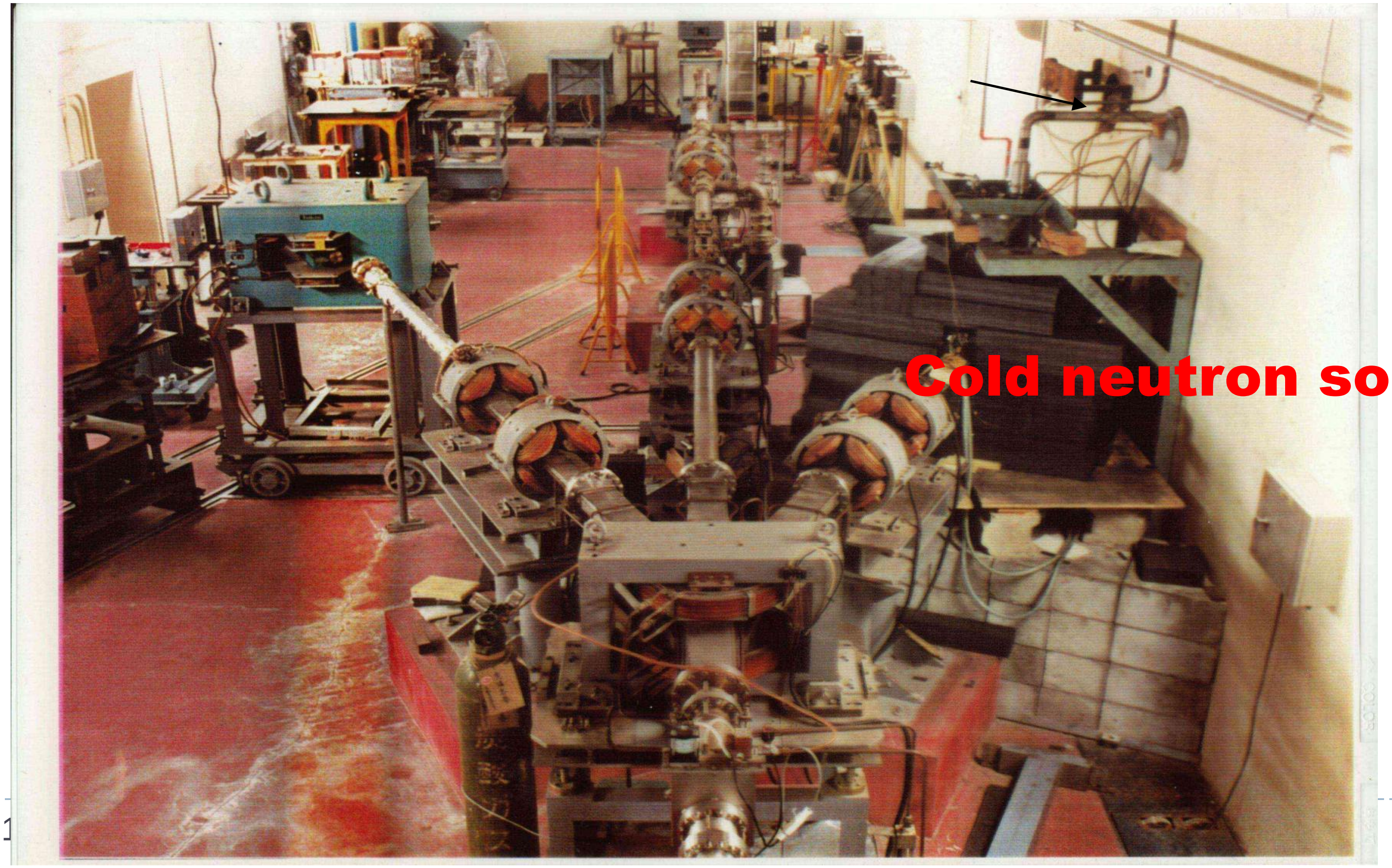
Aim: to observe the change of preferred orientation and crystallite size between welded and original areas.



H. Sato, T. Kamiyama and Y. Kiyanagi (Materials Transactions, 52 (2011) pp.1294-1302.)

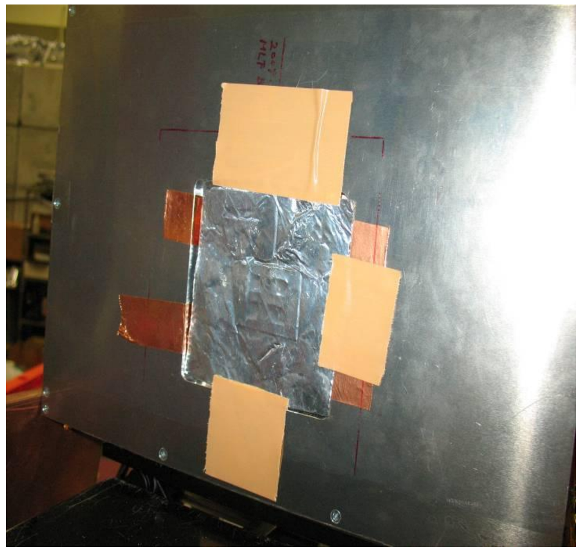
0.6 cm thickness

Hokkaido electron linac neutron source



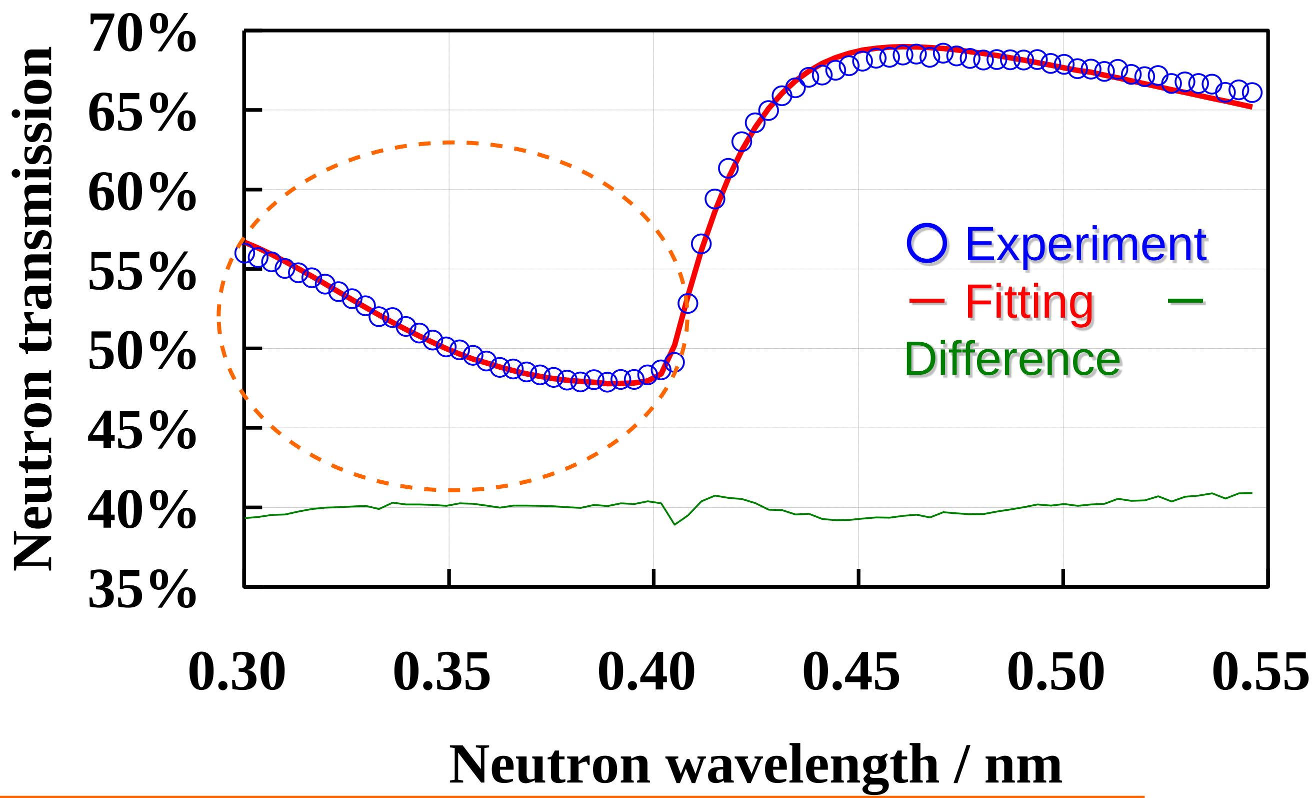
GEM detector

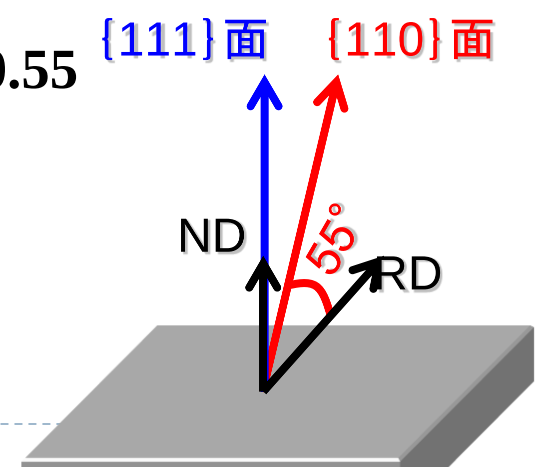
GEM detector developed by S. Uno(KEK)



- Pixel size：0.8mm x 0.8mm, 14400（120×120）
- Effective area：10 cm×10 cm
- Counting rate：>1MHz/Detector
- Time stamp：~20ns

{110} orientation analysis（ND）at outside of the welded region

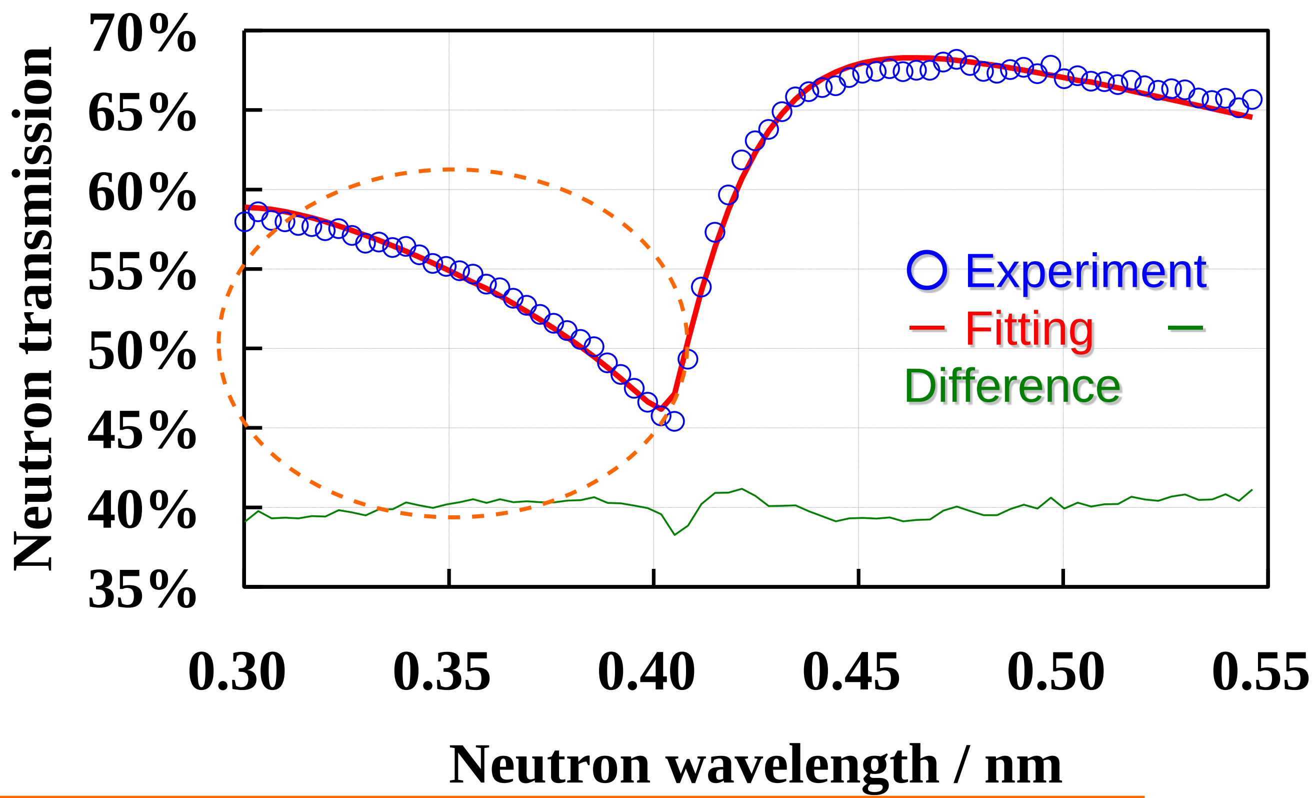


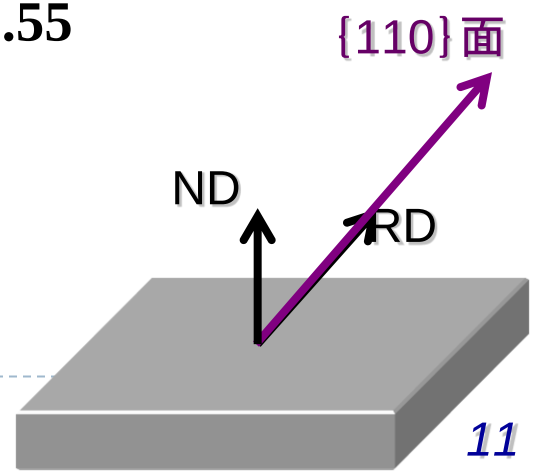


Rietveld refinement parameters obtained by fitting

Degree of anisotropy（March-Dollase coefficient）“ *r* ”：0.54
Preferred orientation “ *A* ”：<111> along ND（∵ $r < 1$）
Surface density “ $\rho \times t$ ”：4.79×10^{22} cm^{-2}
Crystallite size “ *S* ”：4.65 μm

{110} orientation analysis（RD）at outside of the welded region

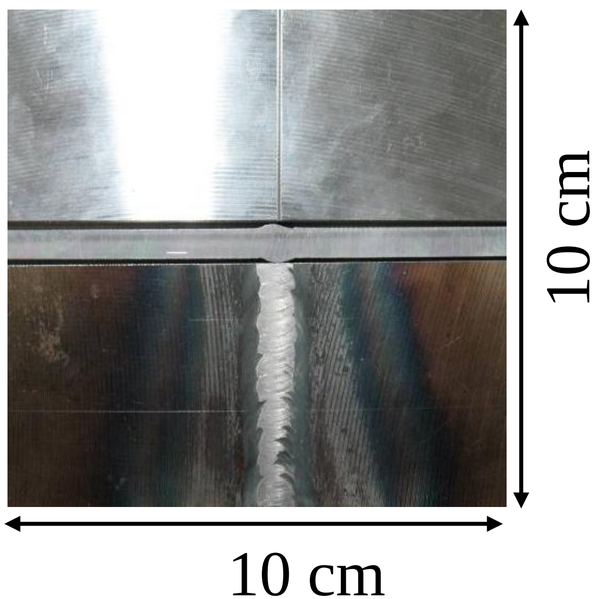




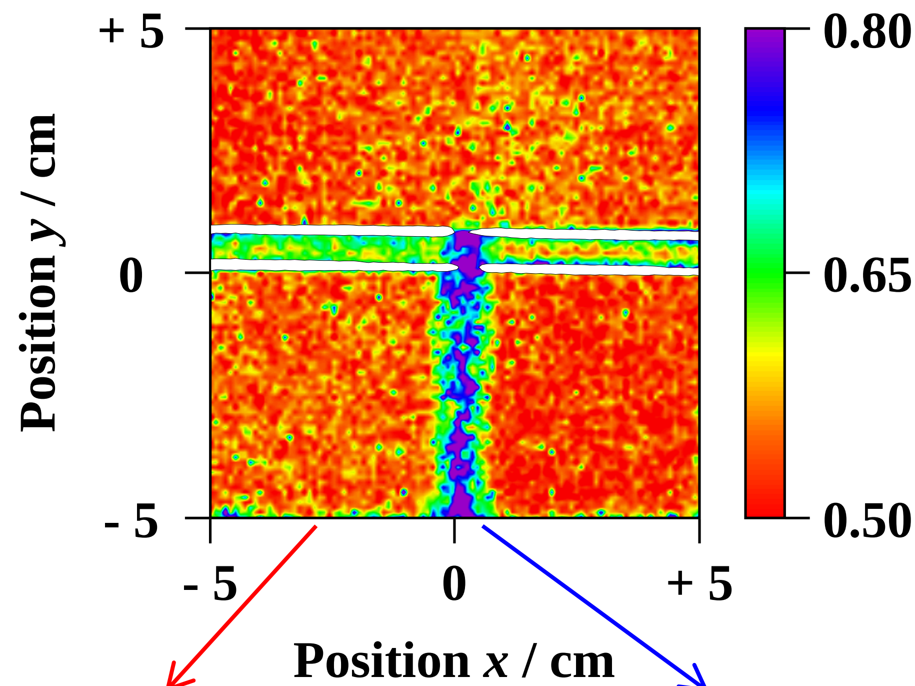
<u>*Rietveld refinement parameters obtained by fitting*</u>

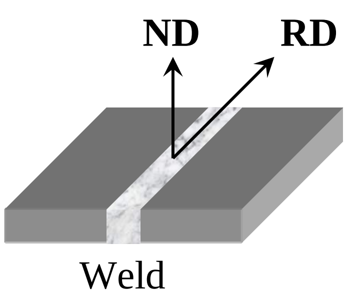
Degree of anisotropy（March-Dollase coefficient）“ *r* ”：0.65
Preferred orientation “ *A* ”：<110> along RD（∵ $r < 1$）
Surface density “ $\rho \times t$ ”：4.90×10^{22} cm^{-2}
Crystallite size “ *S* ”：6.09 μm

Anisotropy

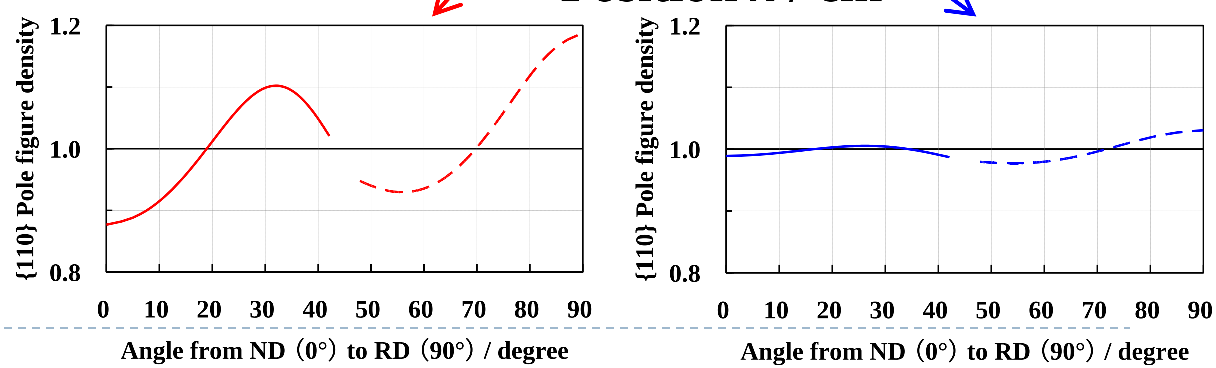


Degree of preferred orientation
(March-Dollase coefficient)



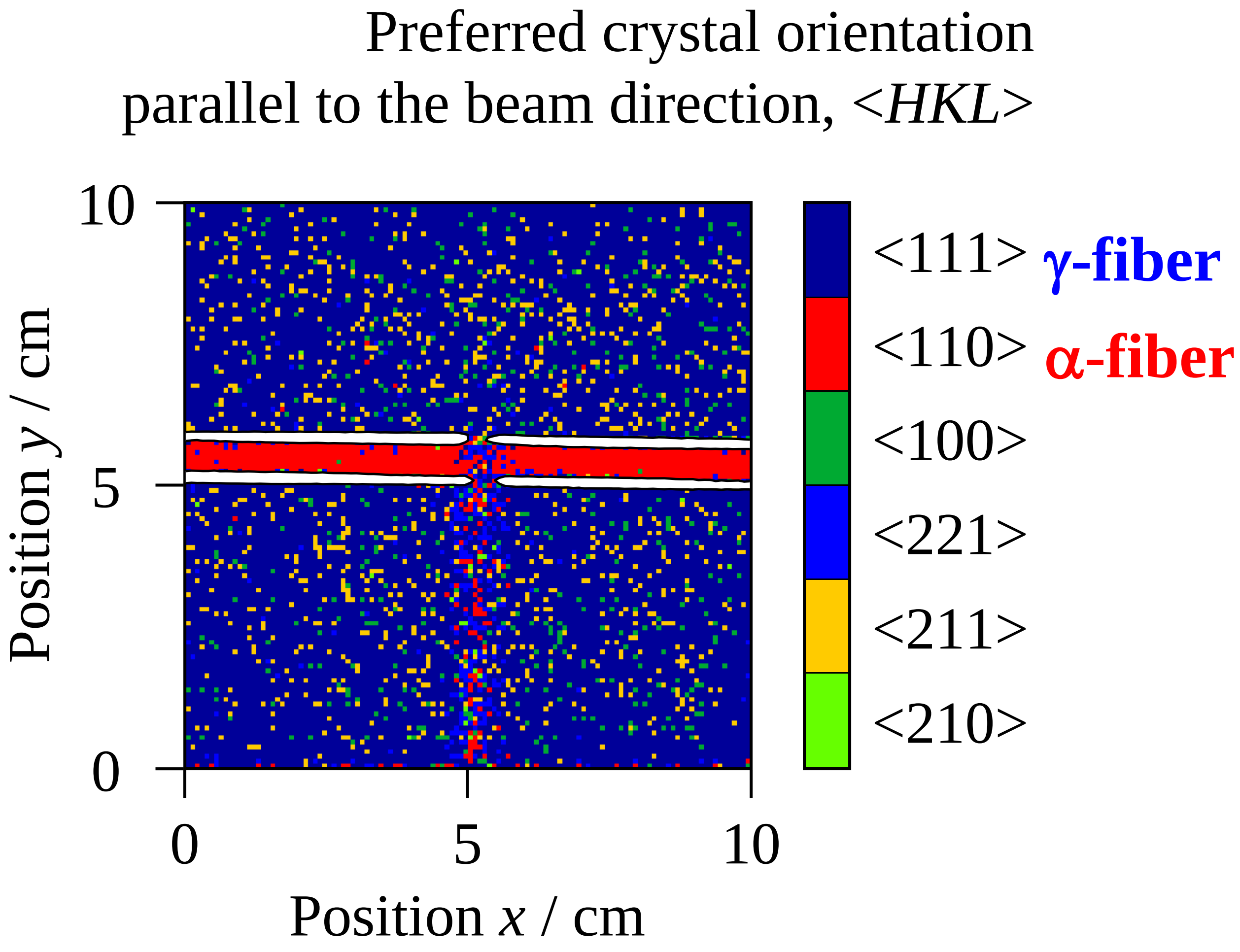


0.6 cm thickness

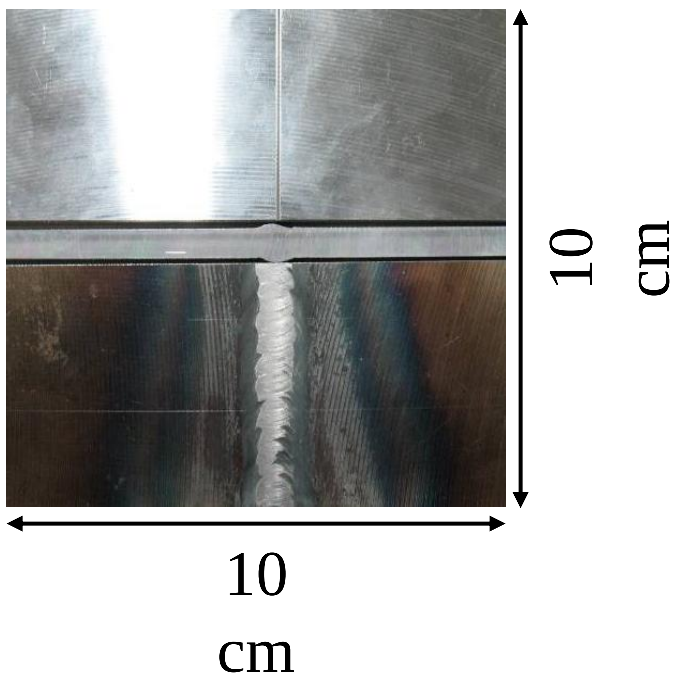


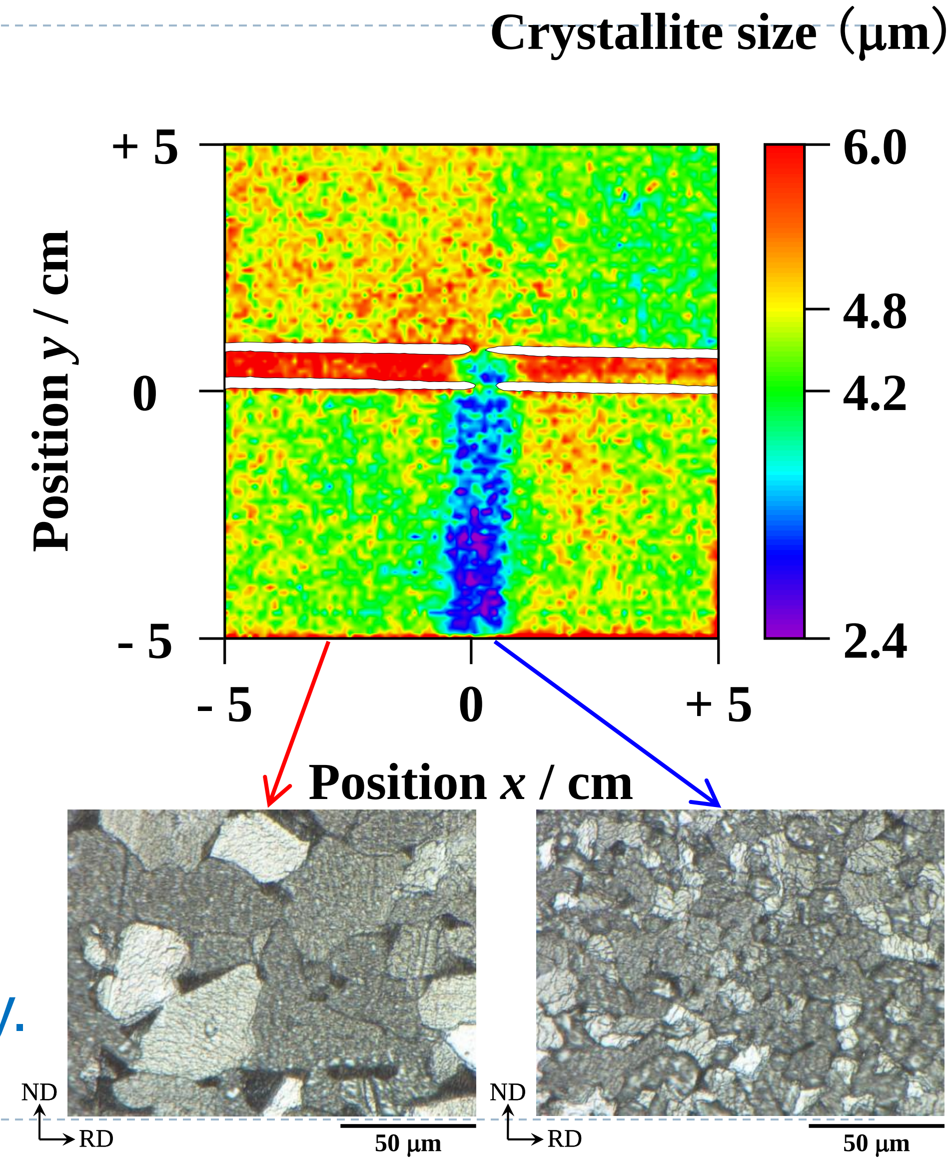
Pole figures extracted from a Bragg edge transmission

Preferred orientation



Crystallite size





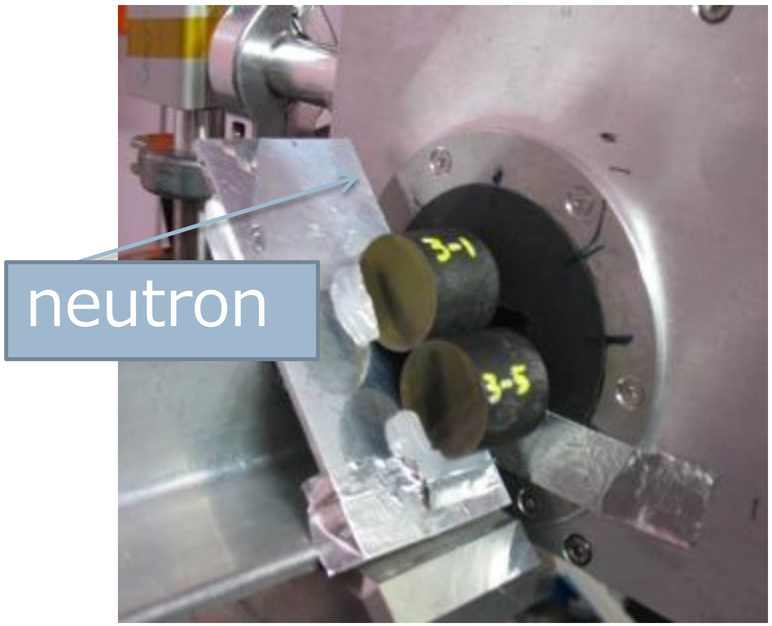
Confirmation of grain size by a microscope

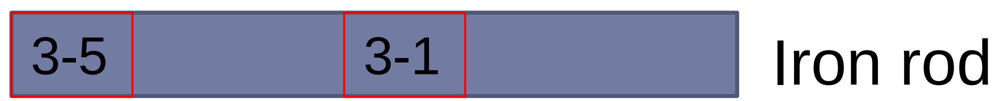
We observed the changes clearly.

b. Quenched iron rod

By T. Kamiyama, R. Takamori, A. Tremsin, N. Ayukawa, T. Shinohara, T. Kai, Y. Kiyanagi

Aim: to confirm the quenched region, 3 mm.

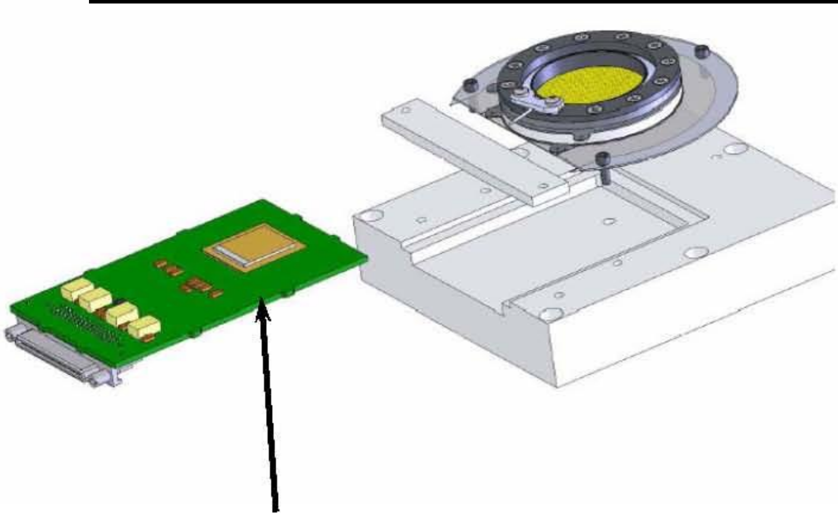


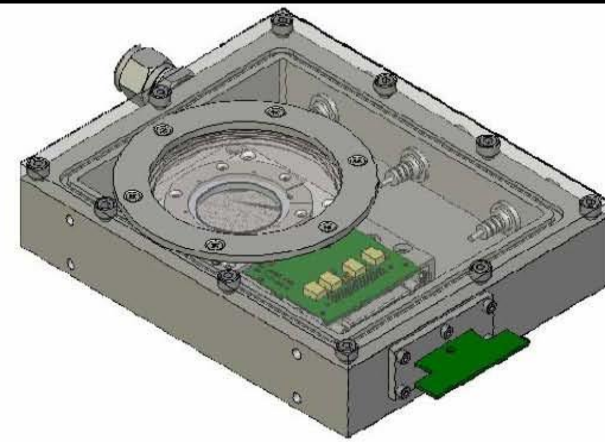


Sample: Quenched iron
Diameter: 26mm
Thickness: 20mm
Quenched thickness: 3mm

MCP detector with Medipix/Timepix readout

MCP detector developed by Dr. A. Tremsin at UC Barclay.



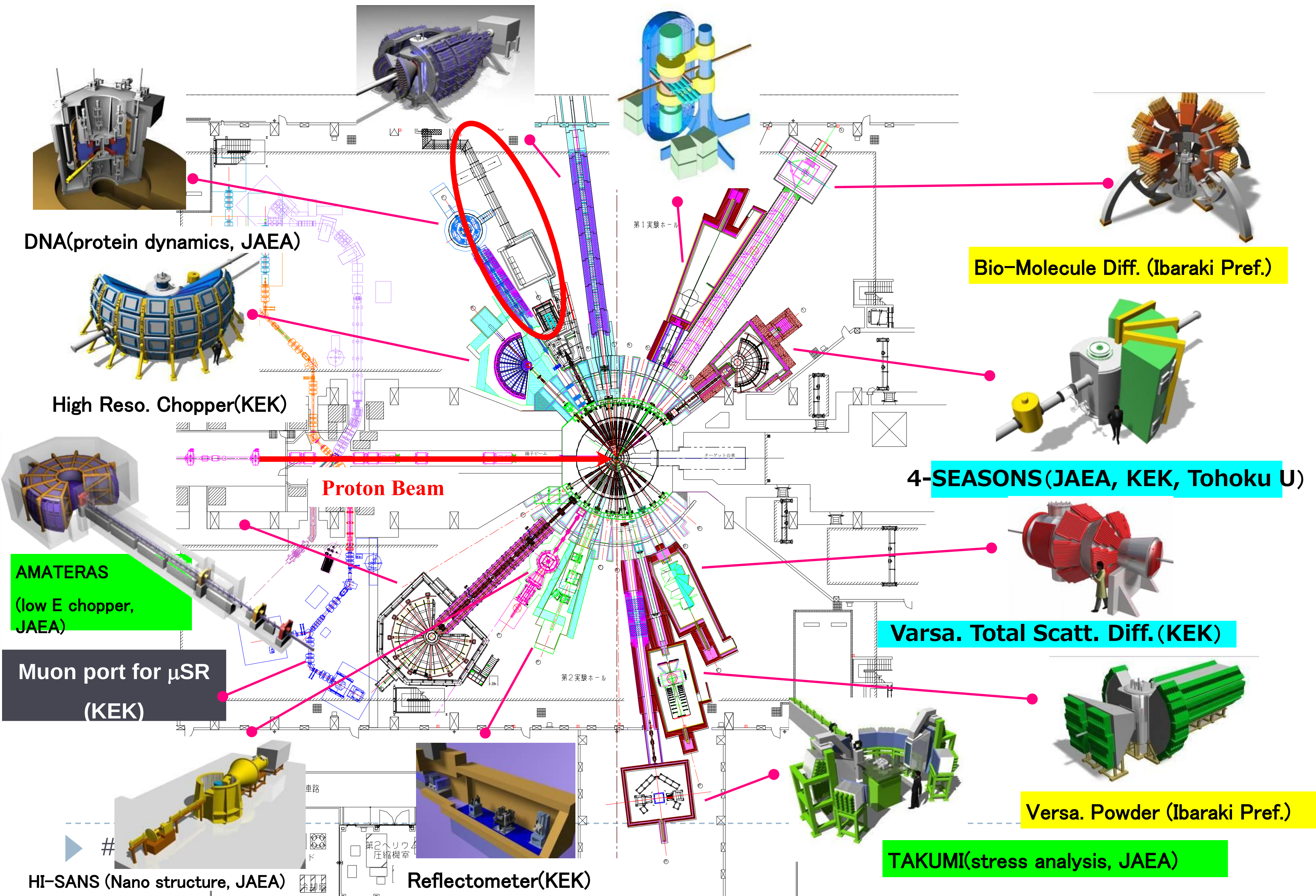


- 256 x 256 array of 55 μm pixels
- 100 kHz/pxl
- Frame rate: 1-3 kHz
- Low noise (<100e$^-$) = low gain operation (10 ke$^-$)
- ~1 W watt/chip, abuttable
- Developed at CERN

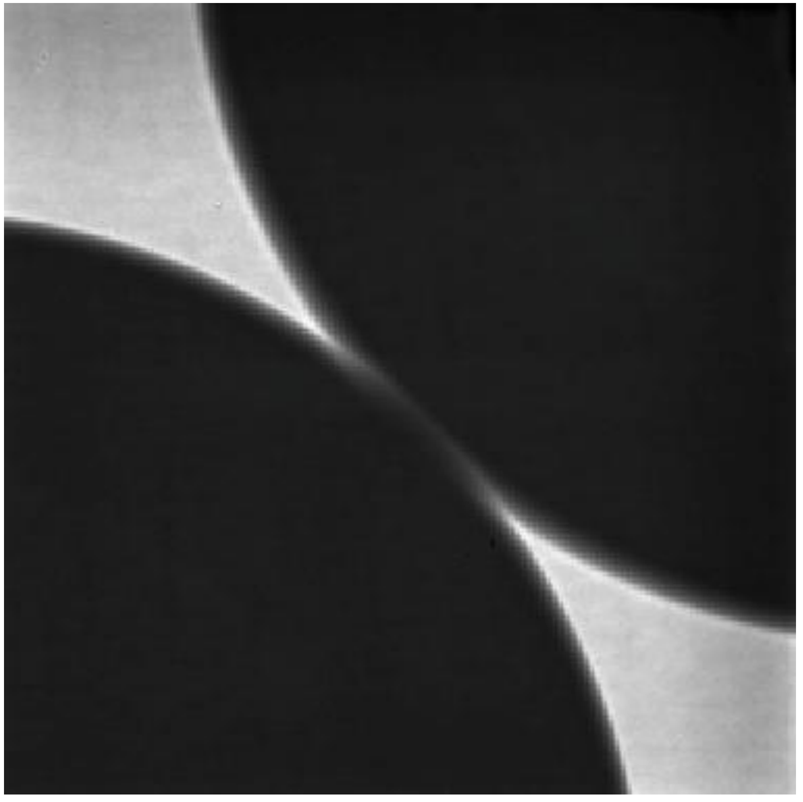
Stack of MCPs is placed above
Medipix2/TimePix readout

Muros readout electronics (NIKHEF) and
PixelMan software (IEAP, CTU
Prague) with
Labview and C++ plugins (UCB)

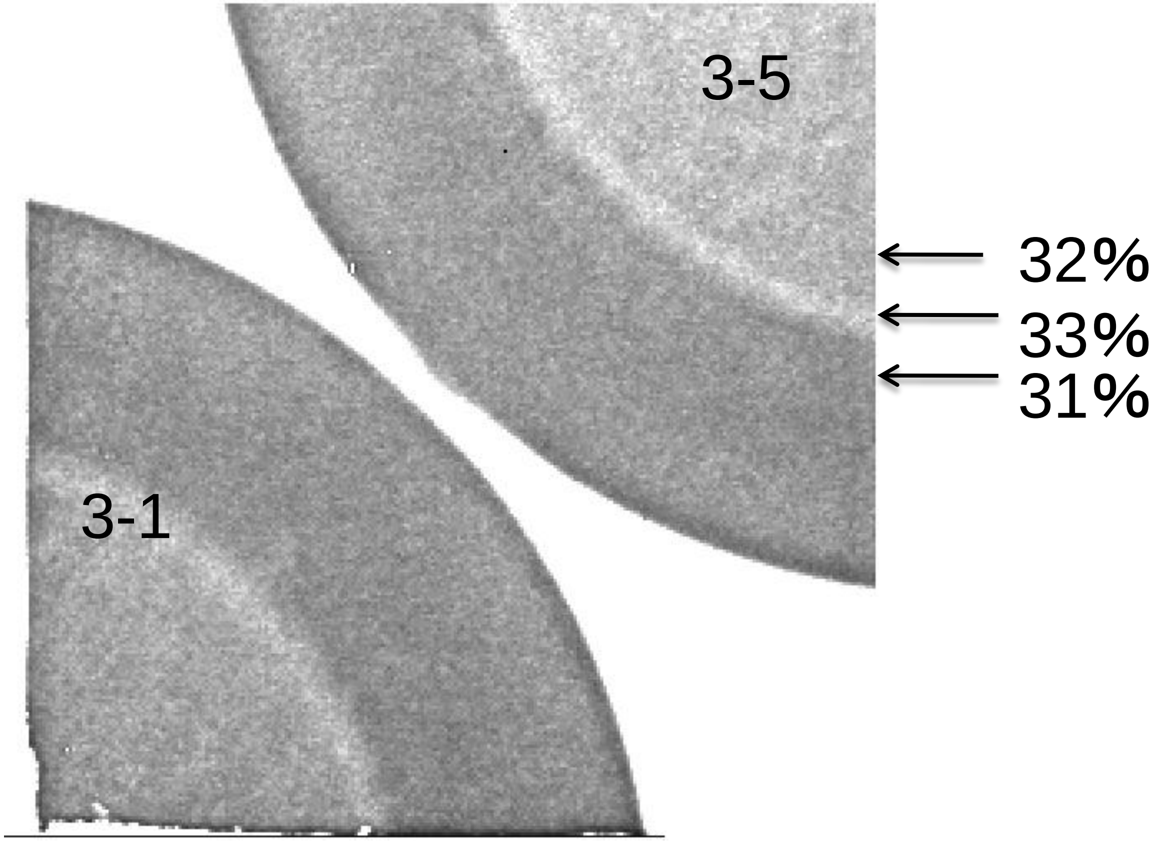
NOBORU beam line at J-PARC



Transmission image obtained by whole neutrons

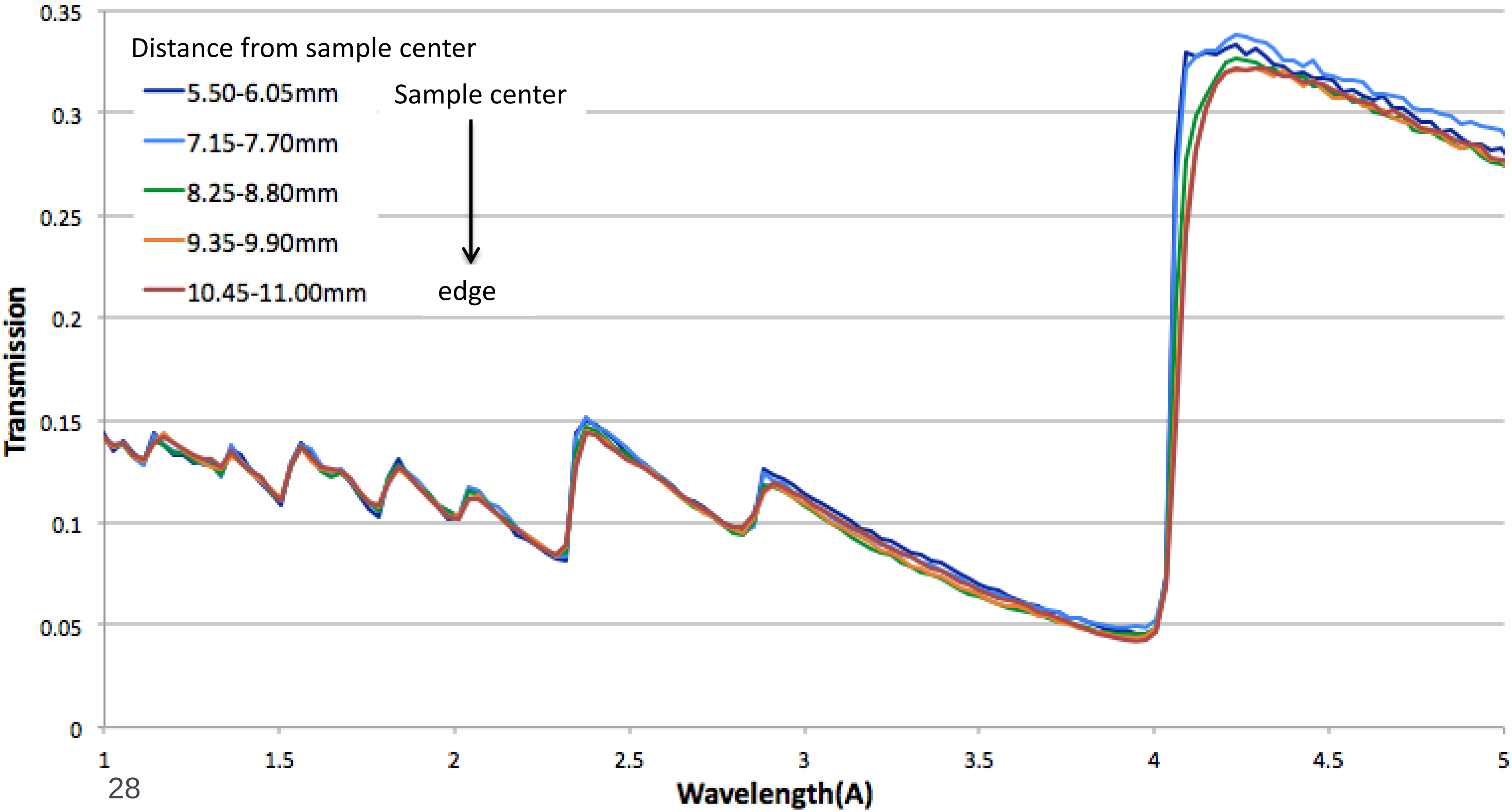


More detailed image of transmission around 4.5 A

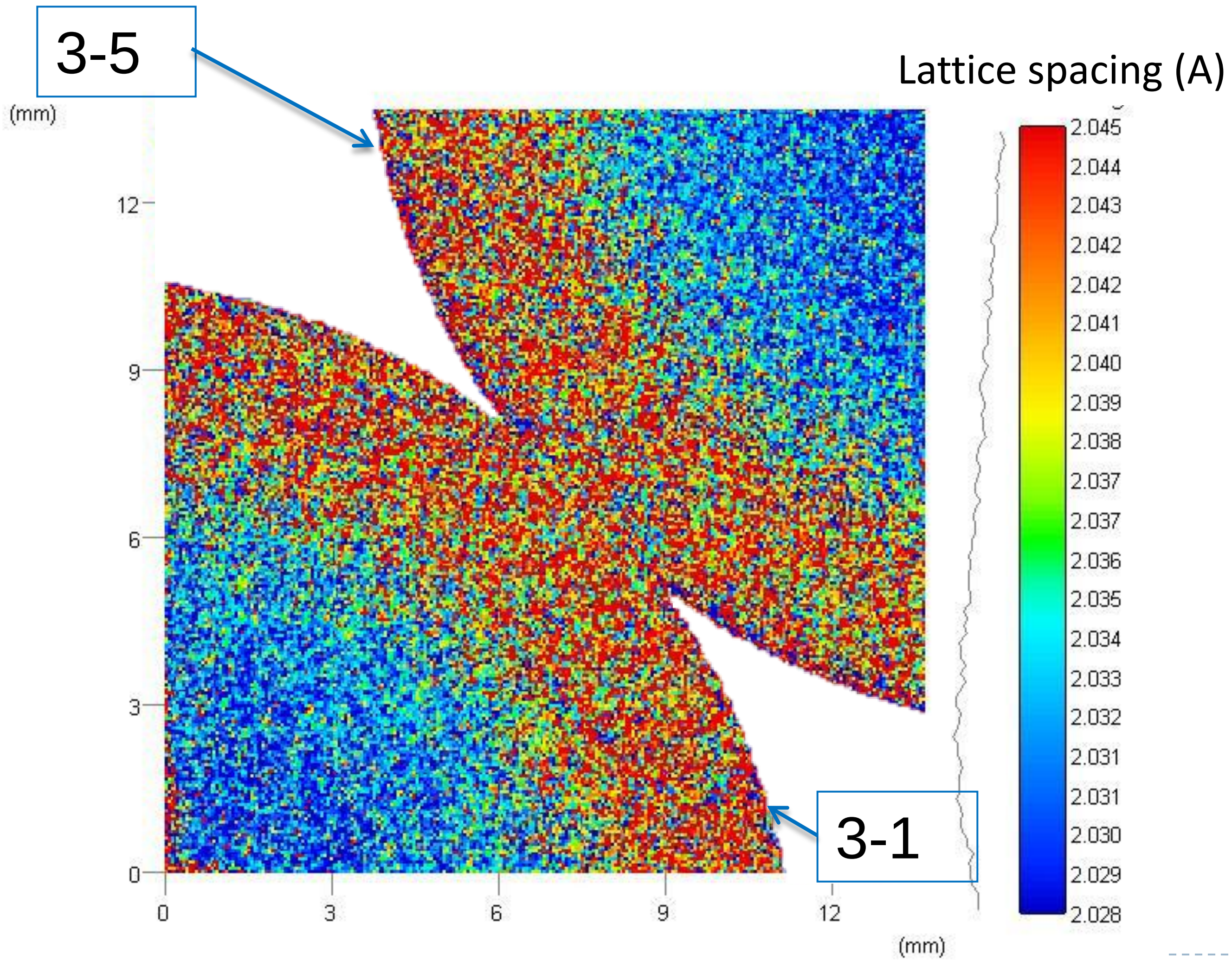


We observed an enhanced image of the boundary between quenched and un-quenched regions.

Transmission spectra



Lattice spacing image (55μm)



Lattice spacing at the quenched area is larger than the un-quenched one

c. In-situ tensile test

(strain, texture, crystallite size of a iron plate during tensile test)

Aim: to see change of the crystallite size, anisotropy, strain depending on load, and check reliability of strain obtained by the transmission.

To be submitted to Journal by

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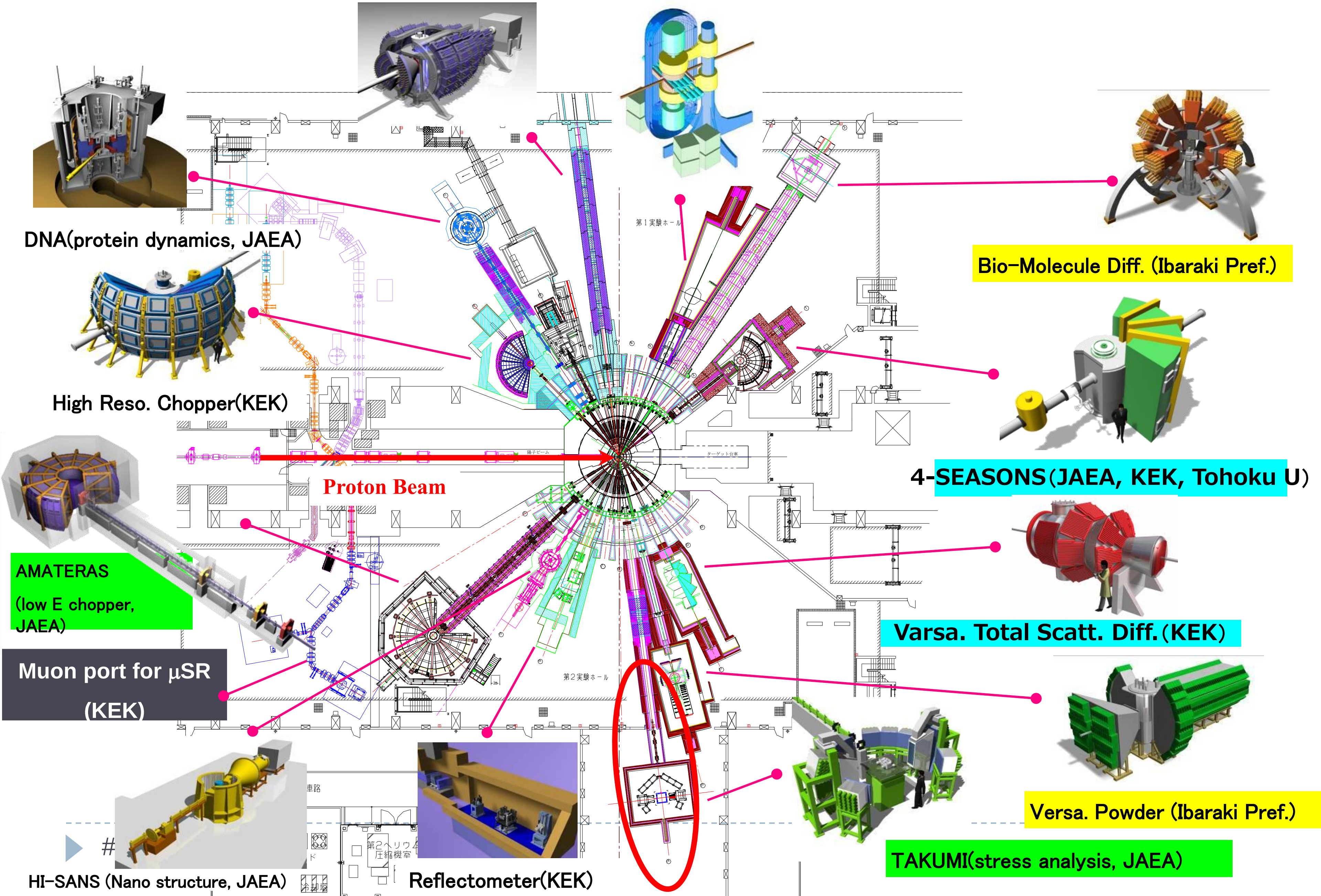
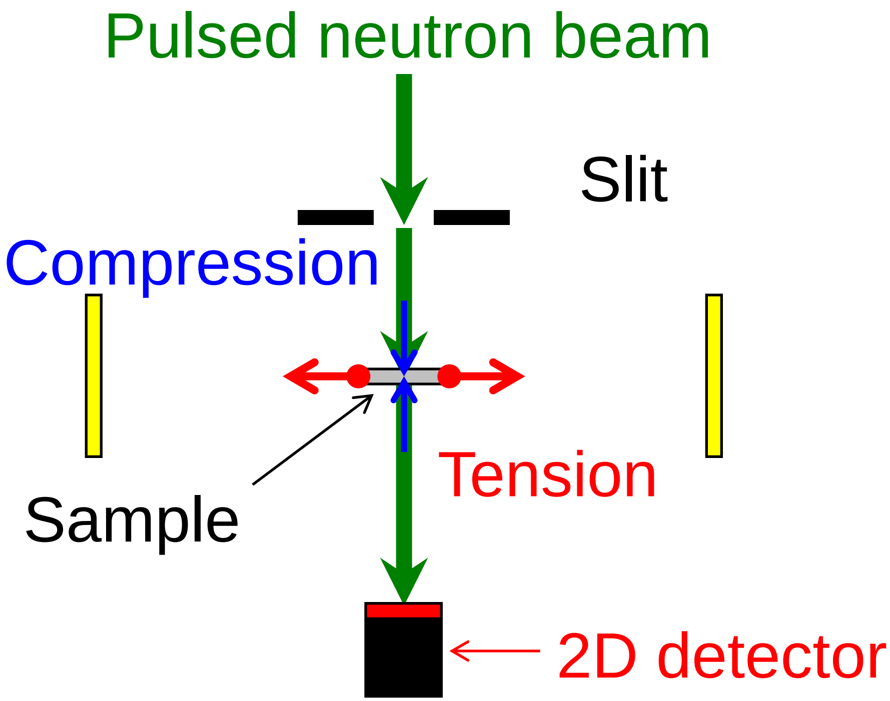
[1]Faculty of Engineering, Hokkaido
[2]Frontier Research Center for Applied Atomic Sciences, Ibaraki University
[3]J-PARC Center, Japan Atomic Energy Agency

‘TAKUMI’ beam line at J-PARC



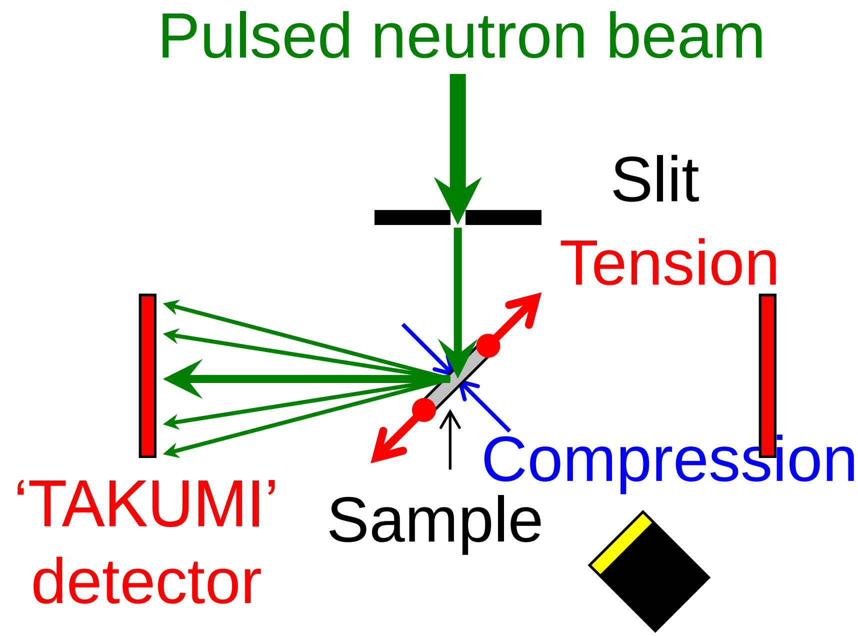
Setup for measurements under the tensile test

(1) Bragg edge transmission



- Slit：2cm width, 4cm height
- Spatial res. ：3mm×3mm
- Sample-detector：9cm
- Meas. time：3hrs/meas. (2min/pos.)

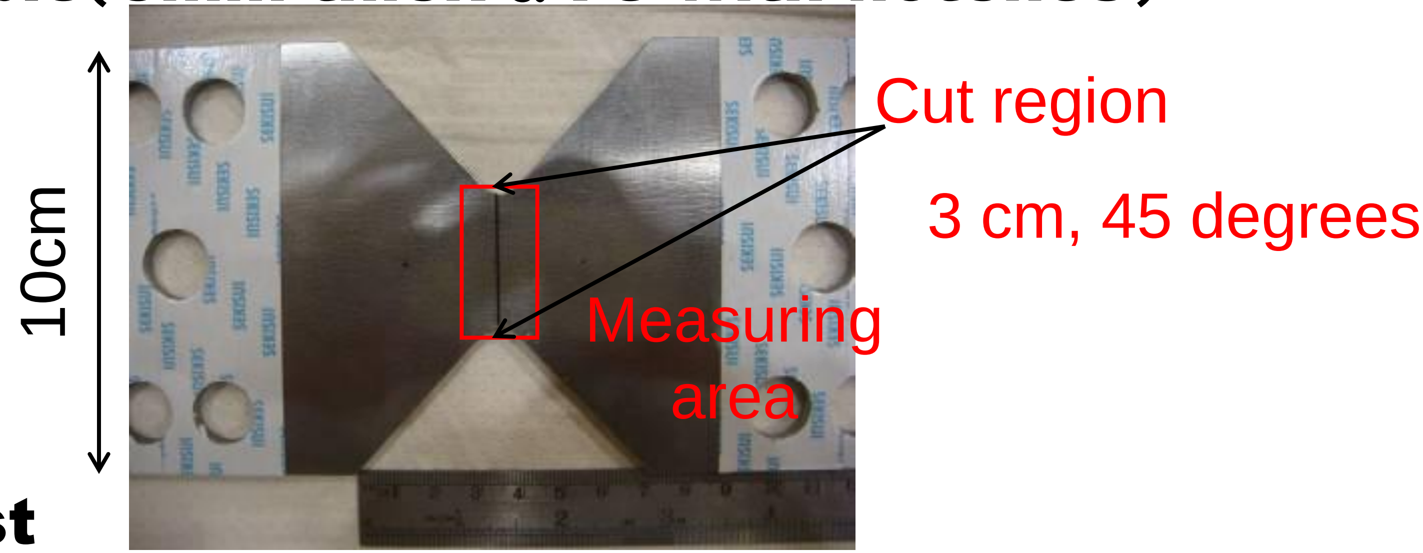
(2) Diffraction and mapping



- Slit:4mm width,4cm height
- slit (mapping) ：4mm×4mm
- Sample-detector：2m
- Meas. time：5min/meas.
- Meas. Time(mapping) ：2.5min/pos.

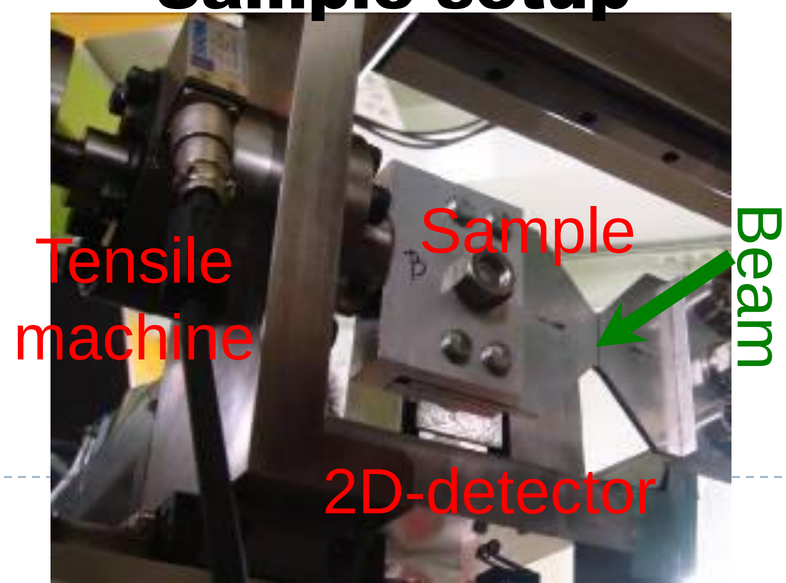
Photos of experimental setup

Sample(5mm thick α-Fe with notches)



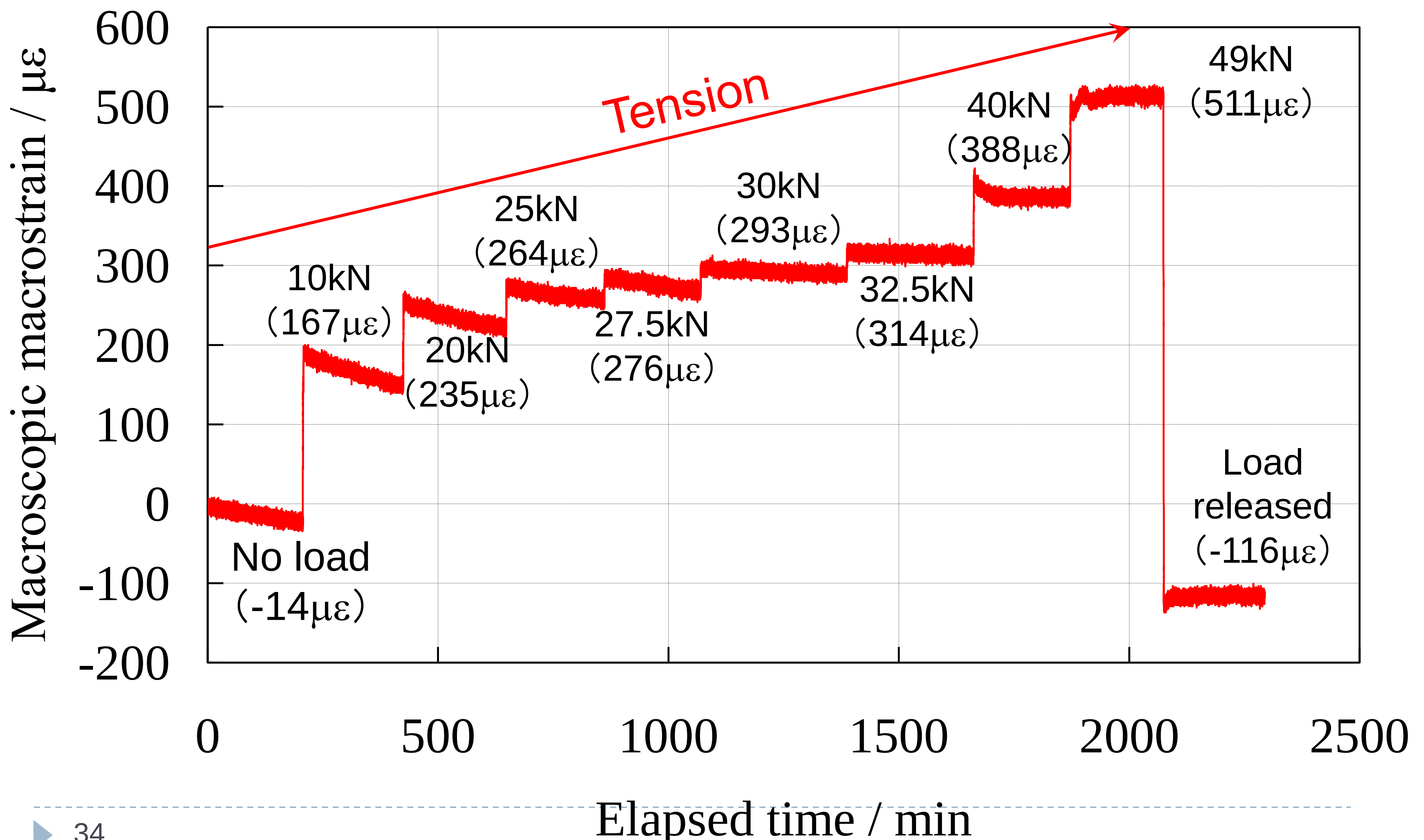
Tensile test machine

Sample setup



Slit(B_4C)

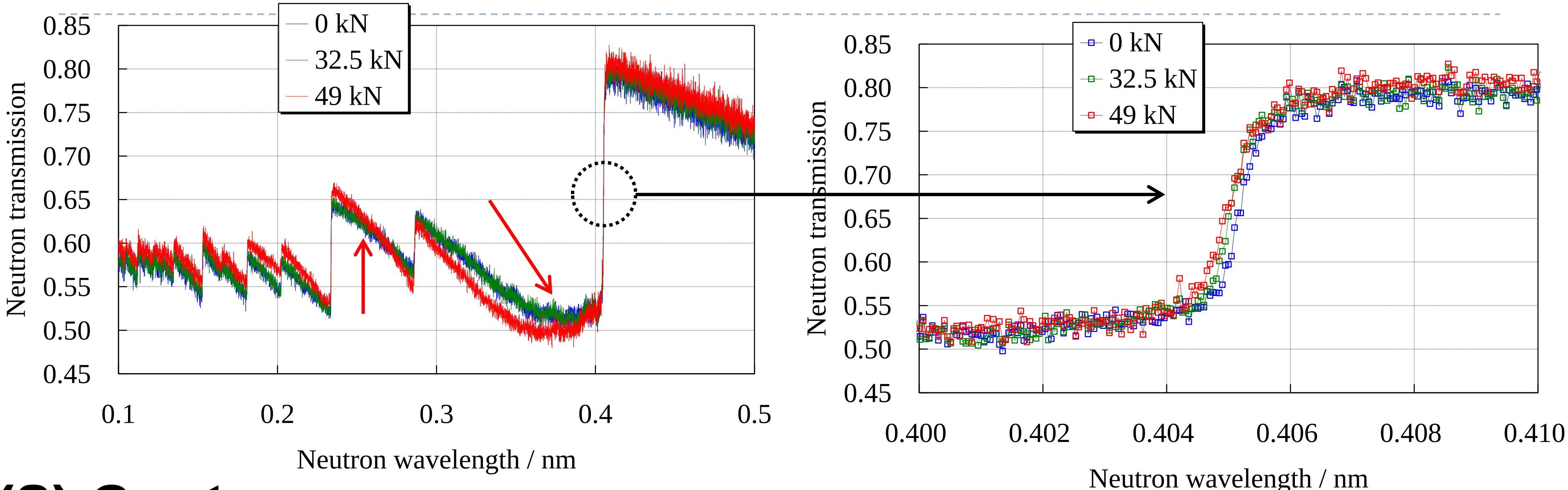
Macroscopic strain measured by a strain gauge



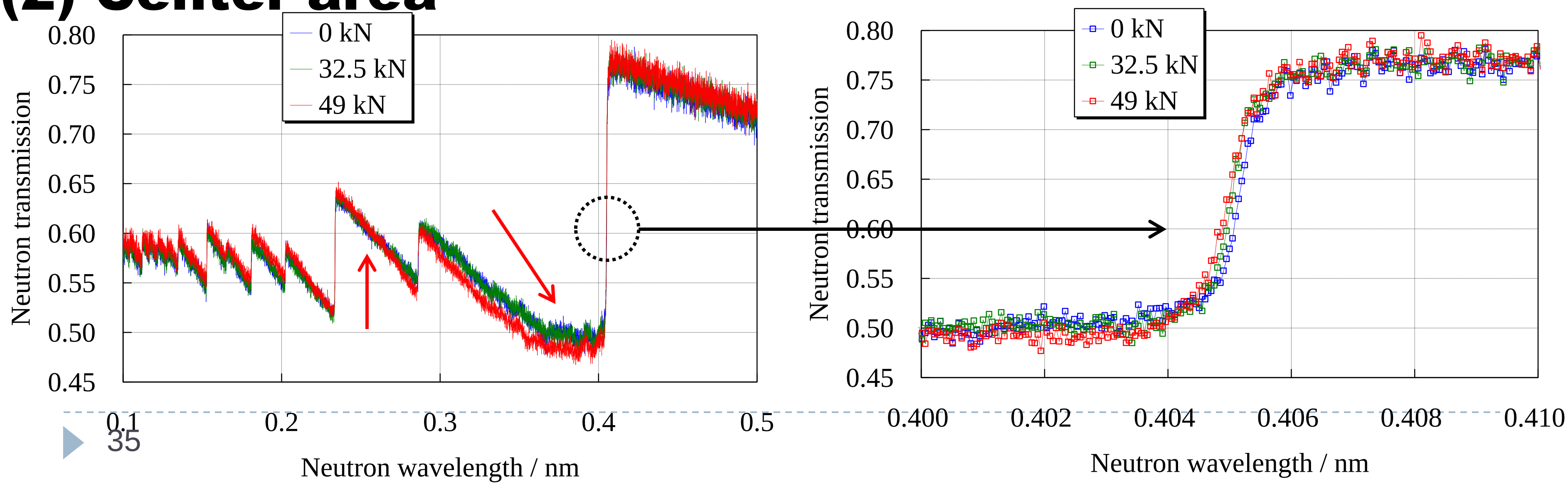
Change of Bragg edge shapes at three loads

(1) Near notch

(Expanded)

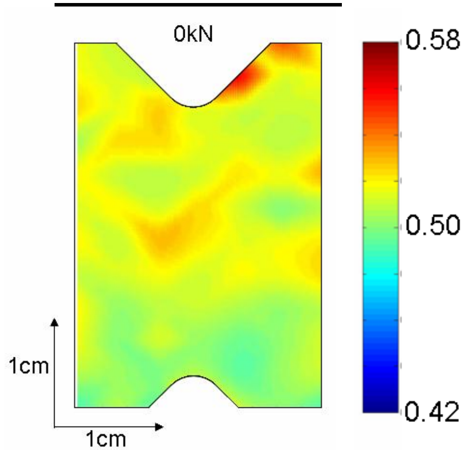


(2) Center area

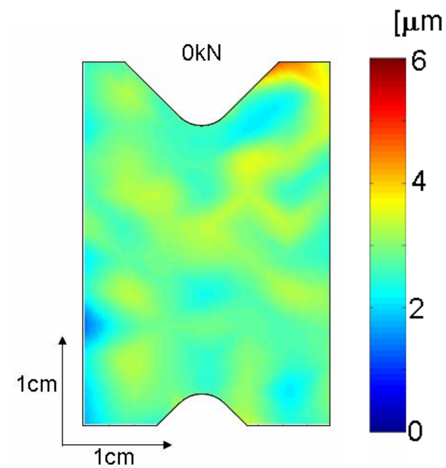


0 kN (before tensile test)

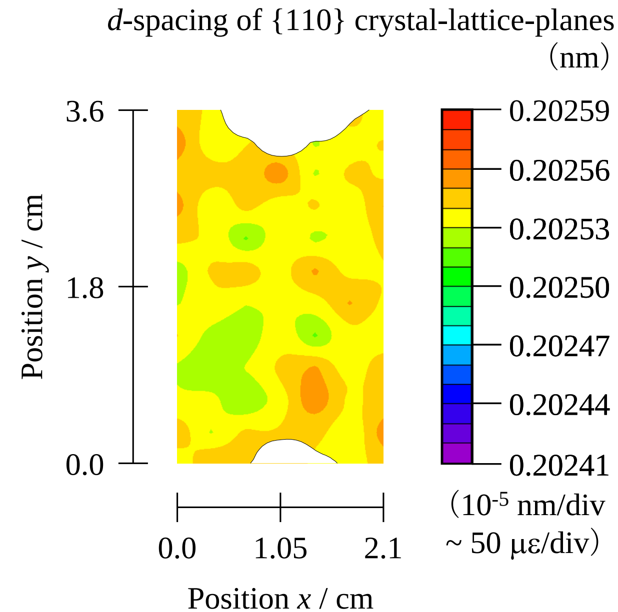
<u>March-Dollase coefficient</u>



<u>Crystallite size</u>



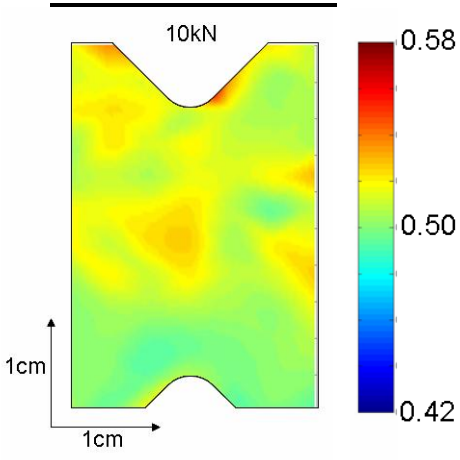
Lattice spacing {110}



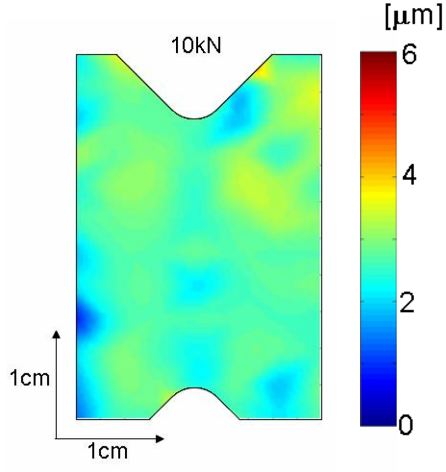
The strain data showed in the following figures were calculated by an assumption of the lattice spacing data obtained here to be d_0.

10 kN

March-Dollase coefficient

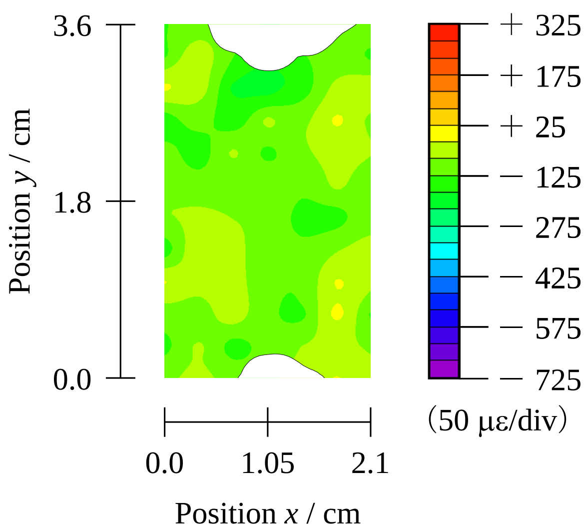


Crystallite size

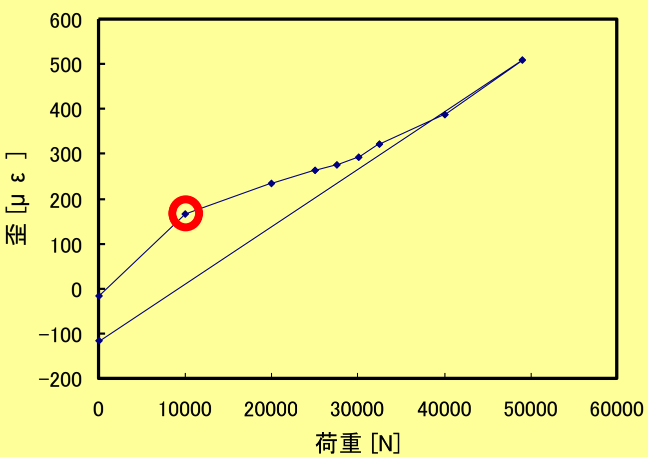


Strain {110}

Macrostrain of {110} crystal-lattice-planes
(με = 10⁻⁴ % = 10⁻⁶)

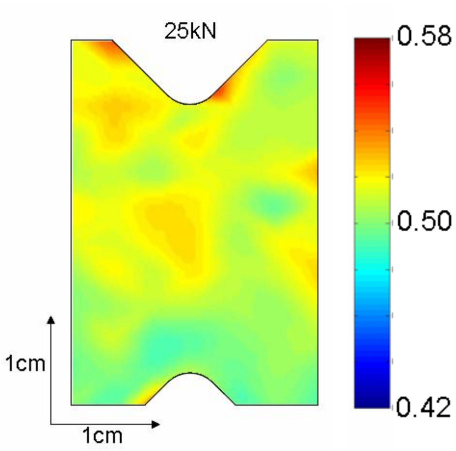


Macroscopic strain obtained by a strain gauge

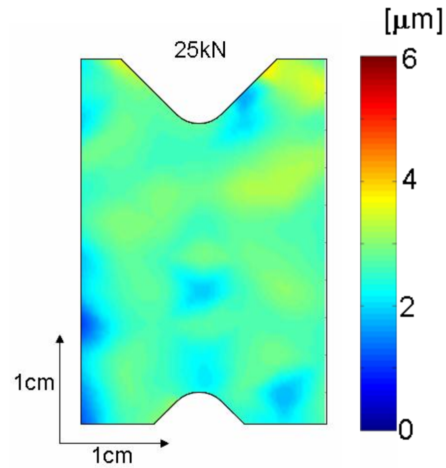


25 kN

March-Dollase coefficient

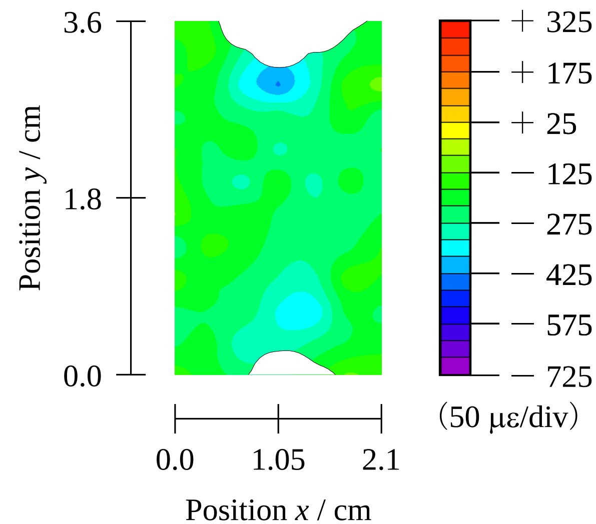


Crystallite size

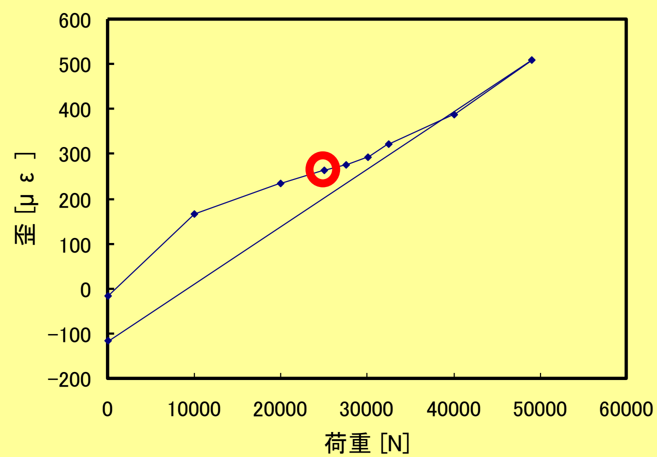


Strain {110}

Macrostrain of {110} crystal-lattice-planes
($\mu\varepsilon = 10^{-4}\ \% = 10^{-6}$)

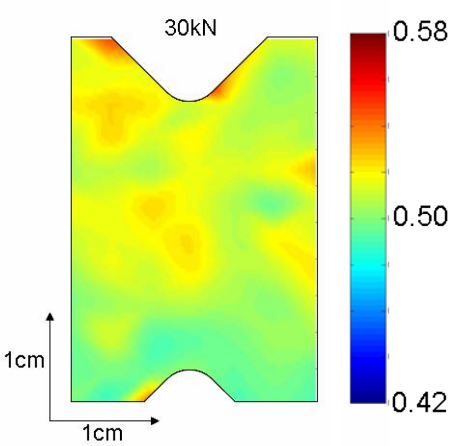


Macroscopic strain obtained by a strain gauge

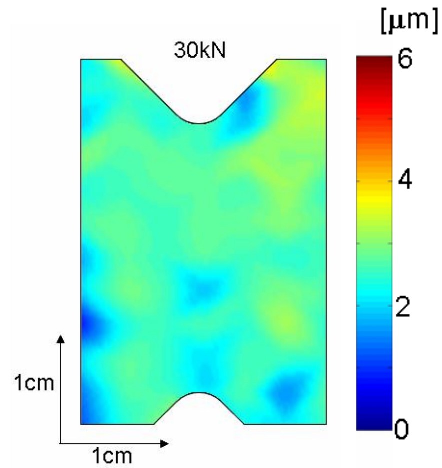


30 kN

March-Dollase coefficient

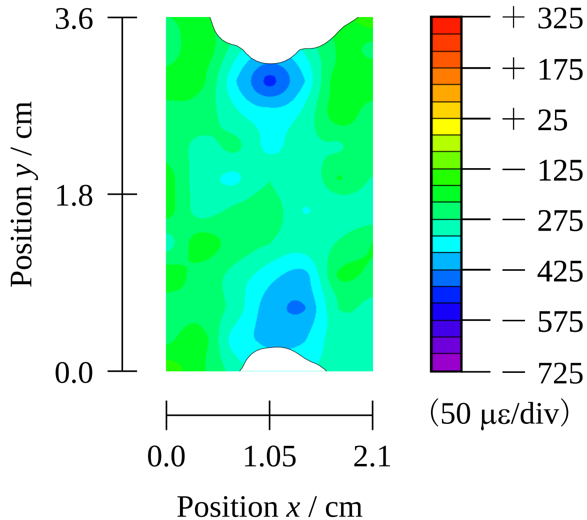


Crystallite size

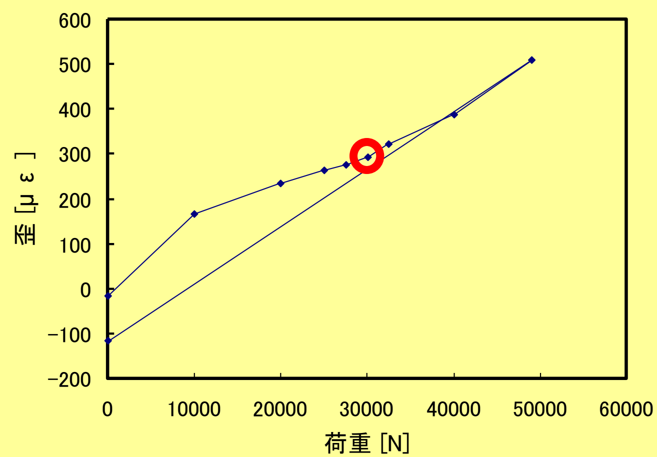


Strain {110}

Macrostrain of {110} crystal-lattice-planes
(με = 10^-4 % = 10^-6)

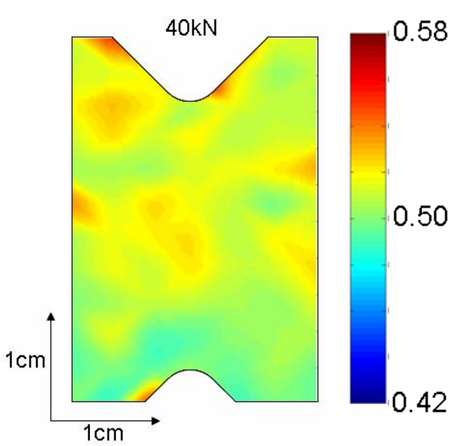


Macroscopic strain obtained by a strain gauge

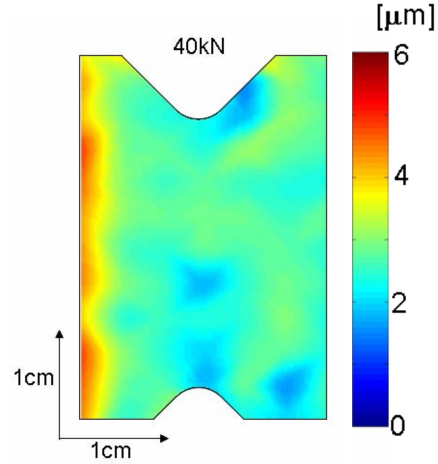


40 kN

March-Dollase coefficient

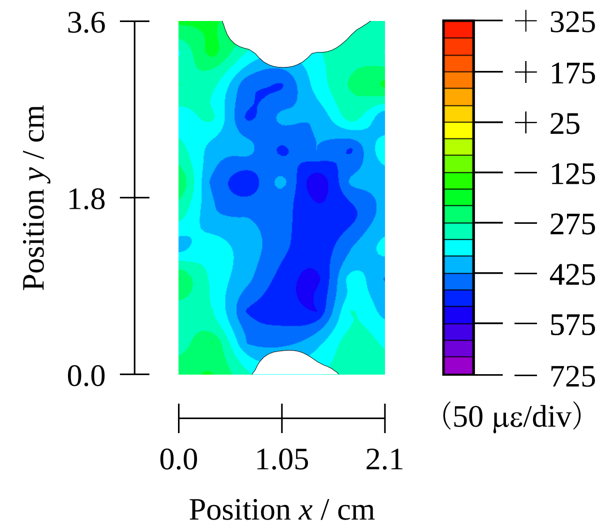


Crystallite size

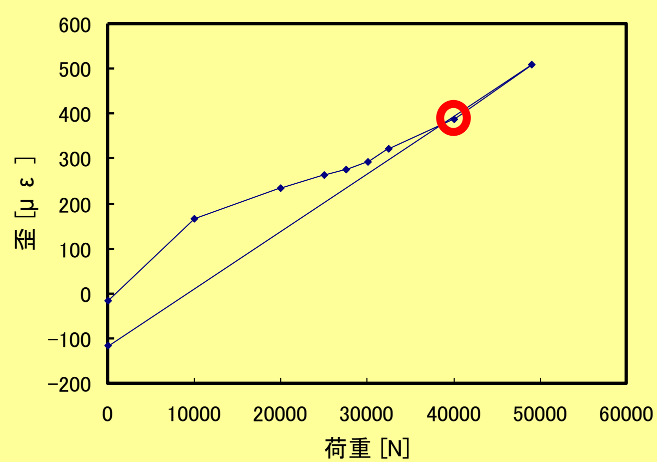


Strain {110}

Macrostrain of {110} crystal-lattice-planes
(με = 10^{-4} % = 10^{-6})

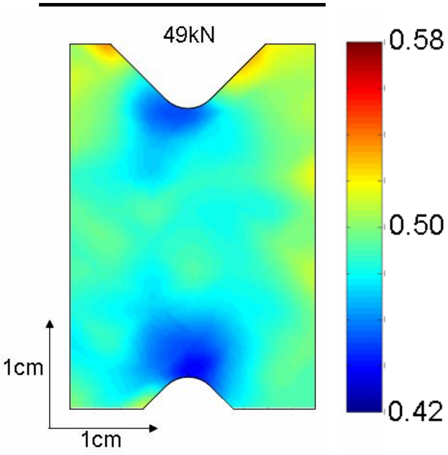


Macroscopic strain obtained by a strain gauge

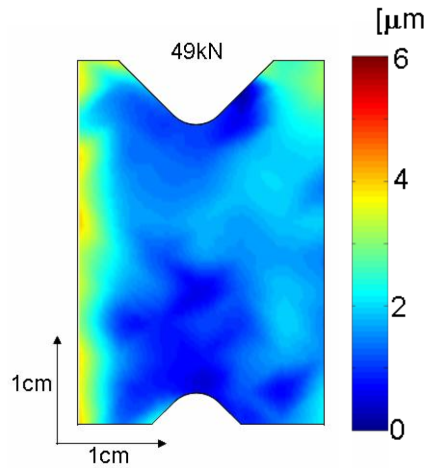


49 kN

March-Dollase coefficient

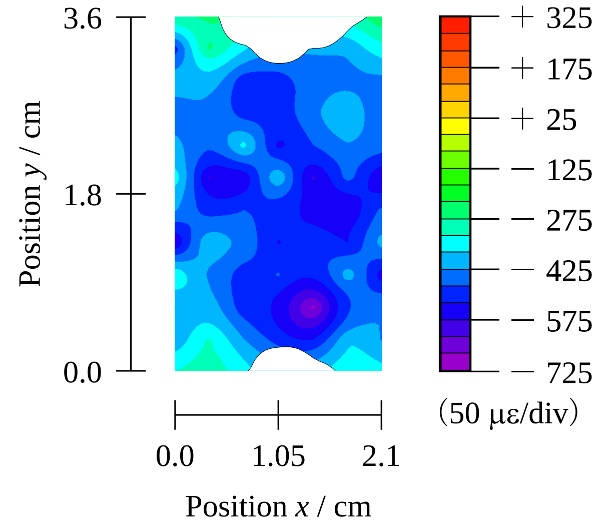


Crystallite size



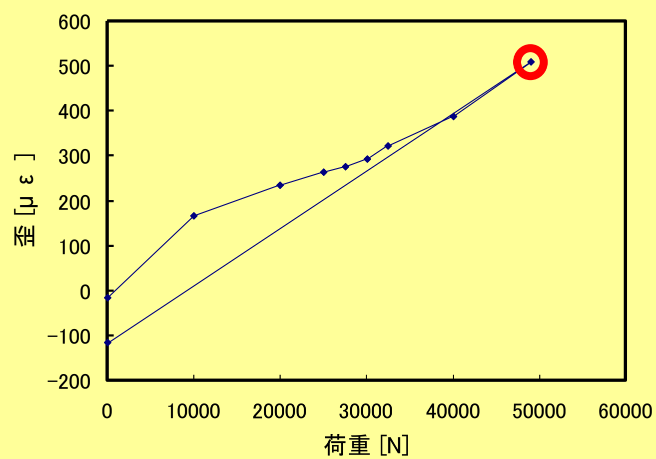
Strain {110}

Macrostrain of {110} crystal-lattice-planes
(με = 10^{-4} % = 10^{-6})



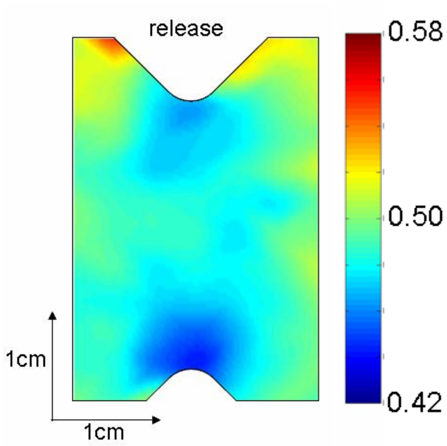
The crystallite size become smaller over the sample at this load.

Macroscopic strain obtained by a strain gauge

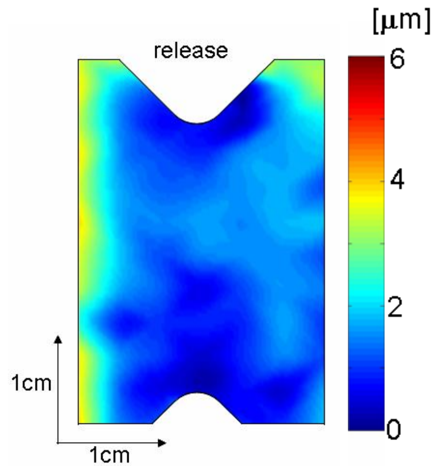


After releasing load

March-Dollase coefficient

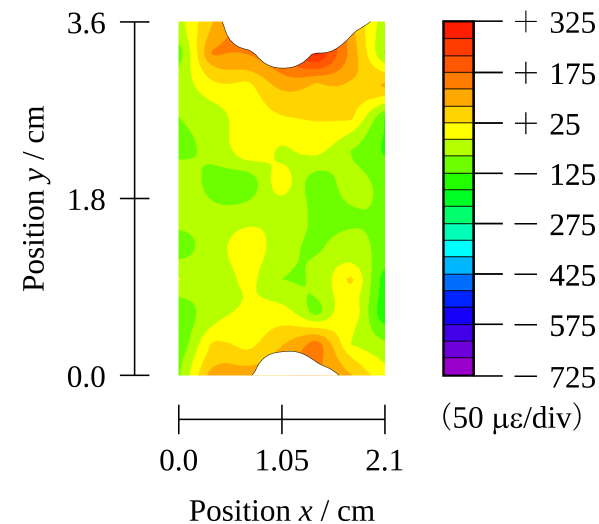


Crystallite size



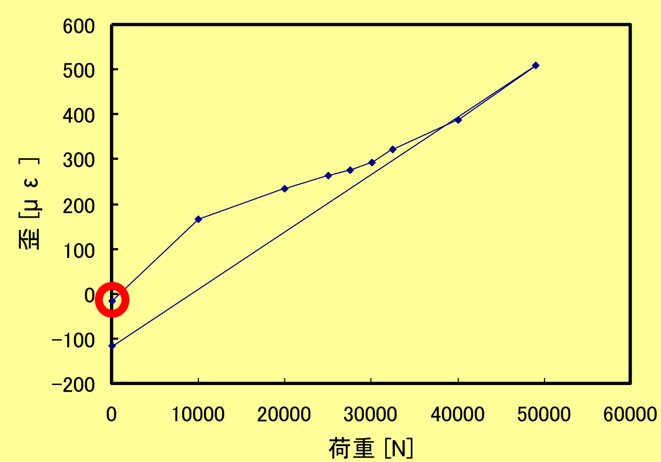
Strain {110}

Macrostrain of {110} crystal-lattice-planes
(με = 10^{-4} % = 10^{-6})



The strain was released and crystallite size was kept small over the sample.

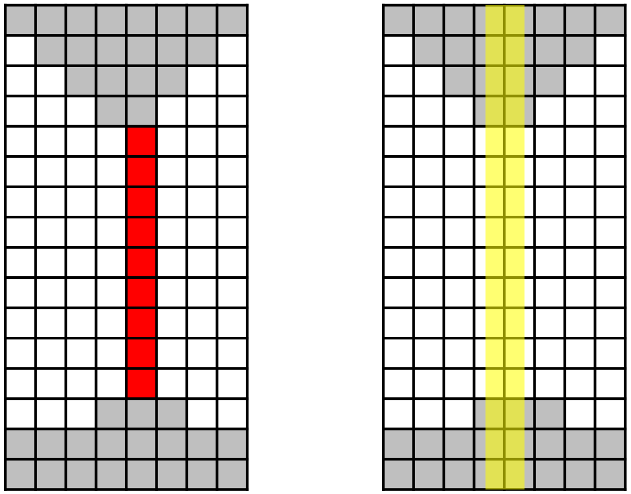
Macroscopic strain obtained by a strain gauge

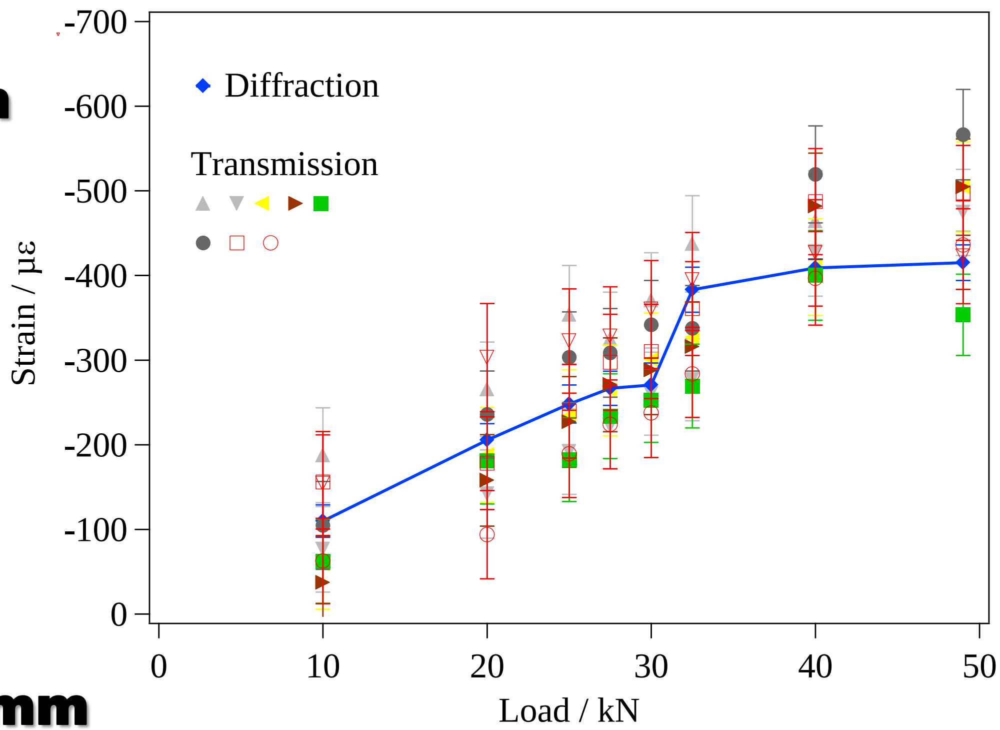


Comparison of compression strain

Measured area

Transmission Diffraction





24mm×3mm 30mm×4mm

The compression strain data obtained by the diffraction sit in the range of the transmission data. The trend is almost the same.

The diffraction data are average value over elastic and plastic deformation areas.

Magnetic field imaging using neutrons

T. Shinohara, K. Sakai, T. Kai, M. Harada, S. Takada, J. Suzuki, F. Maekawa, M. Arai (J-PARC center)

and

Y. Kiyanagi (Hokkaido University)

Principle of magnetic field imaging by neutrons

We detect spin ration in a magnetic field due to precession.

Rotation angle depends on the neutron speed (the wavelength) and path integration over the magnetic field.

Chang e of spin orientation in a magnetic field

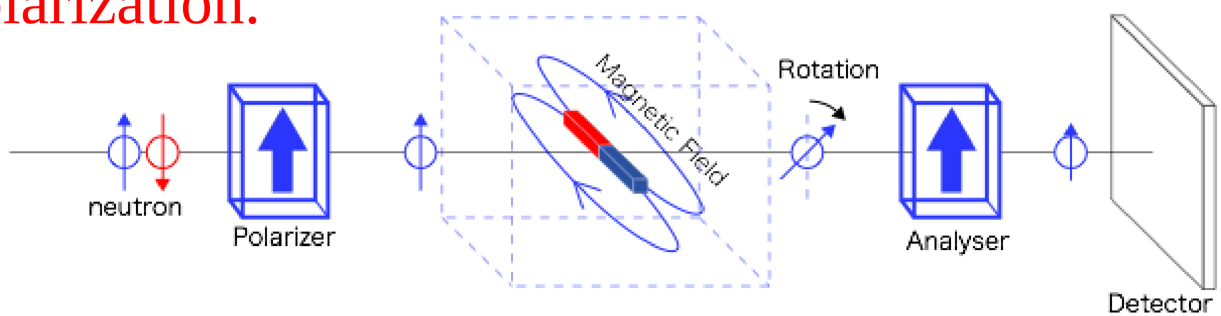
$$\frac{\mathrm{d}}{\mathrm{d}t} S_\alpha(t) = \frac{g\mu_\mathrm{N}}{\hbar} [\mathbf{S}(t) \times \mathbf{B}(t)]_\alpha \,, \quad \alpha = x, y, z$$

Rotation angle due to precession

$$\varphi = \omega_L t = \frac{\gamma_L}{\nu} \int_{path} B \mathrm{d}s$$

$$\omega_L = \frac{g\mu_\mathrm{N}}{\hbar} B = \gamma_L B$$

Spin rotation corresponds to polarization.



Test experiment for magnetic field imaging using a solenoid coil

Beam line：BL10 at J-PARC

Polarizer/analyzer：Bended magnetic mirror

+ Solid collimator（HMI）

Spin flipper：AFP type

（AC field：173kHz, ~20G）

Sample：solenoid(Oiuter diam. 5mm

Detector：5 inch-RPMT

（Schintilator：ZnS(Ag)/^{6}LiF 0.25mm）

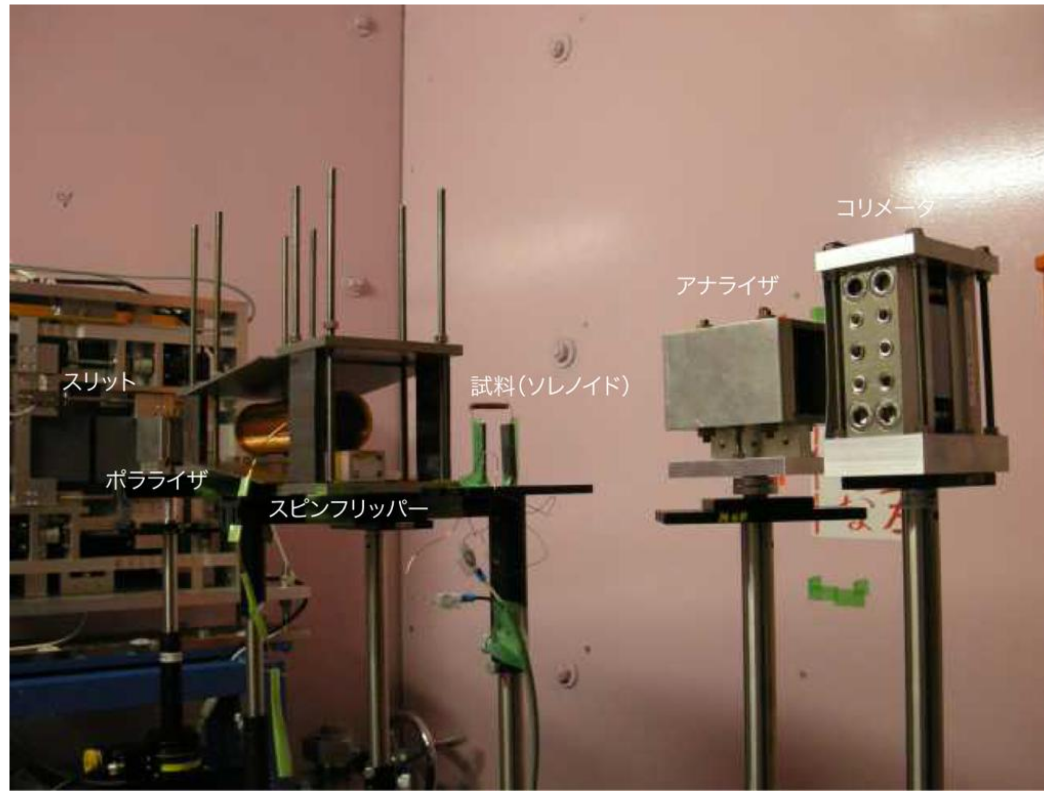
（Δx,Δy=0.5mm, Δt=0.5msec）

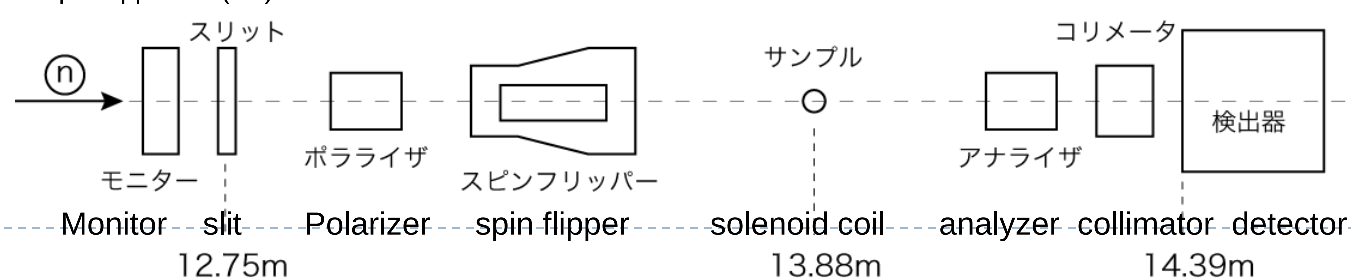
Beam size：20mm(H) x 10mm(W)

Measuring time：~ 30 min.（50000pulses)

Spin flipper OFF(N-) and

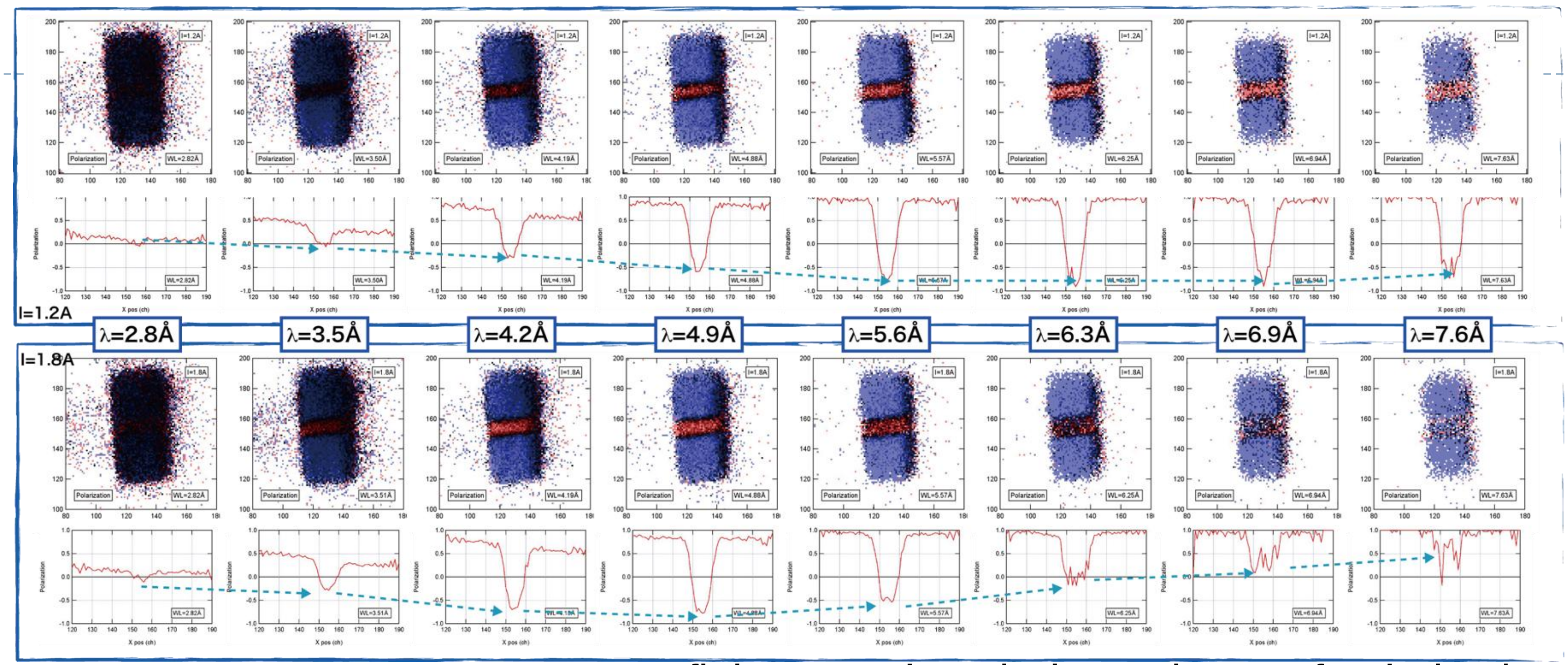
Spin flipper ON(N+)





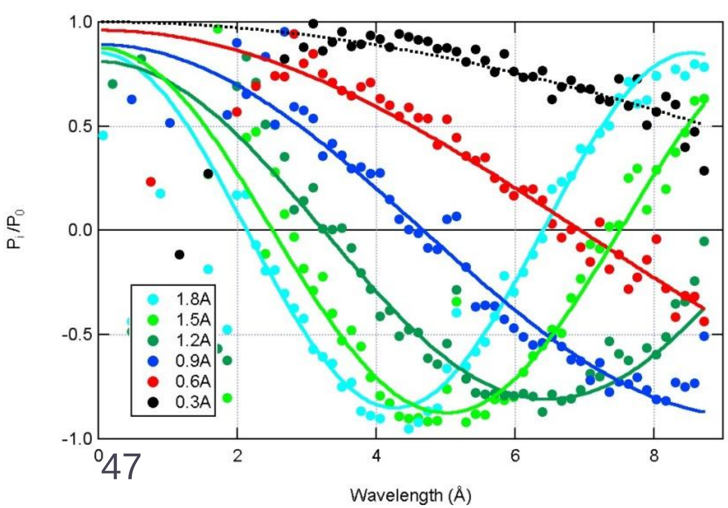
Result of polarization image

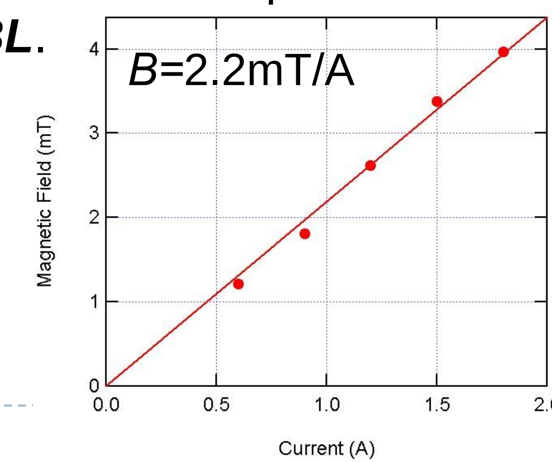
Wavelength dependence



By fitting wavelength dependence of polarization we can get information on ***BL***.

$$P_i/P_0 \propto \cos\phi$$
$$\phi = \gamma B t = \gamma B \frac{L\lambda}{3956}$$





Summary

1. The pulsed neutron transmission method can give crystallographic information such as crystallite size, preferred orientation, strain, phase transition, magnetic field and so on for a thick (bulk) sample over the wide area.

2. The data analysis code ‘RITS’ was developed, and it has been proved to be very useful for deducing quantitative data.

3. The improvement in the experimental method and in the analysis code is required to give more precise data and expand the application field