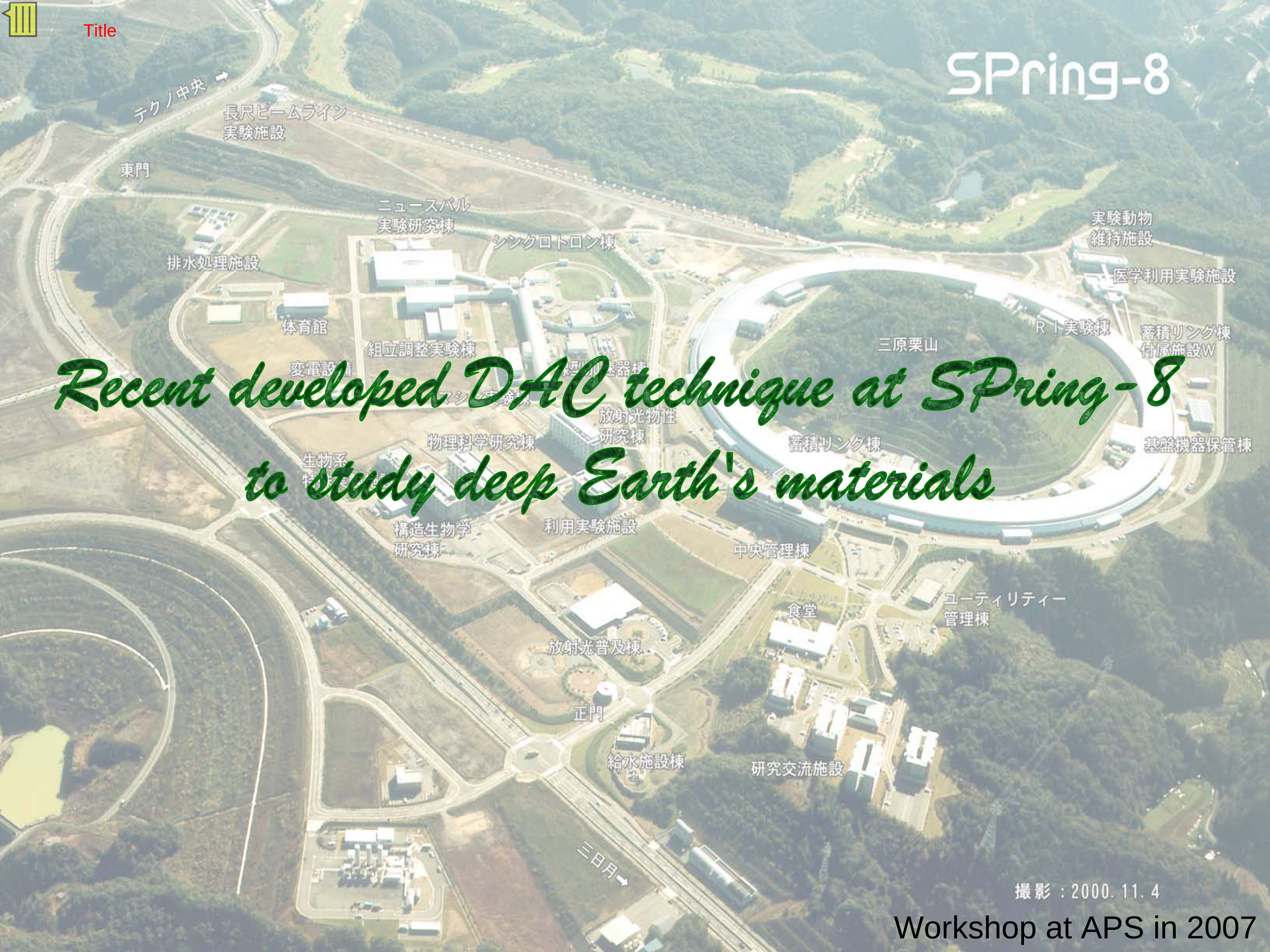
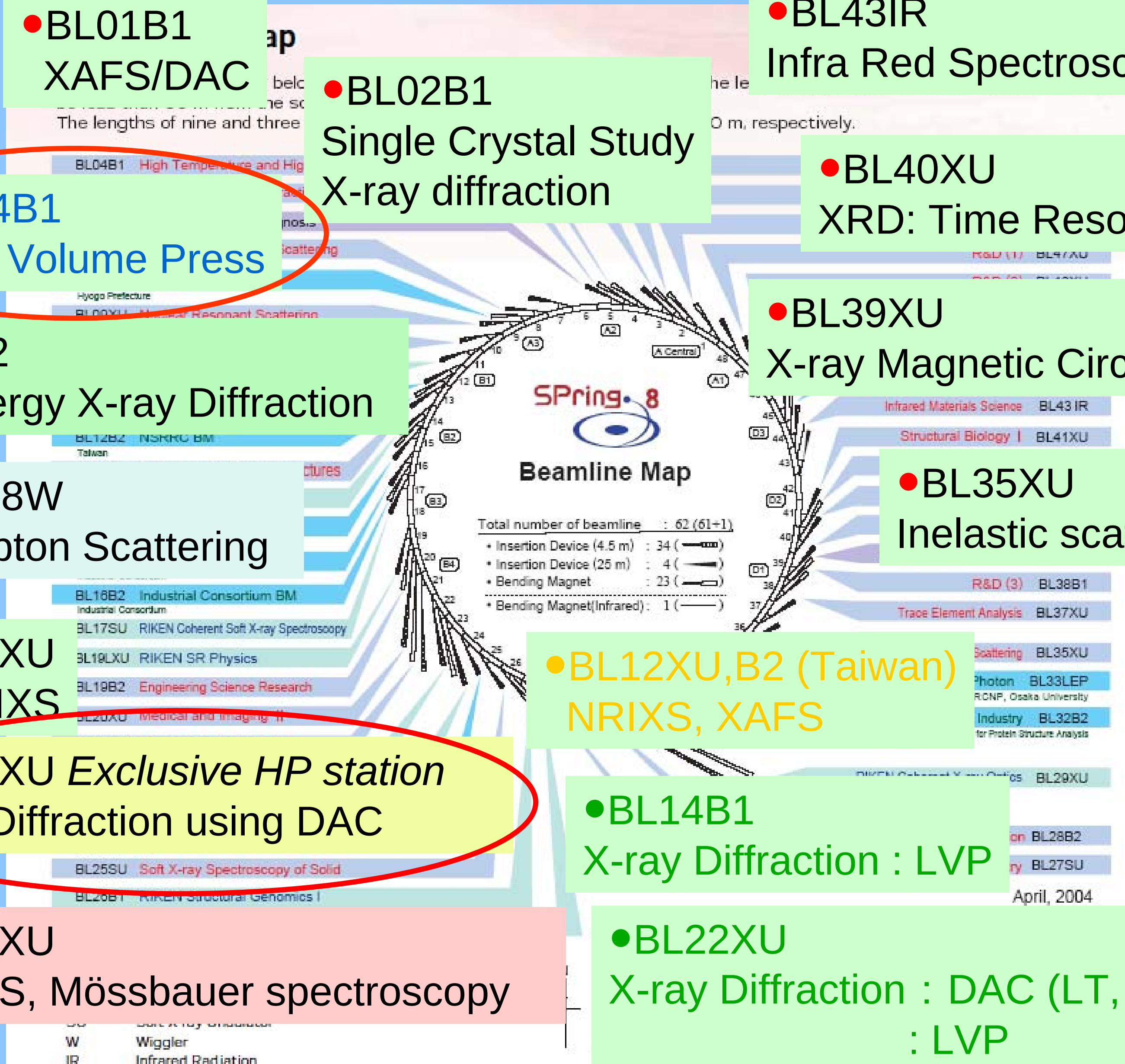
Recent developed DAC technique at SPring-8 to study deep Earth's materials



Workshop at APS in 2007

High Pressure Research at SPring-8



- BL01B1 XAFS/DAC
- BL02B1 Single Crystal Study X-ray diffraction
- BL43IR Infra Red Spectroscopy
- BL40XU XRD: Time Resolved Exp.
- BL04B1 Large Volume Press
- BL04B2 High Energy X-ray Diffraction
- BL39XU X-ray Magnetic Circular Dichroism
- BL08W Compton Scattering
- BL35XU Inelastic scattering
- BL09XU NRIXS
- BL12XU,B2 (Taiwan) NRIXS, XAFS
- BL10XU *Exclusive HP station* X-ray Diffraction using DAC
- BL14B1 X-ray Diffraction : LVP
- BL11XU NRIXS, Mössbauer spectroscopy
- BL22XU X-ray Diffraction : DAC (LT, Single Crystal.) : LVP

High pressure experiments at SPring-8.

Large volume (multi-anvil) press : BL04B1, BL14B1, BL22XU

ED-XRD, radiographic imaging, ultra-sonic technique, etc.

Diamond anvil cell : BL10XU and others

AD-XRD with laser heating, a lot of SR technique

Keys for studying the deep Earth's materials

⇨ ~ experiments under multi-megabar condition

* increase pressure (temperature) limit

** high flux x-ray beam technique*

Outline

- BL10XU : high pressure x-ray diffraction station

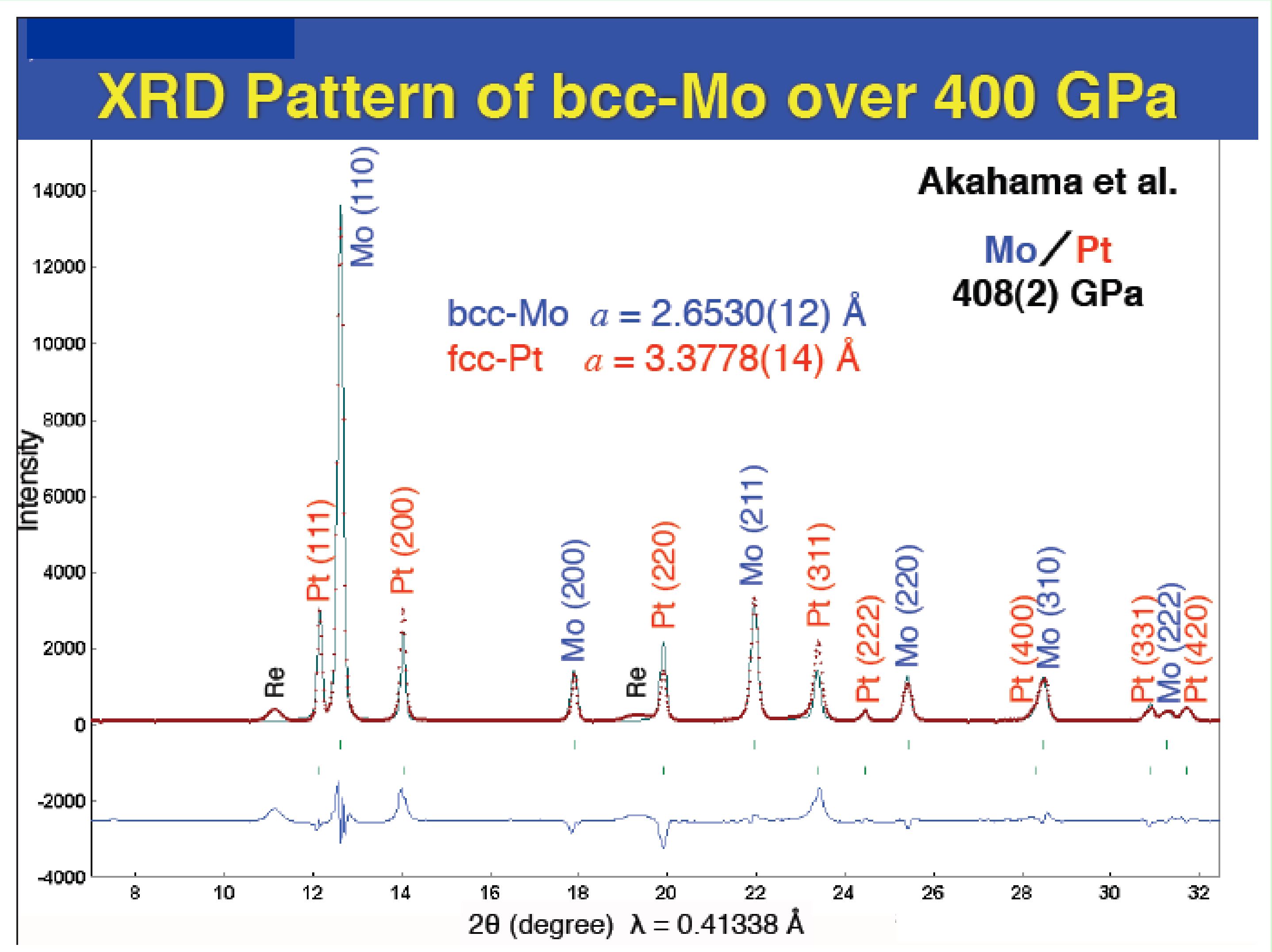
(1) X-ray focusing optics : XRD under multi-megabar

(2) simultaneous measurement system of Brillouin spectroscopy and XRD with LH.

- New attractive measurement technique :

Energy-domain synchrotron radiation Mössbauer spectroscopy using high-flux neV resolution x-ray beam.

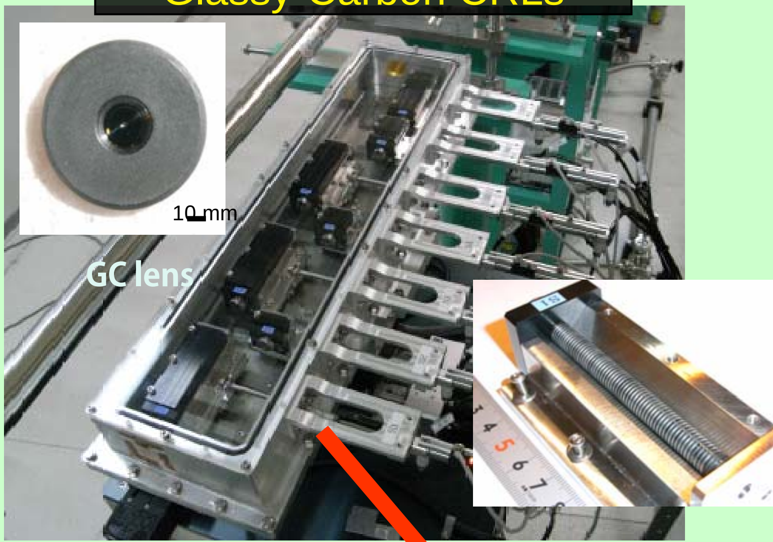
XRD under highest pressure at BL10XU
Mo at 0.4 TPa : X-ray focusing optics



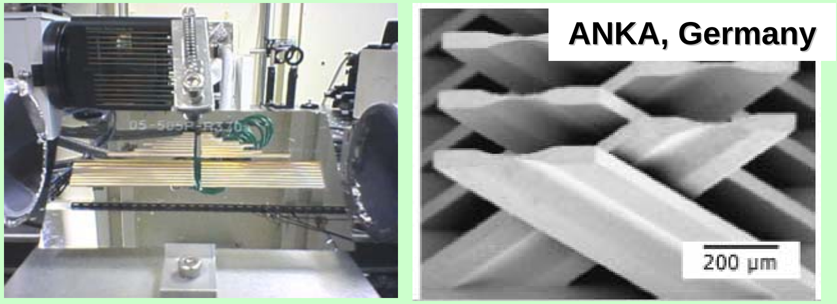
Focusing optics by using double lens system

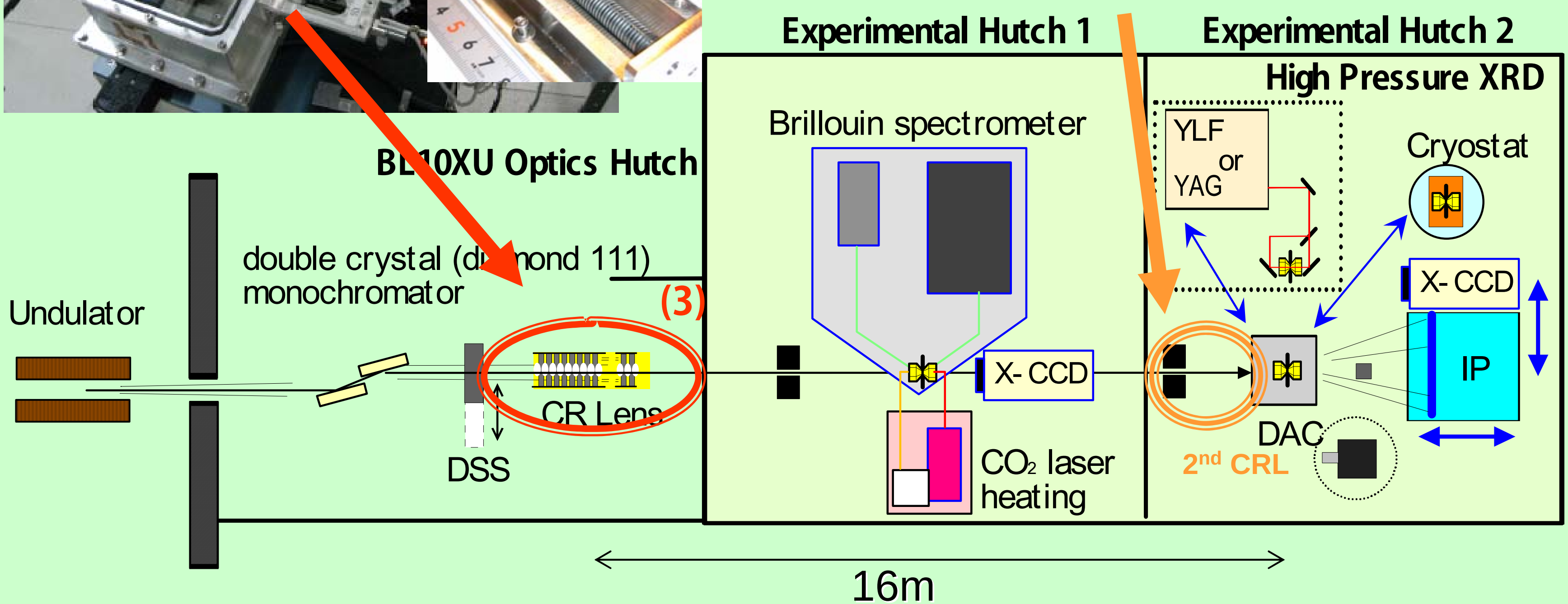
Up-stream focusing lens : large aperture (beam condenser) & long focal distance

Glassy-Carbon CRLs

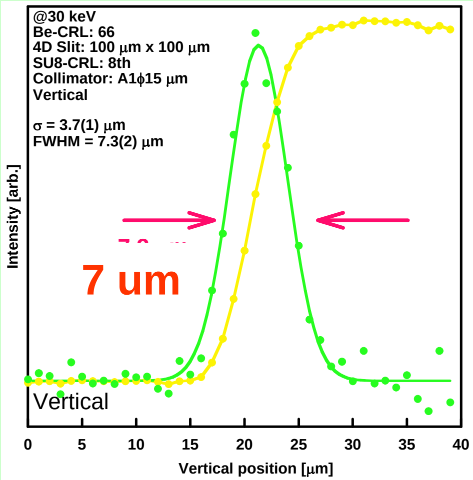


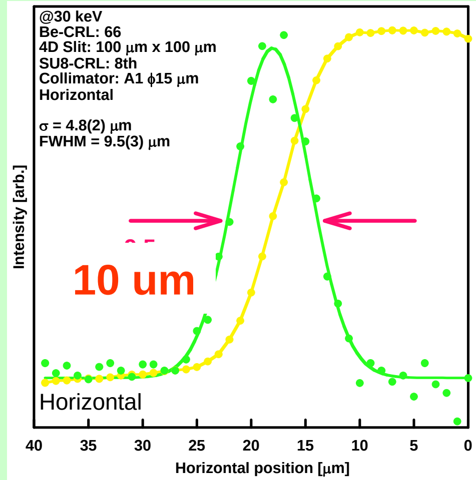
2nd lens : micro-beam & high flux density



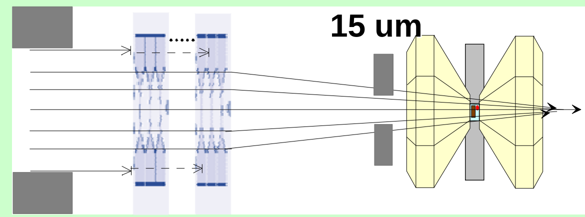


Focused x-ray beam profiles

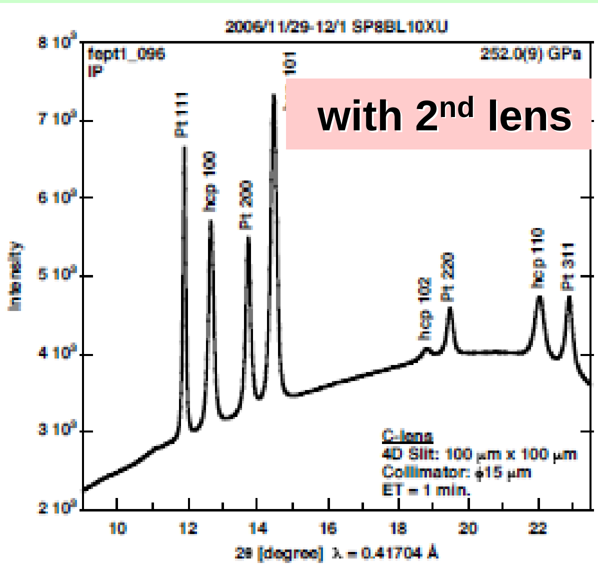


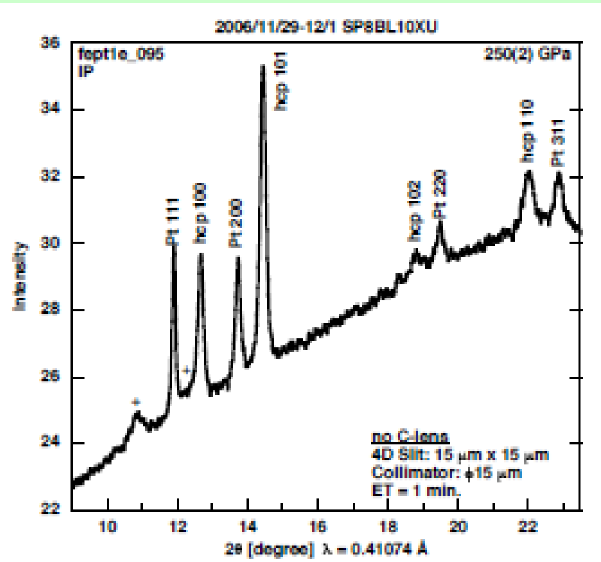


Beam profile : FWHM
~ 7 um (vertical)
~ 10 um (horizontal)
At sample (DAC) position
(knife edge method)



XRD under 250 GPa





Example: Fe (250 GPa)

Effective intensity again

500 times (as the same exposure time)

X-ray divergence (angler resolution)

≒0.01deg. (100μm/55cm, 0.2mrad)

(the case of first lens only)

: 0.003deg. (0.5mm/11m)

for multi-megabar high pressure (and multi-thousand Kelvin) experiments with very good statistic and rapid XRD measurement

High pressure and high temperature *in-situ* Brillouin spectroscopy using infrared laser heating combined with XRD at SPring-8

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Japan Synchrotron Radiation Institute

Nagayoshi Sata

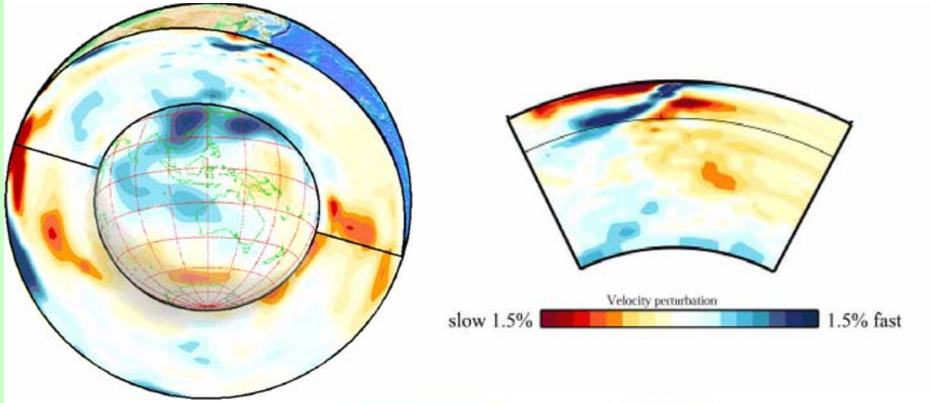
IFREE/AMSTEC

Kei Hirose

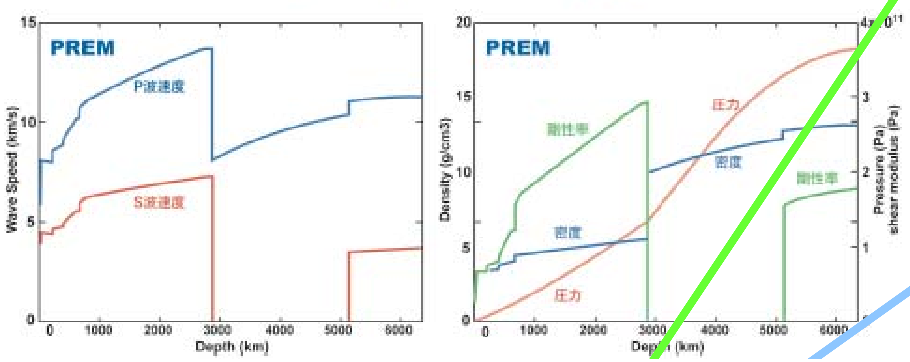
Tokyo Institute of Technology

Brillouin scattering & X-ray diffraction simultaneously measurement

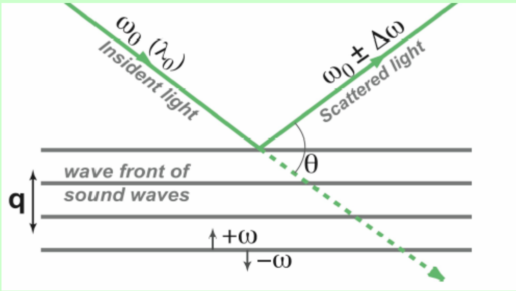
existence of the sound velocity distribution



$$V_p = \sqrt{\frac{K+4G/3}{\rho}} \qquad V_s = \sqrt{\frac{G}{\rho}}$$



elastic properties of minerals under Earth's interiors condition



sound velocity

$V_i = \Delta\omega\lambda/2\sin(\theta/2)$,

i = s (transverse), p (longitudinal)

(in a symmetric scattering geometry)

shear modulus

$G = \rho V_s^2$,

ρ (density from XRD)

adiabatic bulk modulus

$K_s = \rho V_p^2 - 4/3G$

In order to interpret seismic observation in Earth'interior and global seismological models.

New combined system

Performance, Sample/measurement condition

Specimen : **multi crystal (powder, only transparent sample)**

Pressure range : ***~ 150GPa***, **DAC**

Temperature : ***~ 3500K***, **Laser Heating**

Sound velocities measurement : **Brillouin scattering spectroscopy**

Sample density : **x-ray diffraction** (with pressure standard)

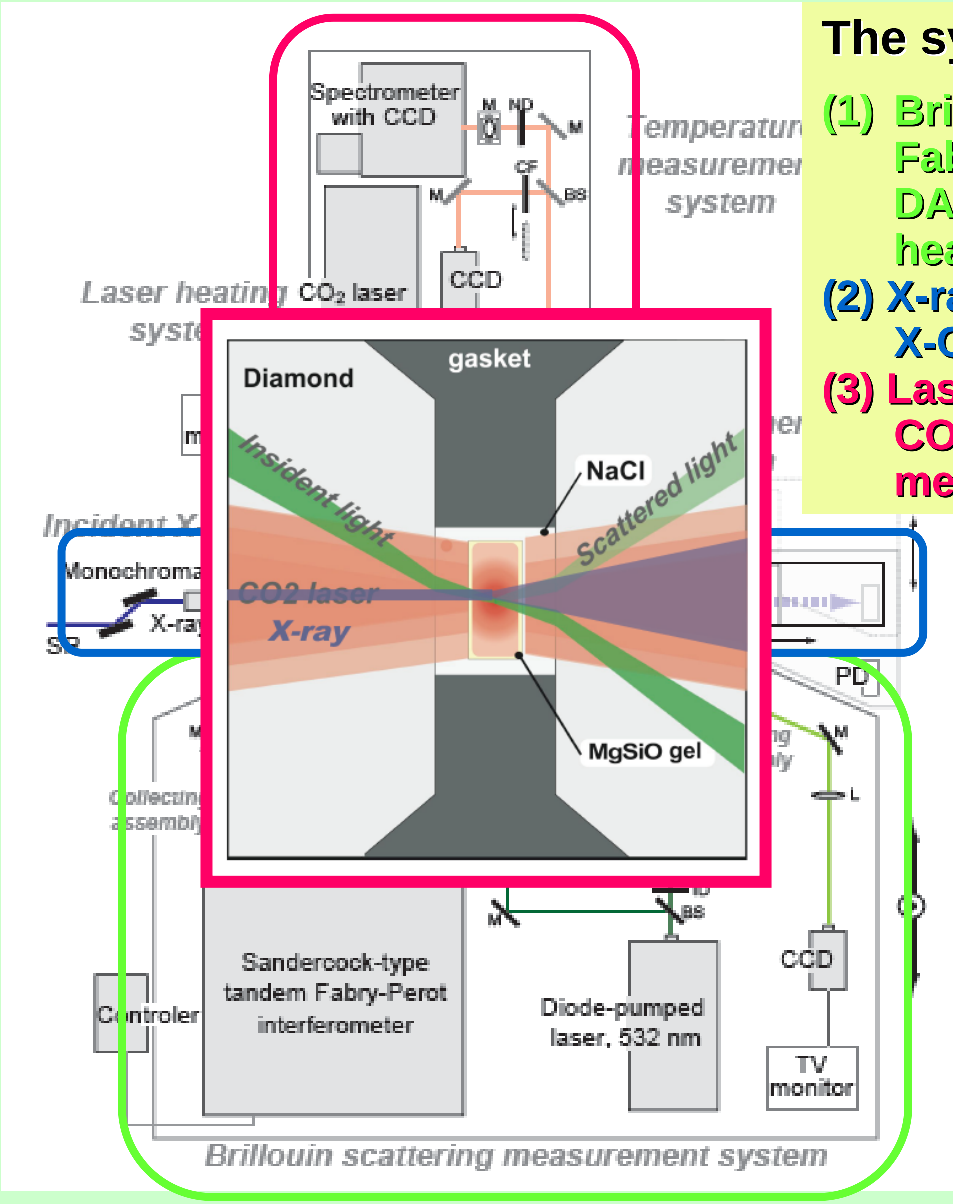
System components

Brillouin scattering : **Fabry-Perot interferometer, symmetric geometry**

Laser Heating : **CO_2 laser** (sample's transparency. Temperature is measured from spectroradiometric method)

X-ray diffraction : **BL10XU** x-ray (50keV) & **x-ray CCD** (lattice parameters, pressure measurement, sample phase monitor)

Combined system at BL10XU/SPring-8

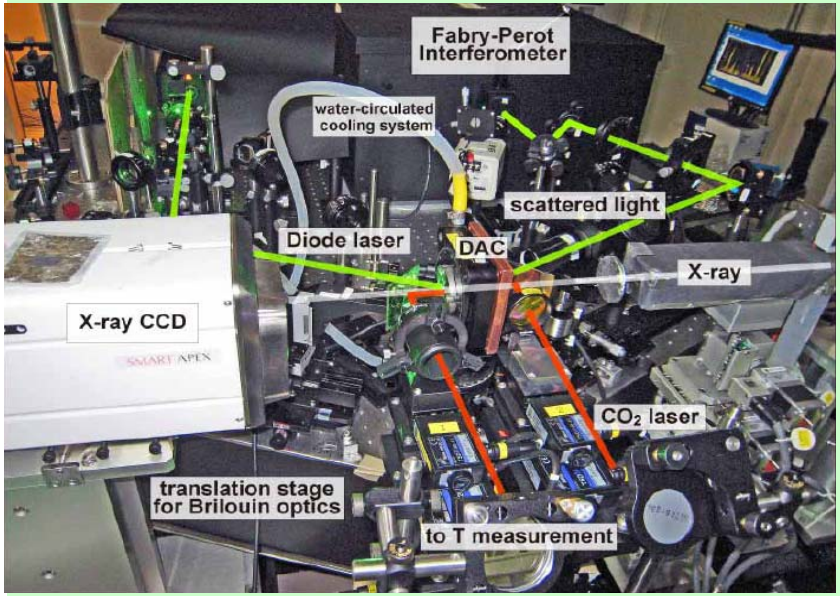


The system consists of three components,

(1) Brillouin specrtometer
Fabre-Perro interferometer
DAC stage (XYZ)
heavy-duty linear translation stages

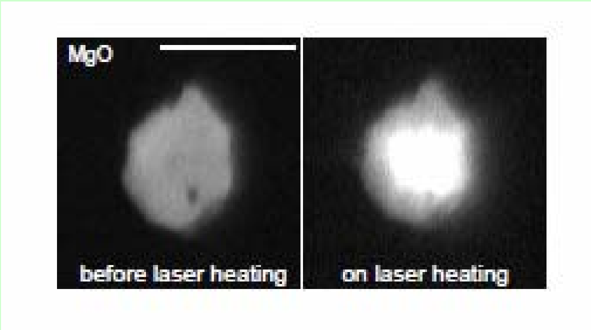
(2) X-ray diffraction system
X-CCD, Focused x-ray beam(50keV)

(3) Laser heating optics
CO_2 laser, Spectroradiometric temperature measurement system

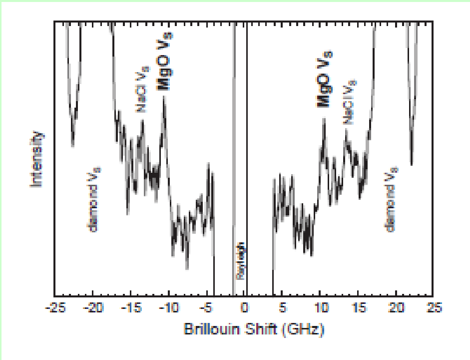


Preliminary Results

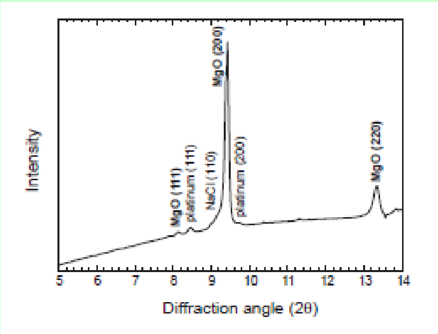
Experiment (1)



Microscopic views of polycrystalline MgO before laser heating (42 GPa) and on laser heating (49 GPa, ~2300 K).

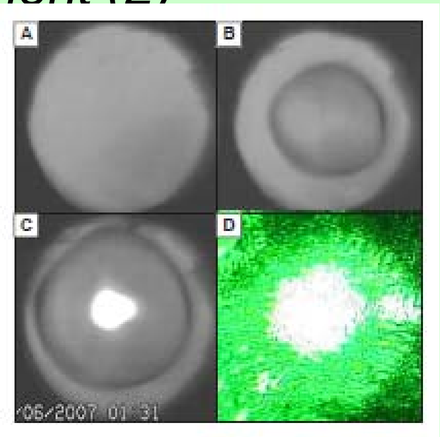


Brillouin scattering spectra of MgO

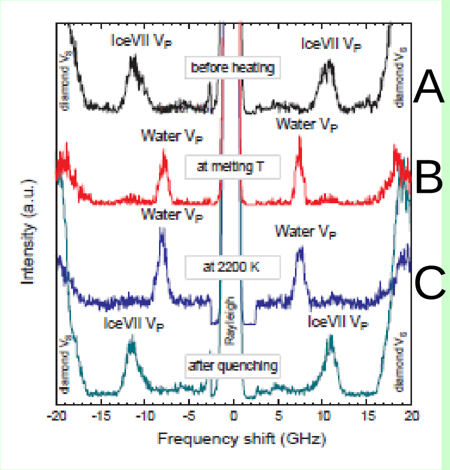


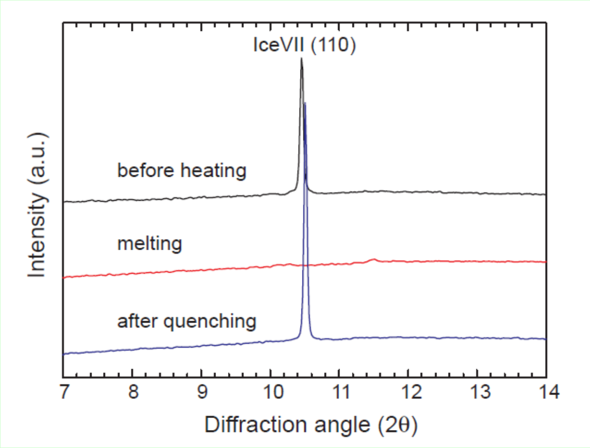
XRD of MgO

Experiment (2)



Microscopic views of H_2O (a) before heating (6 GPa) and (b) at melting temperature, (c), (d) at 2200 K and during Brillouin measurement.





Brillouin scattering spectra and XRD profiles of H_2O before heating (6 GPa) and at melting temperature, at 2200 K and after quenching.

The resent developed energy-domain synchrotron radiation Mössbauer spectroscopy at SPring-8

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- High pressure applications

Naohisa Hirao, Yasuo Ohishi

Japan Synchrotron Radiation Institute

Energy-domain SR Mössbauer spectroscopy

Behavior of iron in deep Earth materials under high pressure

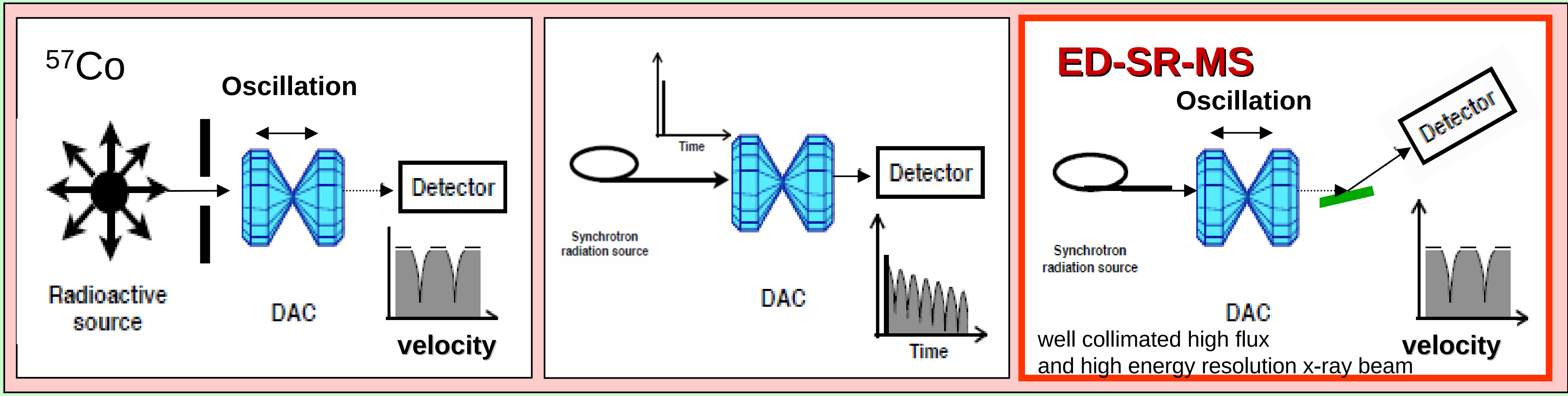
differentiation of the early Earth, subduction and upwelling in the mantle, formation of the Earth's magnetic field

Mössbauer spectroscopy for studying deep Earth's materials

one of powerful methods to study the electronic and magnetic structures of iron and iron containing materials

- **MS from conventional radioactive source**
- **MS from time spectrum by nuclear resonant scattering at SR**
- **Energy-domain synchrotron radiation Mössbauer spectroscopy**

  by collimated SR x-ray and neV high energy resolution technique

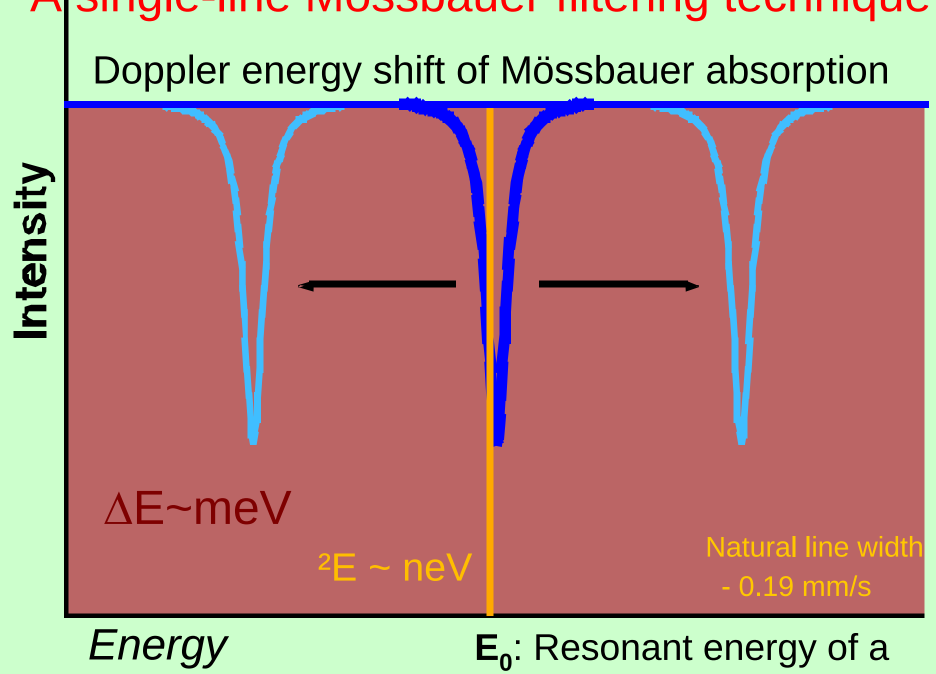


neV resolution X-ray source for Mössbauer spectroscopy

Nuclear resonant filtering of synchrotron radiation by pure nuclear Bragg reflection of $^{57}FeBO_3$ single crystal

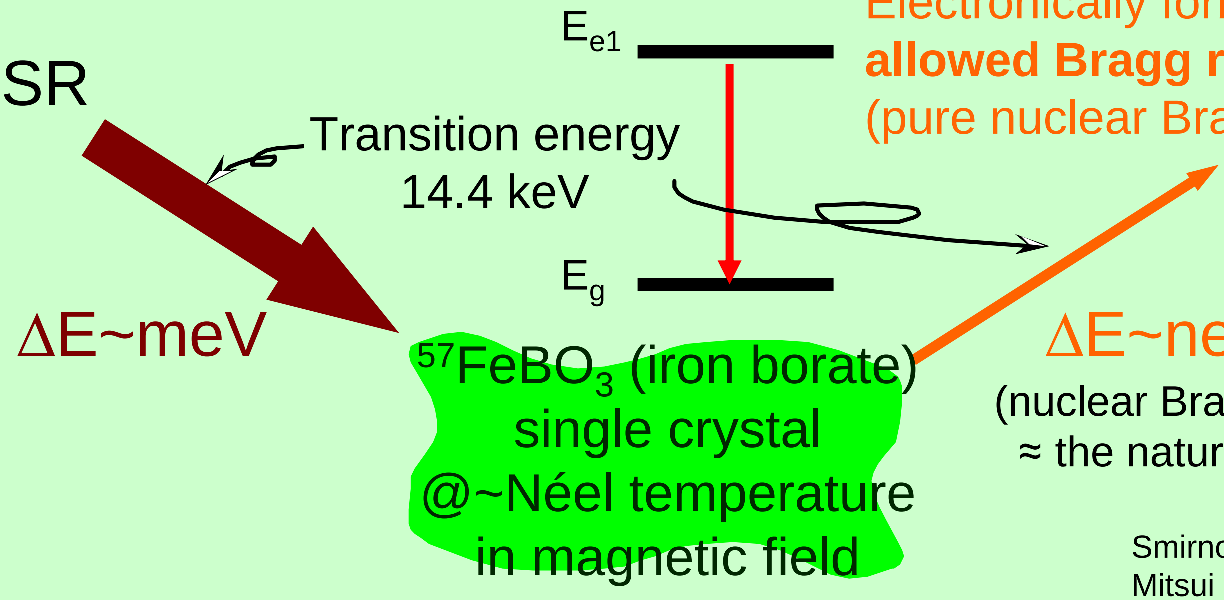
***high collimated and neV energy resolution x-ray beam**

A single-line Mössbauer filtering technique



E$_0$: Resonant energy of a nuclear analyzer crystal

Electronically forbidden and **nuclear allowed Bragg reflection** of $^{57}FeBO_3$ (333) (pure nuclear Bragg reflection)



(nuclear Bragg reflection ≈ the natural width of the nuclear level)

Smirnov et al. (1997) Phys. Rev. B 55, 5811.
Mitsui et al. (2007) Jpn. J. Appl. Phys. 46, L821

Experimental arrangement for energy-domain SR Mössbauer spectroscopy and DAC

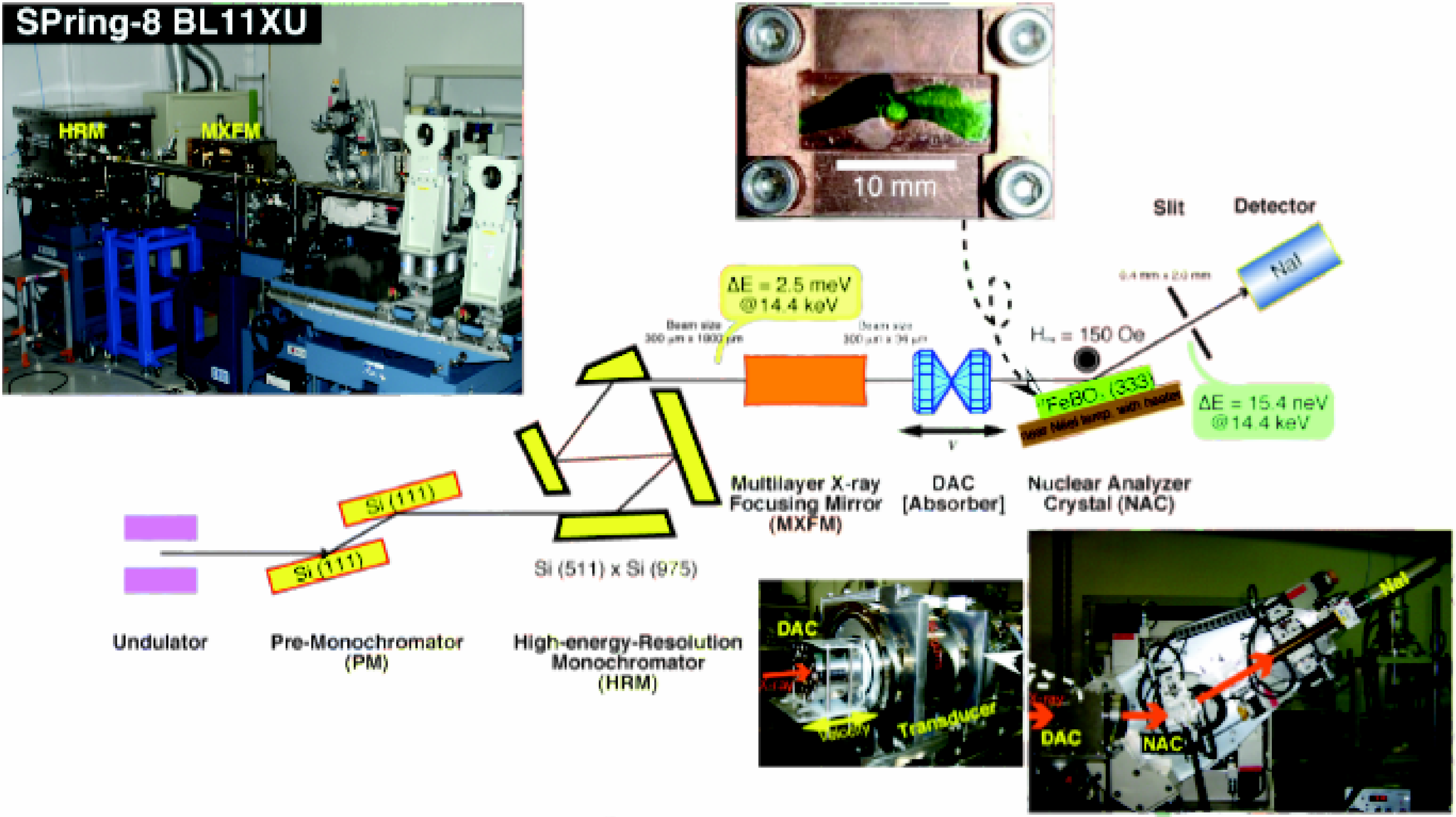


Figure. Experimental setup for the energy-domain SR ^{57}Fe-Mössbauer spectroscopy [Mitsui et al. (2007) Jpn. J. Appl. Phys. 46, L382].

Mössbauer spectrum of Fe_2O_3 under multi-magabar

$\alpha-Fe_2O_3$ (hematite : corundum)

AP(0.1MPa) : paramagnetic NéelTemp. : 955K

HP pha

P ~ 7

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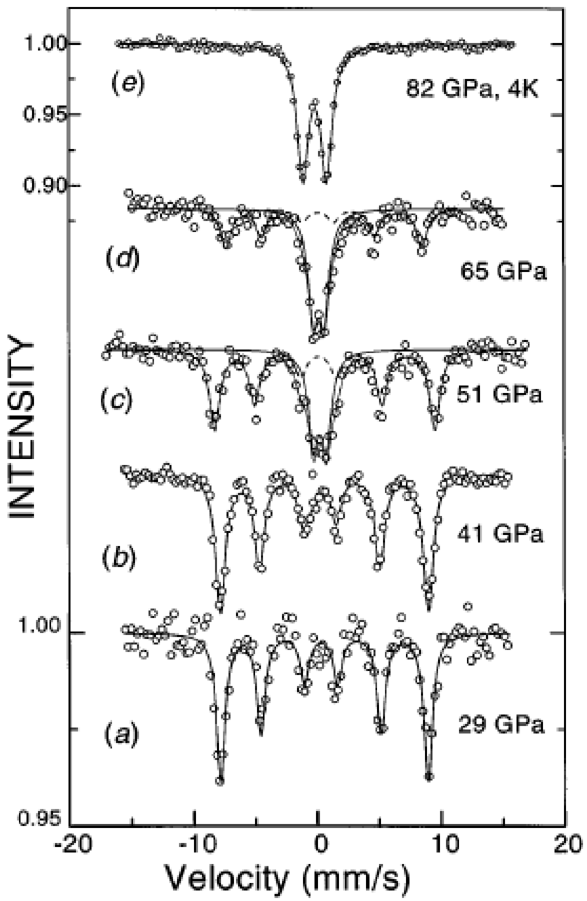
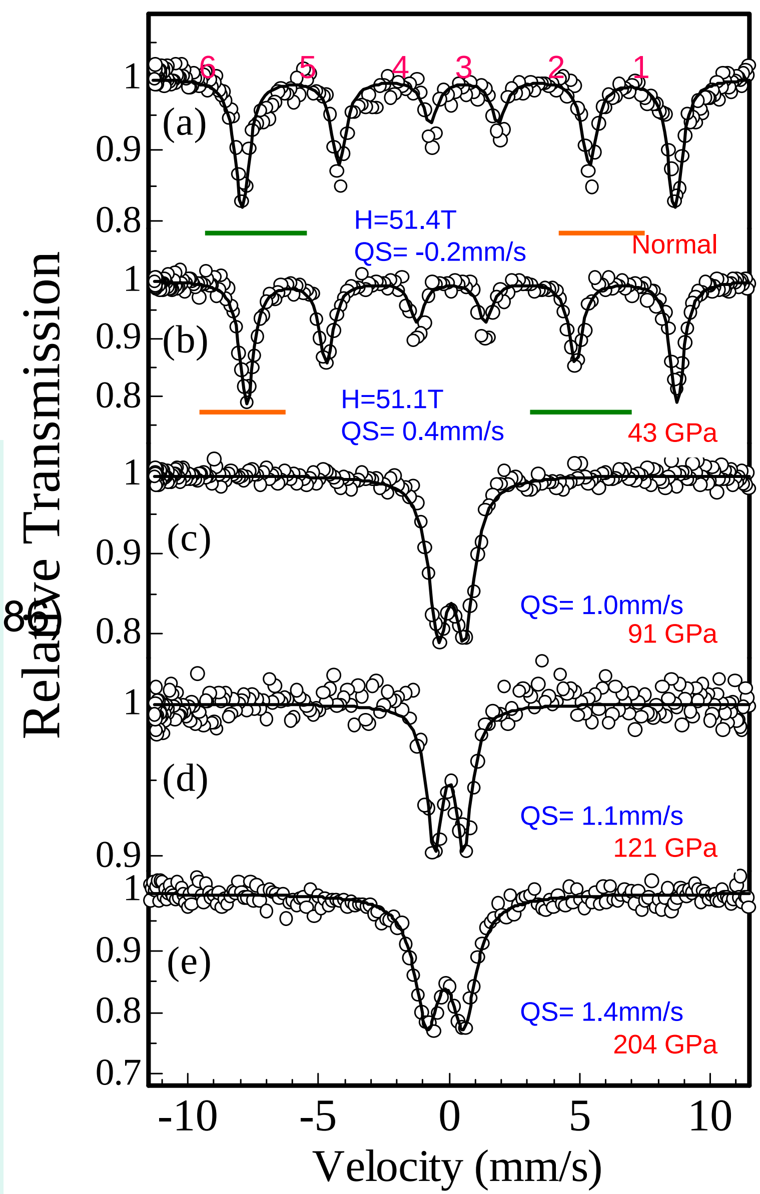


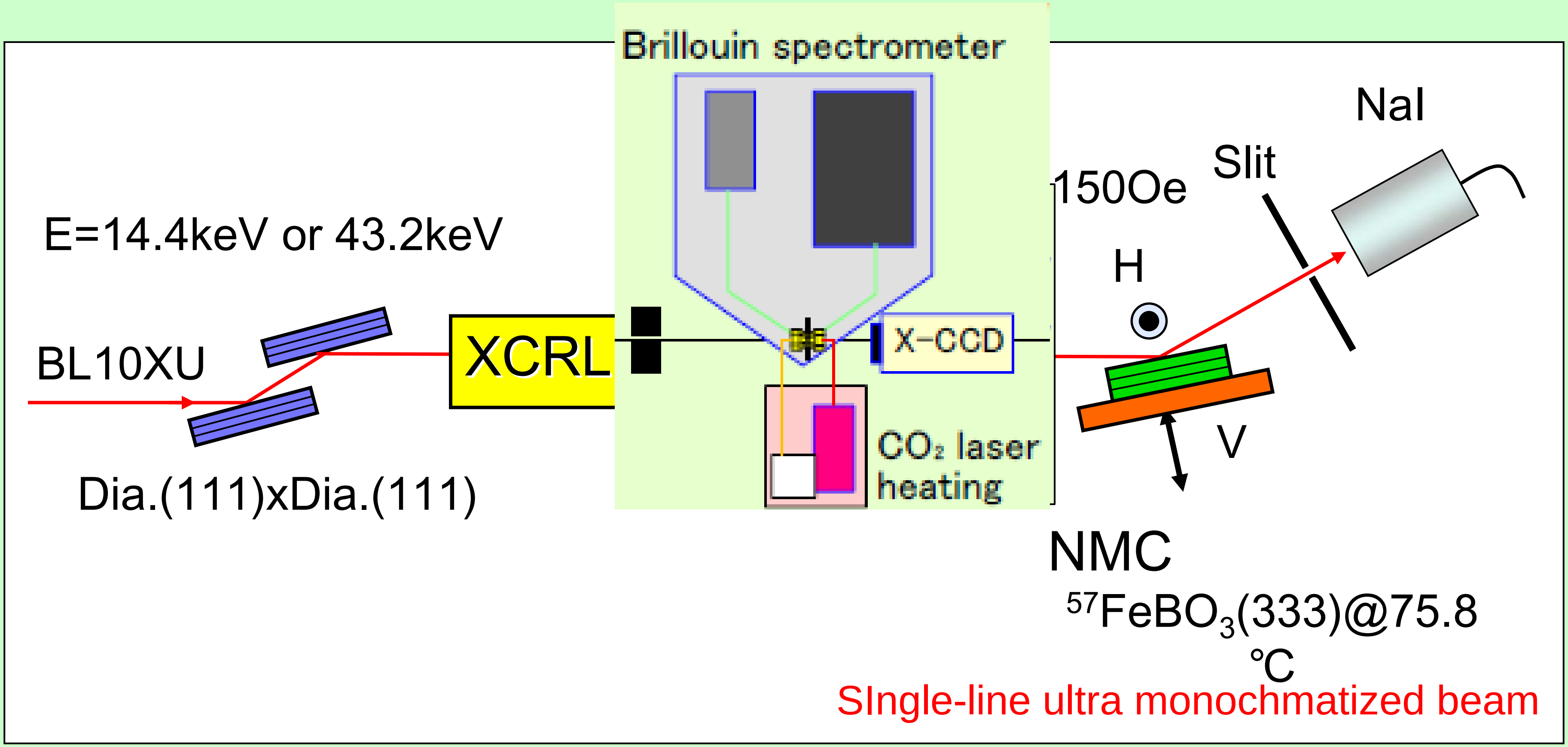
Pasternak, et al. PRL (1999)



8

Exposure time : 1 ~ 2 hrs.

Future Plan : Mössbauer spectroscopy and XRD simultaneous measurement



Summary

High-pressure researches for deep Earth's materials using DAC at SPring-8

BL10XU : *fundamental but still upgraded*

- for higher pressure (and higher temperature)
high flux x-ray beam ⇨ tandem XCRL focusing optics
accurate XRD experiment under multi-megabar
- Simultaneous measurement (XRD & Brillouin spectroscopy)

Resent developed spectroscopy technique at SPring-8

- Energy-domain SR Mössbauer spectroscopy
electronic and magnetic properties for iron containing materials under deep Earth's condition