WD-XRS Imaging with Polycapillary Optics

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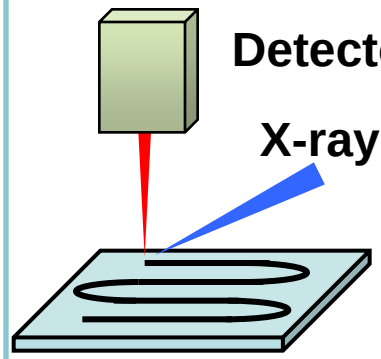
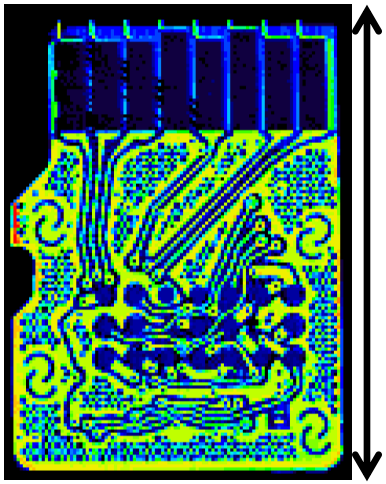
in cooperation with Rigaku Co.

XRF imaging

Scanning mode



Cu Kα image

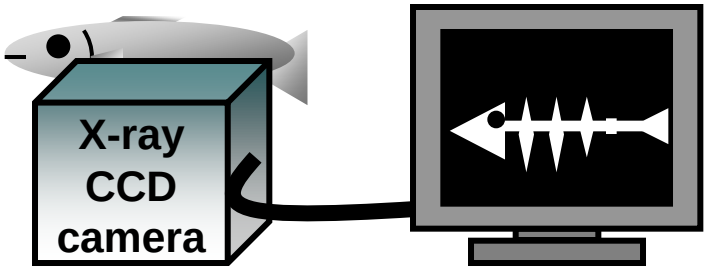


Micro SD card

High resolution image is obtain depending on the diameter of the micro x-ray beam.

Long acquisition time

Full Field mode

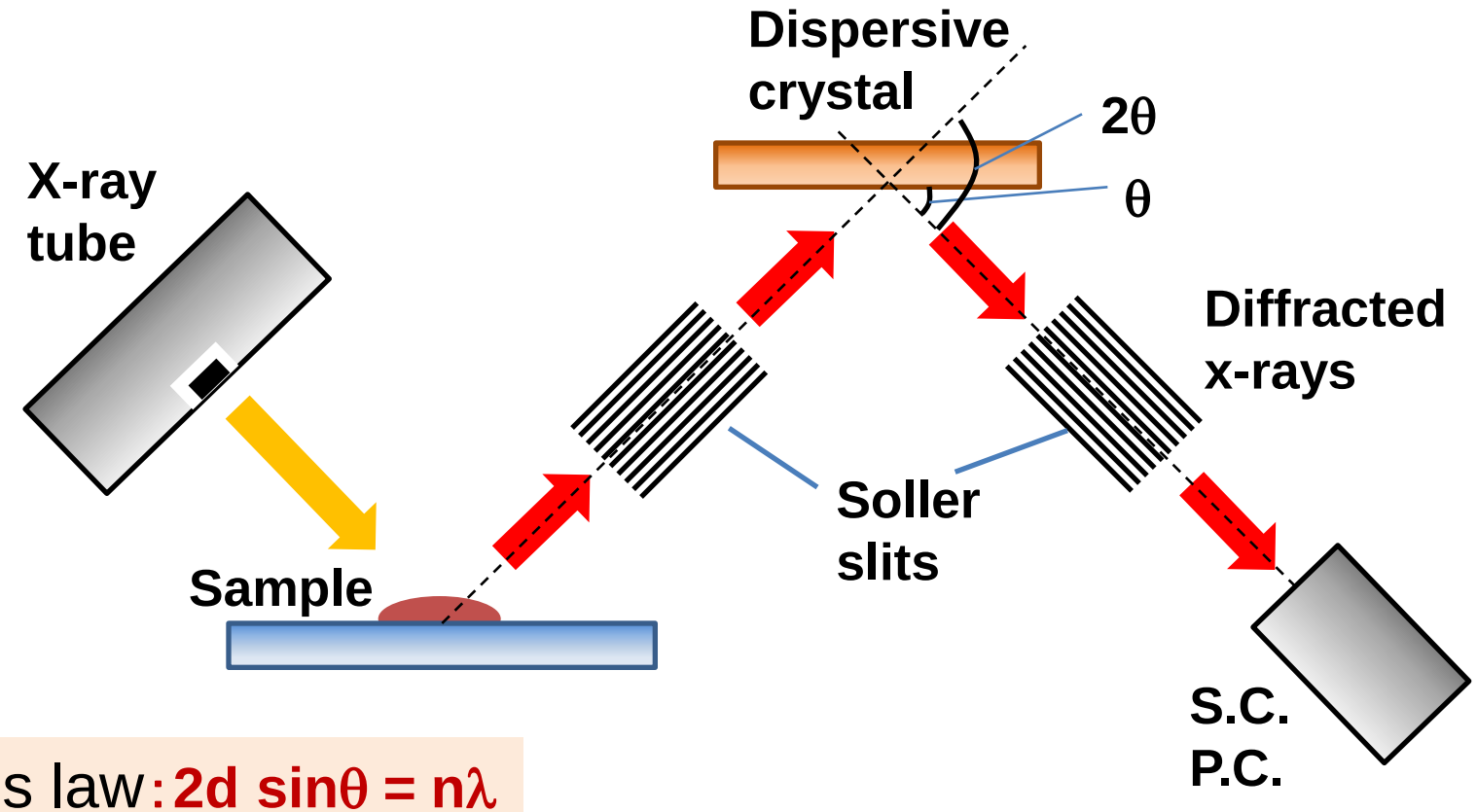


2D detector allows a fast imaging.

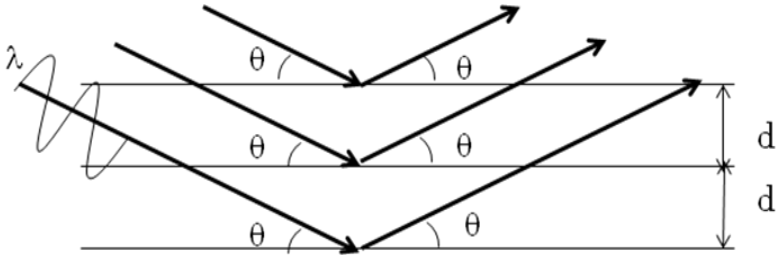
Elemental analysis (imaging) needs energy analysis of XRF.

WD spectrometer

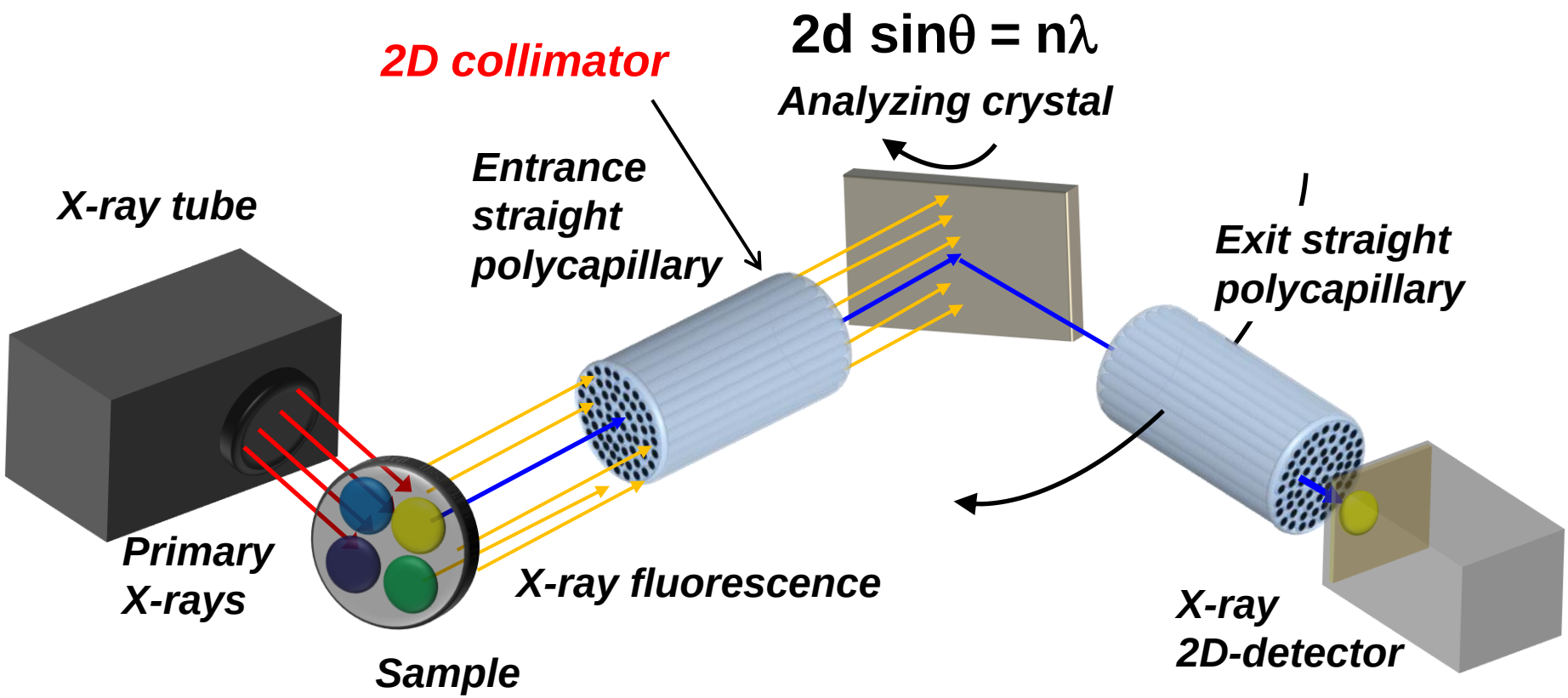
Conventional WD-XRF



Bragg's law : $2d \sin\theta = n\lambda$



Concept of WD-XRF imaging spectrometer

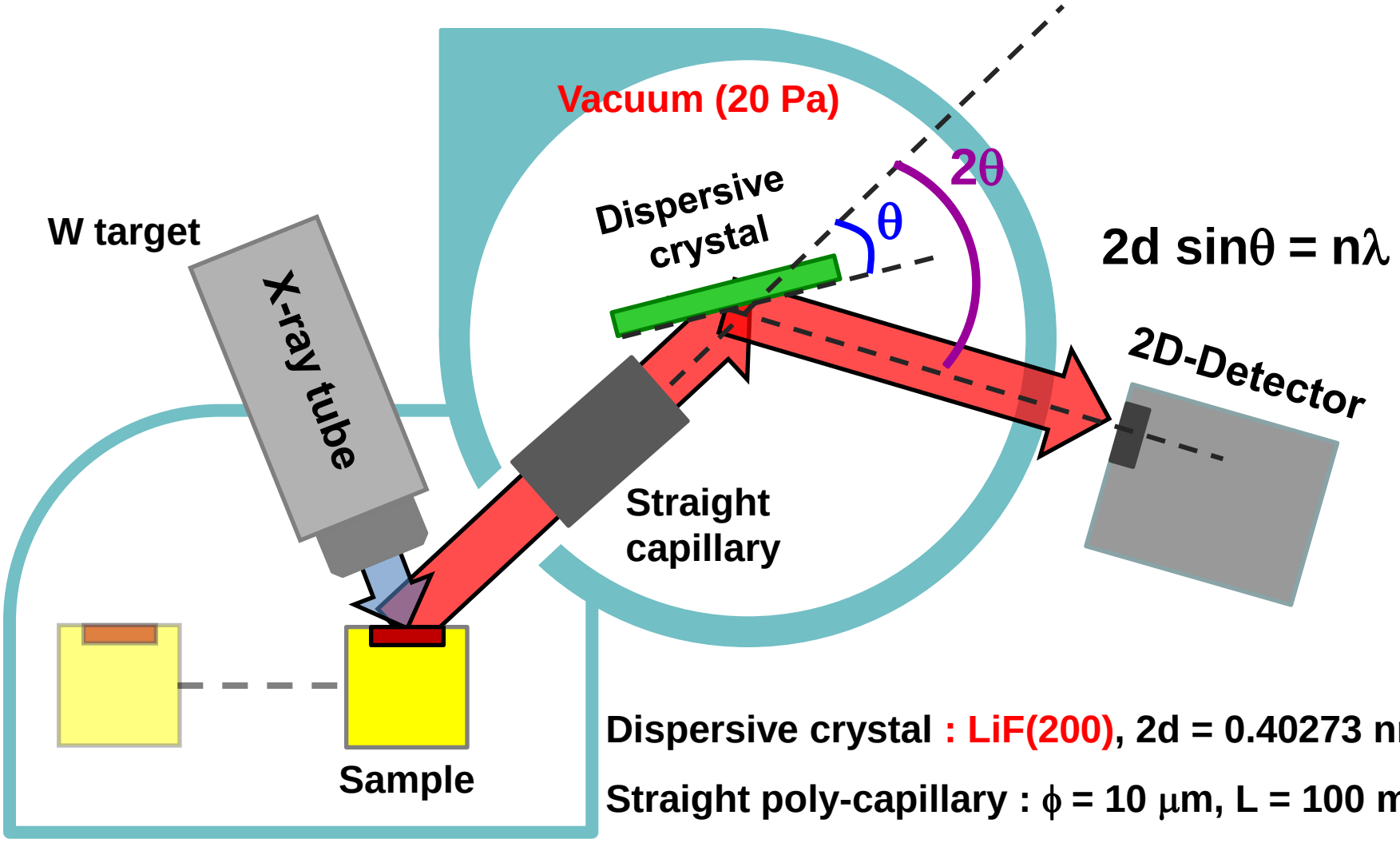


Previous results were reported in

K. Tsuji, et al, *Anal. Chem.*, **83** (2011) 6389-6394.

T. Ohmori, et al., *Spectrochim. Acta, Part B*, **83-84** (2013) 56-60.

WD-XRF Imaging Spectrometer based on Rigaku-RIX1000



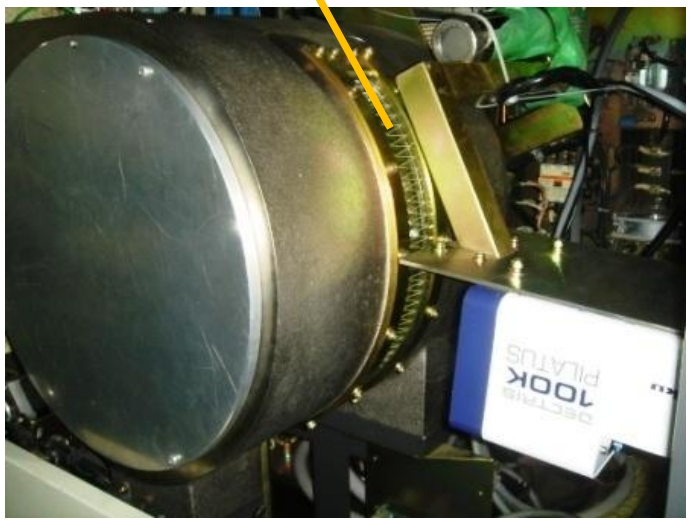
Dispersive crystal : LiF(200), 2d = 0.40273 nm

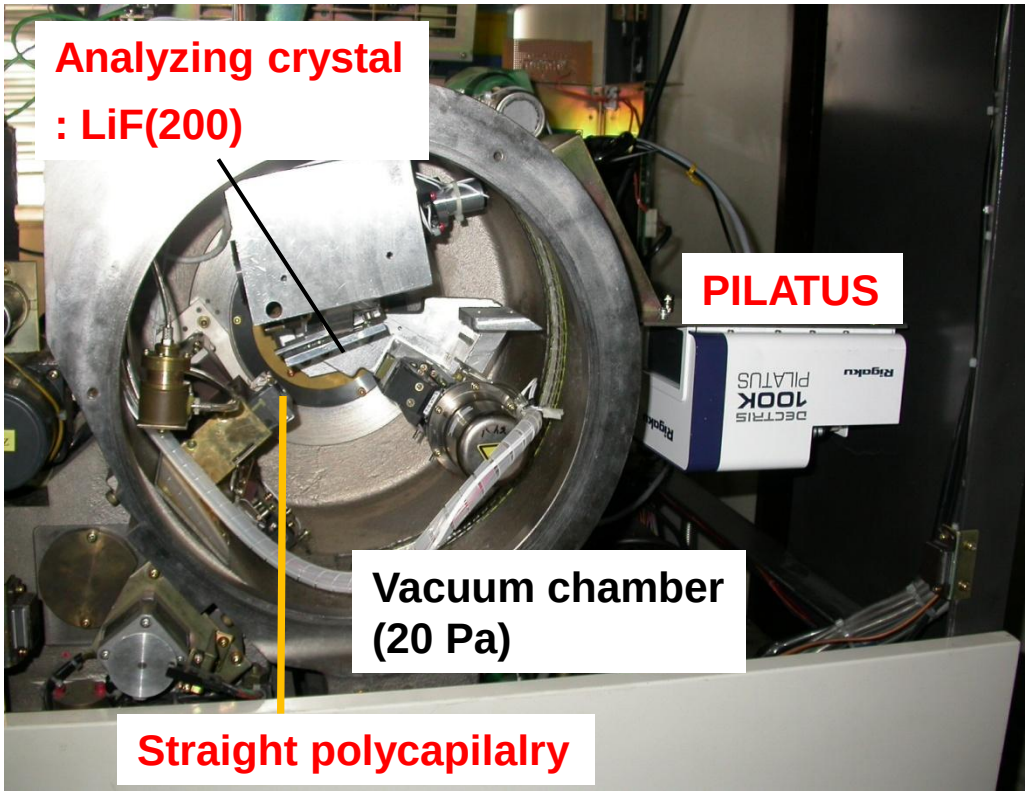
Straight poly-capillary : ϕ = 10 μm, L = 100 mm

WD-XRF Imaging Spectrometer based on Rigaku-RIX1000

Sample – crystal : 218 mm
Crystal – PILATUS: 203 mm

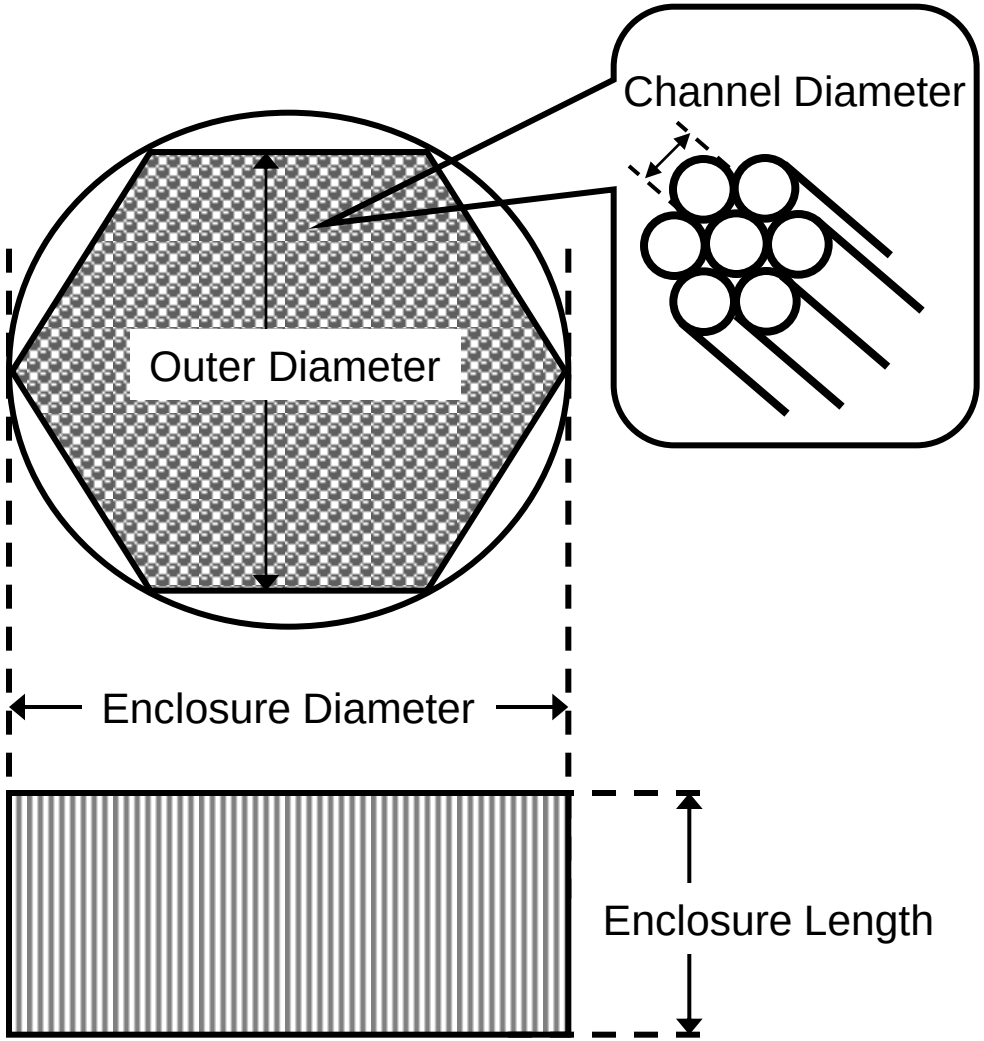
Polymer window supported by wire mesh



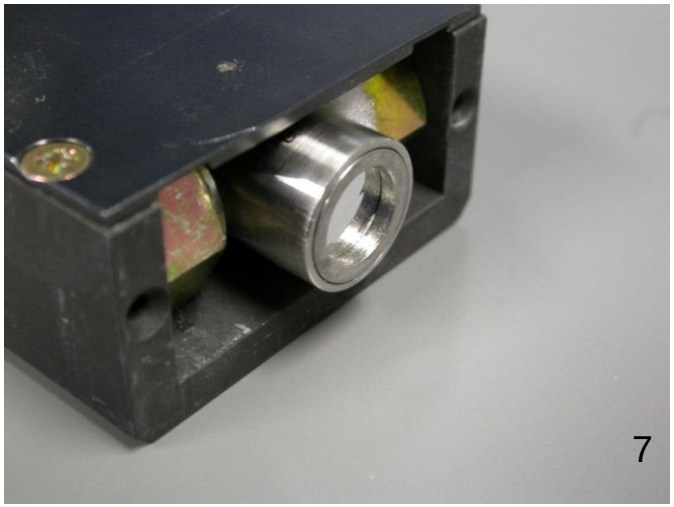


Straight polycapillary optics (by XOS)

Angular filter

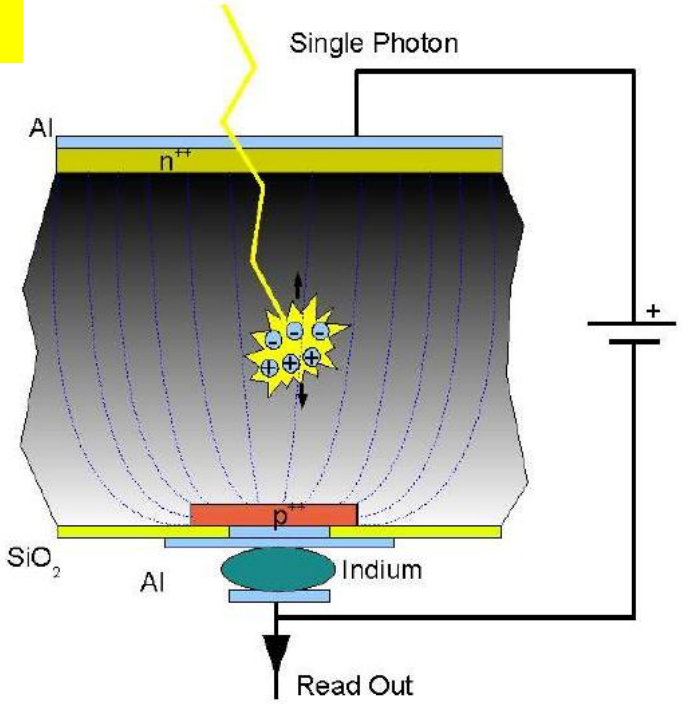


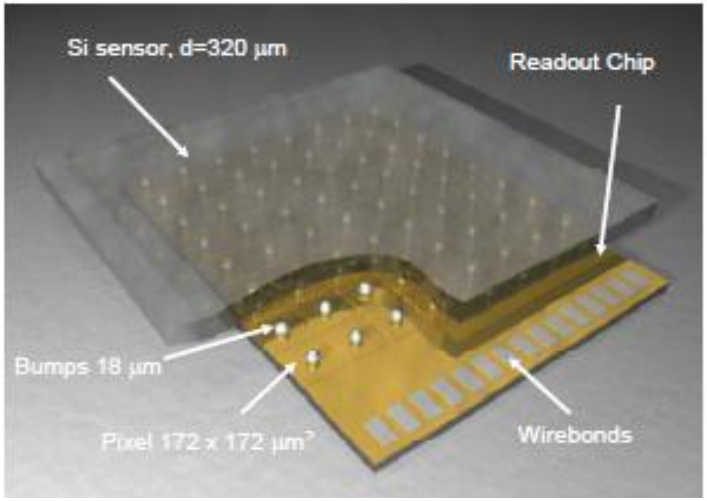
Straight polycapillary	
Outer Diameter	10.0 mm
Channel Diameter	10.0 μm
Enclosure Diameter	16.0 mm
Enclosure Length	105 mm
Open Area	73 %
X-ray transmission efficiency	53 %



Imaging detector (PILATUS, Rigaku)

Sensor : Silicon diode array (320 μm)
Pixel size : 172 x 172 μm^2
Format : 487 x 195 = 94,965 pixels
Area : 83.8 x 33.5 mm^2
Quantum efficiency : 3 keV : 80%, 8 keV : 99%
15 keV : 55 %
Readout time : 2.7 ms
Cooling : Air-cooled
Power consumption 15 W
Dimension 275 x 146 x 85 mm
Weight 4 kg

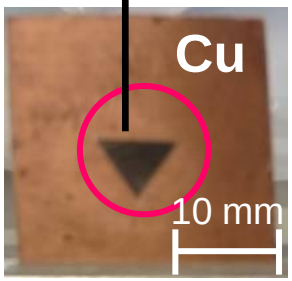




PILATUS: Pixel Apparatus for the SLS

Elemental image and Exposure time

Ni film (50 μm in thickness)

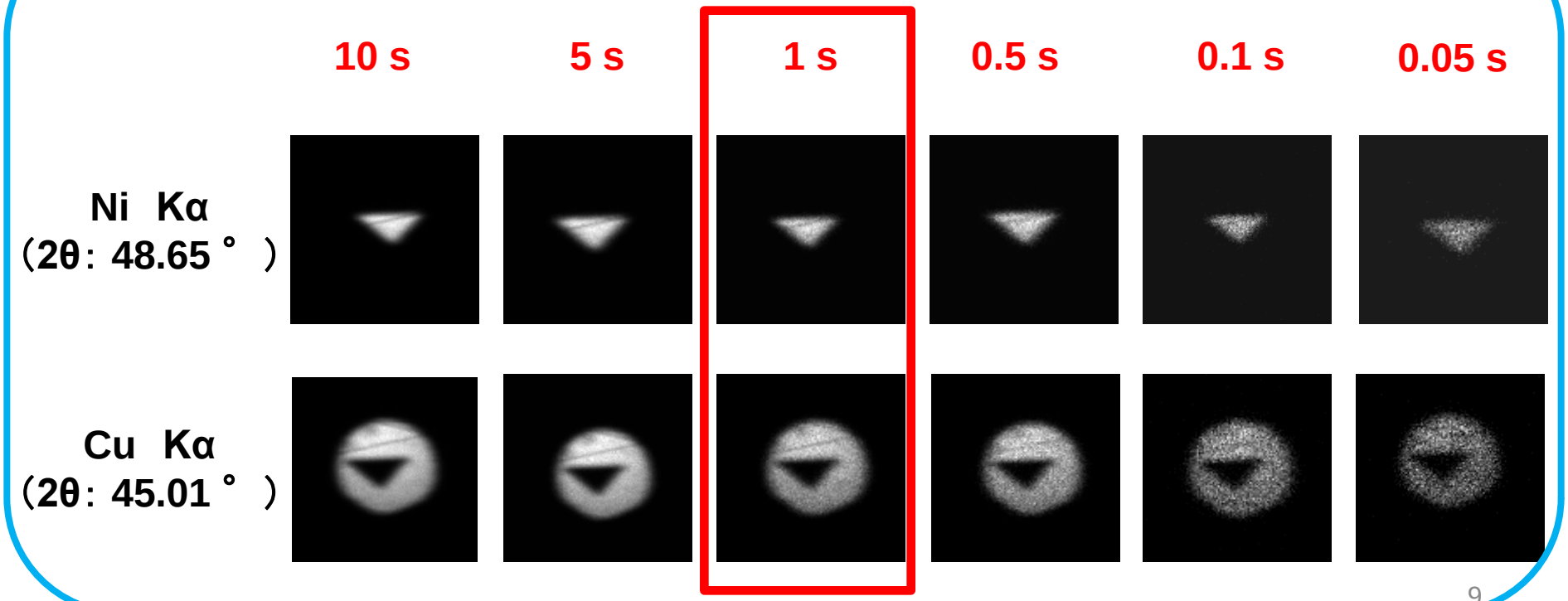


Cu plate

W target (60 kV , 50 mA)

Diffraction angles were adjusted to the corresponding angles of Ni Kα and Cu Kα.

Exposure time:



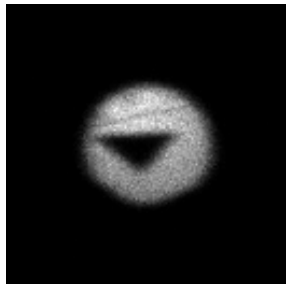
Elemental image and Diffraction angle

Elemental image was obtained,
as diffraction angle was changed.

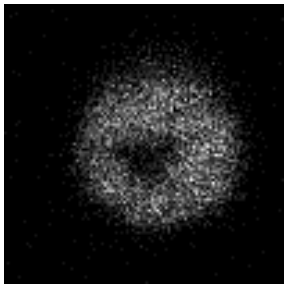
W target (60 kV , 50 mA)

Exposure time: 1 sec.

45.010 ° (Cu Kα)

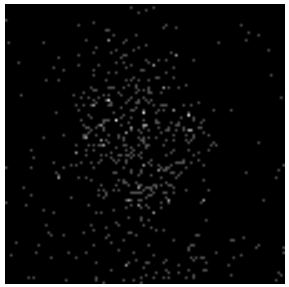


45.5 °



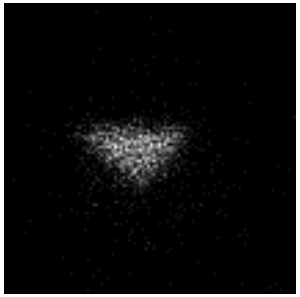
46.0 °

46.5 °

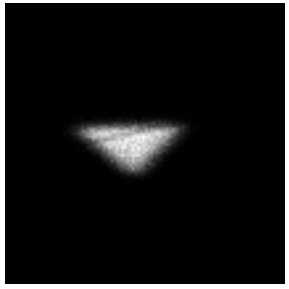


47.5 °

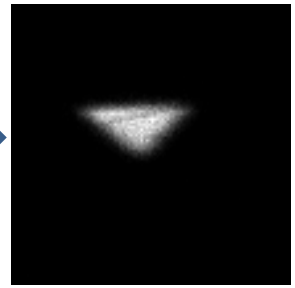
48.0 °



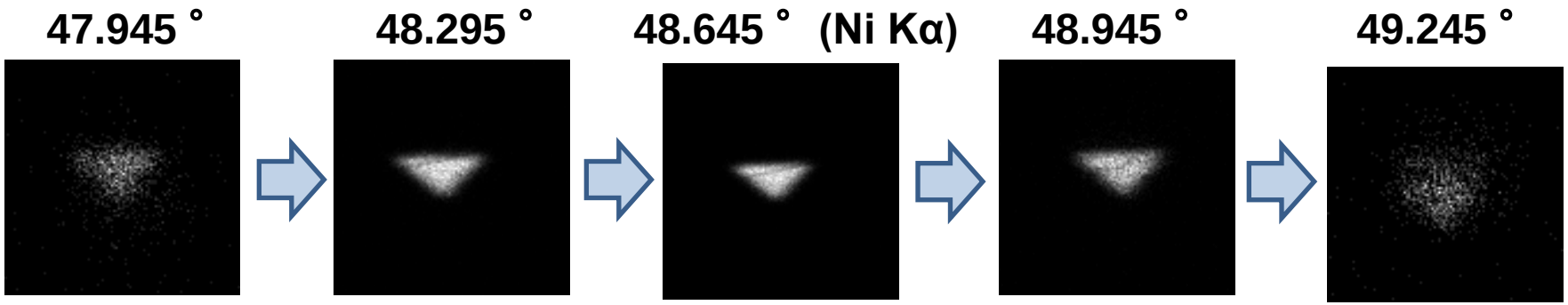
48.5 °



48.645 ° (Ni Kα)



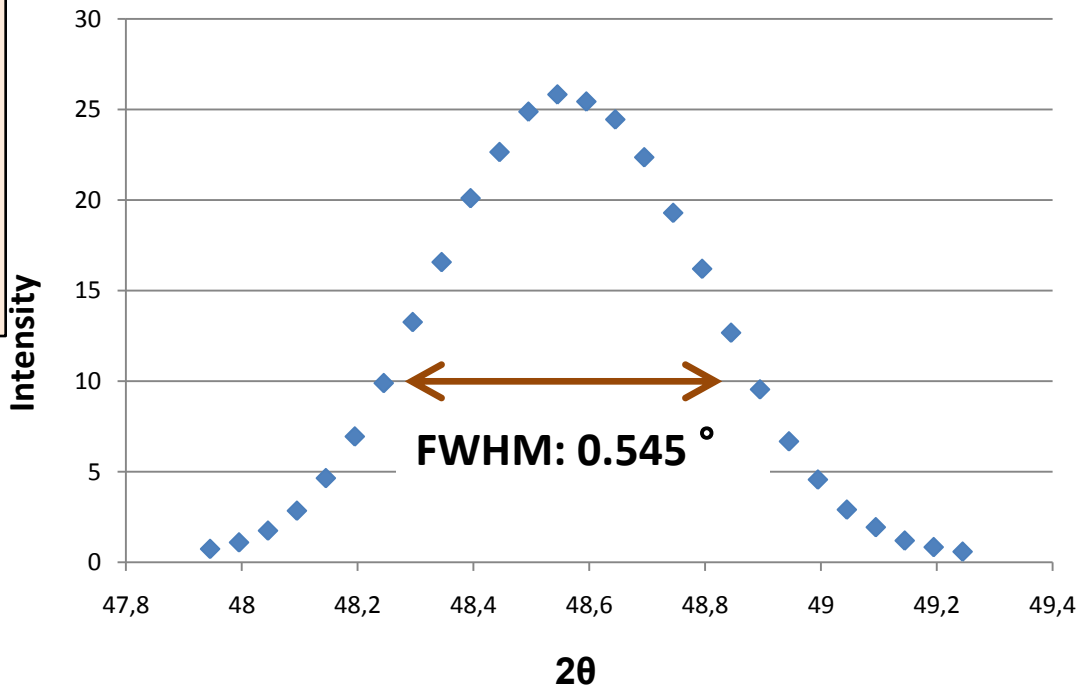
Energy resolution



W target (60 kV , 50 mA)

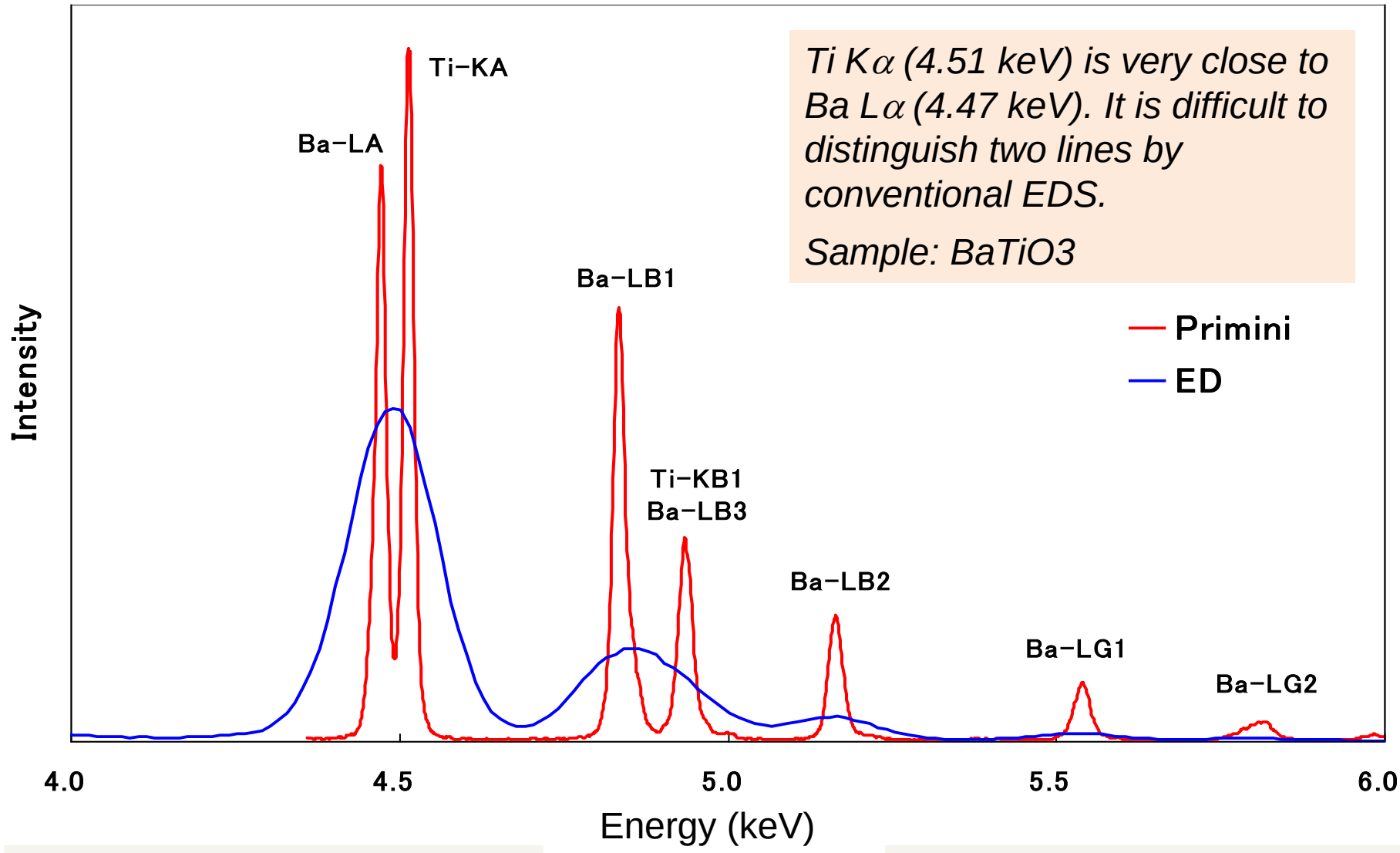
Exposure time: 1 sec.

Integrated intensity in triangle Ni was plotted.



FWHM of 0.545 degree corresponds to 77 eV, at Ni Kα energy.

Energy resolution in WDS and EDS



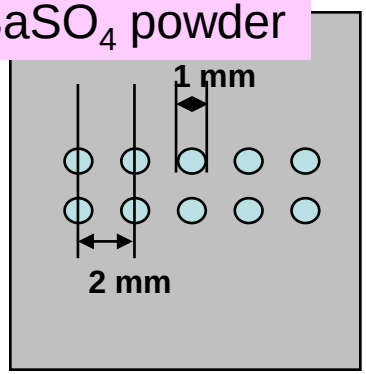
Ti Kα (4.51 keV) is very close to Ba Lα (4.47 keV). It is difficult to distinguish two lines by conventional EDS.

Sample: BaTiO3

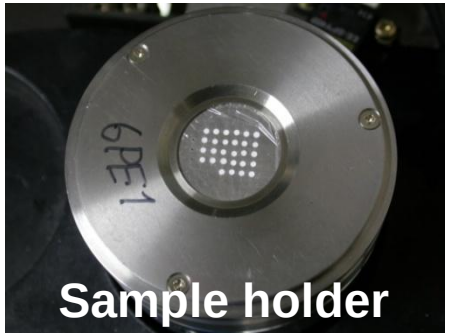
WD-XRF images of Ba-Ti sample

Ti Kα (4.51 keV) is very close to Ba Lα (4.47 keV). It is difficult to distinguish two lines by conventional EDS.

$BaSO_4$ powder

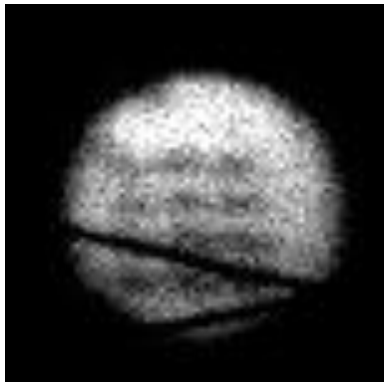
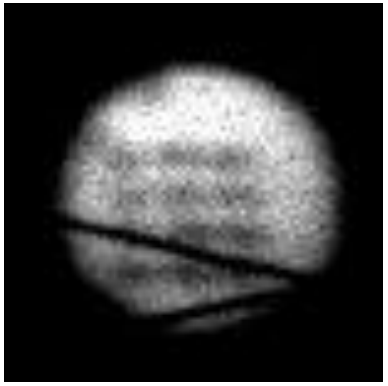
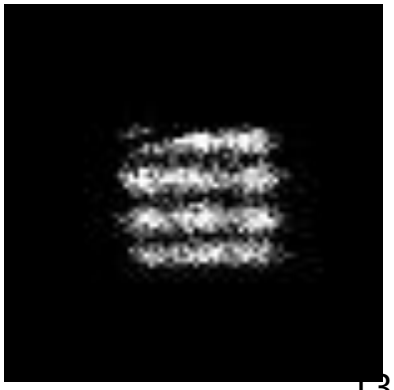


$BaSO_4$ powder was filled into the holes made on Ti plate.



W target
40 kV , 75 mA

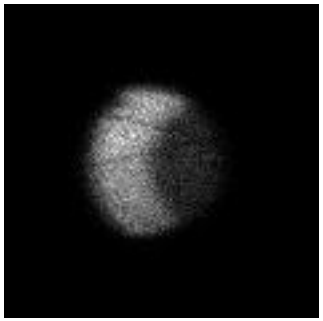
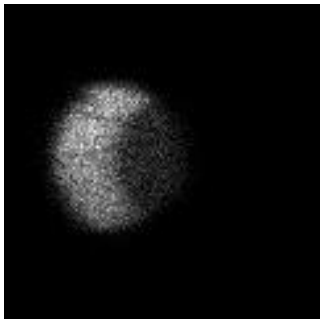
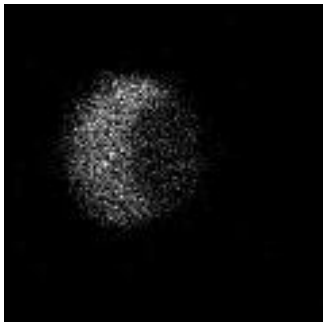
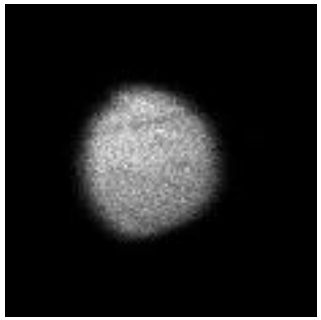
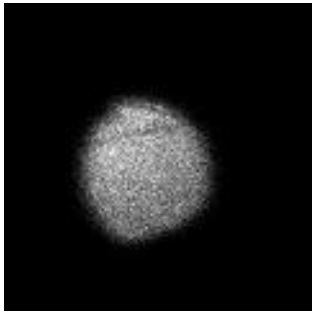
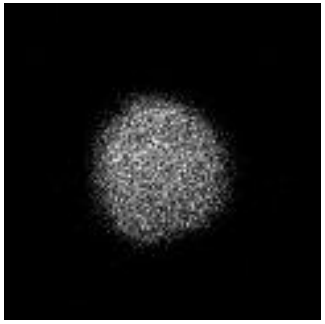
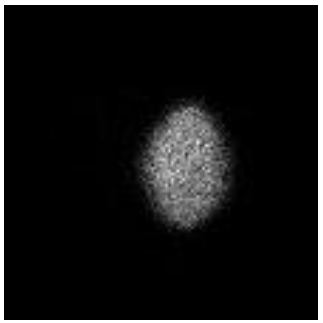
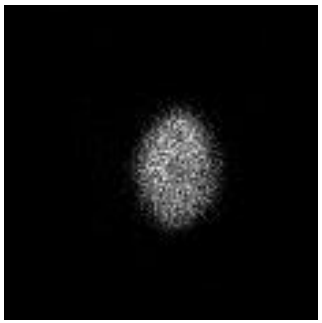
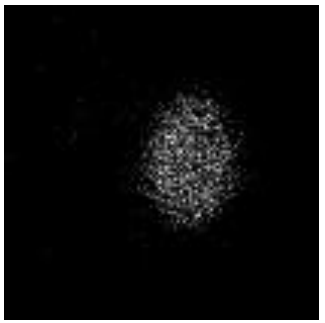
Exposure time:

	30 s	100 s
Ti Kα 86.110°		
40 eV		
Ba Lα 87.135°		

Elemental image of 2 Euro coin

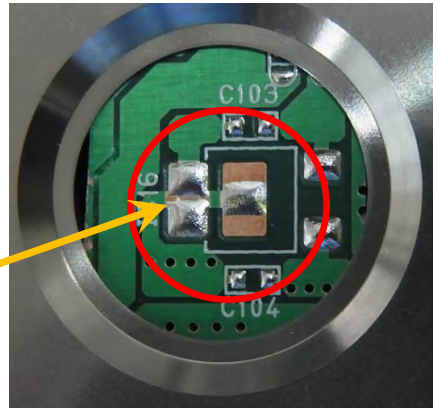
W target,
50 kV , 60 mA

Exposure time:

	1 s	0.5 s	0.1 s
Ni Kα **48.65˚**			
Cu Kα **45.01˚**			
Zn Kα **41.78˚**			

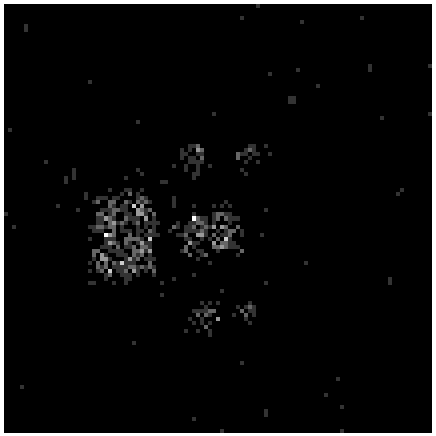
Elemental image of electronic circuit card

W target,
60 kV , 50 mA
2θ : 14.030 °

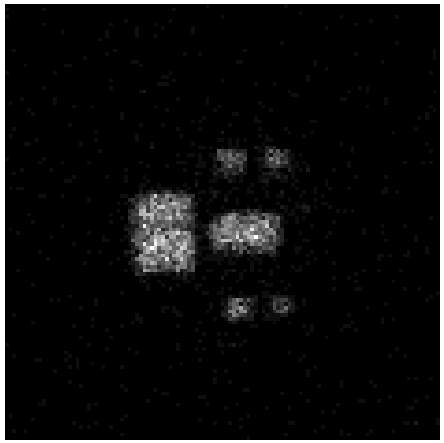


Solder

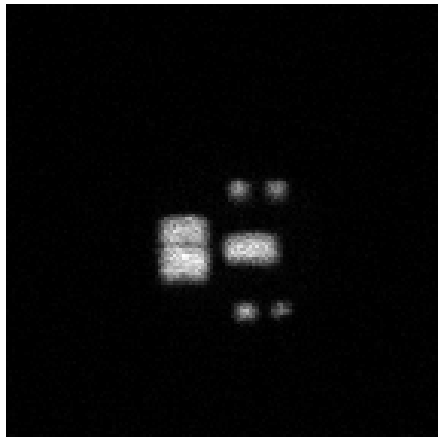
Sn Kα / 1 s



Sn Kα / 10 s



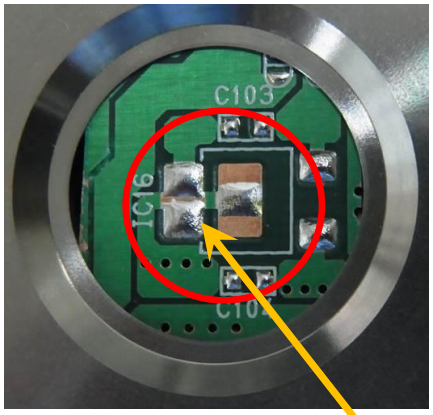
Sn Kα / 100 s



Elemental image of high energy x-rays needed long exposure time.
Sn Ka (25.19 keV)

Elemental image of electronic circuit card

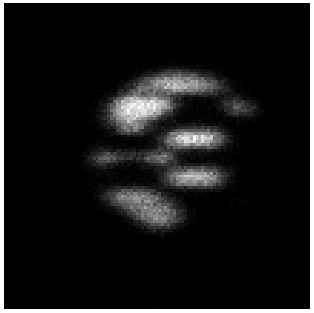
W target,
60 kV , 50 mA



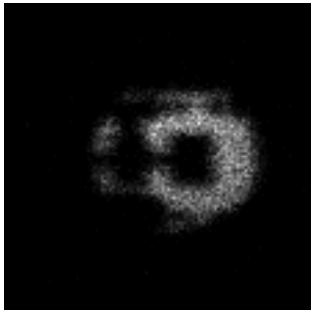
Element / Exposure time (sec.)

Solder

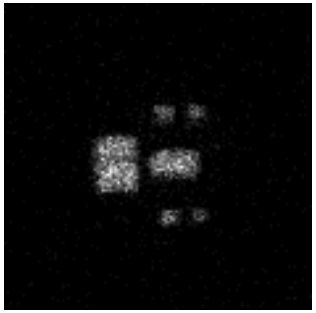
Cu Kα / 1 s
（2θ：45.01°）



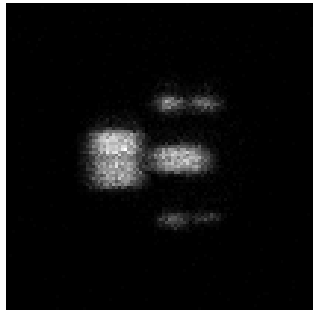
Br Kα / 1 s
（2θ：29.95°）



Sn Kα / 10 s
（2θ：14.03°）



Pb Lα / 10 s
（2θ：33.92°）



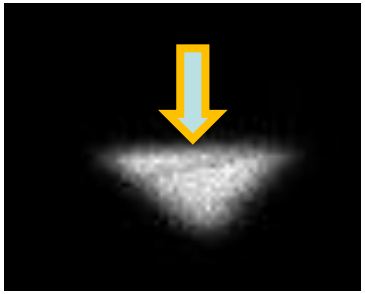
Summary

- ◆WDXRF imaging spectrometer was developed.
- ◆A fast elemental imaging (less than 1 s) was possible by using a highly sensitive imaging detector.
- ◆Energy resolution was 77 eV at Ni Kα.
- ◆Ti Kα and Ba Lα images were separately obtained (difference: 40 eV)
- ◆XRF imaging of Euro coin and electronic circuit was demonstrated.

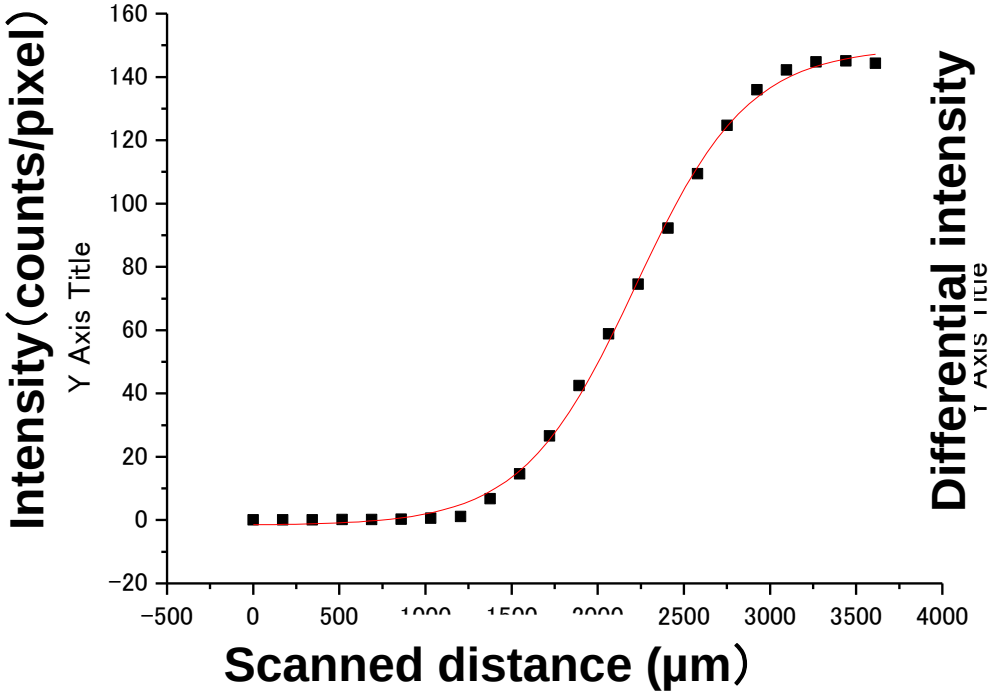
- ***We appreciate Prof. Y. C. Sasaki (Univ. of Tokyo) for his kind offer to use Pilatus detector.***
- ***This work was supported by JSPS Grant-in-Aid for Scientific Research (B) .***

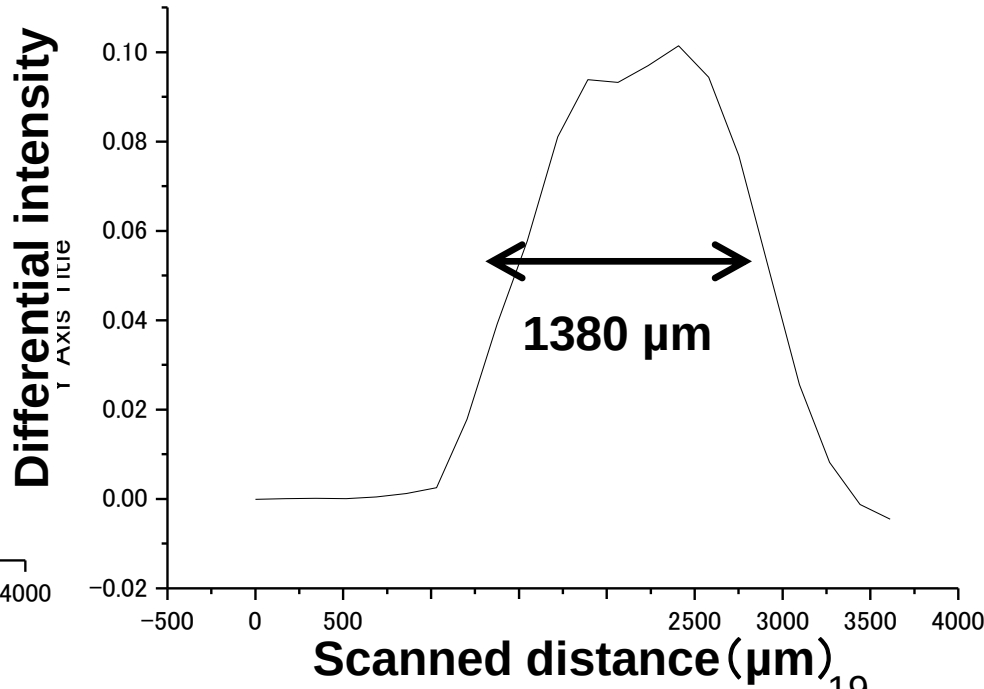
Evaluation of spatial resolution

Ni Kα image



W target,
50 kV , 60 mA
Ni Kα (2θ=48.645°)
Exposure time: 1s

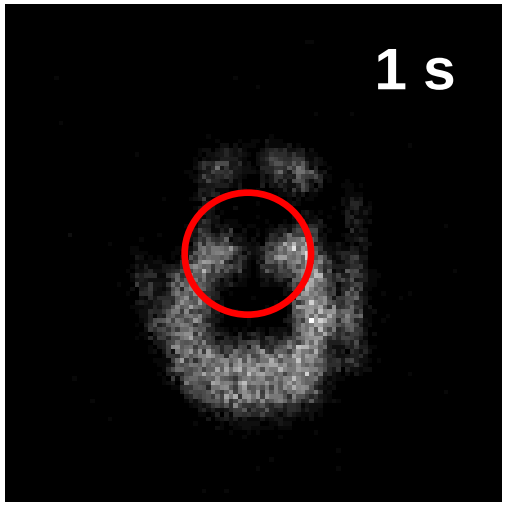


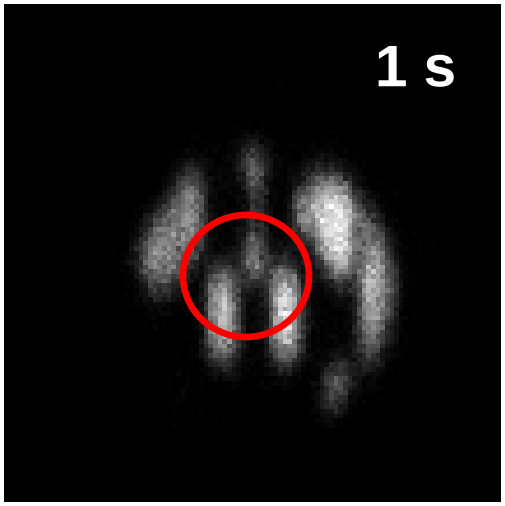


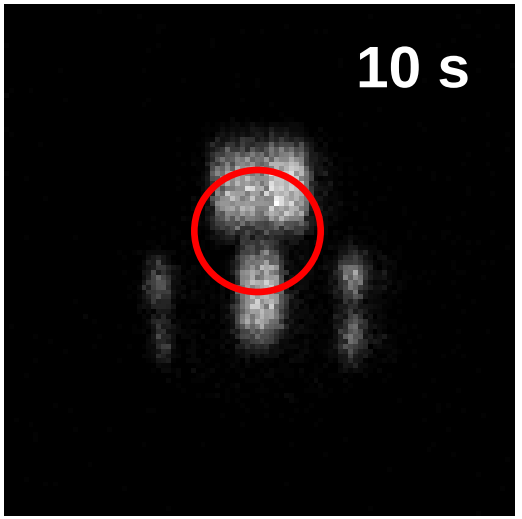
Scanning Micro-XRF (Horiba, XGT2500)

- **X-ray: Rh, 50kV, 1mA**
- **Micro beam of XGT: 100 μm**
- **Total mapping time: *3600 s***
- **Pixel : 256 × 256**
- **Area: *5.12mm × 5.12 mm***

Comparison (ED & WD imaging)



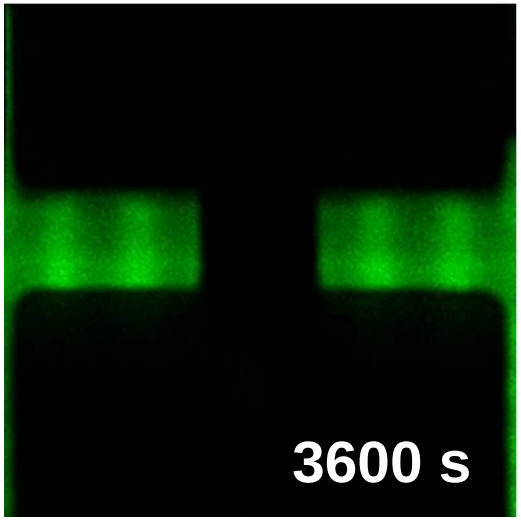


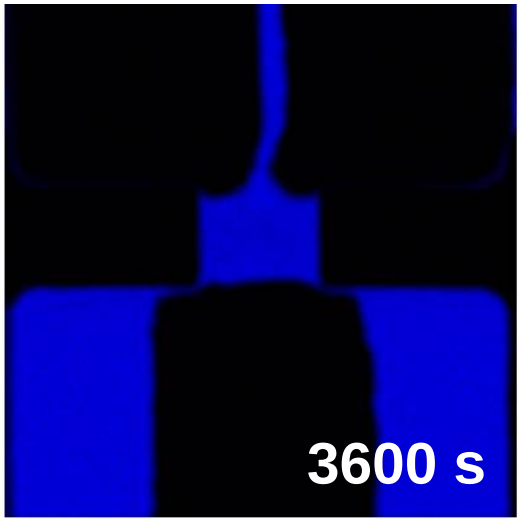


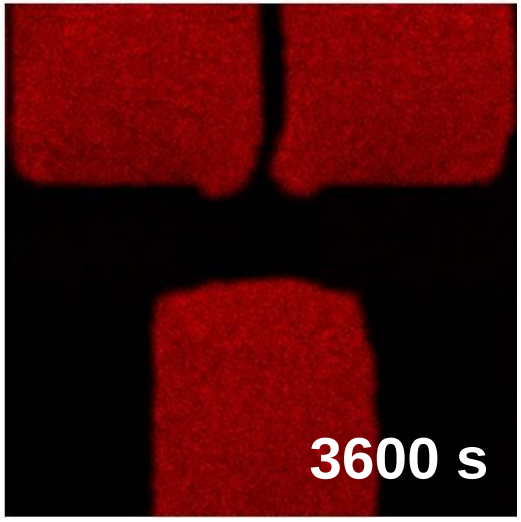
BrKα

CuKα

PbLα



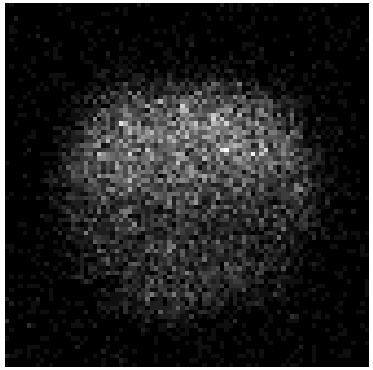




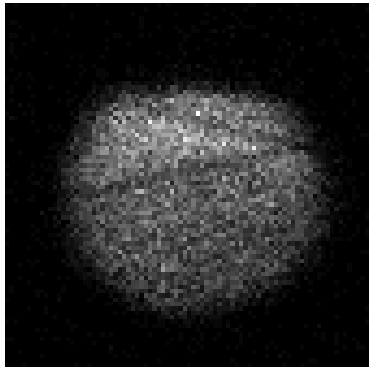
Elemental image of 2 Euro coin

W target, 40 kV , 75 mA, exposure time: 100 s

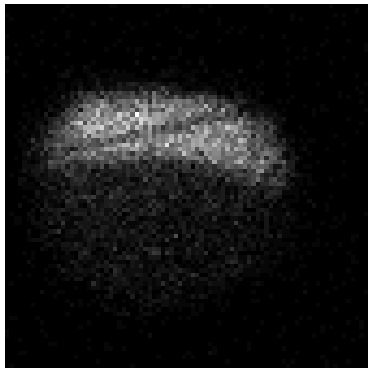
52.78° (Co Kα)

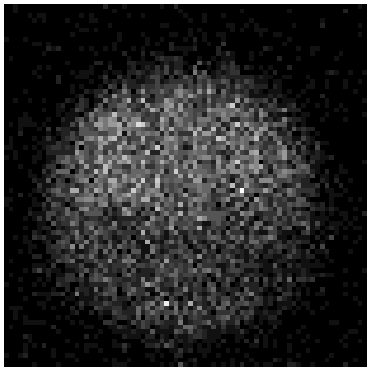


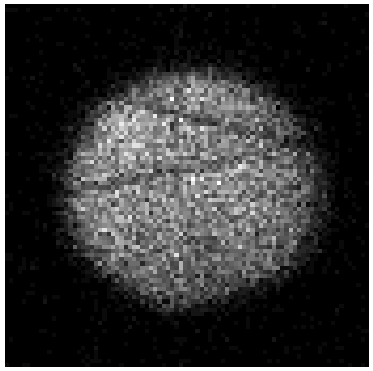
57.50° (Fe Kα)

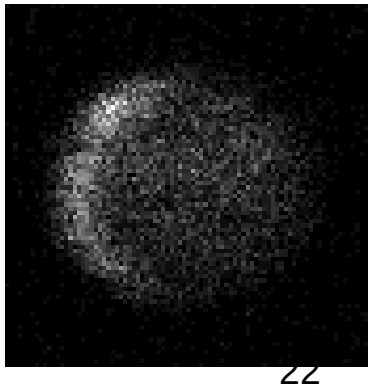


62.95° (Mn Kα)



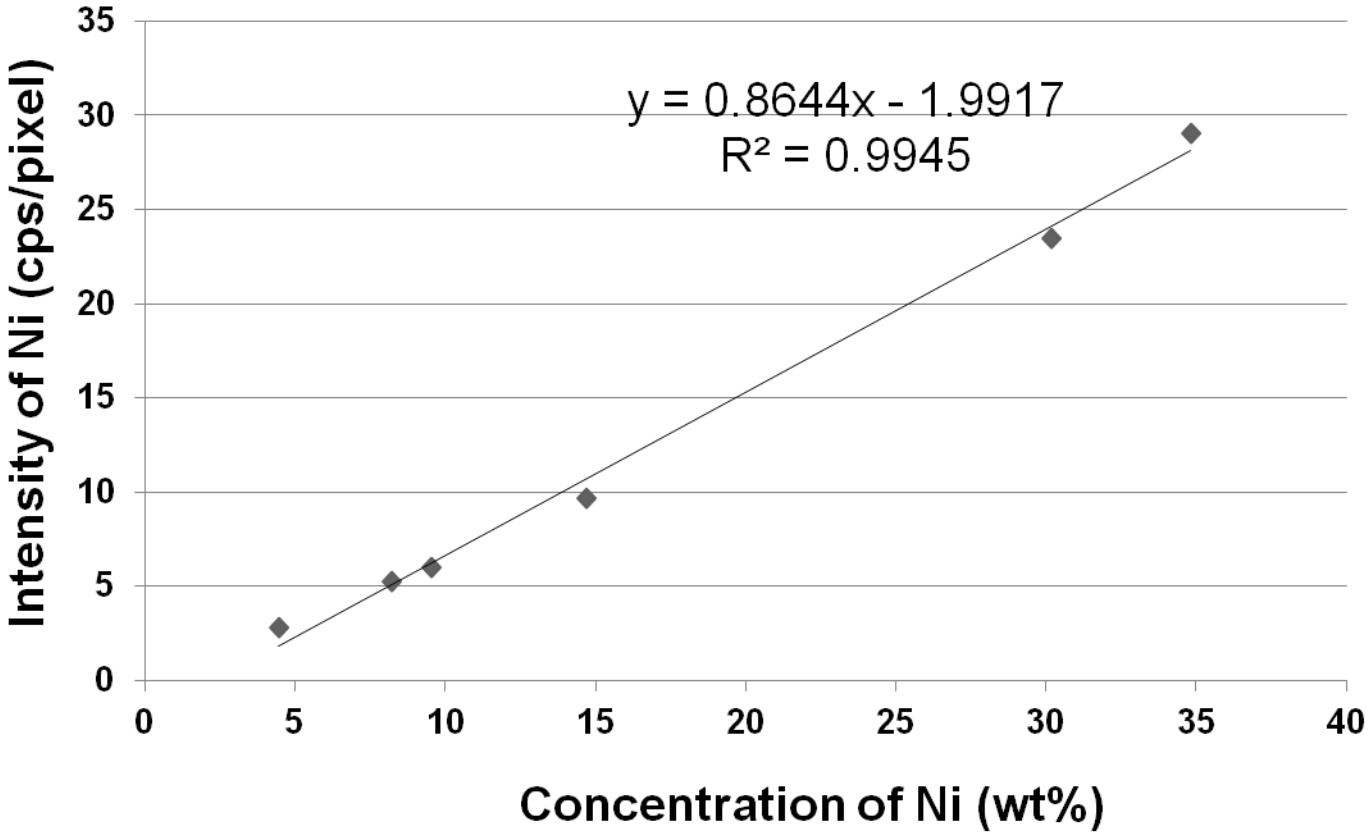






Calibration curve of Ni in steel sample

W target, 50 kV, 60 mA, 100 s



Averaged intensity per sec. per pixel was plotted.