

A transition edge sensor microcalorimeter system for the energy dispersive spectroscopy performed on a scanning-transmission electron microscope

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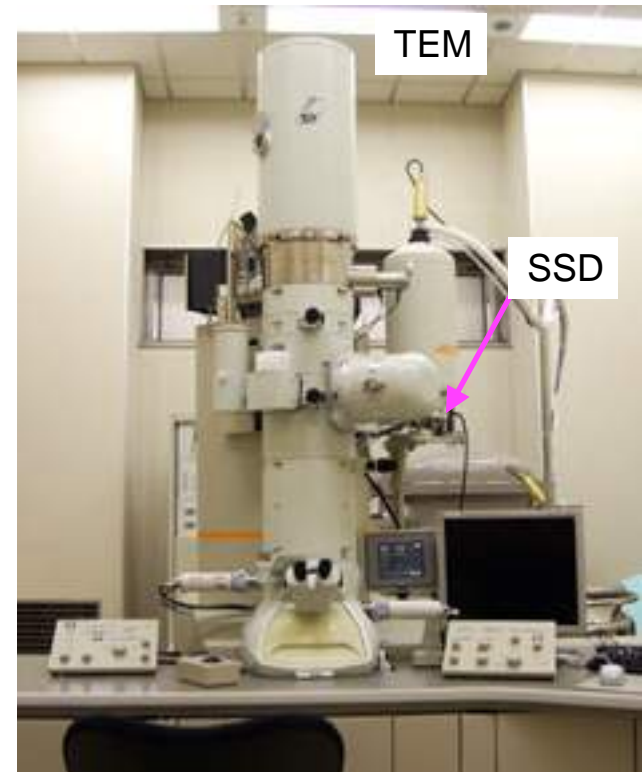
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EDS Performed on TEM

- Transmission Electron Microscope (TEM)
- Spatial resolution ~ 0.1 nm.
- Powerful tool for microscopic structure observation for innovative nano-material development.
- Element analysis with EDS of characteristic X-rays.
- ΔE of the X-ray detector limits element analysis ability.
- ΔE of Si(Li) X-ray detector 130 eV at 5.9 keV X-rays.
- X-ray detector with high energy resolution

Excellent ΔE of TES μ -cal.
 \Rightarrow EDS performed on TEM



http://www.nims.go.jp/tem/images/img-facilities/sengen_JEM2100F-2.jpg

TES μ -cal. EDS Mounted on SEM(1)

Several types have been developed for practical application.

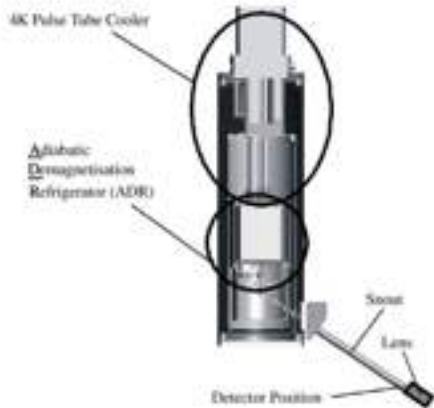


Fig. 2. POLARIS spectrometer.



Fig. 3. Polar microcalorimeter energy dispersive X-ray spectrometer.

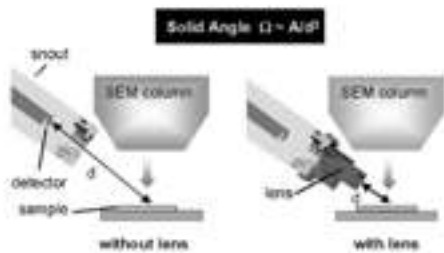
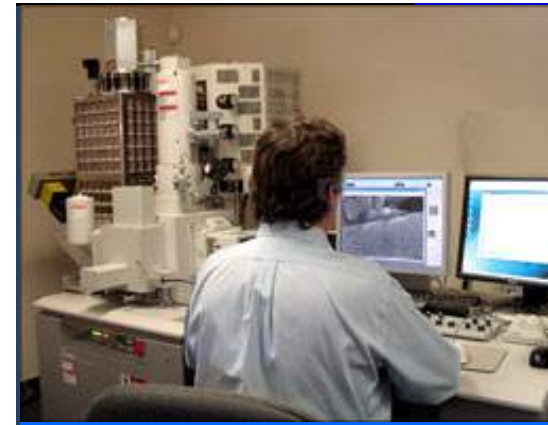
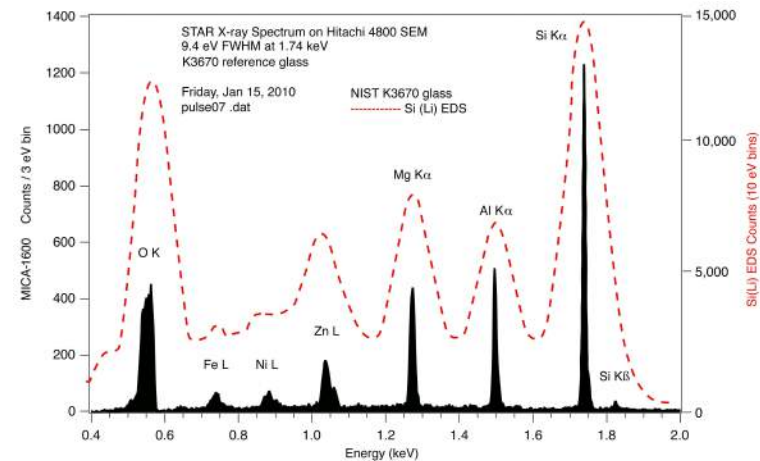


Fig. 4. SEM column and microcalorimeter snout w/o of X-ray lens.

Hollerith et al., NIM. A520 (2004) 606–609



Redfern et al., J. Res. Natl. Inst. Stand. Technol. **107**, 621–626 (2002)

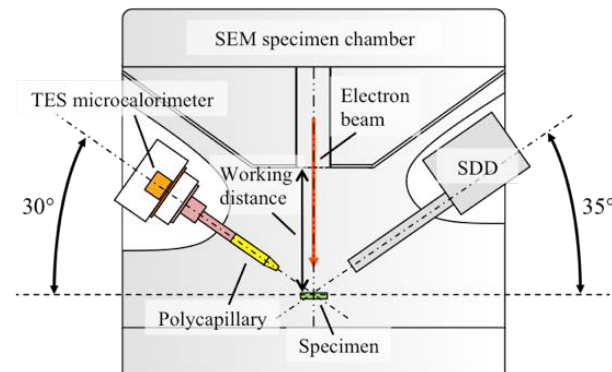
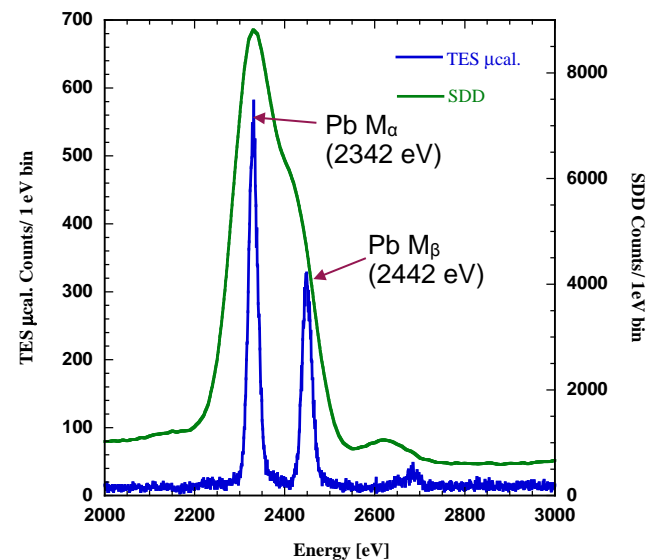
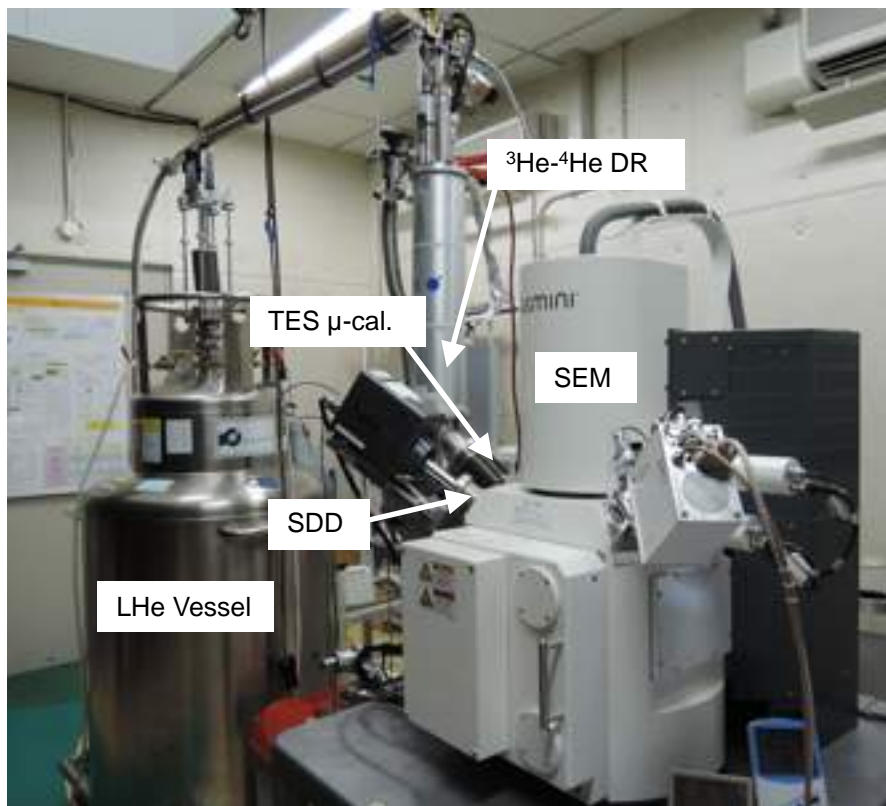


http://www.starcryo.com/microcal_eds.htm

TES μ -cal. EDS Mounted on SEM(2)

One of open advanced facilities for research and education at the Ultramicroscopy Research Center, Kyushu University

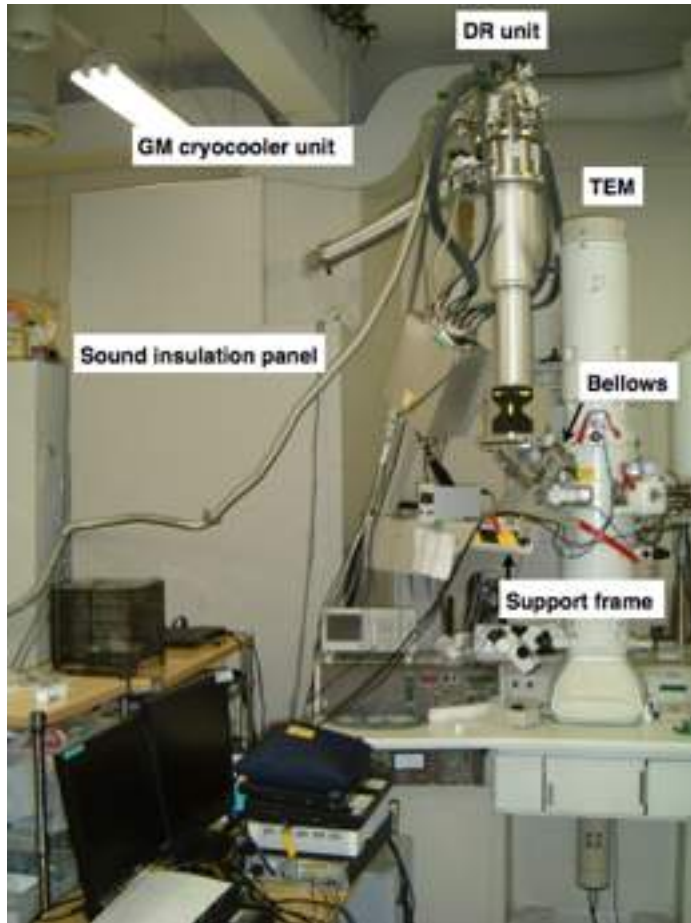
Single pixel TES μ -cal. with a polycapillary X-ray optic
 ΔE of TES μ -cal.: < 20 eV FWHM @Al-K $_{\alpha}$ 1.48 keV
Compact DR pre-cooled by circulating LHe.



Details of TES μ cal. and the DR: Maehata et al., NIM, A648 (2011) 285–2899

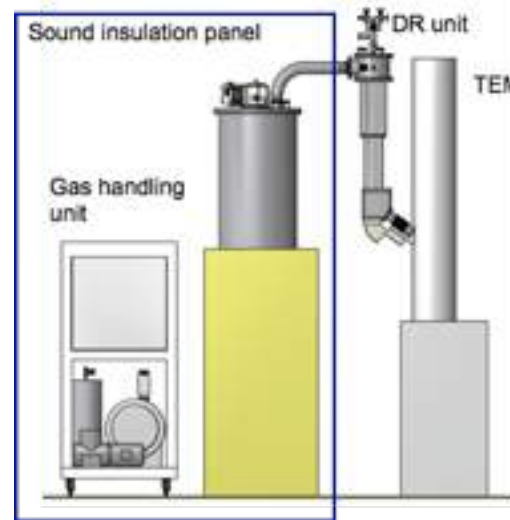
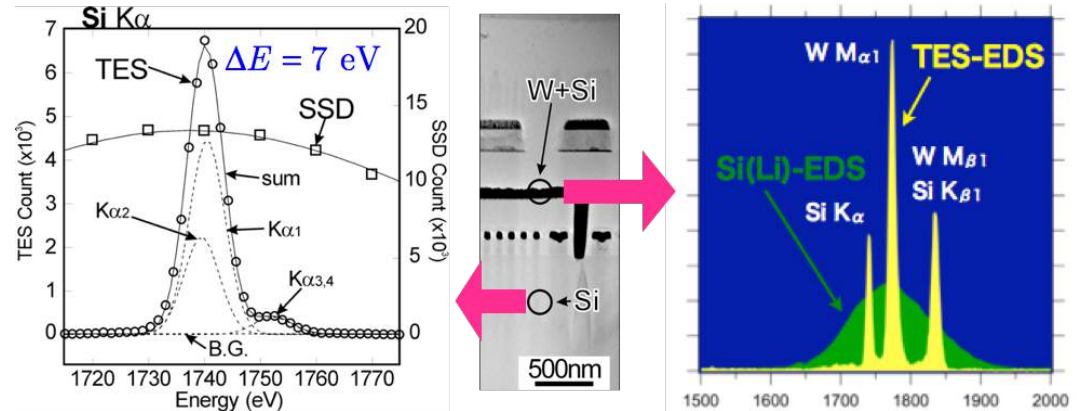
Prototype TES μ -cal. EDS on TEM

Single pixel TES μ -cal. EDS similar to that on SEM at Kyushu Univ.



Hara et al., J. Electron Microsc., 59 (2010) 17-26

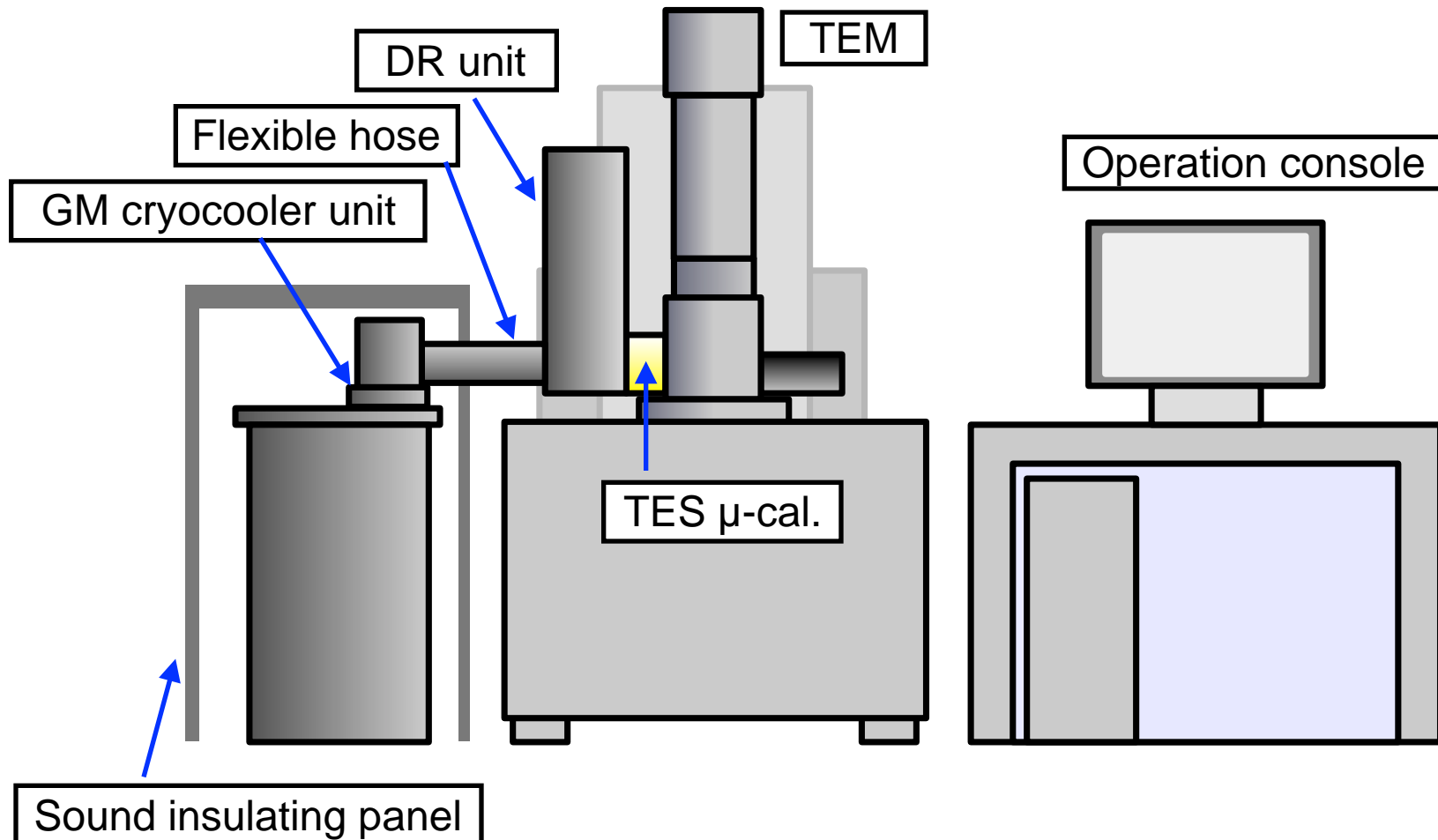
Demonstration of TES μ -cal. EDS on TEM



Remote helium cooling loop for pre-cooling the DR with GM cooler split type. Maehata et al., Cryogenics, 61 (2014) 86-91

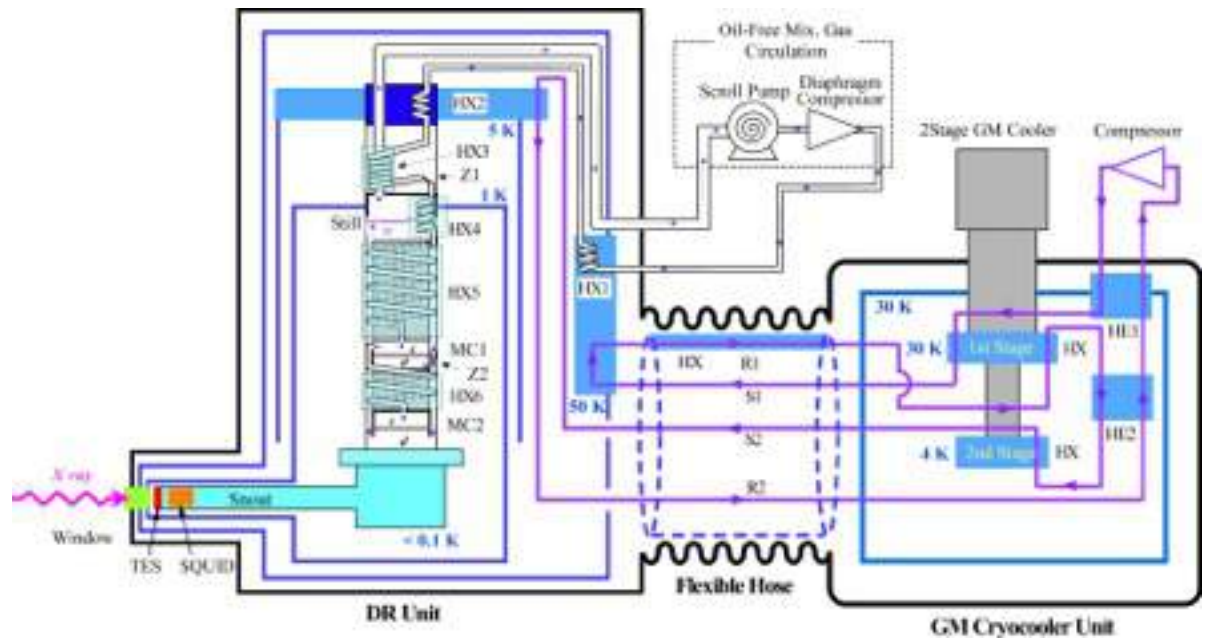
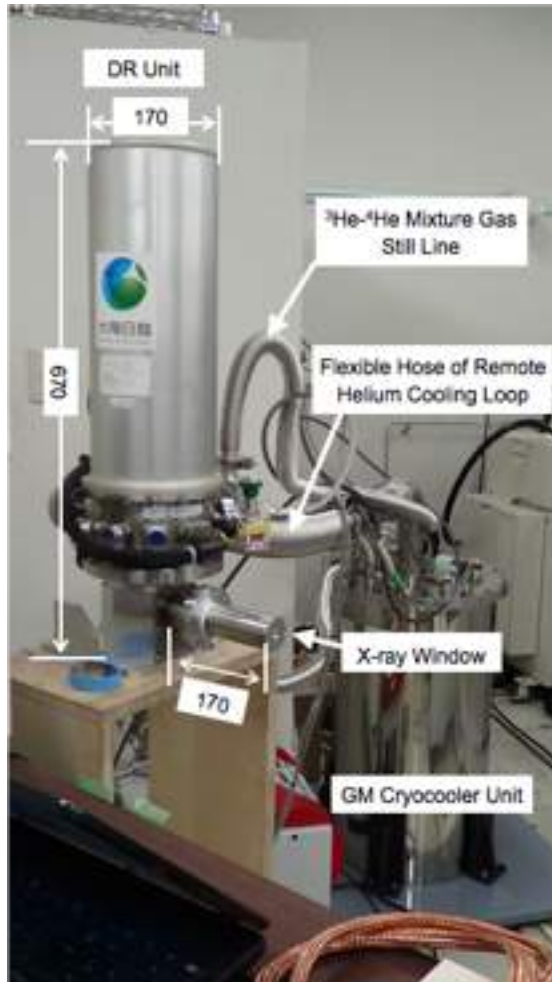
Arrangement of TES μ -cal. EDS on TEM

Compact dry DR located at lower position for practical operation



Compact Dry DR for TES μ -cal. EDS

Remote Helium Cooling Loop for pre-cooling DR

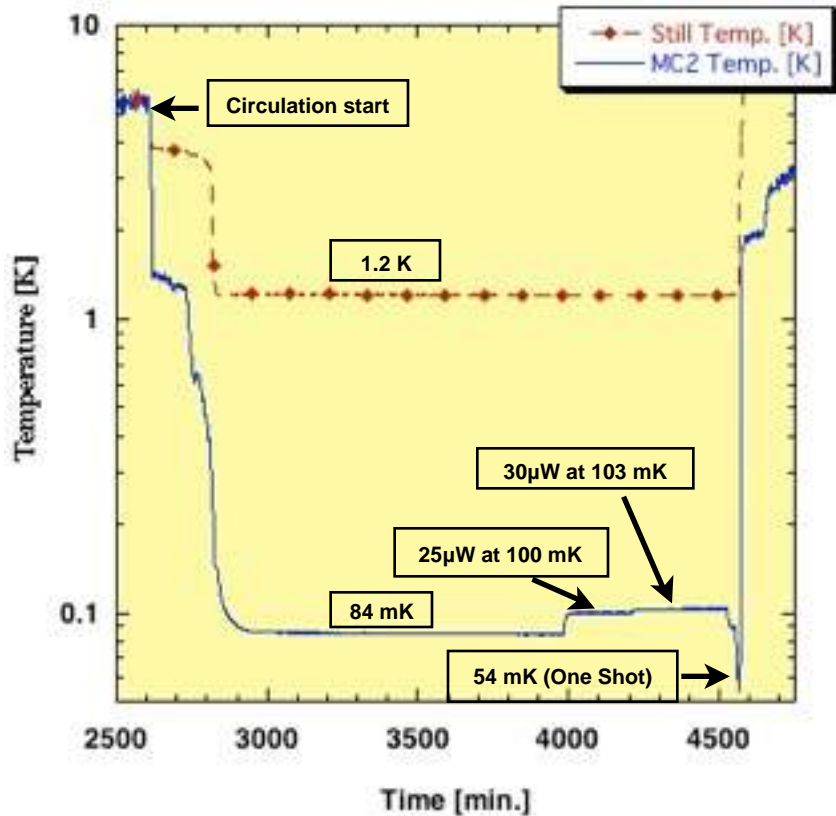


GM-cryocooler;
 SHI RDK-415D
 2nd stage 1.5W @ 4.2 K
 1st Stage 35W @ 50K
 ^4He gas circulation ;
 SHI CAN-11 compressor

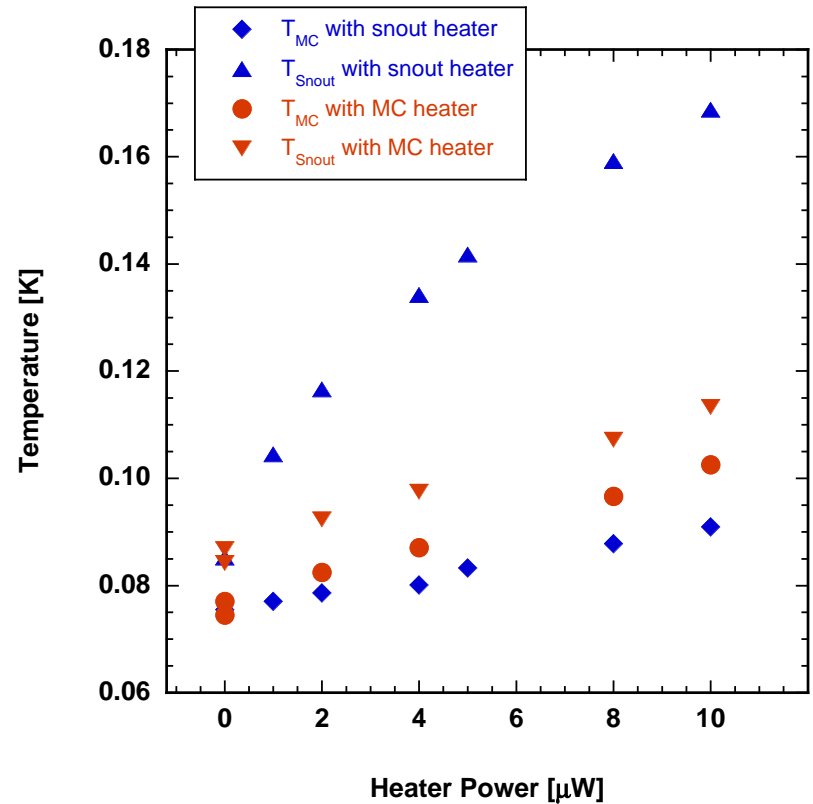
Dilution GHS ;
 XDS35i Dry pump
 KNF diaphragm compressor

Maehata et al., J. Supercond. Nov. Magn., 28 (2015) 1161–1165

Operation Tests of Compact DR Unit



Cool down curve without snout

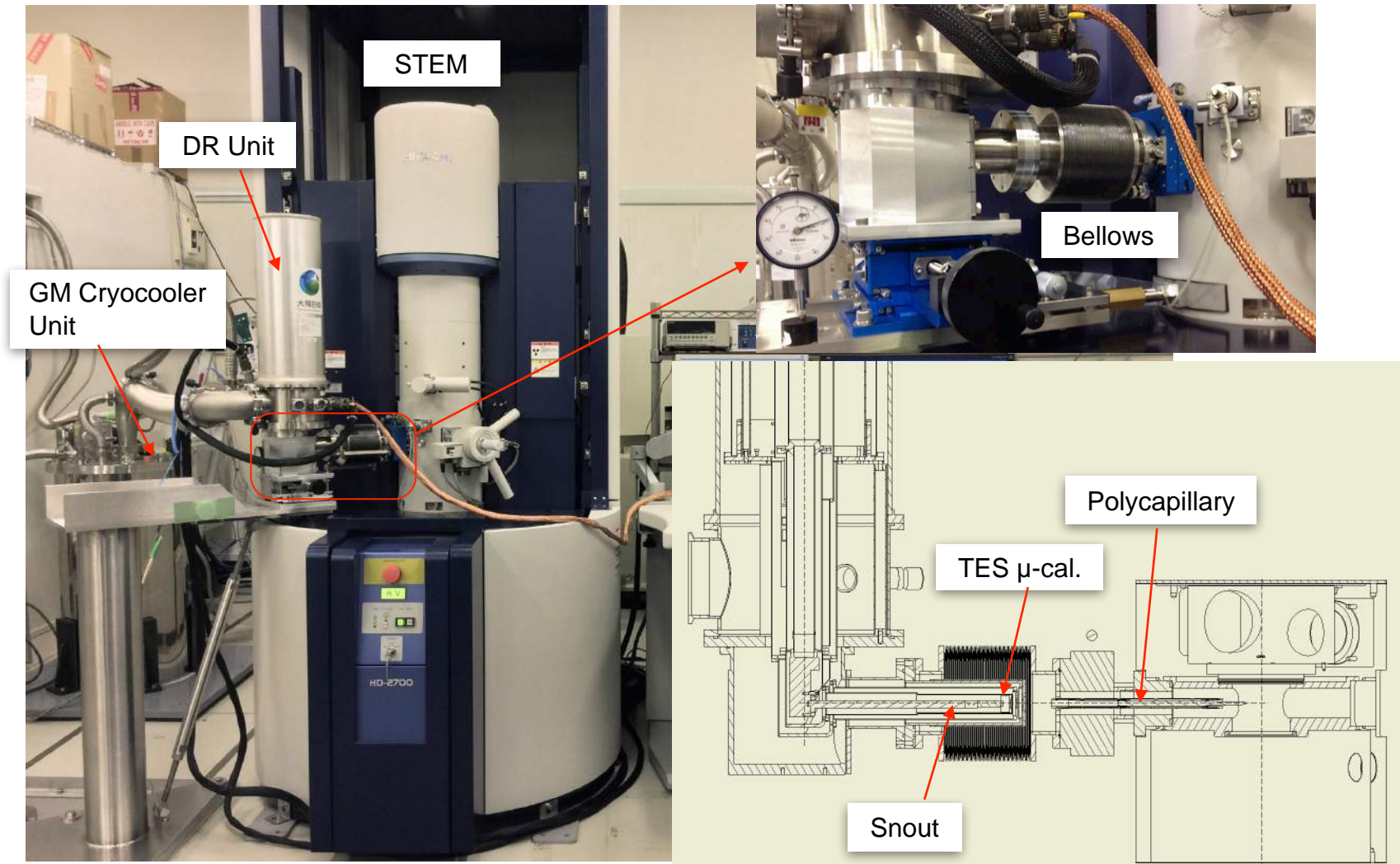


Cooling power with snout

Mount on STEM

STEM (Hitachi HD-2700) for TES μ -cal. EDS at NIFS

Single pixel TES μ -cal. EDS similar to that on prototype (SEM at Kyushu Univ.)



STEM Image with DR Operation

STEM image Si [110]

Magnification

x15000k

15M

Brightness/Contrast

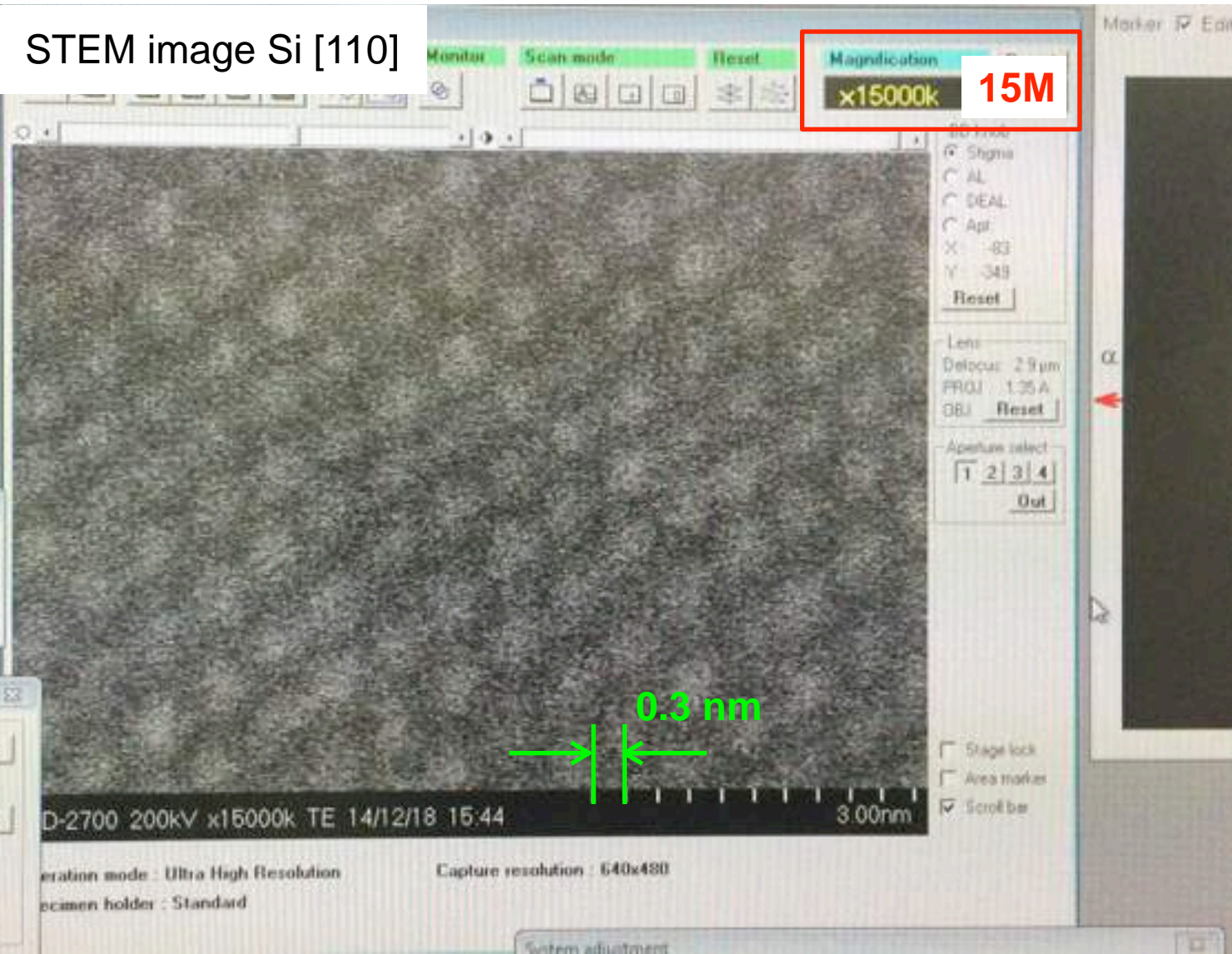
	A	B
Brightness	2473	
Contrast	4065	

Lens control

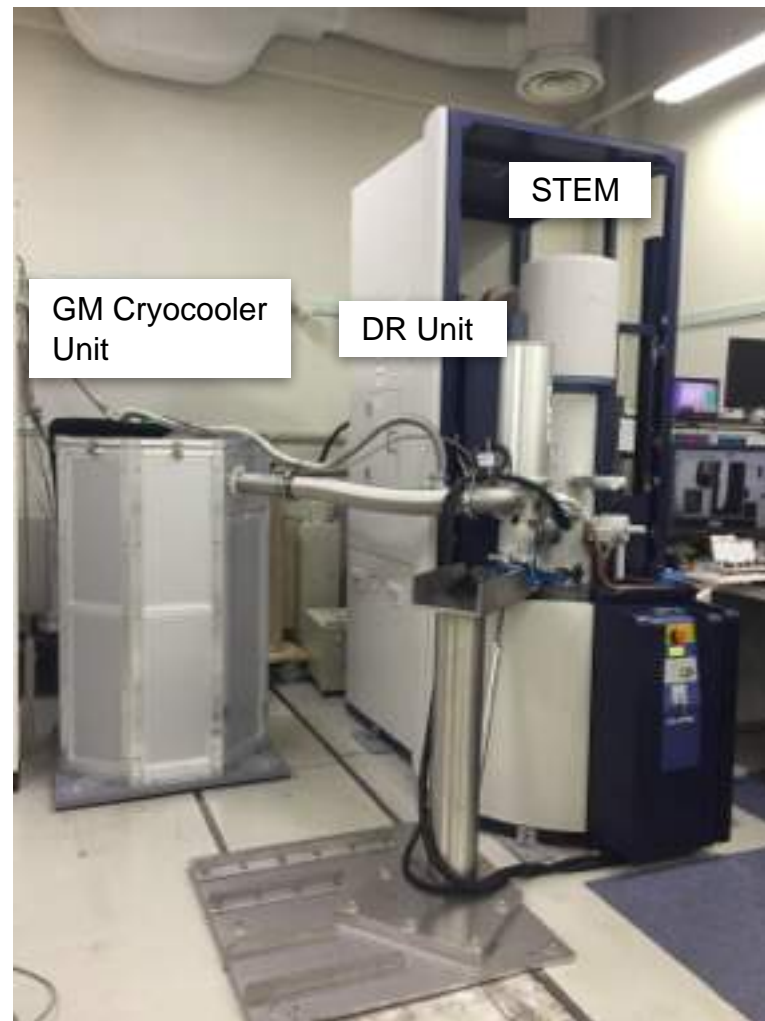
Focus knob select

<input type="radio"/> C1	2.30000	<input type="checkbox"/>	Reset
<input type="radio"/> C2	1.468	<input type="checkbox"/>	
<input checked="" type="radio"/> OBJ	5.87359	<input type="checkbox"/>	
<input type="radio"/> PROJ	1.349	<input type="checkbox"/>	

Protect



Reduction of GM noise



Long term operation > 3 months

Example of TES μ -cal. EDS on STEM

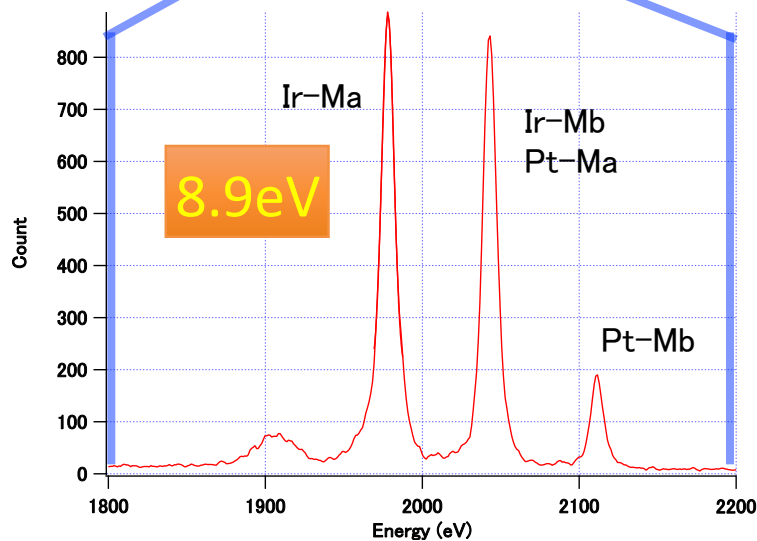
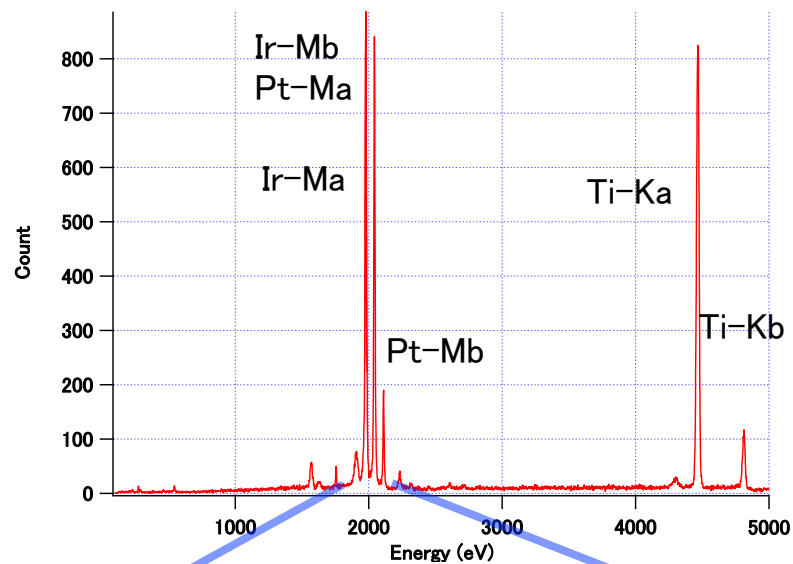
Specimen : $\text{Ti}_{50}\text{Ir}_{12.5}\text{Pt}_{37.5}$



TiIrPt Picture (Detector : TE)

Observation condition

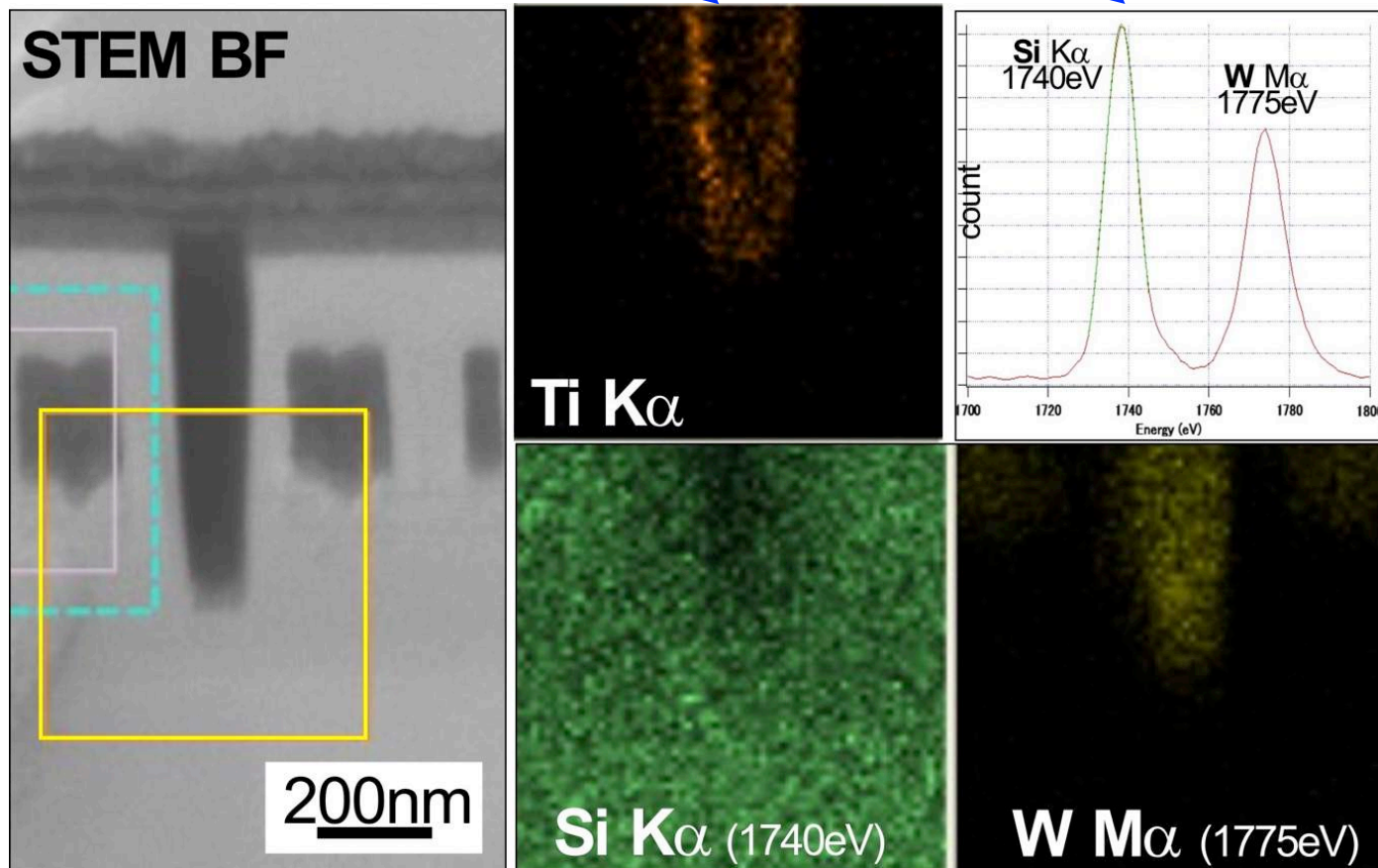
- AP1
- EDX Mode
- Tilt : -30°



Mapping by TES μ -cal. EDS on STEM

Detection of Ti layer of ~ 10 nm wide

$\Delta E = 9.7$ eV FWHM @ Si $K\alpha$



Counting Rate ~ 300 cps \Rightarrow Insufficient

Individual visualization of Si and W



Summary

- ▶ Single pixel TES μ -cal. EDS mounted on STEM
- ▶ Nano-scale STEM image observation with operating DR
- ▶ $\Delta E < 10$ eV in STEM-EDS
- ▶ Element distribution mapping obtained by TES μ -cal. EDS on STEM
- ▶ Insufficient counting rate

Increasing counting rate above 5 kcps \Rightarrow 64 pixel array TES μ -cal. EDS system

In this workshop

- ▶ Fabrication: Muramatsu et al., T1.1
- ▶ Characterization System: Yoshimoto et al., G4.27
- ▶ Probe assembling: Nagayoshi et al. T3.5, G3.33
- ▶ Operation and Readout: Yamamoto et al. T4.3
- ▶ Polycapillary Optics: Takano et al. G4.26

