

# Low temperature irradiation tests on stabilizer materials using reactor neutrons at KUR

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(KEK)

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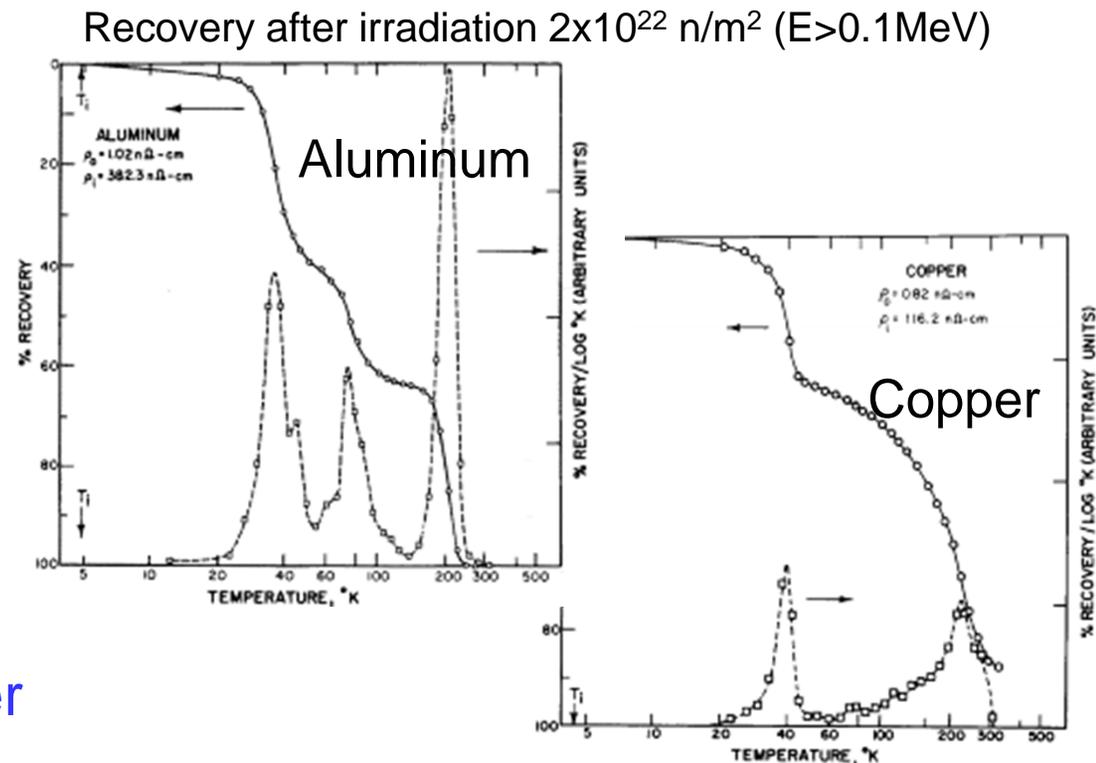
# Irradiation effects on Al, Cu in literature

- pure Al (RRR=2286)
  - Fast neutron  $2 \times 10^{22}$  n/m<sup>2</sup>  
Induces  $\rho_i = 3.8 \text{ n}\Omega \cdot \text{m}$  [1]
    - $\rho_i = 0.02 \text{ n}\Omega \cdot \text{m}$  for  $10^{20}$  n/m<sup>2</sup>
  - **Perfect** recovery by anneal at RT
- pure Cu (RRR=2280)
  - $\rho_i = 1.2 \text{ n}\Omega \cdot \text{m}$  [1]
  - 10% damage remains after annealing at RT

How about cold-worked Al-stabilizer  
→ tests at KUR

Table 3  
Irradiation induced resistivity,  $\rho_i$ , defect concentration,  $C_i$ , and ratio of induced to residual resistivity,  $\rho_i/\rho_0$ .

Element	Induced resistivity, $\rho_i$ (n $\Omega \cdot \text{cm}$ )	Induced concentration a) ( $10^{-4}$ a.f.)	$\rho_i/\rho_0$
Aluminum	382.3	5.6	275
Nickel	363.9	5.6	31
Copper	116.2	4.8	142
Silver	87.9	3.6	54
Gold	102.7	4.0	40
Platinum	264.6	3.6	48
Iron	1137.2	9.1	21
Molybdenum	593.3	6.0	142
Cobalt	794.6	8.0	9



[1] J.A. Horak and T.H. Blewitt, J. Nucl. Materials, Vol. 49 (1973) p161

# Low Temperature Irradiation Facility

[2] M. Okada et al., NIM A463 (2001) pp213-219

- Kyoto Univ. Research Reactor Institute
- 5MW max. thermal power
- Cryostat close to reactor core
- Sample cool down by He gas loop
  - 10K – 20K
- Fast neutron flux
  - Measured by Ni activation in 2010
  - $1.4 \times 10^{15}$  n/m<sup>2</sup>/s (>0.1MeV) @1MW

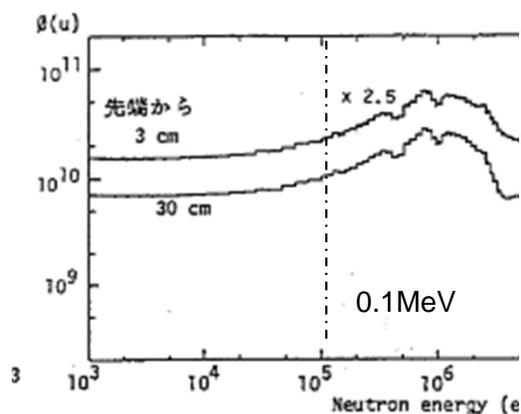
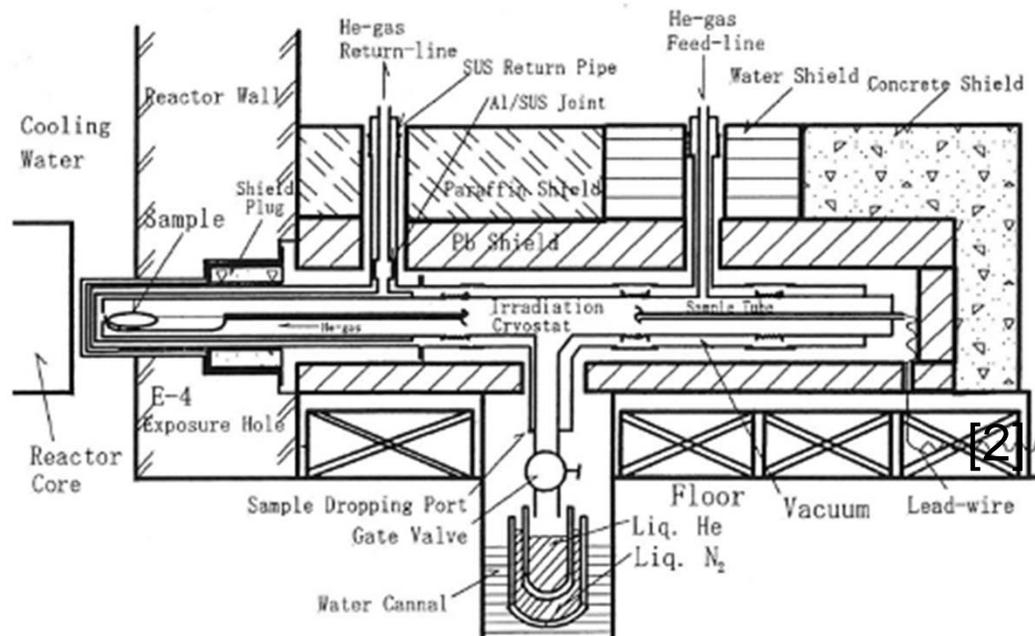
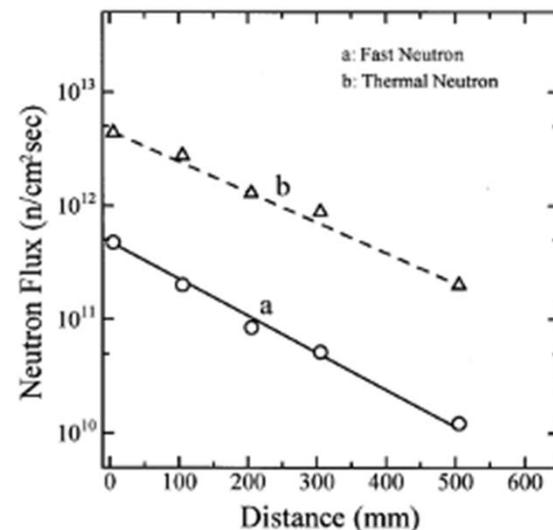


Fig. 15 Neutron energy spectrum in LTL of KUR for ordinary core (above 1000 eV)



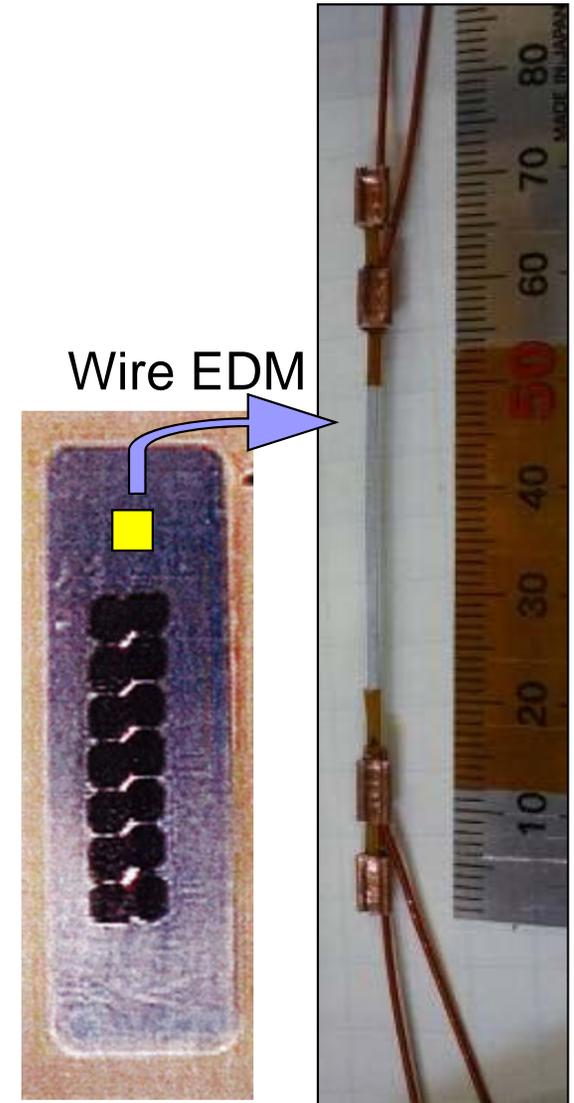
KUR-TR287 ( Fig. 7. Neutron flux distribution as a function of distance from top of sample chamber, (a) fast-neutron and (b) thermal neutron.

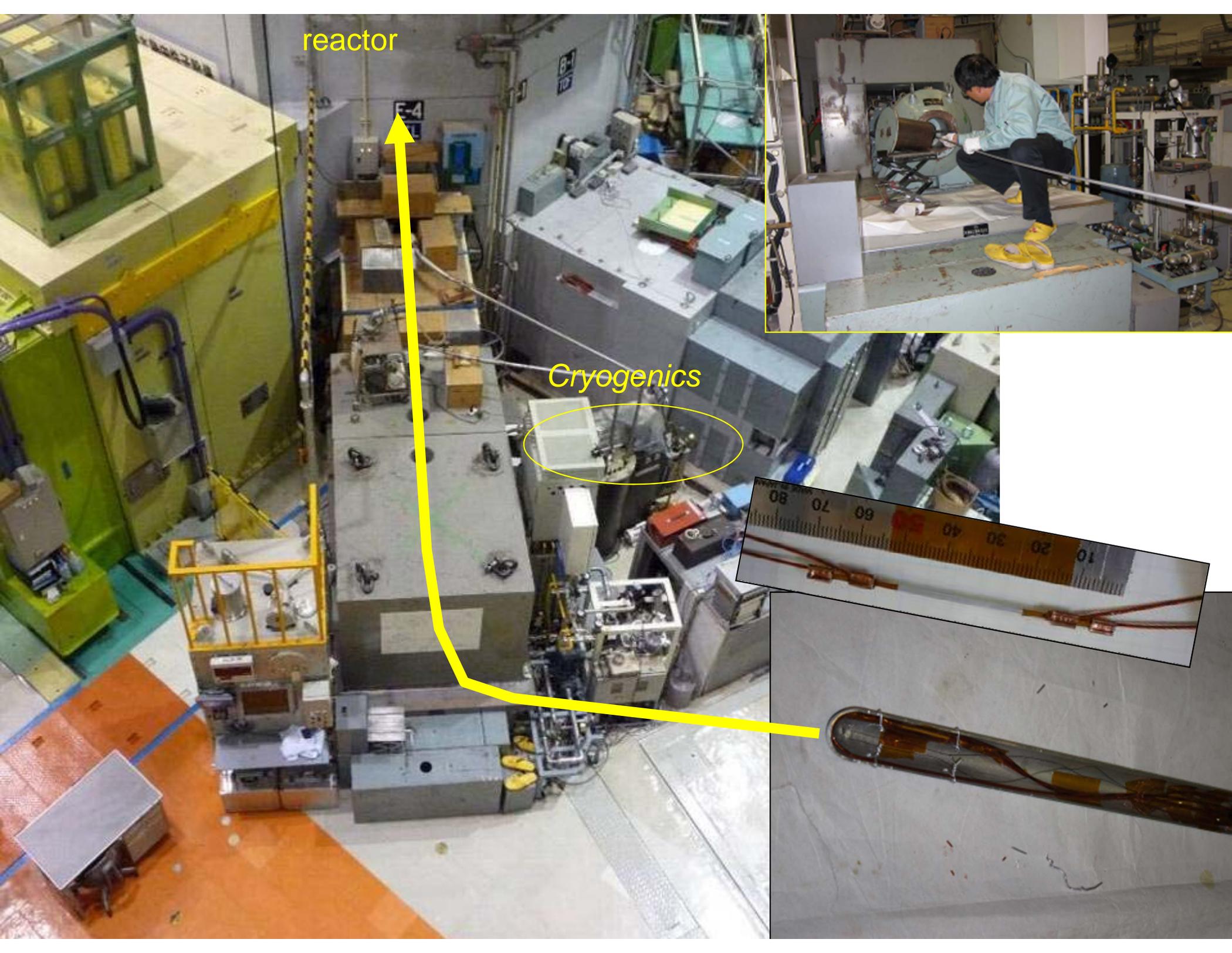
# Irradiation sample

- Aluminum stabilizer sample from the superconductor by **wire electrical discharge machining** in KEK
  - Keep defects by cold-work
- Size: 1mmx1mmx70mm
- Voltage taps with 45mm spacing
- 4 wire resistance measurement by nano-voltmeter
- CERNOX CX-1050-SD close to sample temperature (also irradiated)

## Irradiation sample

- 5N aluminum + Cu, Mg with 10 % cold work
- RRR=450
  - 1.35m $\Omega$  @RT, 3 $\mu\Omega$  @10K





reactor



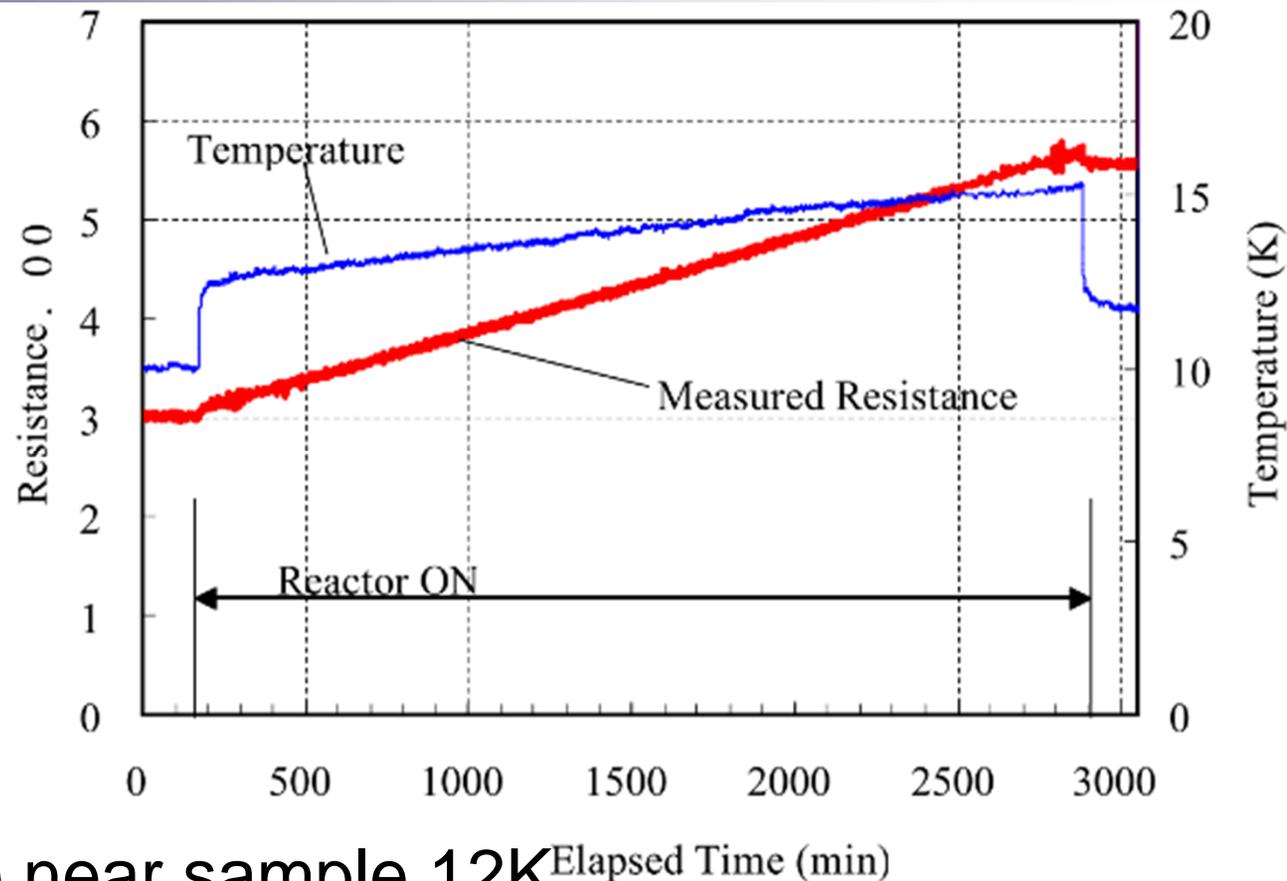
Cryogenics



# Al-Cu-Mg



1mmx1mmx45mm(Vsens)



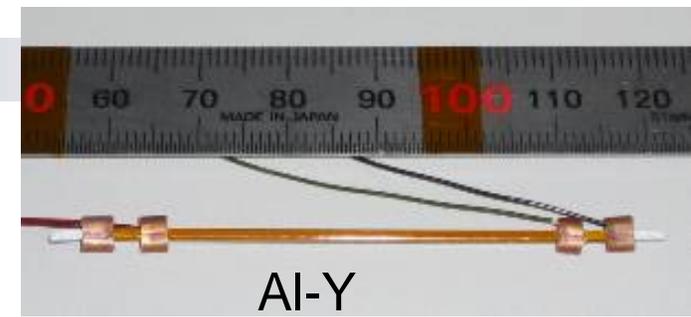
- He gas temperature near sample 12K
- Resistance changes linearly from low dose (No threshold)
  - $3.1\mu\Omega \rightarrow 5.7\mu\Omega$
  - induced  $\rho_i = 0.056 \text{ n}\Omega\cdot\text{m}$  for  $2.3 \times 10^{20} \text{ n/m}^2$  ( $>0.1\text{MeV}$ )
  - As expected (cf. [1]), more due to epithermal neutron?
- In COMET, expected  $\rho_i < 0.2 \text{ n}\Omega\cdot\text{m}$  for  $6 \times 10^{20} \text{ n/m}^2$

Temperature drift 12K→15K due to CERNOX sensor degradation?

# Al-Y, OFHC



OFHC  
 $\phi 1\text{mm} \times 35\text{mm}(\text{Vs})$   
RRR=311



Al-Y  
 $1\text{mm} \times 1\text{mm} \times 45\text{mm}(\text{Vs})$   
RRR=334,361

## ■ Neutron fluence

- $2.6 \times 10^{20}$  n/m<sup>2</sup> in  
1MWx52hrs

## ■ Al-Y1

- $4.0\mu\Omega \rightarrow 7.3\mu\Omega$
- $\rho_i = 0.07 \Omega\text{m}$

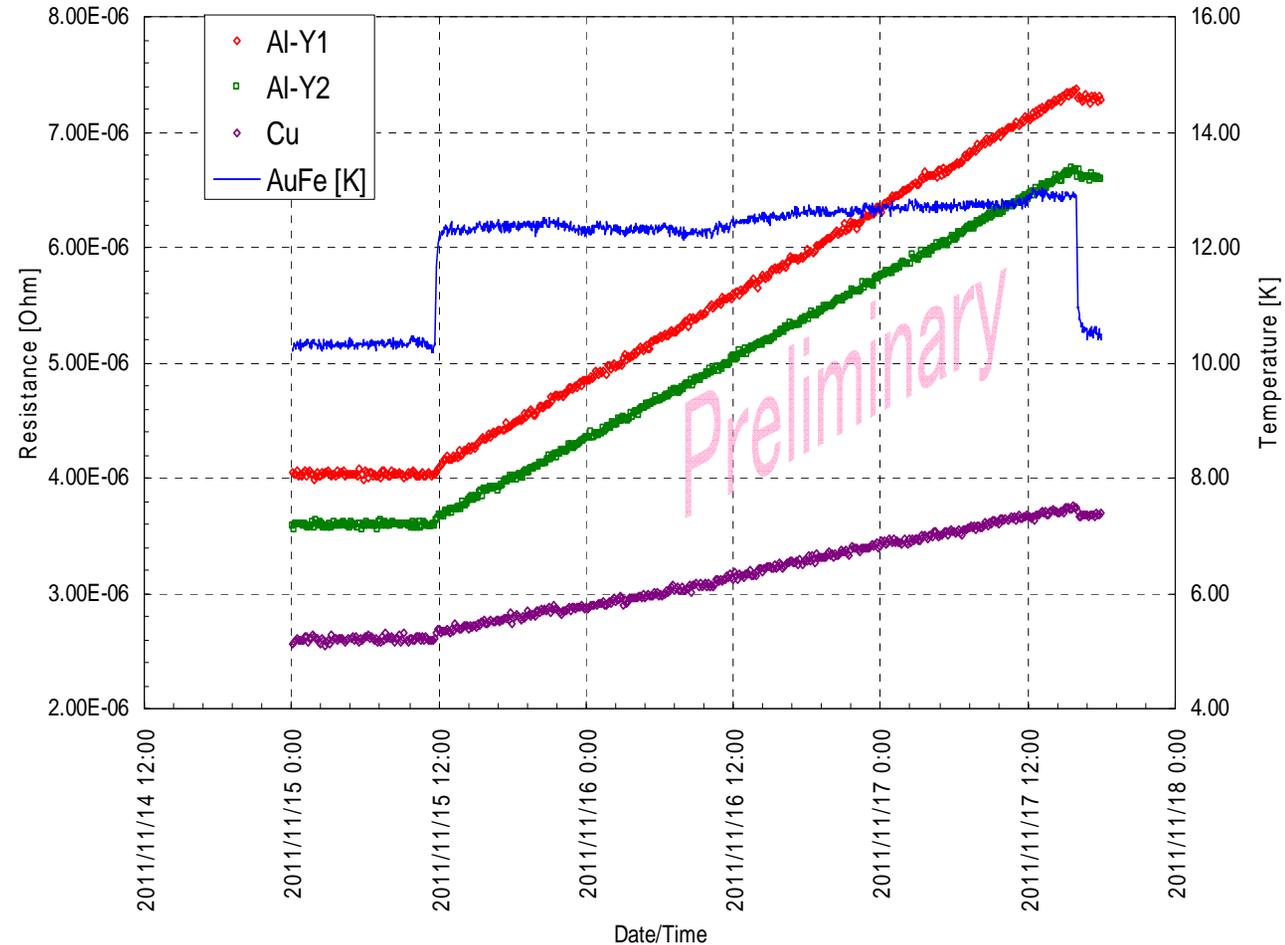
## ■ Al-Y2

- $3.6\mu\Omega \rightarrow 6.6\mu\Omega$
- $\rho_i = 0.07 \Omega\text{m}$

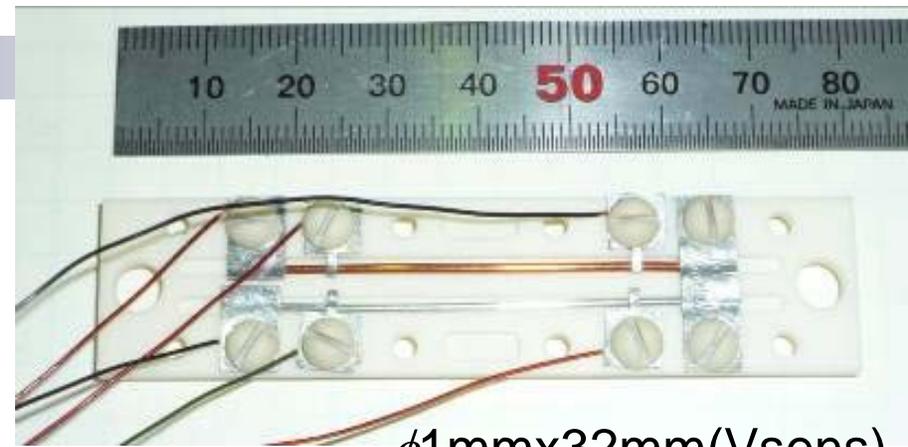
## ■ OFHC

- $2.6\mu\Omega \rightarrow 3.7\mu\Omega$
- $\rho_i = 0.03 \Omega\text{m}$

KUR2011Nov



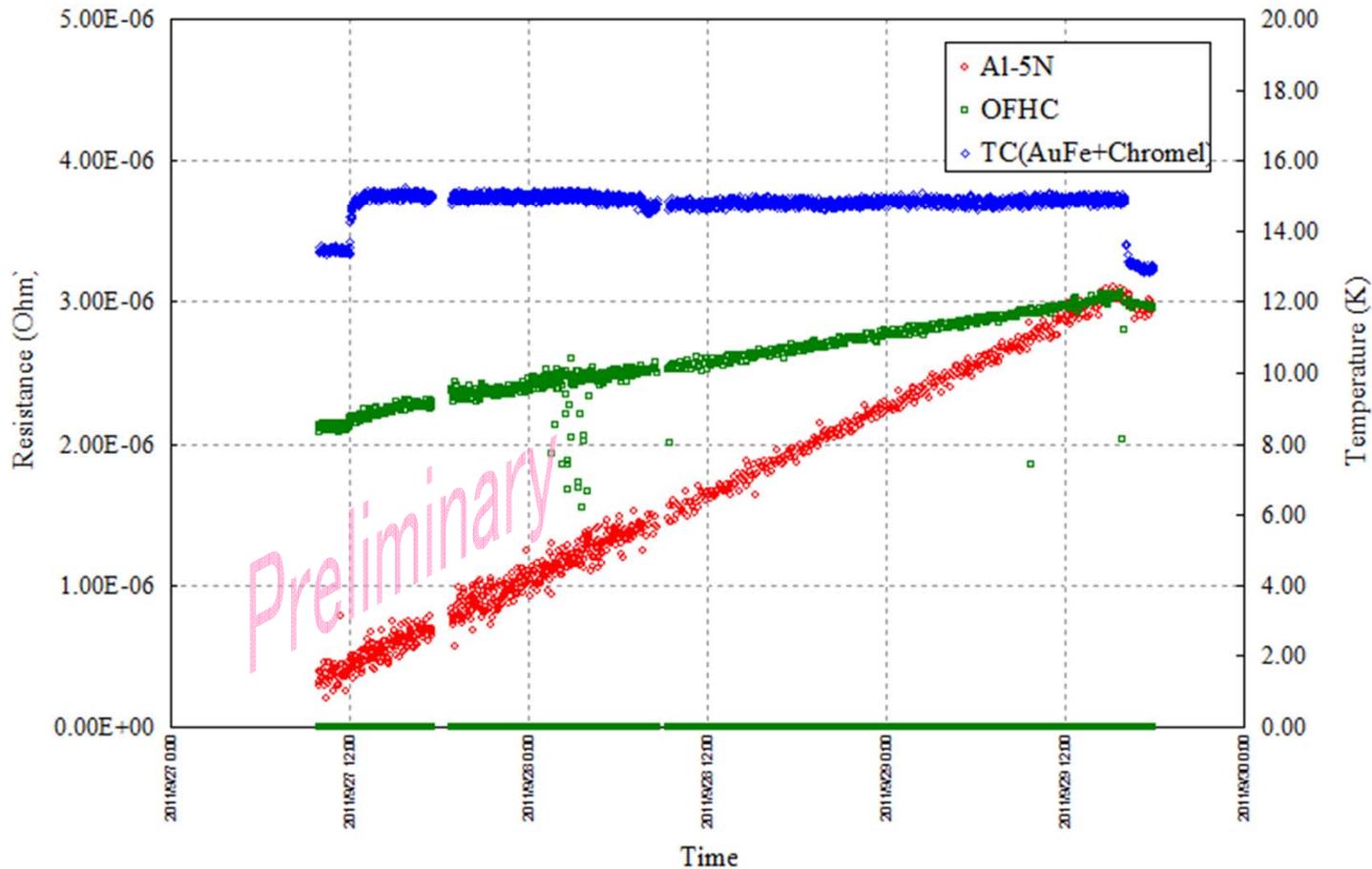
# OFHC, 5N AI



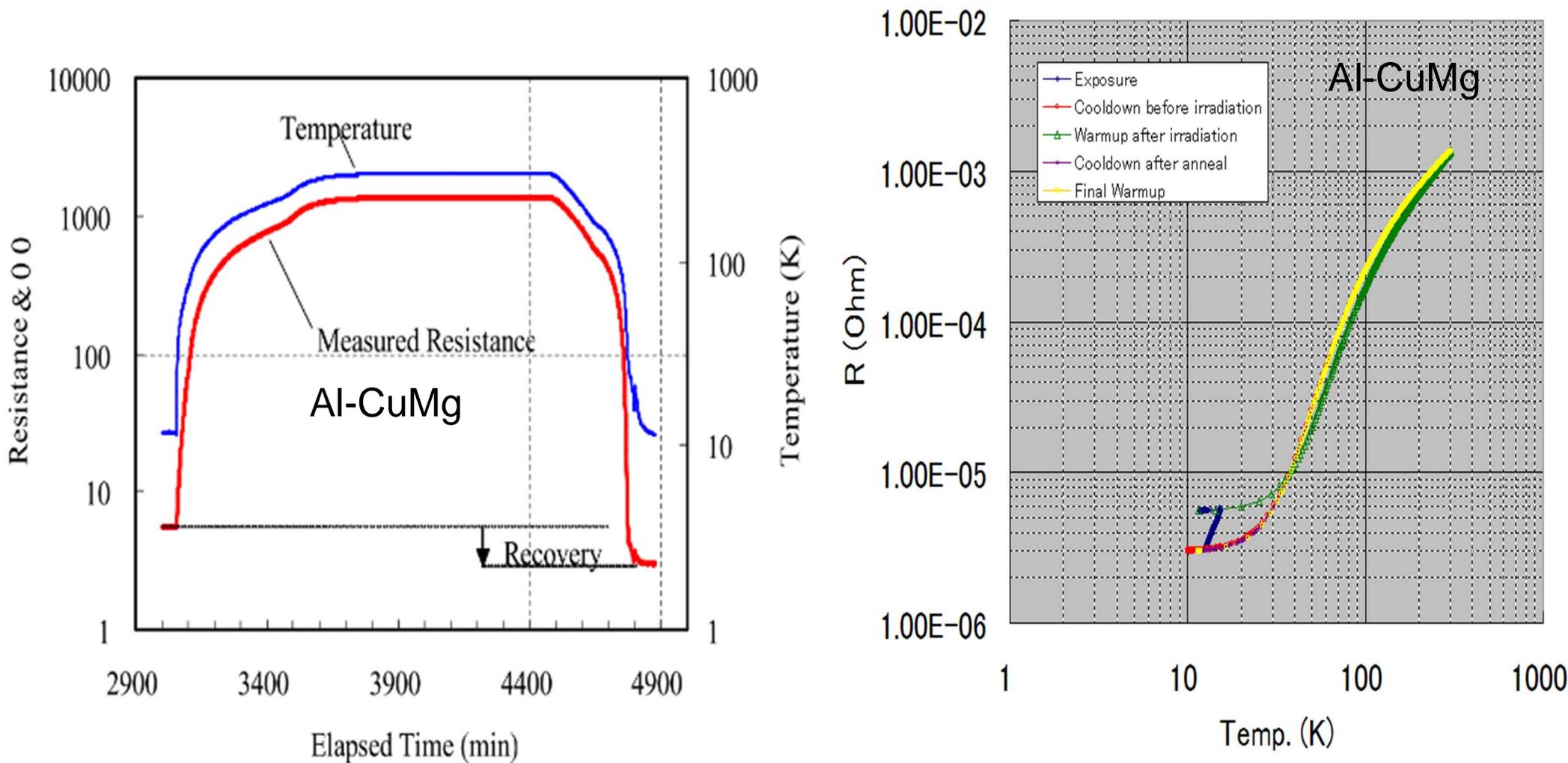
$\phi 1\text{mm} \times 32\text{mm}$  (Vsens)  
5N-AI: RRR~3000

OFHC: RRR~300

2011Sep

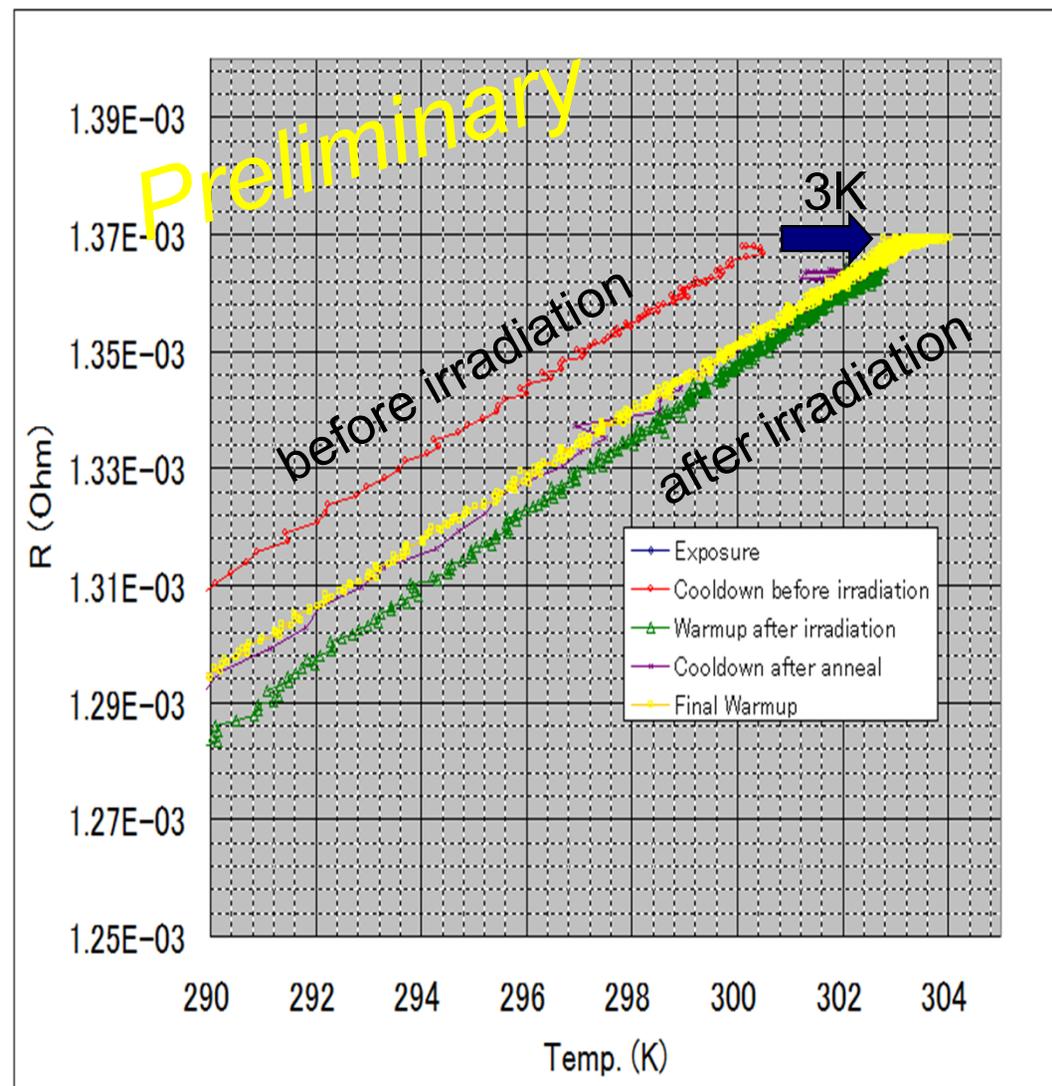
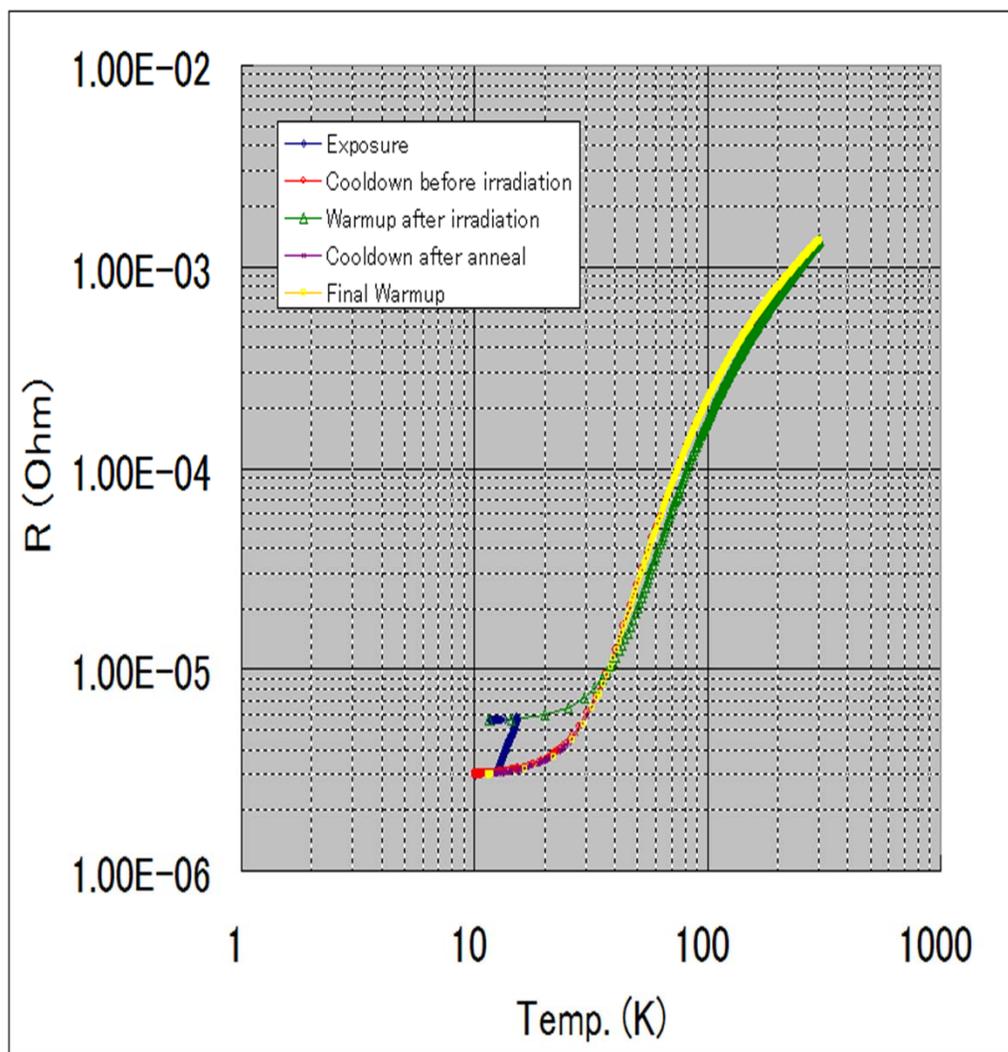


# Annealing at Room Temperature



- Al-CuMg recovered perfectly!

# CERNOX degradation?



- $dR/dT = -0.26 \Omega/K @ 300K$
- $dT = 3K \rightarrow dR = 0.78 \Omega$

# CERNOX sensor

- Lakeshore CERNOX sensor

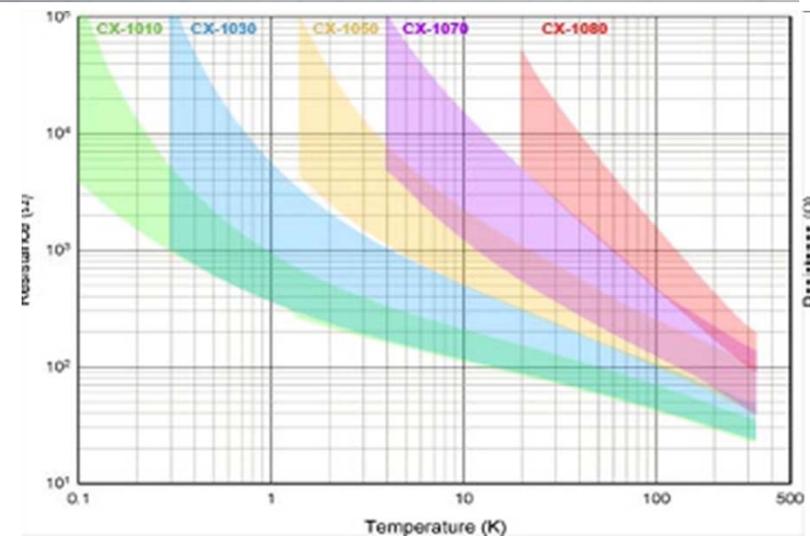
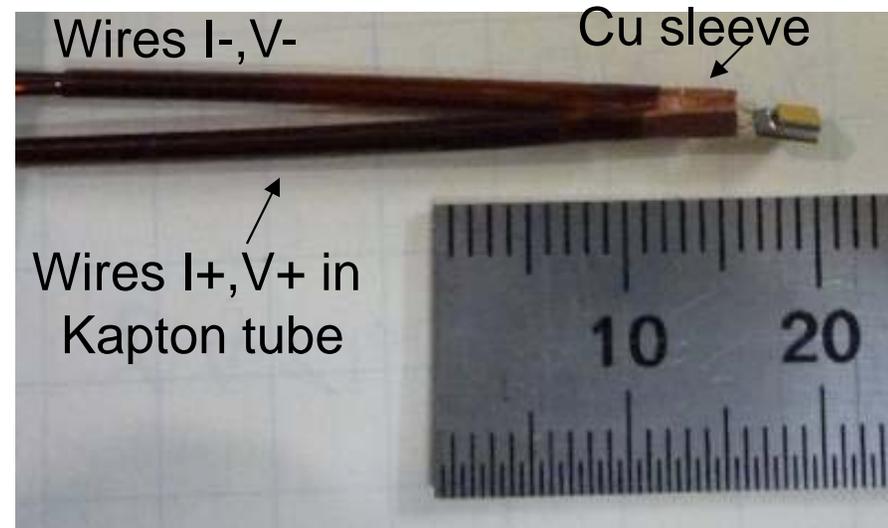
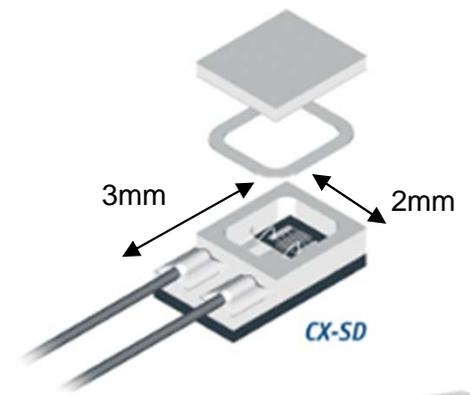
- sputtered zirconium oxy-nitride thin films

SS. Courts and P.R. Swinehart, Advances in Cryogenic Engineering, Vol. 45

- Tested at  $10^{19}$  n/m<sup>2</sup> for LHC

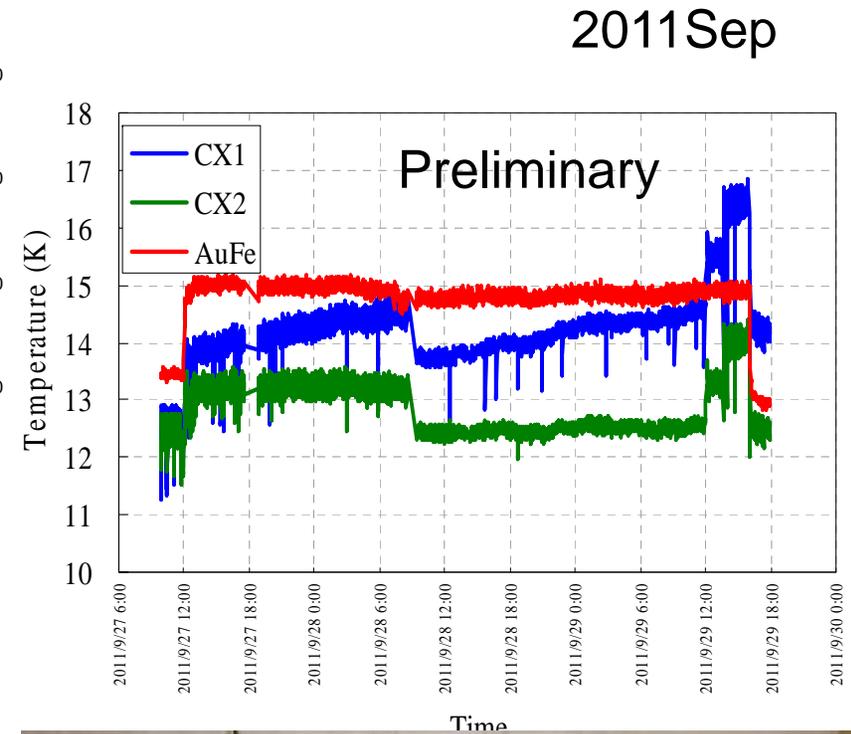
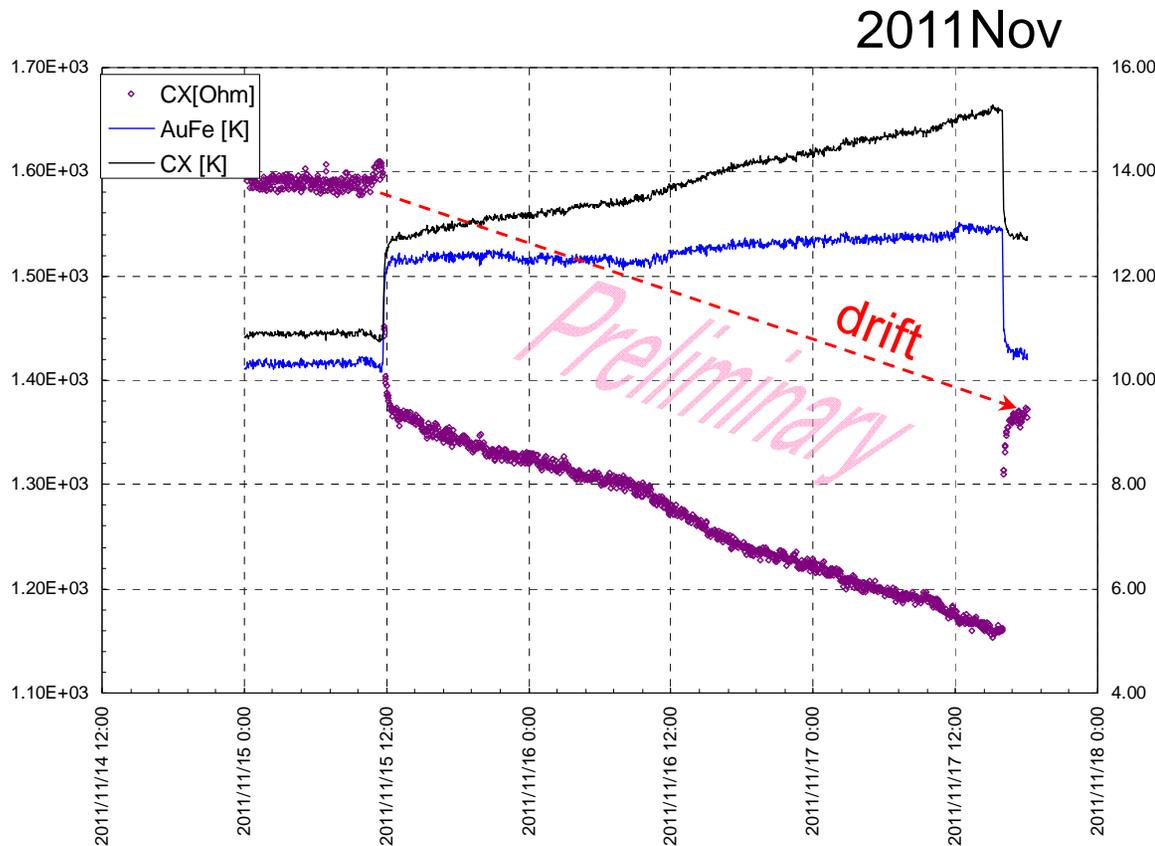
- Drift ~ 2mK@1.8K

LHC Project Report 209



<http://www.lakeshore.com/temp/sen/crtd.html>

# Drift of CERNOX sensor readout



CERNOX drift  $1.6\text{k}\Omega \rightarrow 1.4\text{k}\Omega$  for  $2.6 \times 10^{20} \text{ n/m}^2$   
Irradiation effect? Self heating?

# Summary

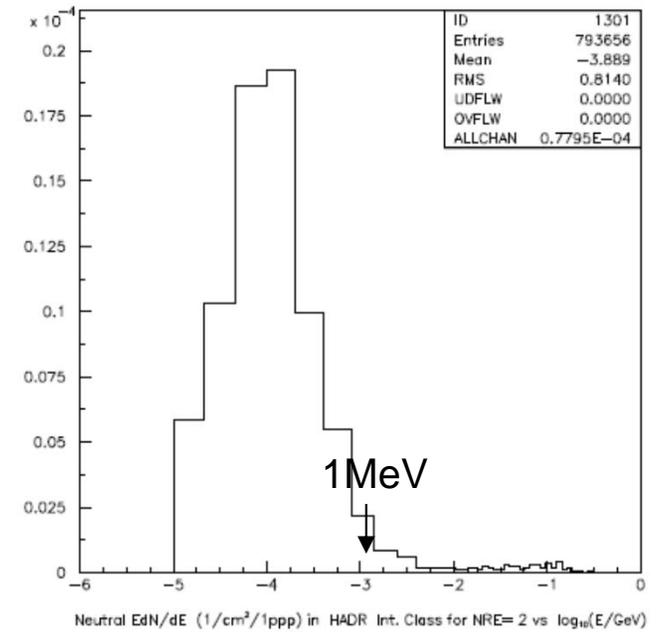
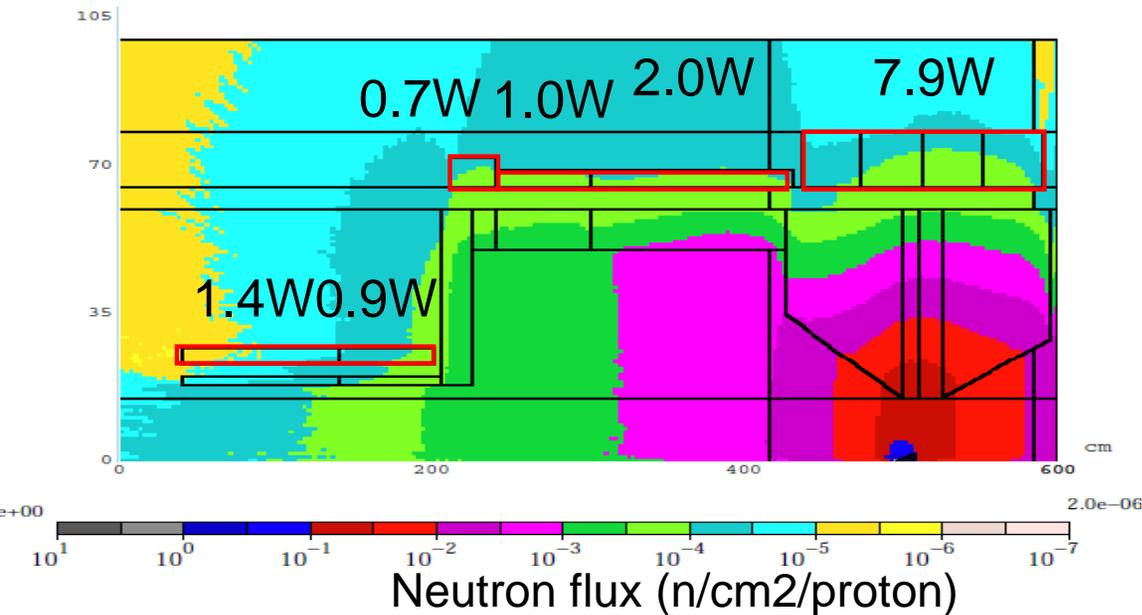
- Stabilizer materials were tested by KUR neutrons
  - Al-CuMg, Al-Y, OFHC
  - Recovery of Al-CuMg has been observed
  - Cycle irradiation effect on Cu will be investigated
- Induced resistance is slightly larger than expected
  - epithermal neutron irradiation? ← Dr. Iwamoto
  - gamma ray?
- CERNOX seems to degrade at  $10^{20}$  n/m<sup>2</sup> in temperature range of 10K-20K
  - Need to check recovery by thermal cycle to RT





# Backup

# Radiation on CS



- Maximum heat deposit
  - 10 mW/kg
- Maximum dose
  - 0.07 MGy/10<sup>21</sup>p
- Neutron flux
  - 1x10<sup>21</sup> n/m<sup>2</sup>/10<sup>21</sup>p
  - fast neutrons 6x10<sup>20</sup> n/m<sup>2</sup>/10<sup>21</sup>p (>0.1MeV)

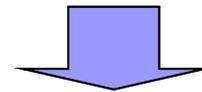
Neutrons penetrates thick 45cm tungsten shield surrounding the target

Neutron fluence for experimental life-time (~10<sup>21</sup> p) approaches a level of ITER magnets (ITER requirement: 10<sup>22</sup> n/m<sup>2</sup>)

# Problematic components

- **Stabilizer**
  - Aluminum alloy
  - Copper
- **Thermal conductor**
  - Pure aluminum
  - Copper
  - Aluminum alloy
- **Thermo sensor**
  - No experience at  $10^{21}$  n/m<sup>2</sup>

- Fast-neutron irradiation induces defects in metal.
- Defects could be accumulated at **Low temperature**,
- and causes degradation of electrical/thermal conductivity



- **Problems in**
  - Quench protection, Stability
  - Cooling

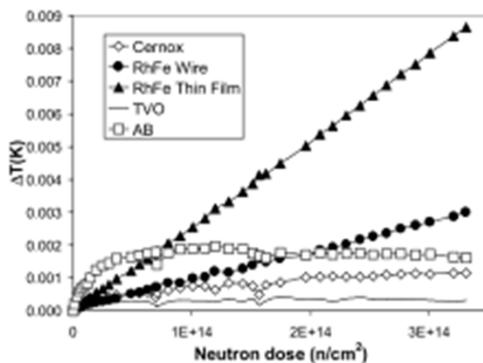
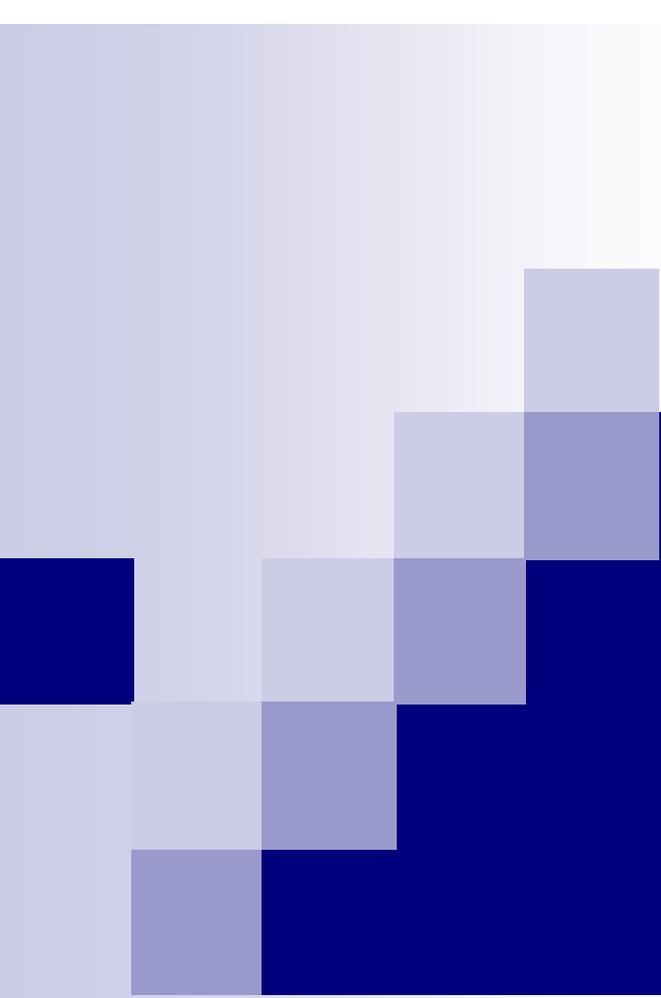


Figure 3 Error on temperature measurement on some sensors during irradiation ( $T_{\text{bath}}=1.8$  K)



# Neutron Flux at KUR-LTL

Makoto Yoshida (KEK)

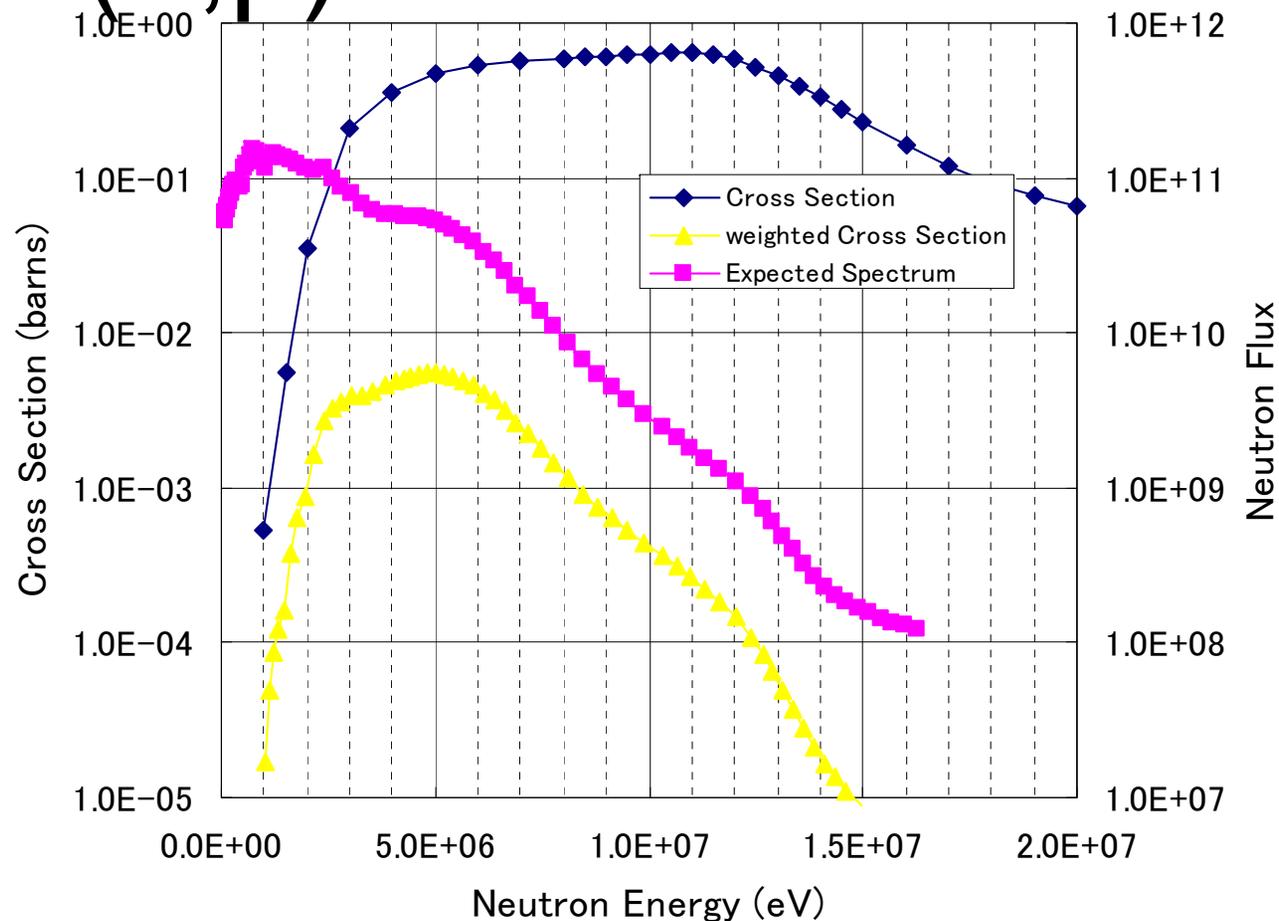
2011/5/25

# Ni activation measurement

- Ni foil (10 mg) was irradiated near aluminum sample in LTL sample chamber in Nov. 2010
  - 1 MW x 45 hours
- After 131 day cooling down, gamma ray from Co58 was measured
- $\text{Ni58}(n,p)\text{Co58}$  reaction rate during neutron exposure was calculated to be  $1.55\text{E-}14 \text{ atm}^{-1}\text{sec}^{-1}$
- Neutron spectrum in LTL sample chamber with old reactor core setup was measured in 1987 [1]

[1] "Measurement of Neutron Spectrum at Irradiation Facilities of Kyoto University Reactor, KUR," K. Kobayashi et al., KURRI Technical Report 287 (1987)

# Ni58(n,p)Co58 cross section



- Average cross section in reactor neutron spectrum  
0.194 barns (>1MeV)
- Average down to 0.1 MeV, although the reaction Ni58(n,p)Co58 is sensitive to energy range above a few MeV ...  
0.108 barns (>0.1MeV)

# Neutron fluence

- Fast neutron fluence  $> 1$  MeV
  - $45 \text{ [hours]} \times 1.55\text{E-}14 \text{ [atm}^{-1}\text{sec}^{-1}] / 0.194 \text{ [barns]}$   
 $= 45\text{[hours]} \times 7.990 \times 10^{14} \text{ [n/m}^2\text{/s]}$   
 $= 1.3 \times 10^{20} \text{ [n/m}^2]$
- Fast neutron fluence  $> 0.1$  MeV
  - $45 \text{ [hours]} \times 1.55\text{E-}14 \text{ [atm}^{-1}\text{sec}^{-1}] / 0.108 \text{ [barns]}$   
 $= 45\text{[hours]} \times 1.435 \times 10^{15} \text{ [n/m}^2\text{/s]}$   
 $= 2.3 \times 10^{20} \text{ [n/m}^2]$

\*assuming reactor neutron spectrum measured in [1]