

Neutron Irradiation Measurement for Superconducting Magnet Materials at Low Temperature

Tatsushi NAKAMOTO

KEK

Collaborators/Supporters

[KEK] M. Yoshida, M. Sugano, M. Iio, S. Mihara,
H. Nishiguchi, K. Yoshimura, T. Ogitsu, A. Yamamoto,

[Osaka Univ.] A. Sato, M. Aoki, T. Itahashi, Y. Kuno,

[KUR] Q. Xu, K. Sato, T. Yoshiie, Y. Kuriyama, Y. Mori,

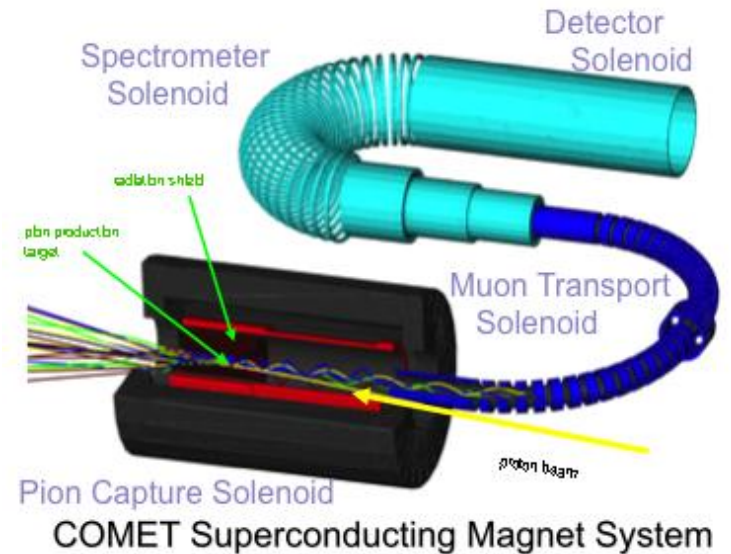
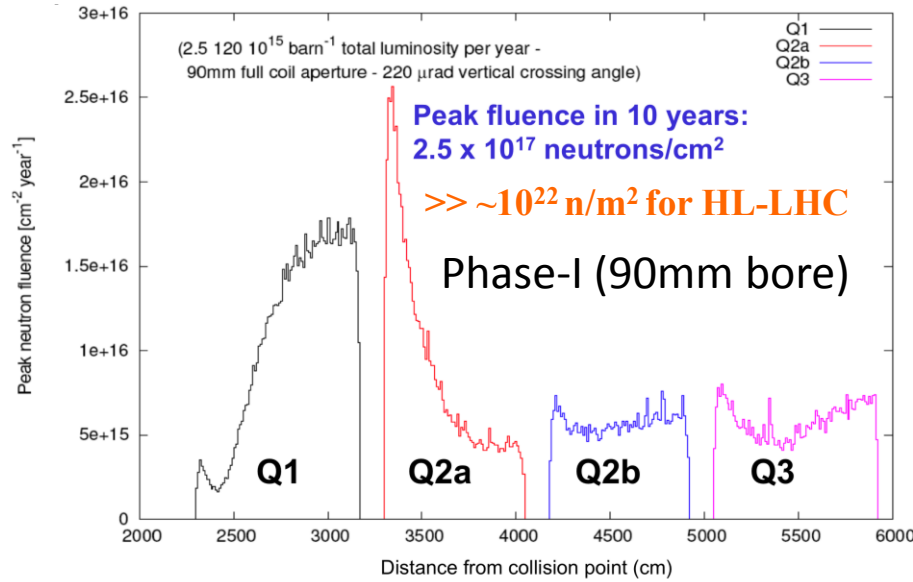
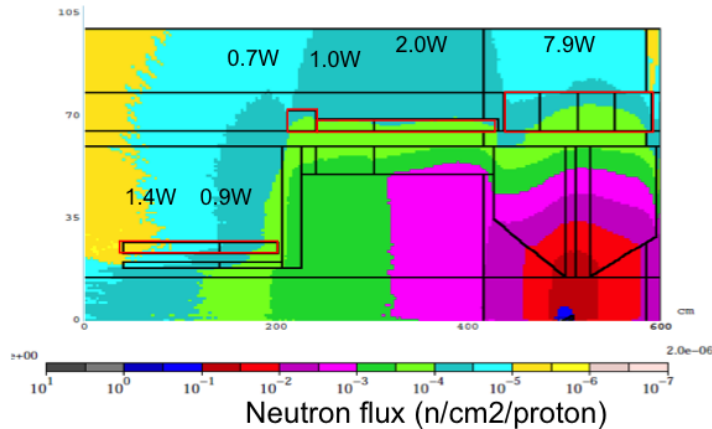
[Fermilab] M. Lamm and Mu2e collaboration

[CERN] G.D. Rijk, E. Todesco, L. Bottura, L. Rossi.

High Radiation Environment for SC

Magnets

- HiLumi LHC: 10^{21} - 10^{22} n/m²
- COMET & Mu2e experiments (J-PARC, Fermilab)
 - Search for μ -e conversion
 - Pion capture solenoids w/ Al stabilized NbTi SC cable.
 - Same spec as ATLAS-CS.
 - Neutron fluence: $> 10^{21}$ n/m²



Neutron irradiation effects at low temperature need be studied.

Why ρ of stabilizer? Why at low temperature?

- **Electrical resistivity ρ** of stabilizers (aluminum, copper) is one of the most sensitive property in the SC magnet materials with respect to the radiation.
 - Induced resistivity is remarkable at LT.
 - Recovery effect starts at 20 K or higher.
- The induced ρ by the radiation will **compromise the quench stability and protection scheme**. Coil temperature will be increased in the in-direct cooling magnets.
- Anneal effect and full-recovery during warm-up to RT would be expected in aluminum, but only **80-90% recovery in copper (??)**.
- Questions to be studied:
 - Samples from the practical SC wire/cable: RRR of 100 to 500.
 - Degradation may start even below 10^{20} n/m² ?
 - Fluence threshold ?
 - Full recovery by full thermal cycle?
 - Accumulated resistivity after multiple irradiation?

Previous Work in Literature: $\Delta\rho_{irr}$

Neutron irradiation at 4K, and warm-up stepwise.

Horak et.al., J. Nucl. Materials, 49, p161 (1973&74)

Guinan et.al., J. Nucl. Materials, 133&134, p357 (1985)

Reactor n
on Al

ρ_0 : 0.0102
 $\Delta\rho_{irr}$: 3.823
(n Ω m)

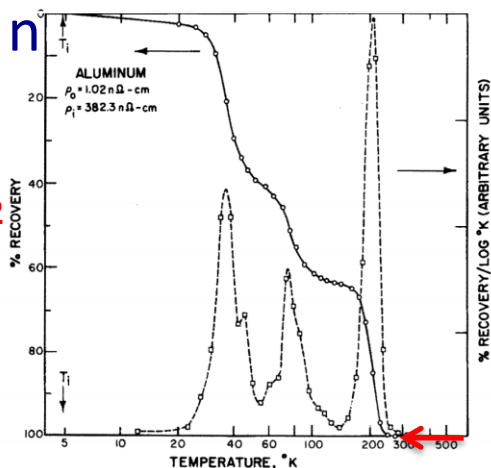
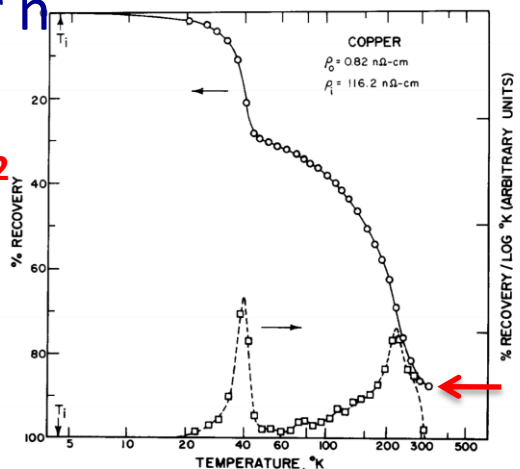


Fig. 3. Recovery and differential recovery versus logarithm of absolute temperature for aluminum irradiated at 4.5 K to 2×10^{18} n/cm² of $E > 0.1$ MeV.

Reactor n
on Cu

ρ_0 : 0.0082
 $\Delta\rho_{irr}$: 1.162
(n Ω m)



90% recovery

Fig. 5. Recovery and differential recovery versus logarithm of absolute temperature for copper irradiated at 4.5 K to 2×10^{18} n/cm² of $E > 0.1$ MeV.

- fluence up to 2×10^{22} n/m² (En>0.1MeV)
- RRR of ~2000

14MeV n
on Al

ρ_0 : 0.386
 $\Delta\rho_{irr}$: 0.336
(n Ω m)

14MeV n
on Cu

ρ_0 : 0.098
 $\Delta\rho_{irr}$: 0.191
(n Ω m)

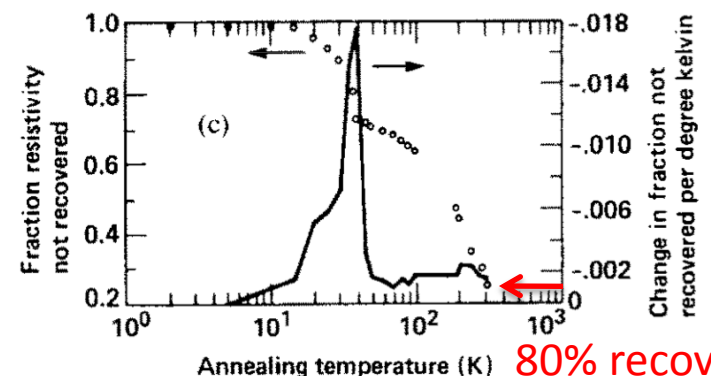
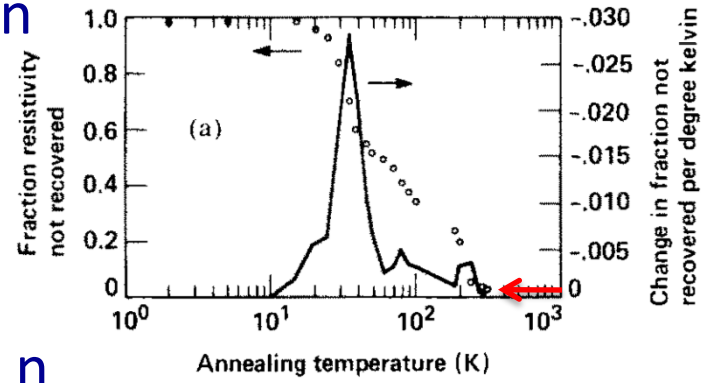


Fig. 2. Post-irradiation, isochronal annealing results for (a) Al, (b) Ni, (c) Cu and (d) Pt. Annealing results below 50 K for Ni and Pt were lost because of warming.

- fluence up to 1×10^{21} n/m²
- RRR of ~100

- Double of resistivity observed at 10^{21} n/m².
- Full recovery in Al expected by T.C.
- Degradation in Cu will be accumulated even after T.C.

Neutron Irradiation at KUR

- Kyoto Univ. Research Reactor Institute
- 5MW max. thermal power
- Irradiation cryostat close to reactor core
- Sample cool down by He gas loop: 10K – 20K
- Fast neutron flux ($E_n > 0.1\text{MeV}$): $1.4 \times 10^{15} \text{ n/m}^2/\text{s}$ @ 1MW

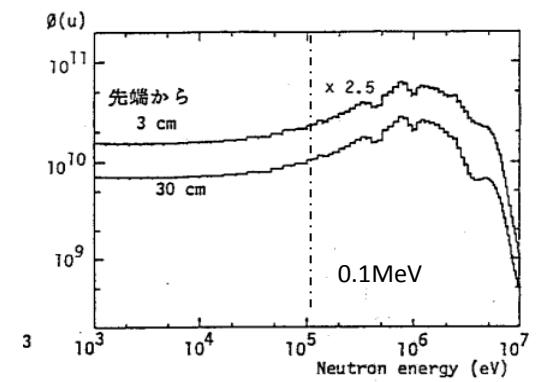
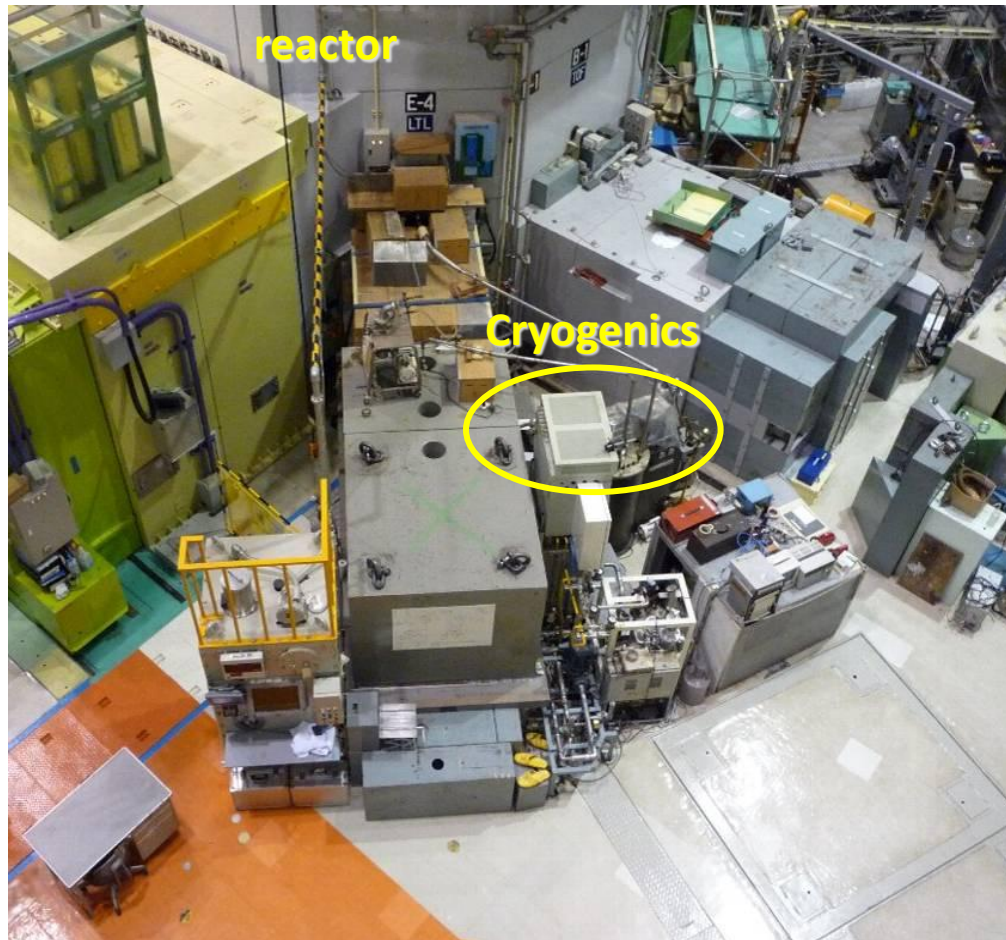
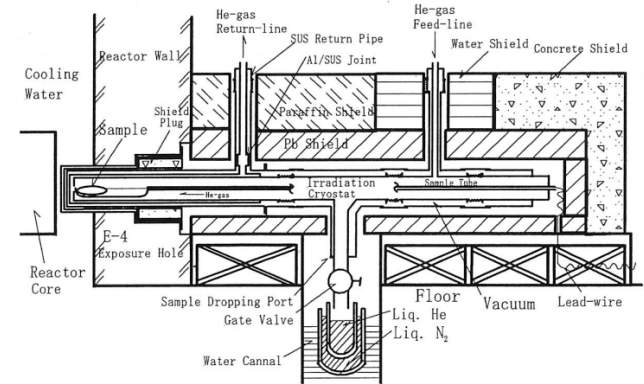


Fig. 15 Neutron energy spectrum in LTL of KUR for ordinary core (above 1000 eV) KUR-TR287 (1987)

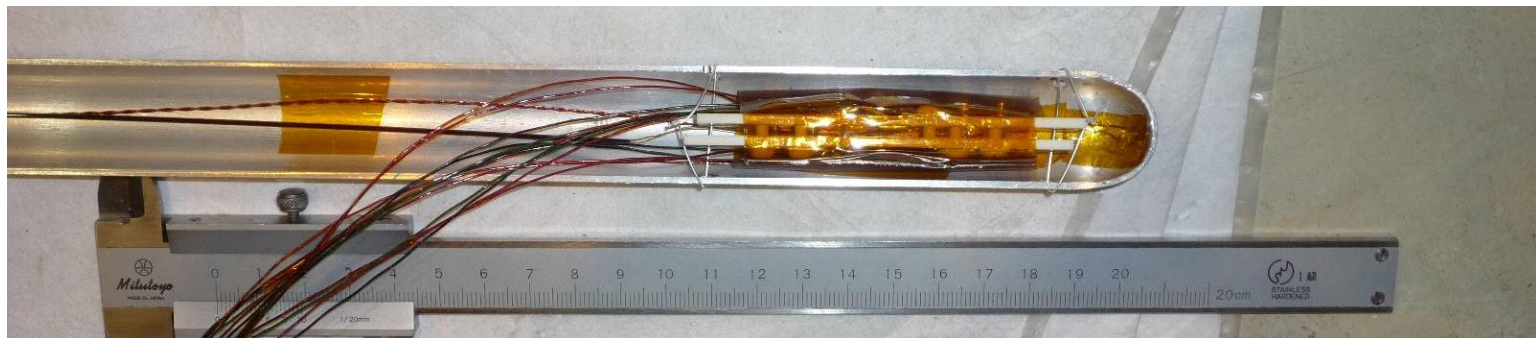
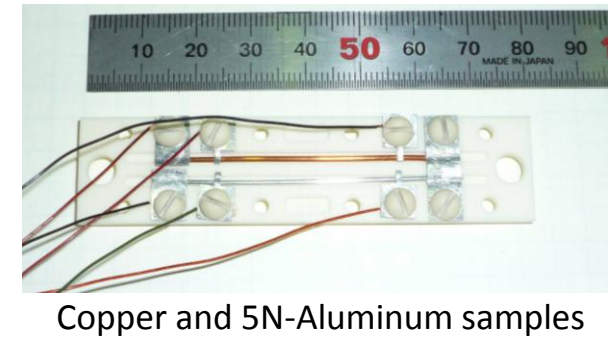
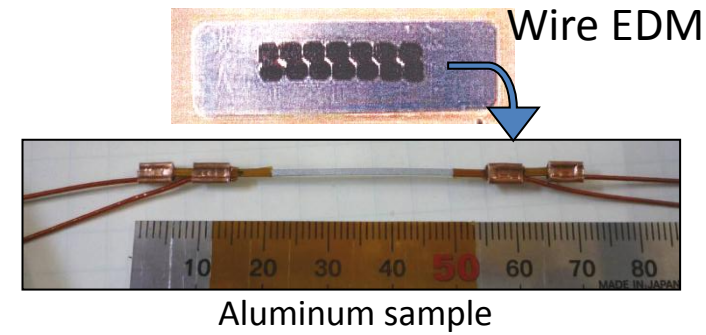


M. Okada et al., NIM A463 (2001) pp213-219



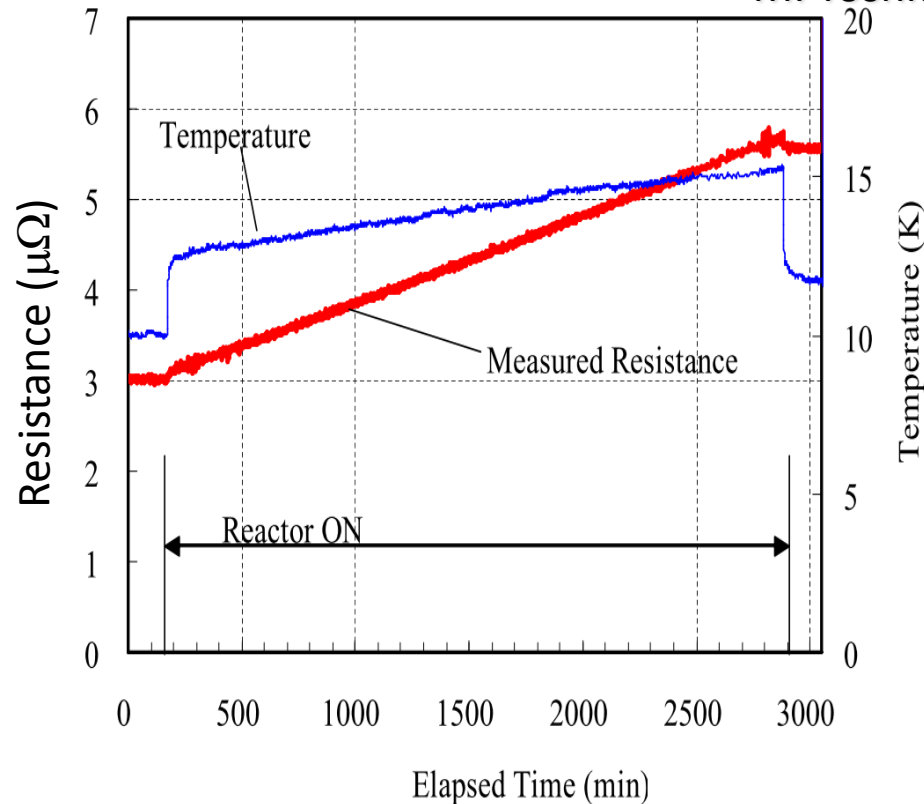
Sample and Measurement

- Aluminum:
 - Cut by EDM from Al stabilized NbTi cable.
 - 5N Al + Cu(20ppm), Mg(40ppm) with 10% cold work. RRR of ~500.
 - 1mmx1mmx70mm, $L_{v-taps} = 45$ mm
- Copper:
 - Provided by Hitachi Cable. Material for SC wire. RRR of ~300.
 - $\phi 1\text{mm} \times 60\text{mm}$, $L_{v-taps} = 32$ mm
- 5N Aluminum (for reference):
 - Provided by Sumitomo Chemical. RRR of ~3000.
 - $\phi 1\text{mm} \times 60\text{mm}$, $L_{v-taps} = 32$ mm
- 4 wire resistance measurement by nano-voltmeter: Keithley 6221+2182A
- Thermometers: CERNOX CX-1050-SD, TC (AuFe-Chromoel)
- Neutron fluence determined by Ni foil activation method.



Result: $\Delta\rho_{\text{irr}}$ for Aluminum

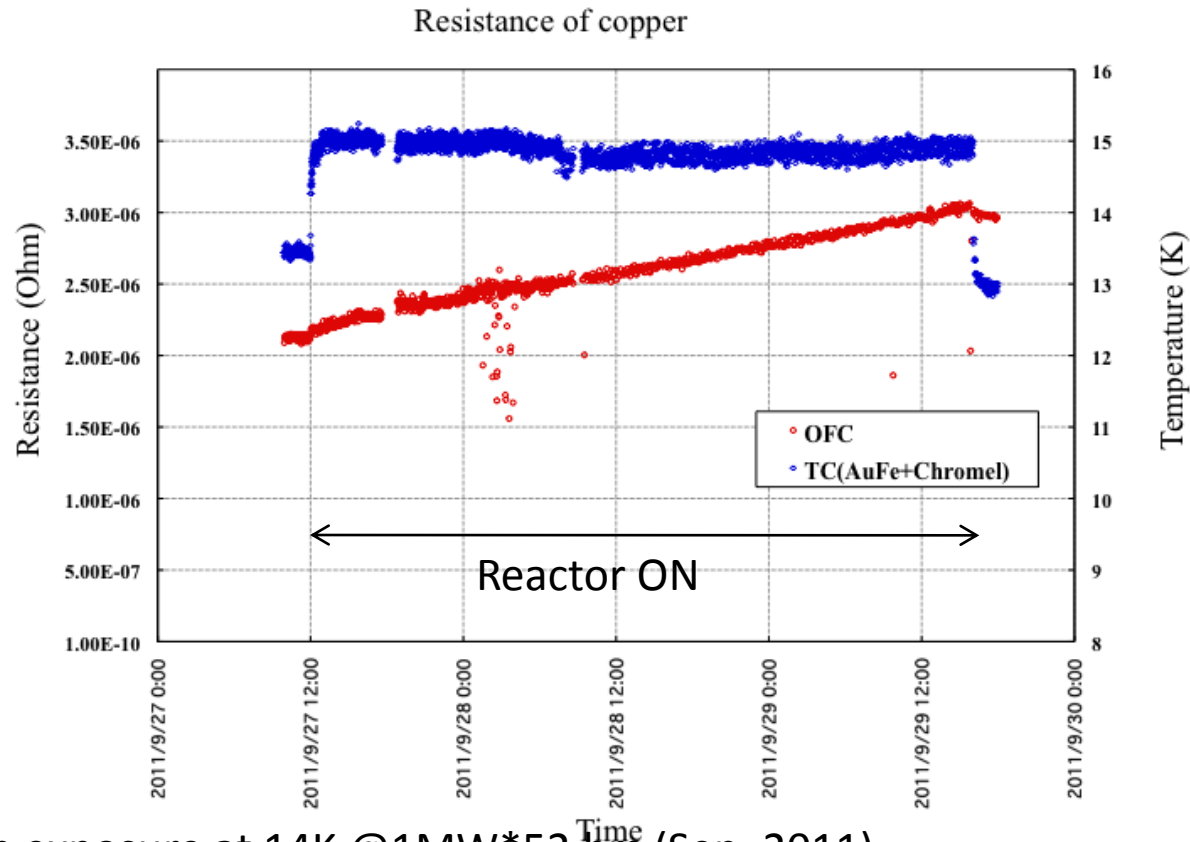
M. Yoshida et al., ICMC2011



- Fast neutron exposure at 12K @1MW*45 hrs (Nov. 2010)
- Resistance was measured *in situ*.
- Resistance increased in proportional to neutron fluence in the range of 10^{19} - 10^{20} n/m²
 - No threshold at low neutron fluence
- Observed $\Delta\rho_{\text{irr}} = 0.056$ n Ω .m for 2.3×10^{20} n/m² (>0.1MeV)
 - Fairly good agreement with the previous work.
 - Present work: $\Delta\rho_{\text{irr}} / \Phi_{\text{tot}} = 2.4 \times 10^{-22}$ n Ω m³ (RRR 500, 2.3×10^{20} n/m²)
 - Previous: $\Delta\rho_{\text{irr}} / \Phi_{\text{tot}} = 1.9 \times 10^{-22}$ n Ω m³ (RRR 2000, 2×10^{22} n/m²)

Result: $\Delta\rho_{irr}$ for Copper

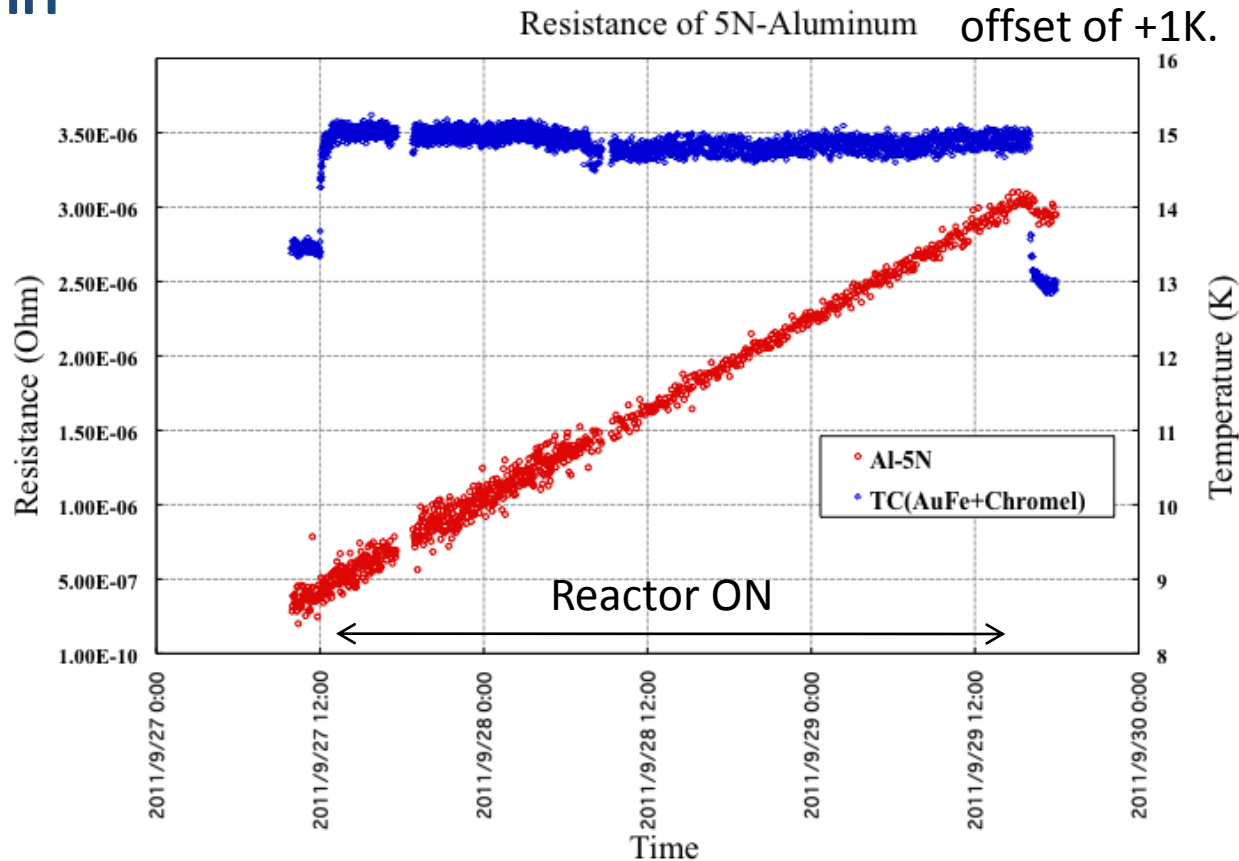
*TC reading includes the offset of +1K.



- Fast neutron exposure at 14K @1MW*52 hrs (Sep. 2011)
- Resistance increased in proportional to neutron fluence in the range of 10^{19} - 10^{20} n/m²
 - No threshold at low neutron fluence
- Observed $\Delta\rho_{irr} = 0.022$ n Ω .m for 2.7×10^{20} n/m² (>0.1MeV)
 - Agreed with the previous work within a factor of 2.
 - Present work: $\Delta\rho_{irr} / \Phi_{tot} = 0.82 \times 10^{-22}$ n Ω m³ (RRR 300, 2.7×10^{20} n/m²)
 - Previous: $\Delta\rho_{irr} / \Phi_{tot} = 0.58 \times 10^{-22}$ n Ω m³ (RRR 2000, 2×10^{22} n/m²)

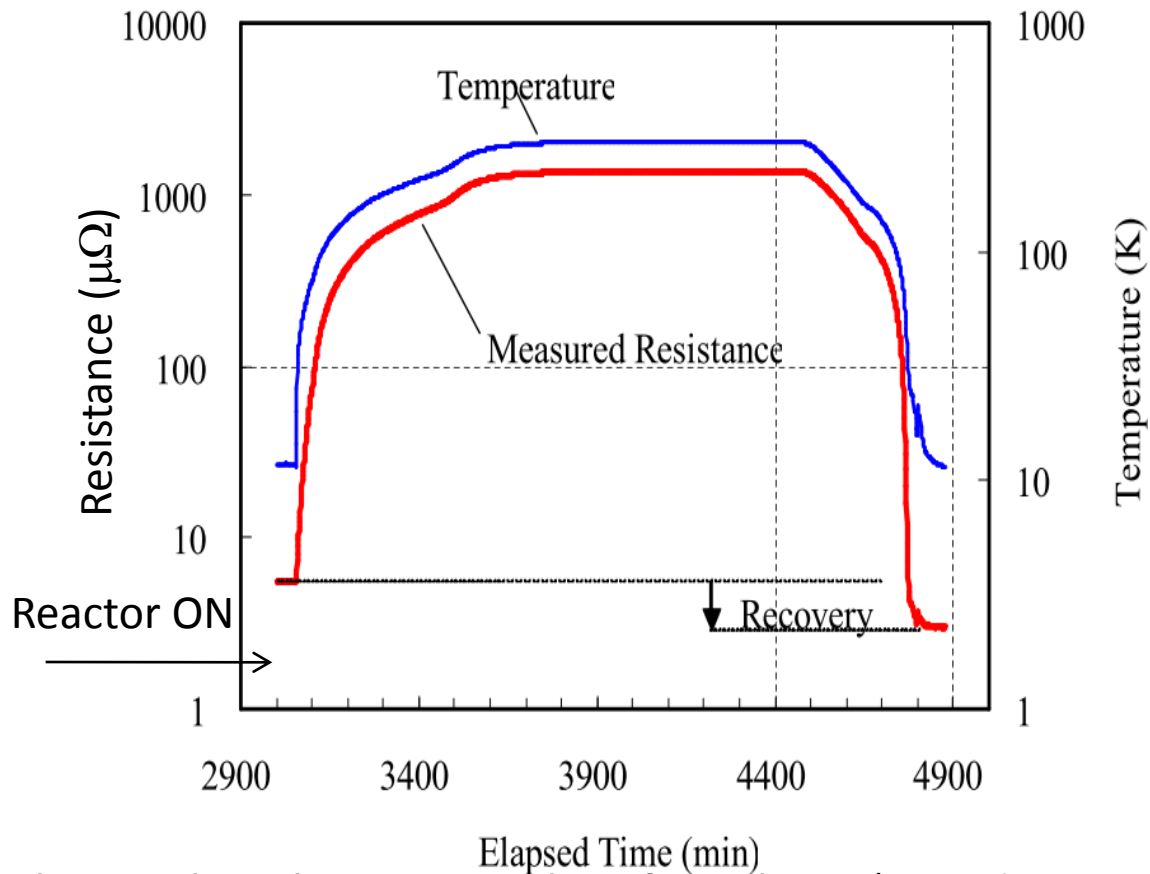
Result: $\Delta\rho_{irr}$ for 5N-Aluminum

*TC reading includes the offset of +1K.



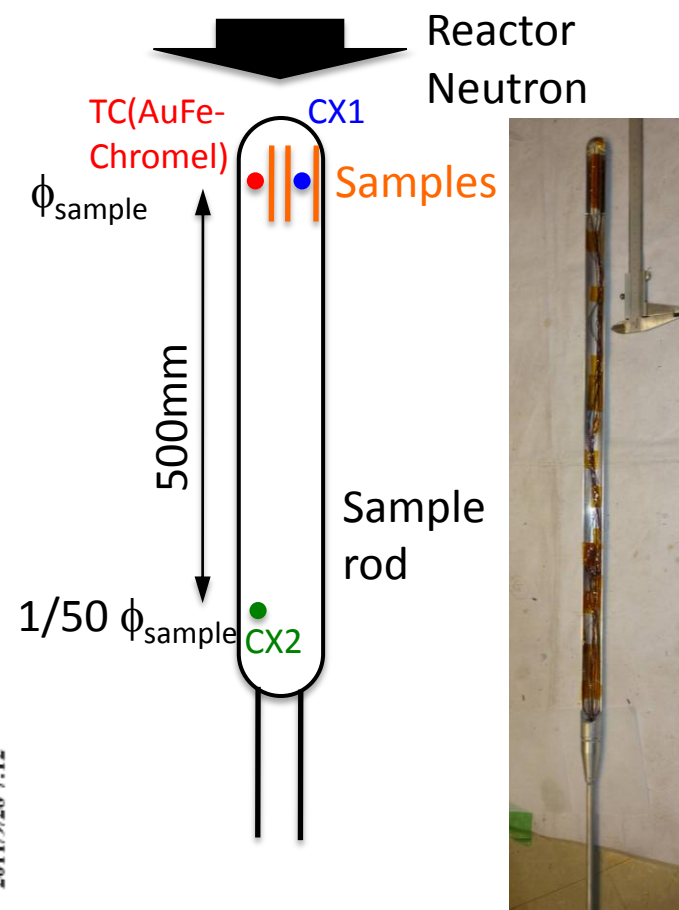
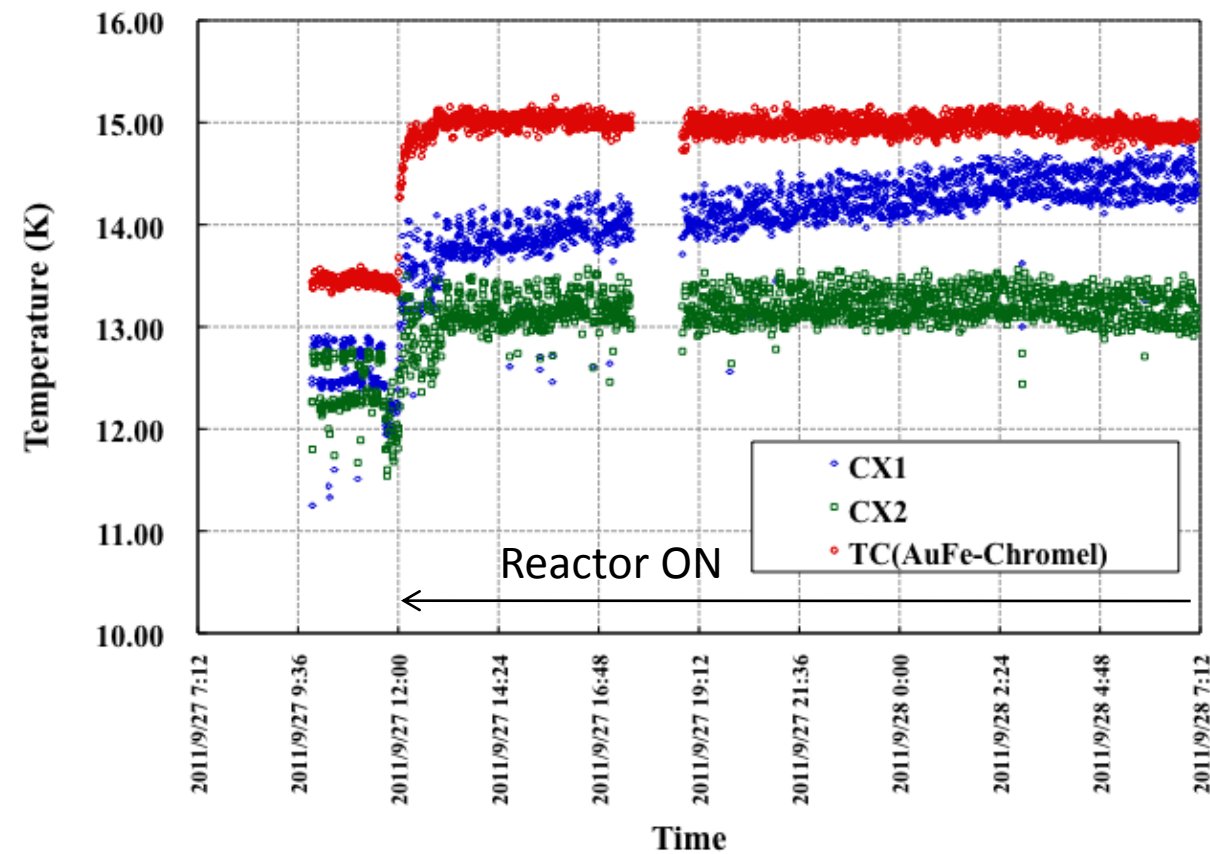
- Fast neutron exposure at 14K @1MW*52 hrs (Sep. 2011)
- Resistance increased in proportional to neutron fluence in the range of 10^{19} - 10^{20} n/m²
 - No threshold at low neutron fluence
- Observed $\Delta\rho_{irr} = 0.064$ n Ω .m for 2.7×10^{20} n/m² (>0.1MeV)
 - Agreed with the previous work within a factor of 2.
 - Present work: $\Delta\rho_{irr} / \Phi_{tot} = 2.4 \times 10^{-22}$ n Ω m³ (RRR 3000, 2.7×10^{20} n/m²)
 - Previous: $\Delta\rho_{irr} / \Phi_{tot} = 1.9 \times 10^{-22}$ n Ω m³ (RRR 2000, 2×10^{22} n/m²)

Result: Anneal and Recovery for Aluminum



- A thermal cycle to RT right after the 1st irradiation (2.3×10^{20} n/m²) in Nov. 2010.
 - Before irradiation: $3.0 \mu\Omega$ @10K
 - After irradiation: $5.7 \mu\Omega$ @12-15K
 - After TC: $3.0 \mu\Omega$ @12K
- **Full recovery of $\Delta\rho_{\text{irr}}$ was confirmed.**

Result: Thermometers



- 2 Cernox sensors and TC (AuFe-Chromel) irradiated together with samples in Sep. 2011.
- Sudden jump right after the reactor start is due to energy deposition by gamma rays and neutrons.
- CX1 reading seems to drift with a rate of 1K/day while TC at the same position shows constant temperature.
 - Likely cause of temperature reading rise in CX1 was degradation.
- Temperature rise in CX2 under low neutron flux is negligibly small.

Discussion

Materials	Aluminum				Copper		
	Horak	Guinan	Present	Present	Horak	Guinan	Present
RRR	2286	74	450	3007	2280	172	319
T_{irr} (K)	4.5	4.2	12	14	4.5	4.2	14
Neutron Source	Reactor	14 MeV	Reactor	Reactor	Reactor	14 MeV	Reactor
Φ_{tot} (n/m ²) (>0.1MeV)	2×10^{22}	$1-2 \times 10^{21}$	2.3×10^{20}	2.7×10^{20}	2×10^{22}	$1-2 \times 10^{21}$	2.7×10^{20}
$\Delta\rho_{irr} / \Phi_{tot} \times 10^{-31}$ (Ωm^3)	1.9	4.09	2.4	2.4	0.58	2.29	0.82
Recovery by thermal cycle	100%	100%	100%	TBD	90%	80%	TBD

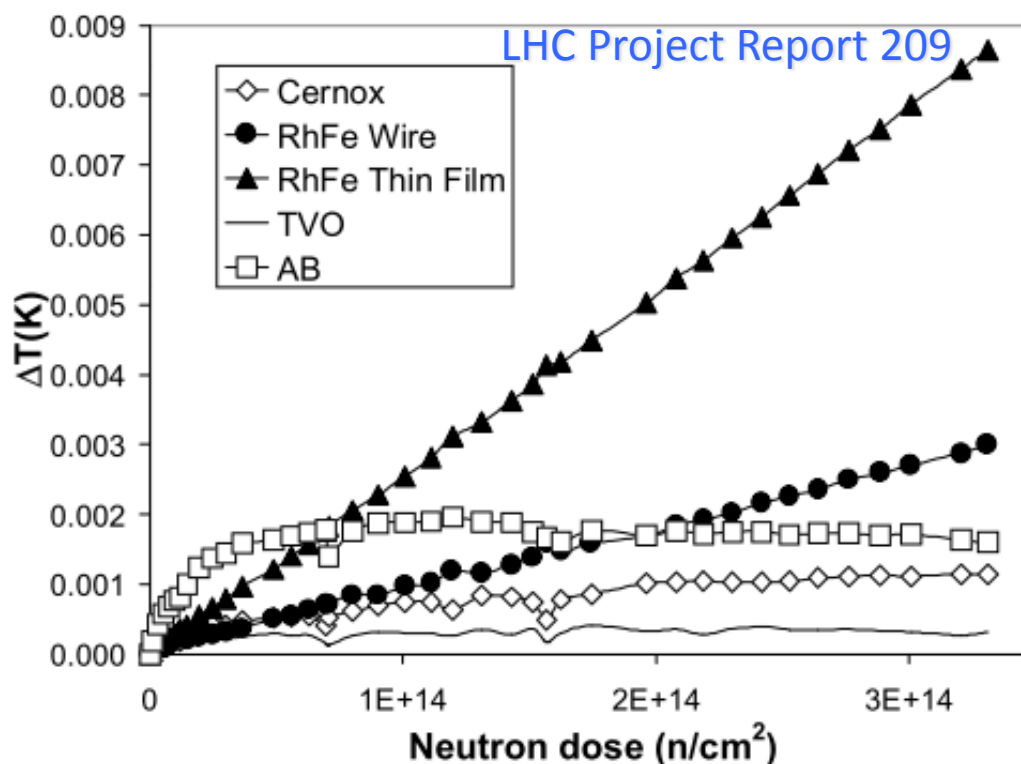
- Degradation rate ($\Delta\rho_{irr} / \Phi_{tot}$) seems to be higher in 14 MeV neutron irradiation. Evaluation using a common index such as DPA would be necessary.
- Present work shows that difference in RRR of Al doesn't influence the degradation rate.
- For copper, degradation rates ($\Delta\rho_{irr} / \Phi_{tot}$) are ranged from 0.58 to 2.29 $10^{-31} \Omega m^3$. What if SC cables with the initial RRR of 200 are irradiated to 10^{20} or 10^{21} n/m²?
 - 10^{20} n/m²: RRR of 160 – 190
 - 10^{21} n/m²: RRR of 50 – 120
- Recovery by annealing in copper sample and its multiple irradiation are planned in 2012.

Summary and Further Plan

- Reactor neutron irradiation tests for SC stabilizers (Al, Cu) at low temperature have been carried out to study the degradation behavior. Recovery by annealing to RT have been also studied.
- Irradiation of aluminum and copper samples up to $2-3 \times 10^{20}$ n/m² below 20 K showed that the degradation rates ($\Delta\rho_{\text{irr}}/\Phi_{\text{tot}}$) agreed with the previous work within a factor of 2.
- Full recovery of resistivity degradation by annealing was confirmed in the aluminum sample.
 - For the copper sample, the recovery behavior during the repeated irradiation and annealing will be studied in 2012.
- Cernox thermometers irradiated up to $2-3 \times 10^{20}$ n/m², which is 20 times as high as that for the previous work. The induced resistance per neutron fluence was consistent with the previous work.
- Further neutron irradiation tests for other SC magnet materials will be made at KUR.

Why thermometers?

- Irradiation effects of thermometer including Cernox studied for the LHC at 1.8 K. Fluence up to 10^{19} n/m².
 - What happens at the level of 10^{20} or higher?



X60947

$R_0 = 1960 \Omega$ @ 12K

$\Delta R / \Delta T = -170 \Omega / K$

$\Delta R_{irr} = 340 \Omega$ @ $2 \cdot 10^{20}$ n/m²

$R_0 = 12600 \Omega$

$\Delta R / \Delta T = -12000 \Omega / K$

$\Delta R_{irr} = 24 \Omega$ @ 10^{19} n/m²

$\gg 480 \Omega$ for $2 \cdot 10^{20}$

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

SC: NbTi (1)

Degradation on Tc: 0.15 K to 0.6 K @up to $10^{23}/m^2$

Adv. Cryo. Engineering, 32, p853 (1986)

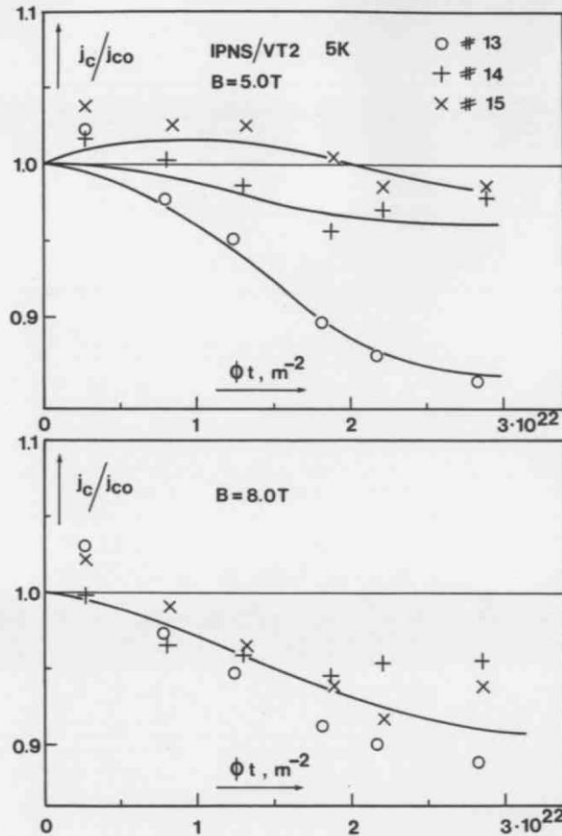


Fig. 2. Change of critical current densities with fast neutron fluence ($E > 0.1$ MeV) at 5 and 8 T. #13, 14, 15: 42, 49, 54 wt%Ti; annealing temperature: 350°C ; final cold work: 71%.

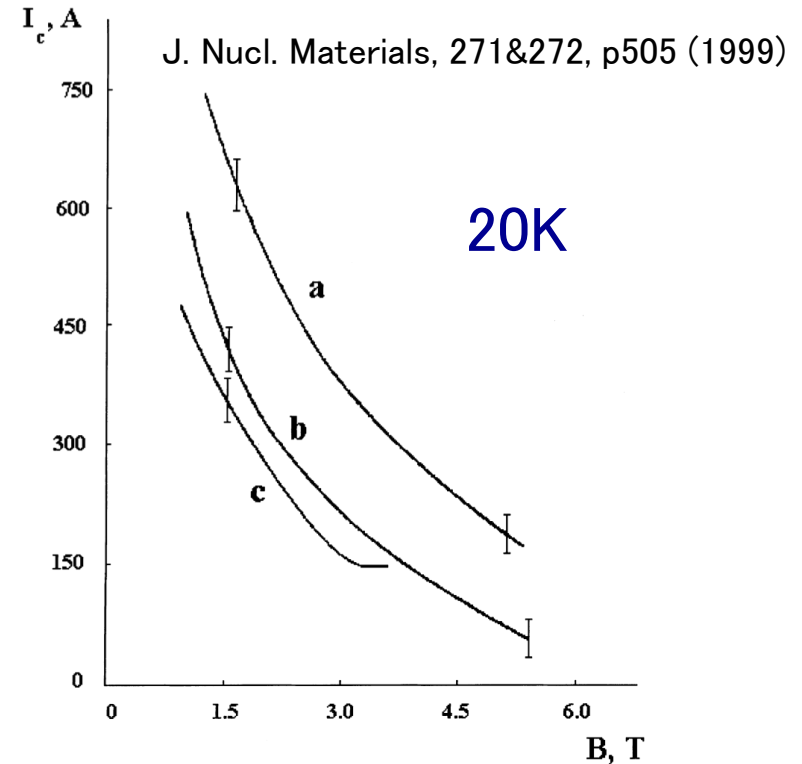


Fig. 2. Dependence of critical current vs magnetic field for 37-core industrial superconducting NbTi wire: (a) before irradiation; (b) after irradiation by the neutron fluence of $8.6 \times 10^{17} \text{ cm}^{-2}$; (c) after irradiation by the neutron fluence of $1.6 \times 10^{18} \text{ cm}^{-2}$.

J_c : < 10% reduction up to $10^{22}/m^2$

I: Significant reduction at 5T @ $10^{22}/m^2$

SC: NbTi (2)

RT, 77K w/ T.C.

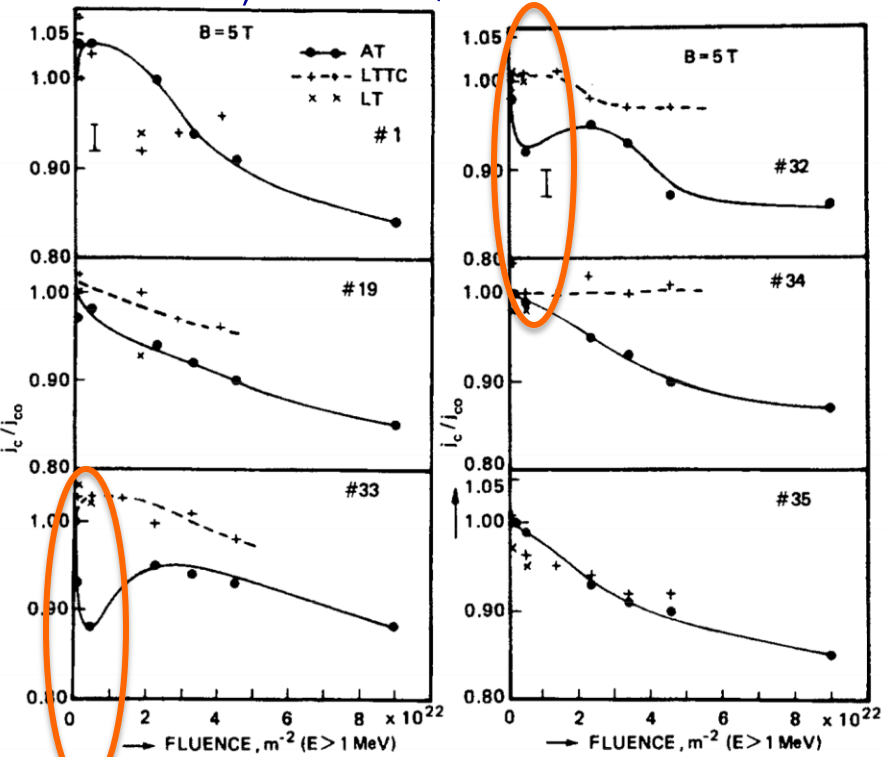


Fig. 11. Changes of critical current densities measured at 5 T with fast neutron fluence. AT, LT: irradiation at ambient temperature and 77 K, respectively; LTTC: irradiation at 77 K and thermal cycle to room temperature. No. 1: Nb-42 wt% Ti, lowest j_{c0} ; Nos. 19, 32, 33: Nb-42, 49, 54 wt%Ti, highest j_{c0} of each series; Nos. 34, 35: Nb-49 wt%Ti, Multifilamentary conductors [41].

J. Nucl. Materials, 108&109, p572 (1982)

RT

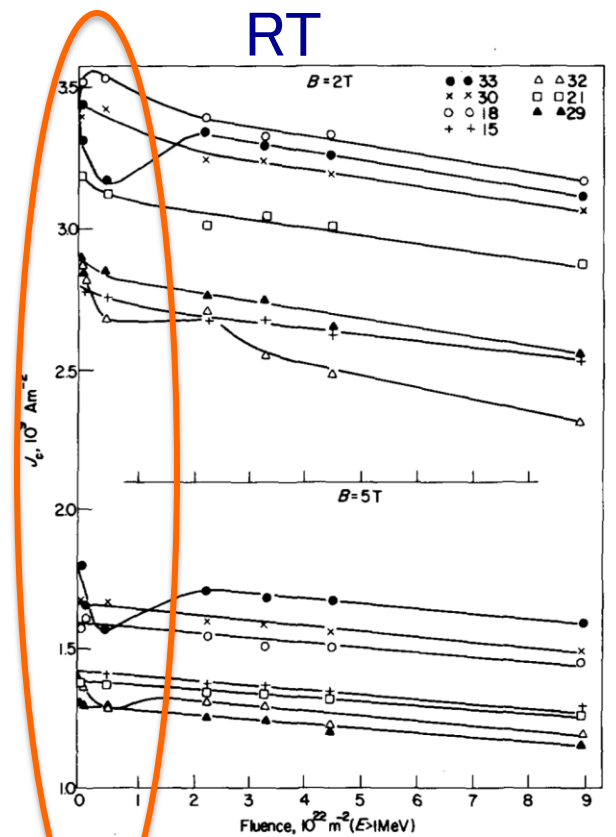
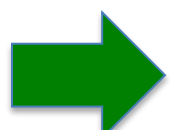


Fig. 9 Critical current densities as a function of fast neutron fluence for the seven highest j_c conductors of the present investigation

Cryogenics, 21, No.4, p223 (1981)

**Jc: Drop and recovery observed to $10^{22}/m^2$.
 10-20% reduction up to $10^{23}/m^2$.
 Recovery by annealing to RT is observed.**



NbTi would be OK up to $10^{22}/m^2$.

SC: Nb₃Sn

Adv. Cryo. Engineering, 32, p853 (1986)

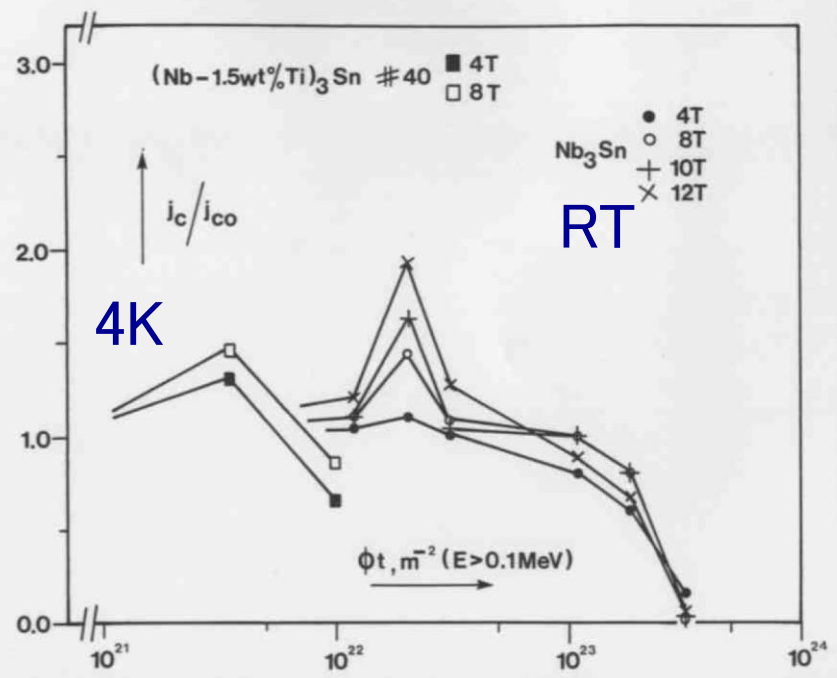
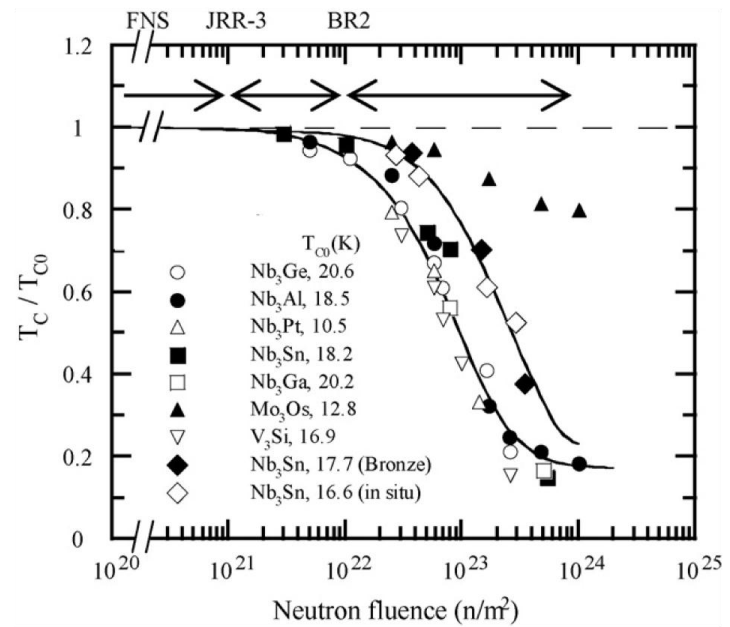


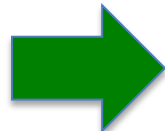
Fig. 6. Change of critical current densities with neutron fluence. The scaling is not completely accurate because of slight differences in damage energy cross sections. Low temperature irradiations on an alloyed conductor are compared to ambient temperature irradiations of pure Nb₃Sn²⁹.

**Jc: Improvement bwn 10²² and 10²³/m².
Significant degradation beyond 10²³/m².**

Fusion Eng. Design, 84, p1425 (2009)



**Tc: -10% @ 10²²/m².
-30% @ 10²³/m².**



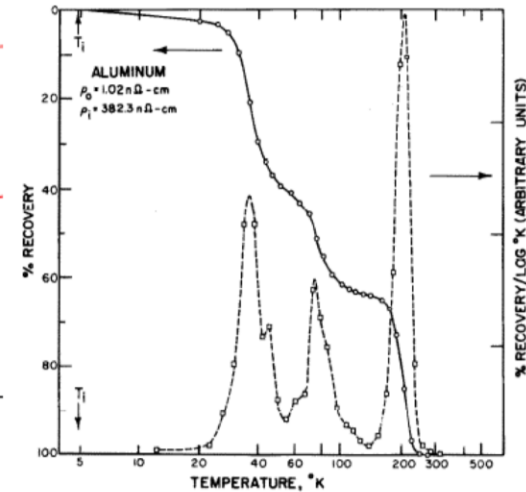
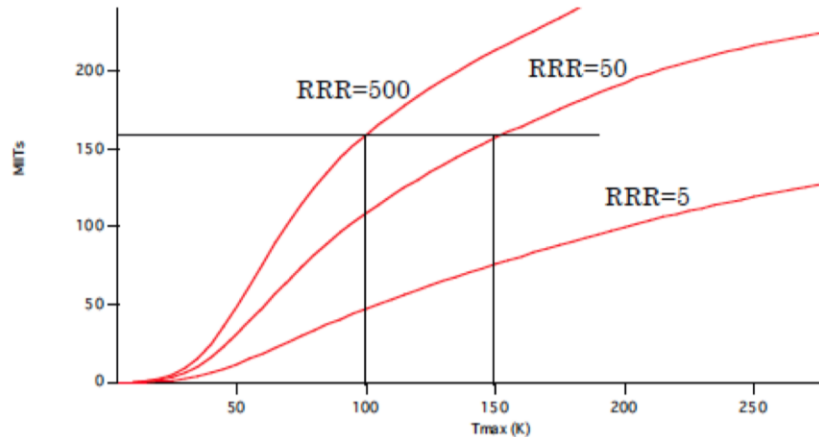
NbSn would be OK up to 10²²/m² as well.

Why is ρ of Stabilizer Important?

>> very concerned with quench protection.

- MIITs:
$$\int_{t_{quench}}^{t_{end}} I^2 dt = \int_{T_0}^{T_{max}} \frac{C_p A}{\rho / A} dT$$

- ρ increase \rightarrow temperature increase



Neutron irradiation test for stabilizers (copper, aluminum) is undoubtedly necessary.

minimum fluence to start of degradation
anneal effect on recovery
R&D of witness sample for the operation