Neutron Irradiation Measurement for Superconducting Magnet Materials at Low Temperature

Tatsushi NAKAMOTO
KEK
Collaborators/Supporters


[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

High Radiation Environment for SC Magnets

- HiLumi LHC: $10^{21}$-$10^{22}$ n/m$^2$
- COMET & Mu2e experiments (J-PARC, Fermilab)
  - Search for $\mu$-e conversion
  - Pion capture solenoids w/ Al stabilized NbTi SC cable.
  - Same spec as ATLAS-CS.
  - Neutron fluence: > $10^{21}$ n/m$^2$

Neutron irradiation effects at low temperature need be studied.
Why $\rho$ of stabilizer? Why at low temperature?

- Electrical resistivity $\rho$ 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 $\rho$ 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/m$^2$?
  - Fluence threshold?
  - Full recovery by full thermal cycle?
  - Accumulated resistivity after multiple irradiation?
Previous Work in Literature: $\Delta \rho_{\text{irr}}$

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


**Reactor n on Al**

- $\rho_0$: 0.0102
- $\Delta \rho_{\text{irr}}$: 3.823 (nΩm)

**14MeV n on Al**

- $\rho_0$: 0.386
- $\Delta \rho_{\text{irr}}$: 0.336 (nΩm)

**Reactor n on Cu**

- $\rho_0$: 0.0082
- $\Delta \rho_{\text{irr}}$: 1.162 (nΩm)

**14MeV n on Cu**

- $\rho_0$: 0.098
- $\Delta \rho_{\text{irr}}$: 0.191 (nΩm)

- 80% recovery
- 90% recovery

- Double of resistivity observed at $10^{21}$ n/m$^2$.
- 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 (En>0.1MeV): $1.4 \times 10^{15} \text{n/m}^2\text{s}@1\text{MW}$

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

Fig. 15 Neutron energy spectrum in LTL of KUR for ordinary core (above 1000 eV) KUR-TR287 (1987)
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.
  - φ1mmx60mm, $L_v$-taps = 32 mm

- **5N Aluminum (for reference):**
  - Provided by Sumitomo Chemical. RRR of ~3000.
  - φ1mmx60mm, $L_v$-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

- Fast neutron exposure at 12K @1MW*45 hrs (Nov. 2010)
- Resistance was measured \textit{in situ}.
- Resistance increased in proportional to neutron fluence in the range of $10^{19}$-$10^{20}$ n/m$^2$
  - No threshold at low neutron fluence
- Observed $\Delta\rho_{\text{irr}} = 0.056$ n$\Omega$.m for $2.3\times10^{20}$ n/m$^2$ (>0.1MeV)
  - Fairly good agreement with the previous work.
    - Present work: $\Delta\rho_{\text{irr}}/\Phi_{\text{tot}} = 2.4\times10^{-22}$ n$\Omega$m$^3$ (RRR 500, $2.3\times10^{20}$ n/m$^2$)
    - Previous: $\Delta\rho_{\text{irr}}/\Phi_{\text{tot}} = 1.9\times10^{-22}$ n$\Omega$m$^3$ (RRR 2000, $2\times10^{22}$ n/m$^2$)
Result: $\Delta \rho_{\text{irr}}$ 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/m$^2$
  - No threshold at low neutron fluence
- Observed $\Delta \rho_{\text{irr}} = 0.022$ nΩ.m for $2.7\times10^{20}$ n/m$^2$ (>0.1MeV)
  - Agreed with the previous work within a factor of 2.
    - Present work: $\Delta \rho_{\text{irr}}/\Phi_{\text{tot}} = 0.82\times10^{-22}$ nΩm$^3$ (RRR 300, $2.7\times10^{20}$ n/m$^2$)
    - Previous: $\Delta \rho_{\text{irr}}/\Phi_{\text{tot}} = 0.58\times10^{-22}$ nΩm$^3$ (RRR 2000, $2\times10^{22}$ n/m$^2$)
Result: $\Delta \rho_{\text{irr}}$ for 5N-Aluminum

- 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/m$^2$
  - No threshold at low neutron fluence
- Observed $\Delta \rho_{\text{irr}} = 0.064$ n$\Omega$.m for $2.7\times10^{20}$ n/m$^2$ (>0.1MeV)
  - Agreed with the previous work within a factor of 2.
    - Present work: $\Delta \rho_{\text{irr}} / \Phi_{\text{tot}} = 2.4 \times 10^{-22}$ n$\Omega$ m$^3$ (RRR 3000, $2.7\times10^{20}$ n/m$^2$)
    - Previous: $\Delta \rho_{\text{irr}} / \Phi_{\text{tot}} = 1.9 \times 10^{-22}$ n$\Omega$ m$^3$ (RRR 2000, $2\times10^{22}$ n/m$^2$)

*TC reading includes the offset of +1K.
Result: Anneal and Recovery for Aluminum

- A thermal cycle to RT right after the 1st irradiation (2.3x10^{20} n/m^2) in Nov. 2010.
  - Before irradiation: 3.0 μΩ @10K
  - After irradiation: 5.7 μΩ @12-15K
  - After TC: 3.0 μΩ @12K
- Full recovery of Δρ_{irr} was confirmed.
Result: Thermometers

- 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.
Degradation rate ($\frac{D\rho_{irr}}{F_{tot}}$) 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 ($\frac{D\rho_{irr}}{F_{tot}}$) 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/m$^2$?

- $10^{20}$ n/m$^2$: RRR of 160 – 190
- $10^{21}$ n/m$^2$: RRR of 50 – 120

Recovery by annealing in cooper 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/m$^2$ 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/m$^2$, 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/m$^2$.
  
  - What happens at the level of $10^{20}$ or higher?

![Graph showing temperature change with neutron dose](image)

Figure 3 Error on temperature measurement on some sensors during irradiation (Tbath=1.8 K)

$\Delta R_0 = 1960 \Omega$ @12K
$\Delta R/\Delta T = -170 \Omega/K$
$\Delta R_{irr} = 340 \Omega$ @ $2 \times 10^{20}$ n/m$^2$

$\Delta R_0 = 12600 \Omega$
$\Delta R/\Delta T = -12000 \Omega/K$
$\Delta R_{irr} = 24 \Omega$ @ $10^{19}$ n/m$^2$

$>> 480 \Omega$ for $2 \times 10^{20}$
SC: NbTi (1)

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


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%.

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


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 $8.6 \times 10^{17}$ cm$^{-2}$; (c) after irradiation by the neutron fluence of $1.6 \times 10^{18}$ cm$^{-2}$.

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 $j_c$; Nos. 1 32, 33: Nb–42, 49, 54 wt% Ti, highest $j_c$ of each series; Nos. 34, 35: Nb–49 wt% Ti, Multifilamentary conductors [41].


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 $10^{22}$/m2.
10–20% reduction up to $10^{23}$/m2.
Recovery by annealing to RT is observed.

NbTi would be OK up to $10^{22}$/m2.
SC: Nb3Sn


Jc: Improvement bwn $10^{22}$ and $10^{23}$/m².
Significant degradation beyond $10^{23}$/m².

Tc: $-10\% @ 10^{22}$/m².
$-30\% @ 10^{23}$/m².

NbSn would be OK up to $10^{22}$/m² as well.
Why is $\rho$ of Stabilizer Important?

>> very concerned with quench protection.

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

- $\rho$ 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