
NIMS
1020
RIKEN
KOBELCO
JEOL

Development of 1020 MHz NMR superconducting magnet using Bi-2223 innermost coil

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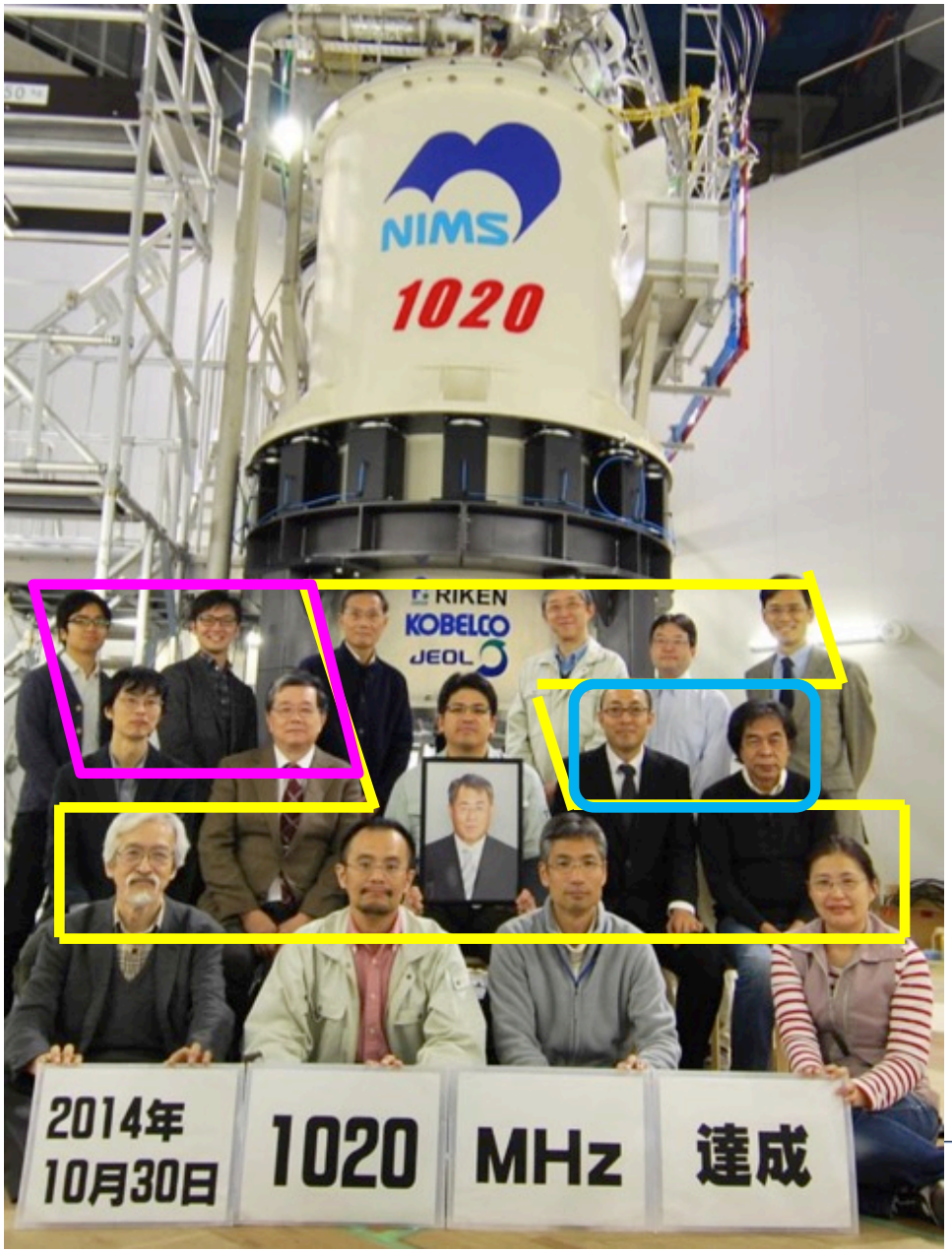
This work is supported by the System Development Program for Advanced Measurement and Analysis (SENTAN) (Program-S), JST.

UHF workshop @ NIH, USA Nov. 12th – 13th 2015

Collaborators



NIMS

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Kobe Steel

T. Miki and K. Saito

JASTEC SUPERCONDUCTOR

Collaborators

… and the founder of this project,

the late
Dr. Tsukasa Kiyoshi.

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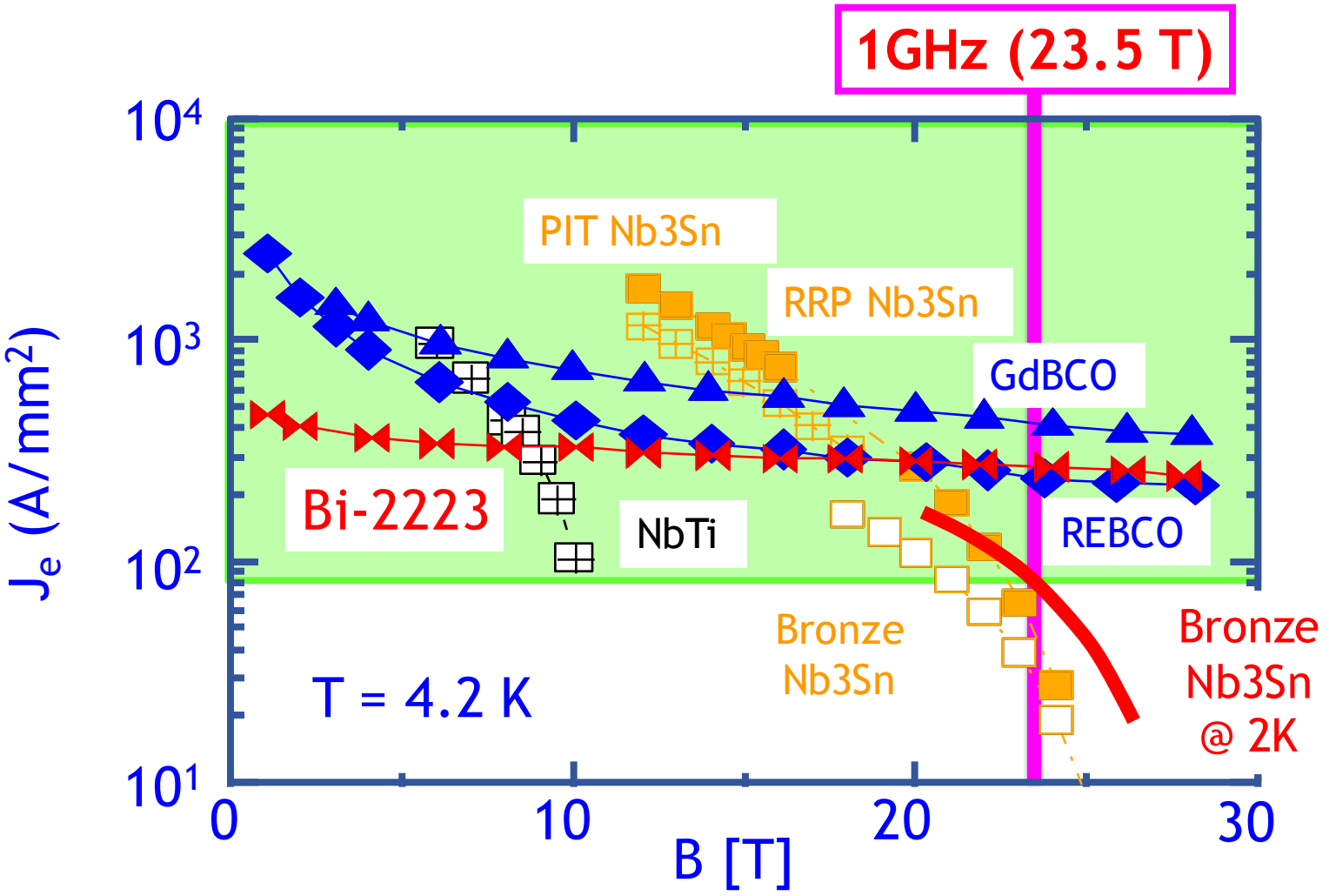
Outline

We succeeded in upgrading 920 MHz superconducting magnet to 1020 MHz using Bi-2223 innermost coil.

- Bi-2223 innermost coil development
- Damage by the huge earthquake
- Restoration
- Cool down
- Ramp up and operation at 1020 MHz (24.0 T)
- NMR data acquisition
- Ramp up to 1030 MHz (24.2 T)

Critical Current of superconducting materials



Y. Miyoshi et al., Physica C, 516 (2015) 31

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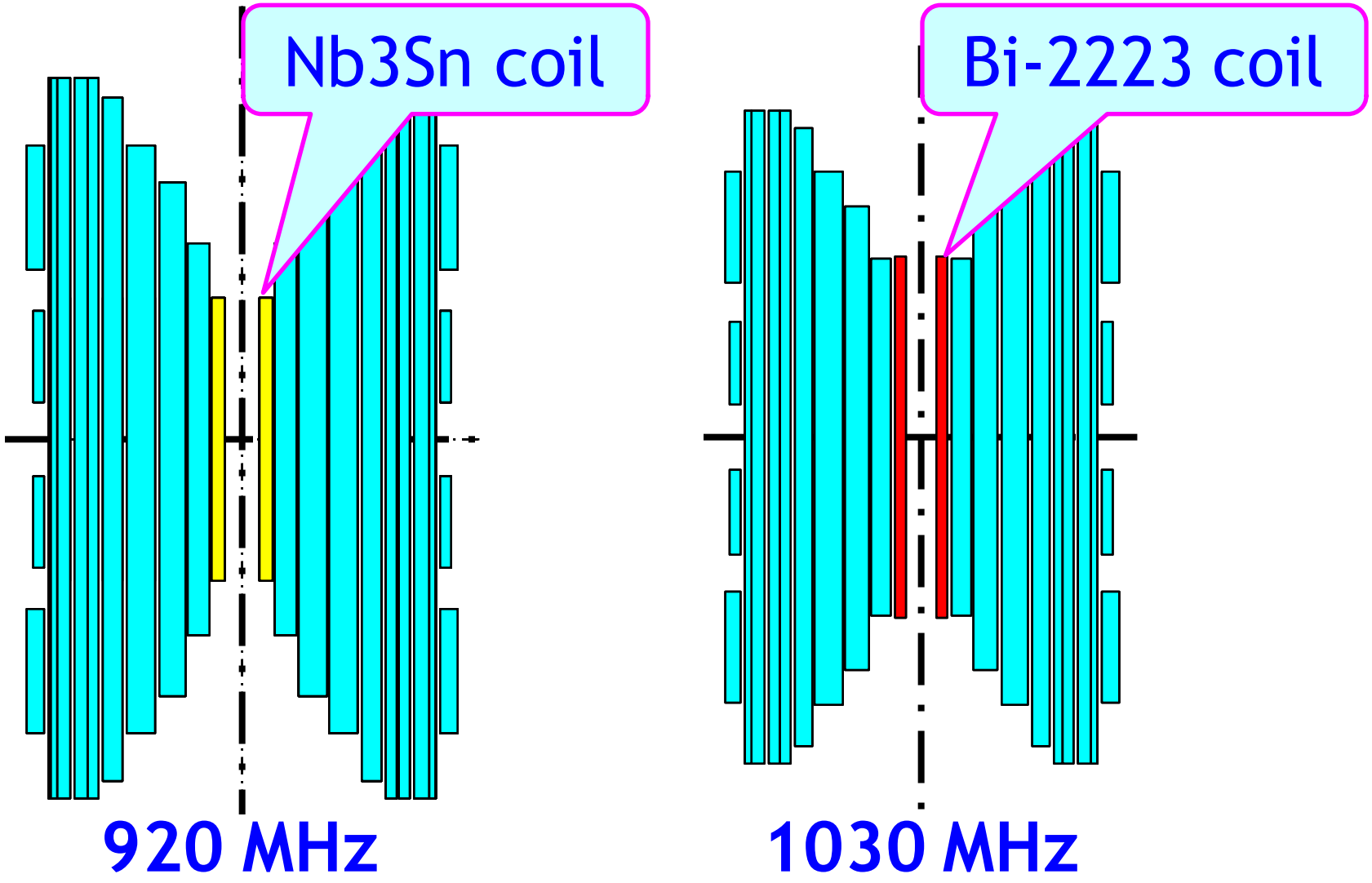
Concept of upgrading 920 MHz magnet to 1030 MHz



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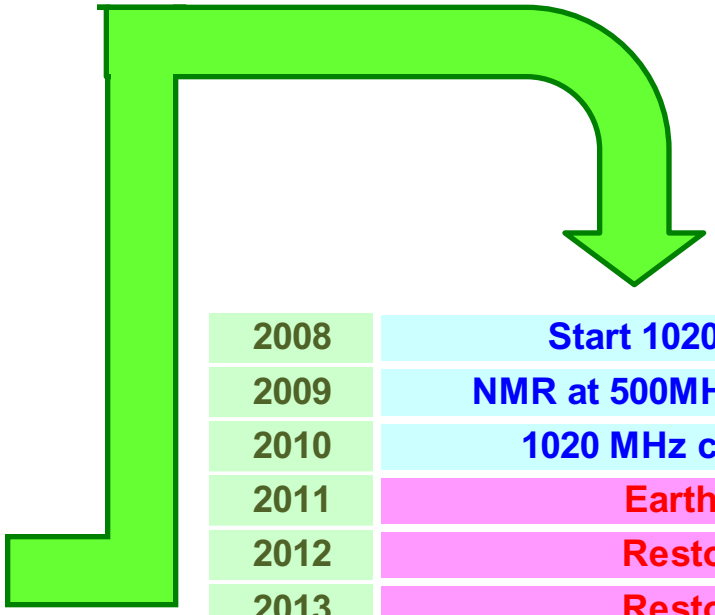
Progress of 1020 MHz development

	920 MHz
1995	Start R&D of 1 GHz project
1996	Design
1997	Wire development
1998	Magnet manufacturing
1999	Magnet manufacturing To give up to use Bi-2212
	900 MHz achieved
2000	Performance check @ 900 MHz
2001	920 MHz achieved
2002	NMR measurement
2003	NMR measurement
2004	930 MHz achieved by 2nd magnet
2005	NMR measurement
2006	NMR measurement
2007	NMR measurement



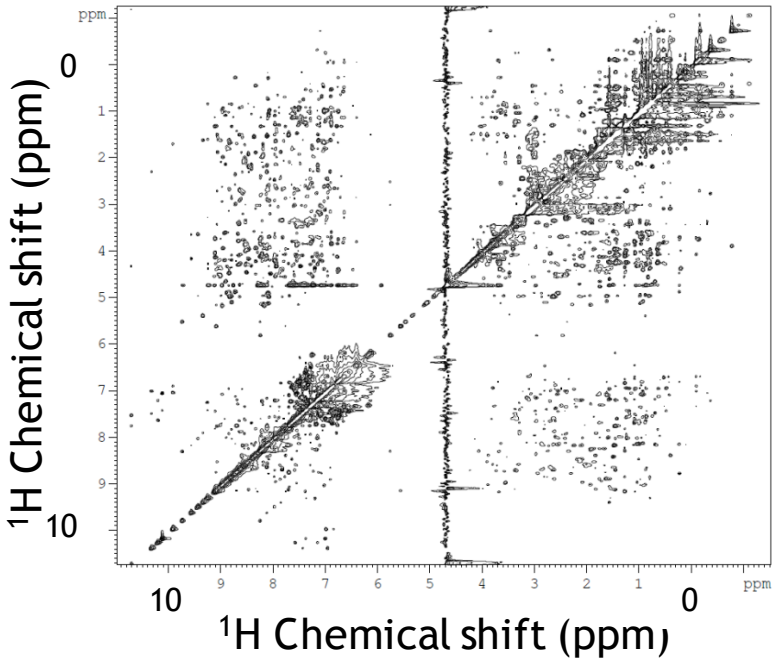
	1020 MHz
2008	Start 1020 MHz R&D
2009	NMR at 500MHz driven mode
2010	1020 MHz coil assembly
2011	Earthquake
2012	Restoration
2013	Restoration
2014	1020 MHz achieved
2015	NMR measurement
	1030 MHz achieved

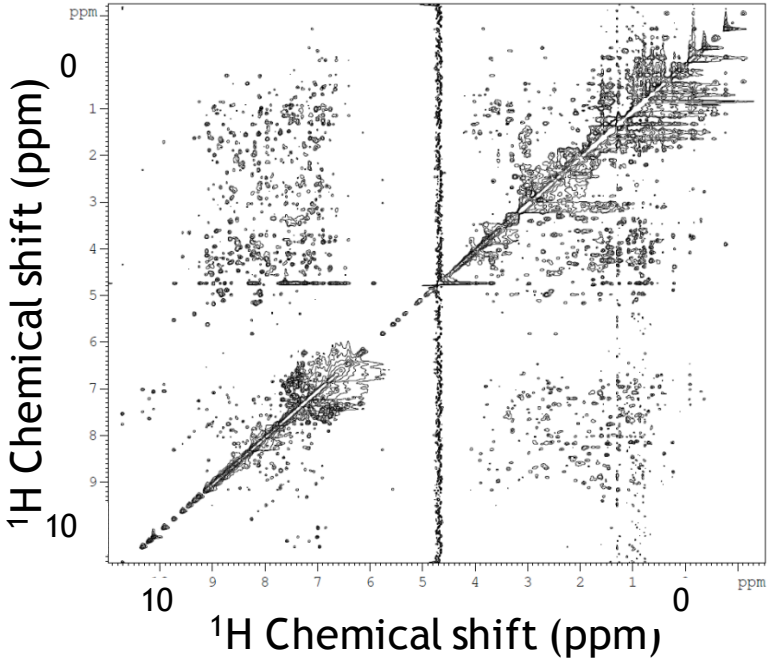
Feasibility of HTS by PS-driven mode

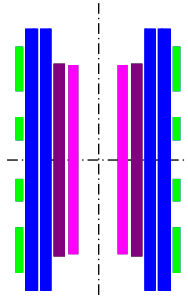
500 MHz Bi-2223/LTS driven mode NMR magnet

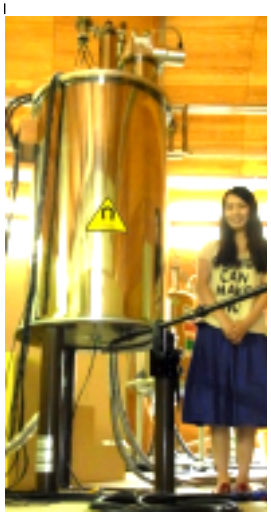
HTS/LTS PS-driven mode is feasible for NMR

WATERGATE NOESY (2mM lysozyme in 90%H_2O and 10%D_2O)









LTS-NMR / Persistent mode

HTS/LTS-NMR / Driven mode

T. Kiyoshi et al., IEEE Trans. Appl. Supercond. 20, 714–717, 2010

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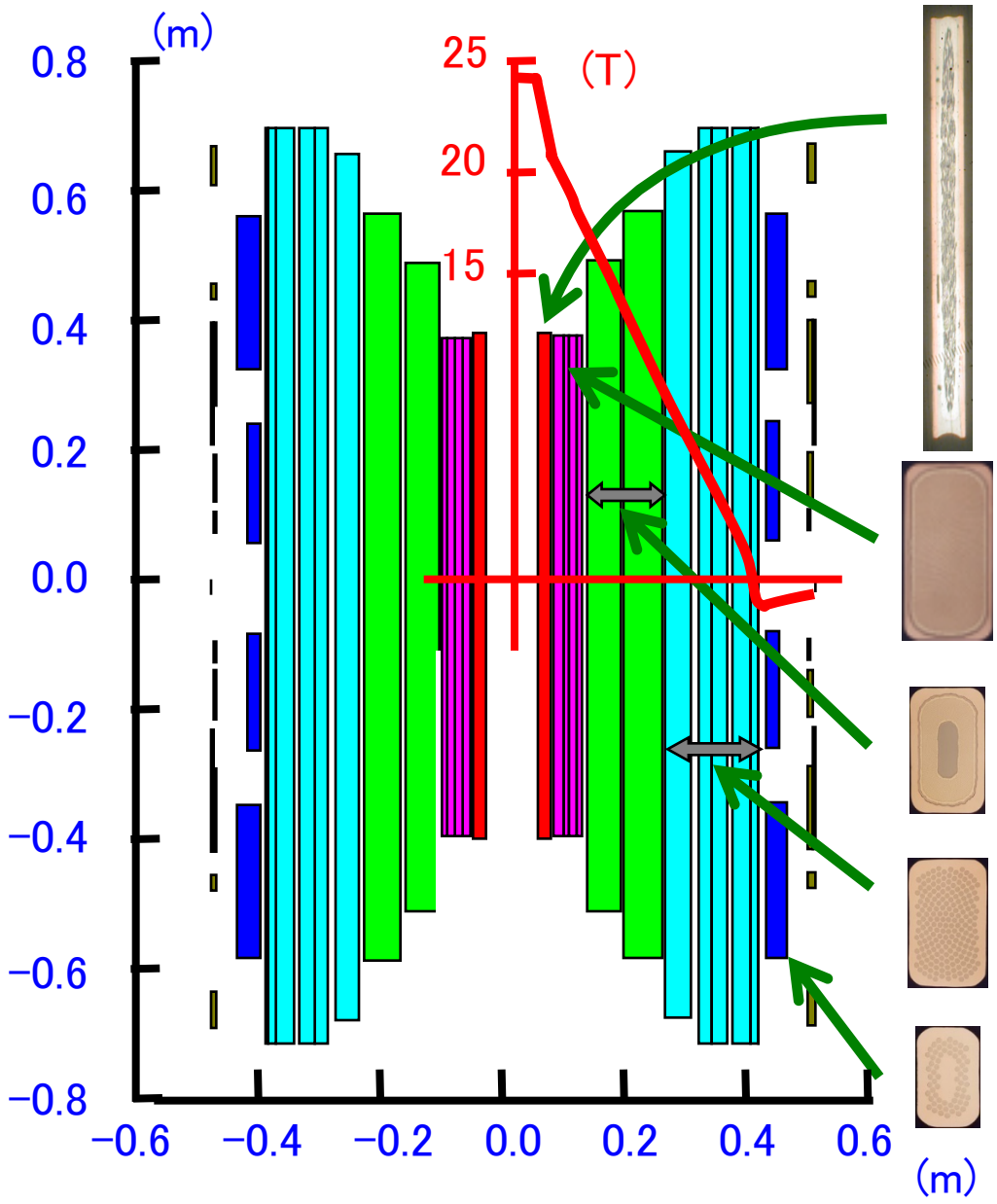
Coil configuration of 1030 MHz NMR magnet



Cu-Sn laminated
Bi-2223
Sumitomo Electric, Ltd.

High-Jc Nb3Sn
(15%Sn bronze)

High-strength Nb3Sn
(Ta reinforced)

High-strength Nb-Ti
(Cold-worked)

Nb-Ti

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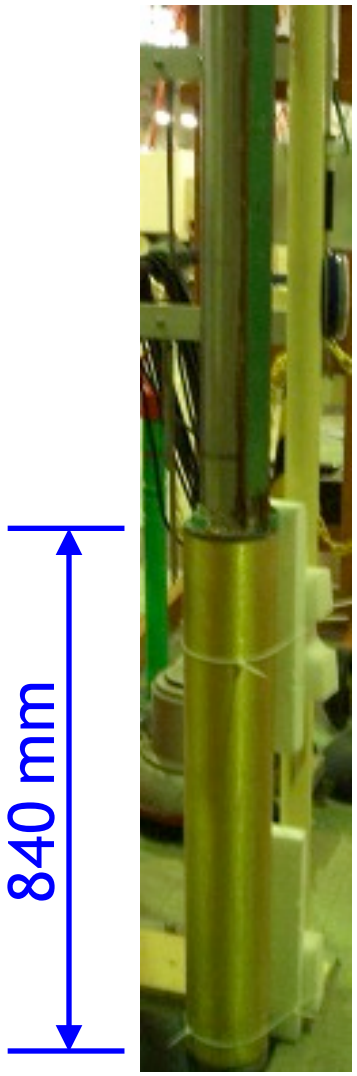
Bi-2223 innermost coil parameters

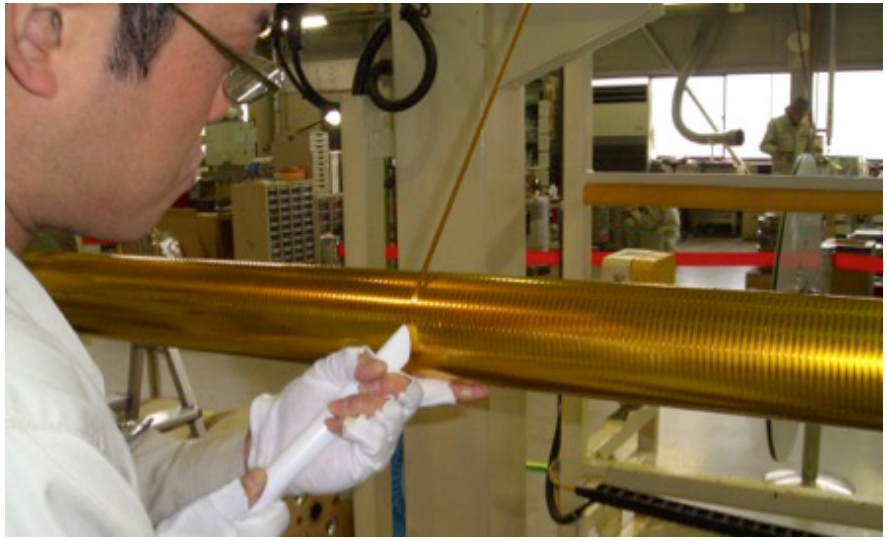
	920 MHz	1030 MHz
Superconductor	Nb_3Sn	Bi-2223
Lamination material	n/a	Cu-Sn
Conductor size [mm]	3.5×1.75	4.45×0.36
Insulation material	Glass yarn	Polyimide tapes
Insulated conductor size [mm]	3.65×1.90	4.50×0.41
Inner diameter [mm]	78.4	78.4
Outer diameter [mm]	124.3	124.0
Height [mm]	599.1	840.0
Number of layers	12	54
Number of turns	1,953	10,094
Conductor length [m]	622	3,209
Operating current [A]	244.2	242.8
Contribution field [T]	0.989	3.66



Bi-2223 coil

Bi-2223 wire by Sumitomo

Bi-2223 innermost coil manufacturing

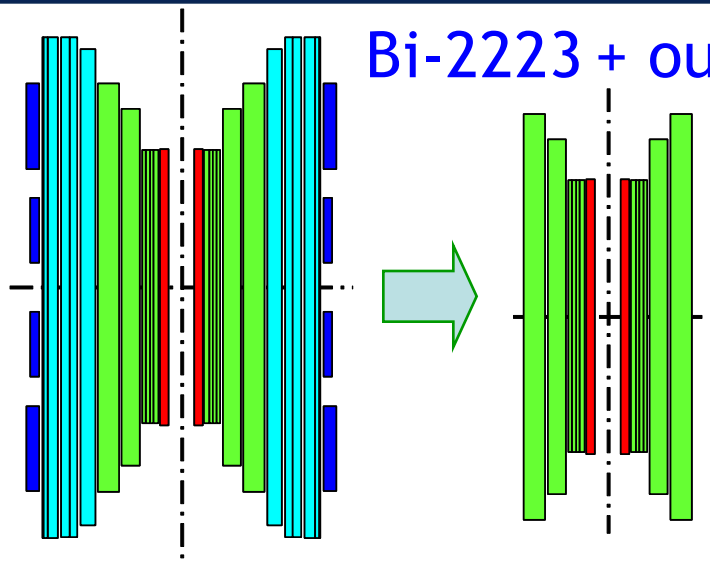


Including 5 joints

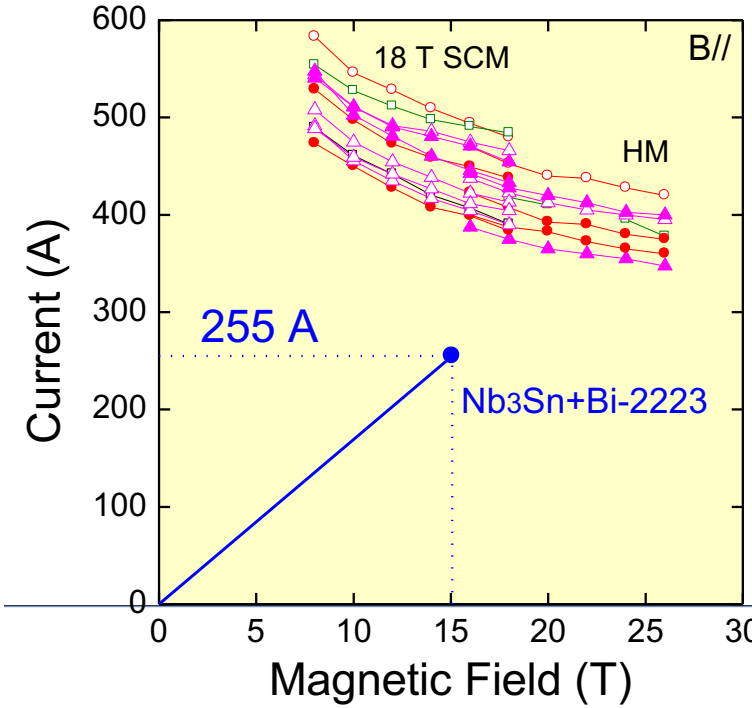
Bi-2223 coil

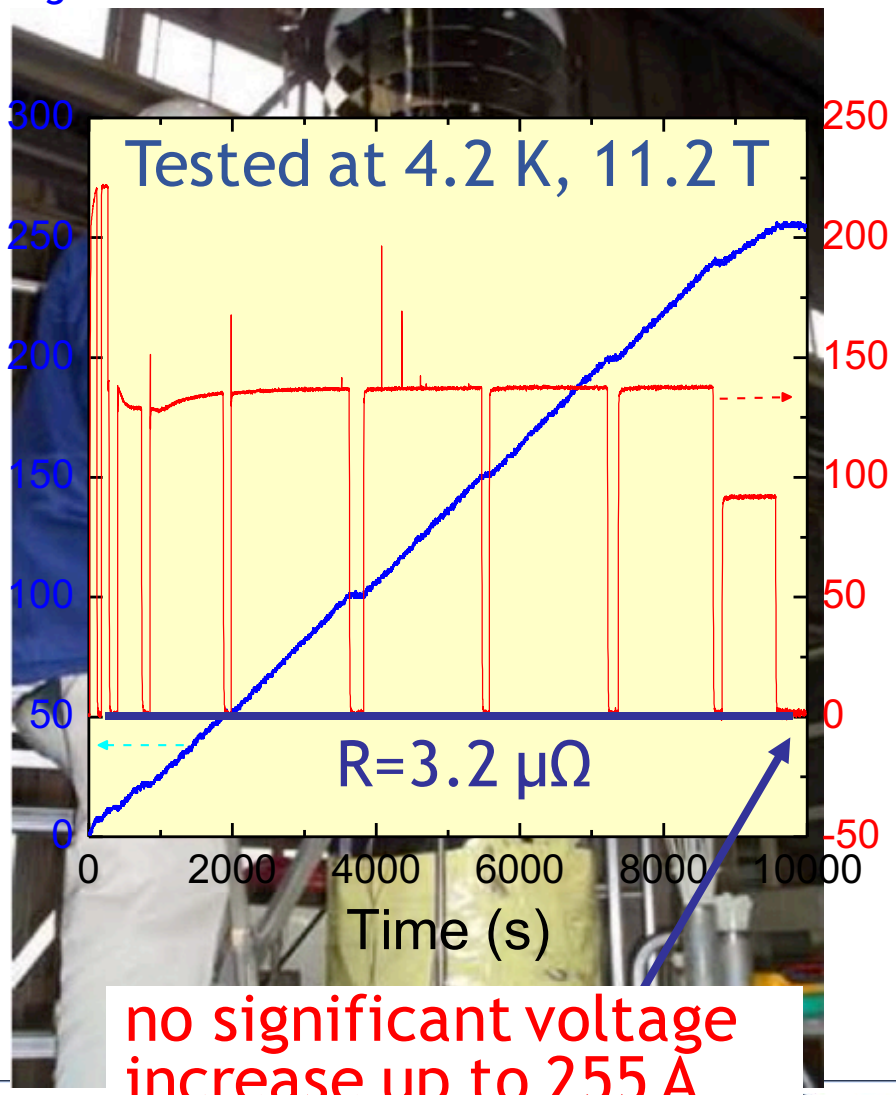
Bi-2223 wire
by Sumitomo

Design confirmation test of Bi-2223 coil



Bi-2223 + outer Nb_3Sn coils





no significant voltage increase up to 255 A

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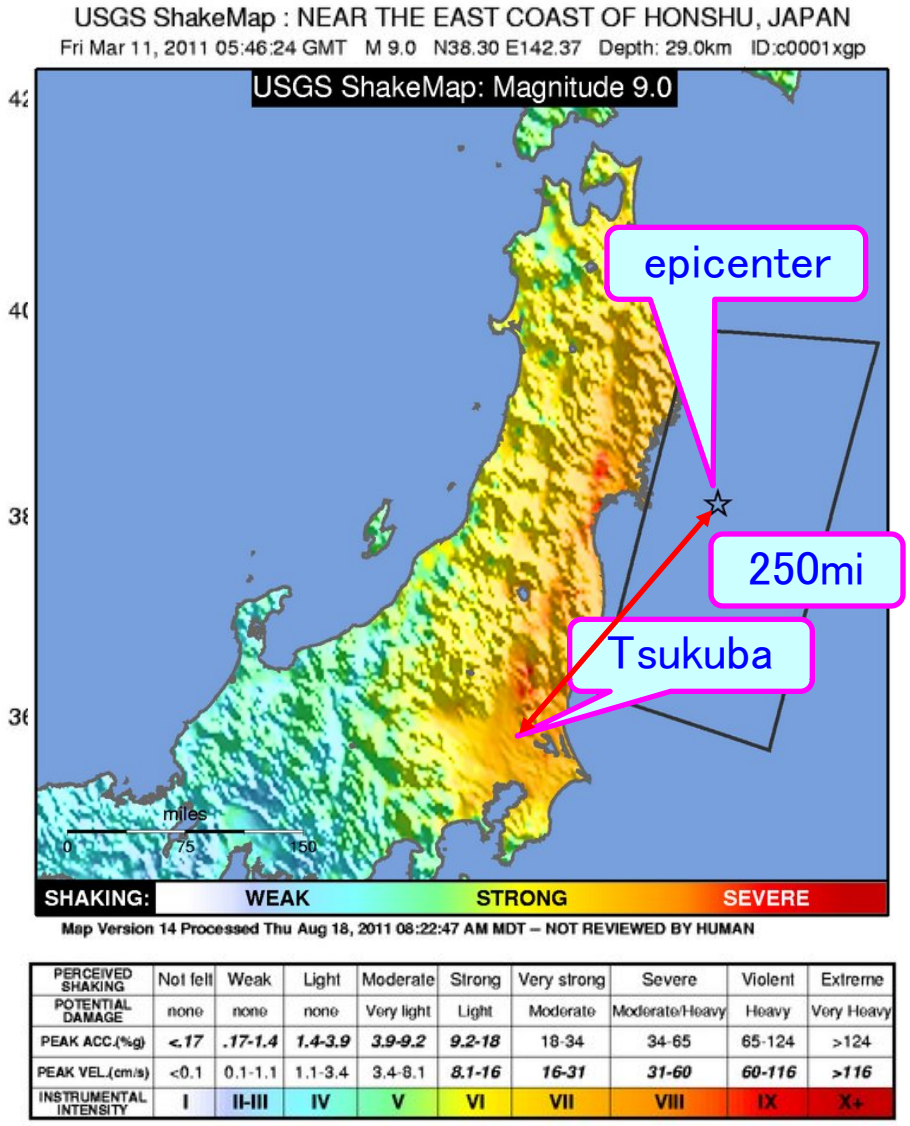
The Great East Japan Earthquake



PERCEIVED SHAKING	Not felt	Weak	Light	Moderate	Strong	Very strong	Severe	Violent	Extreme
POTENTIAL DAMAGE	none	none	none	Very light	Light	Moderate	Moderate/Heavy	Heavy	Very Heavy
PEAK ACC.(%g)	*<.17*	*.17-1.4*	*1.4-3.9*	*3.9-9.2*	*9.2-18*	18-34	34-65	65-124	>124
PEAK VEL.(cm/s)	<0.1	0.1-1.1	1.1-3.4	3.4-8.1	*8.1-16*	*16-31*	*31-60*	*60-116*	*>116*
INSTRUMENTAL INTENSITY	I	II-III	IV	V	VI	VII	VIII	IX	X+

- **Status of the magnet**
  - Cooled down to 4.2 K, waiting for pumping.
  - No current, no field.
- 14:46 JST March 11th 2011
- The biggest earthquake ever recorded in Japan
- Seismic intensity
  - Lower-6 in Tsukuba
  - (upper-6 in Sendai)

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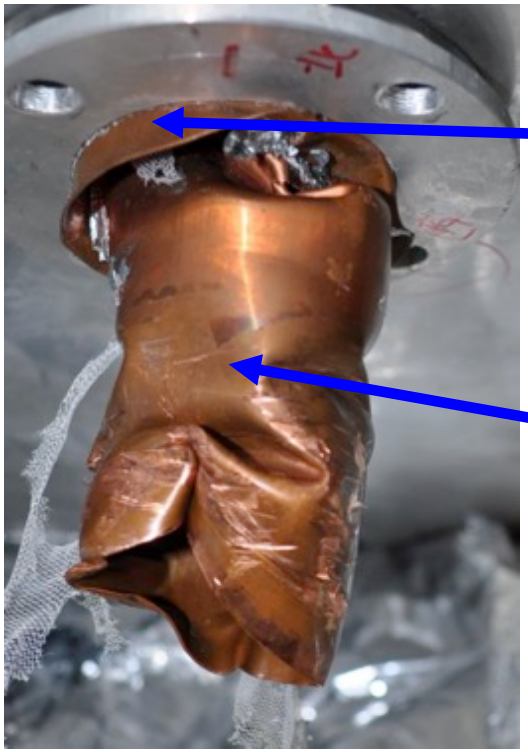
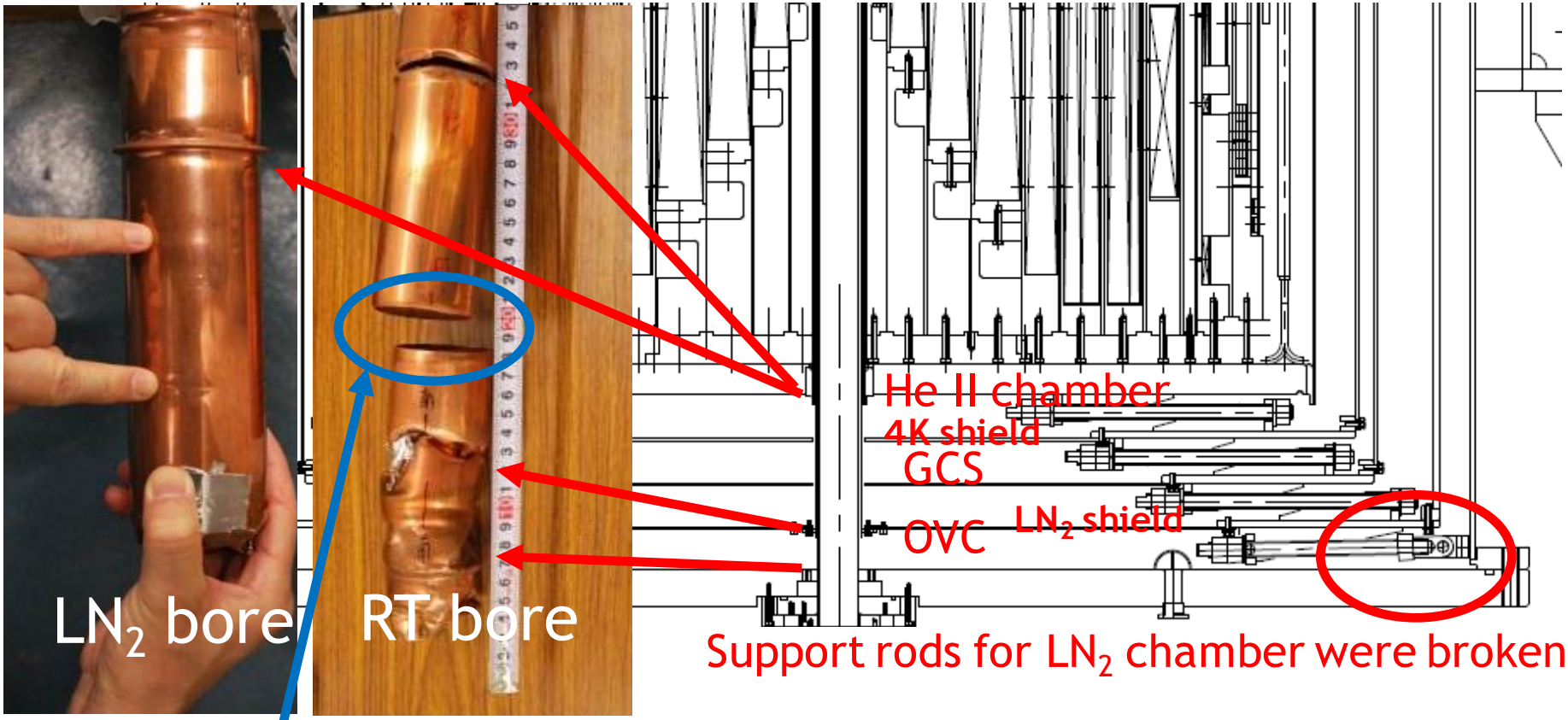
Major damages



Major damages

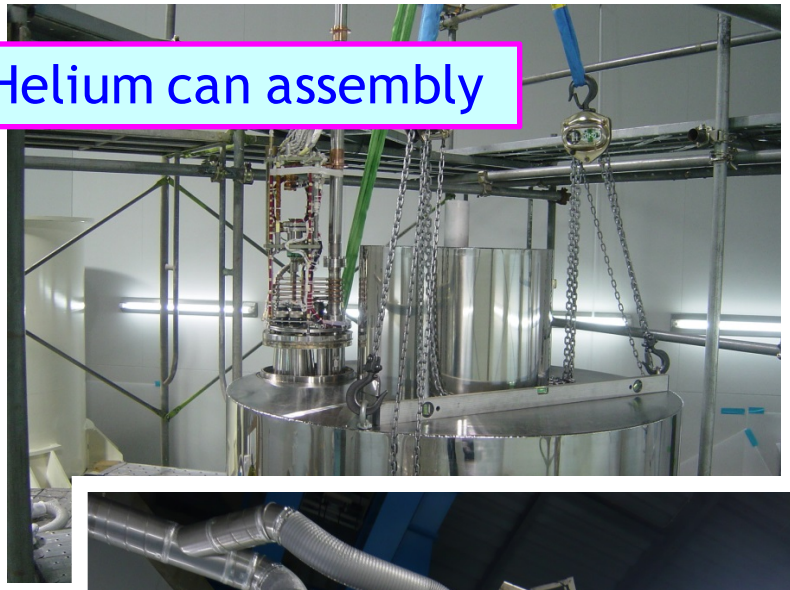


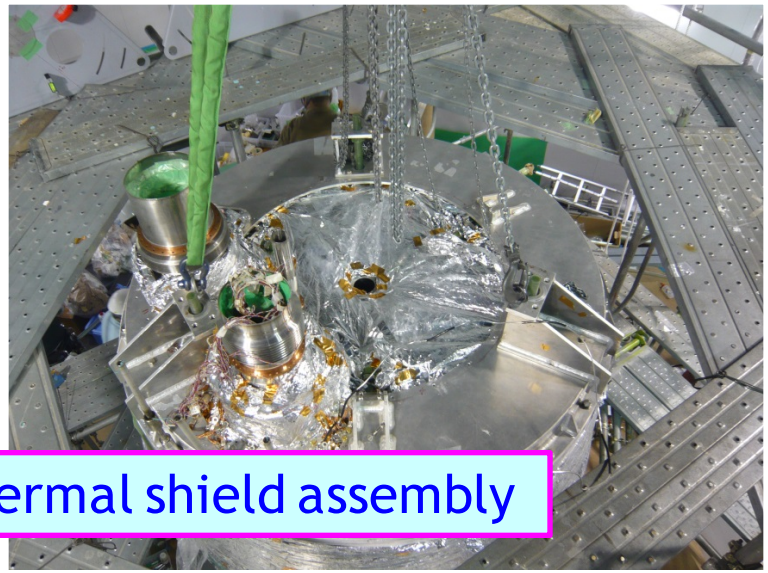
cut by a pipe cutter

Restoration activity (2011–2013)

Resistance check

Helium can assembly

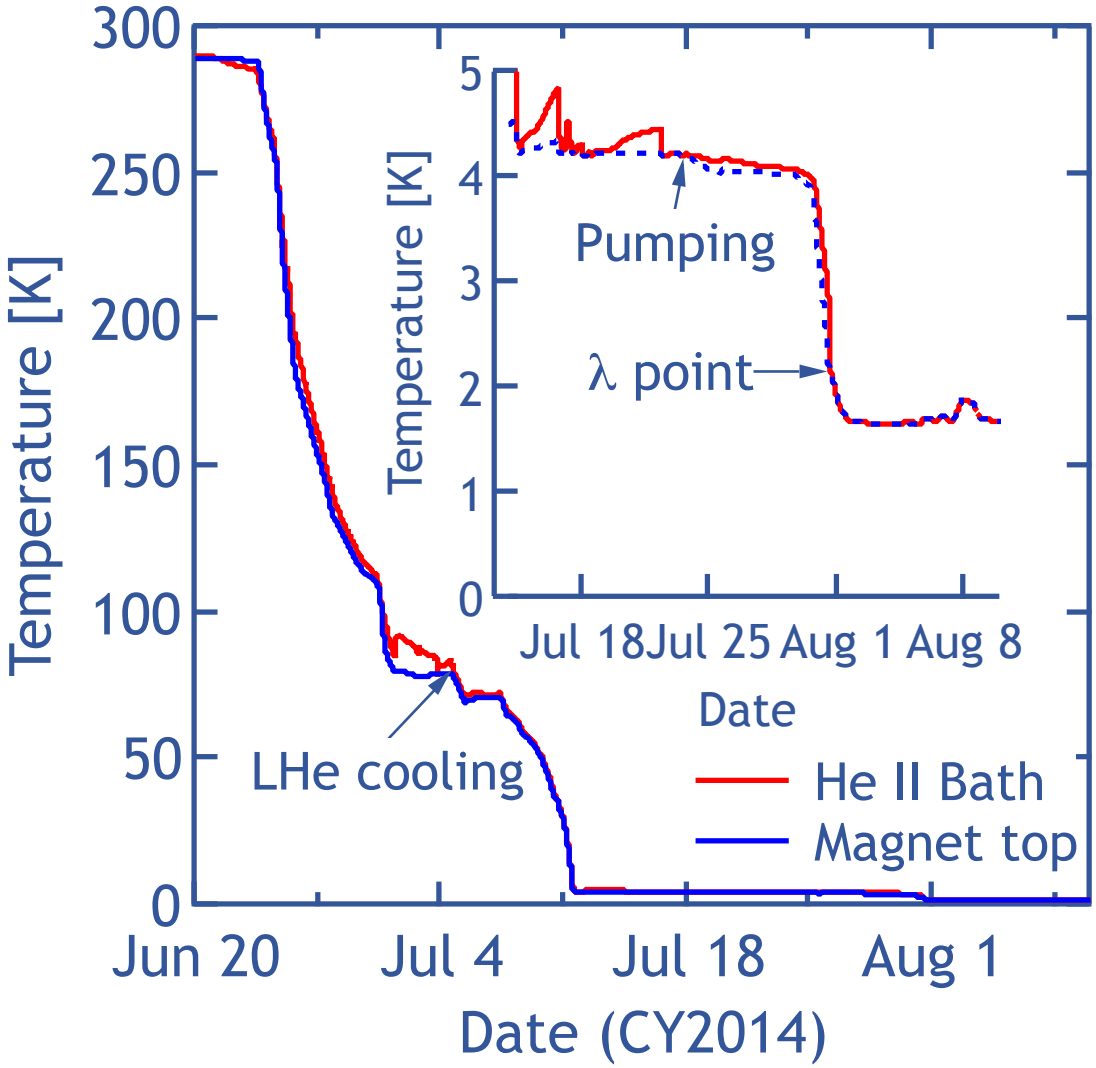




Thermal shield assembly

Restoration completion

Cool down



Cryogen consumption

LN_2 (290-80 K): 10,000 L

LHe (80K-2 K): 6,500 L

80K-4 K : 2,500 L

Storage : 3,000 L

4 K-2 K : 1,000 L

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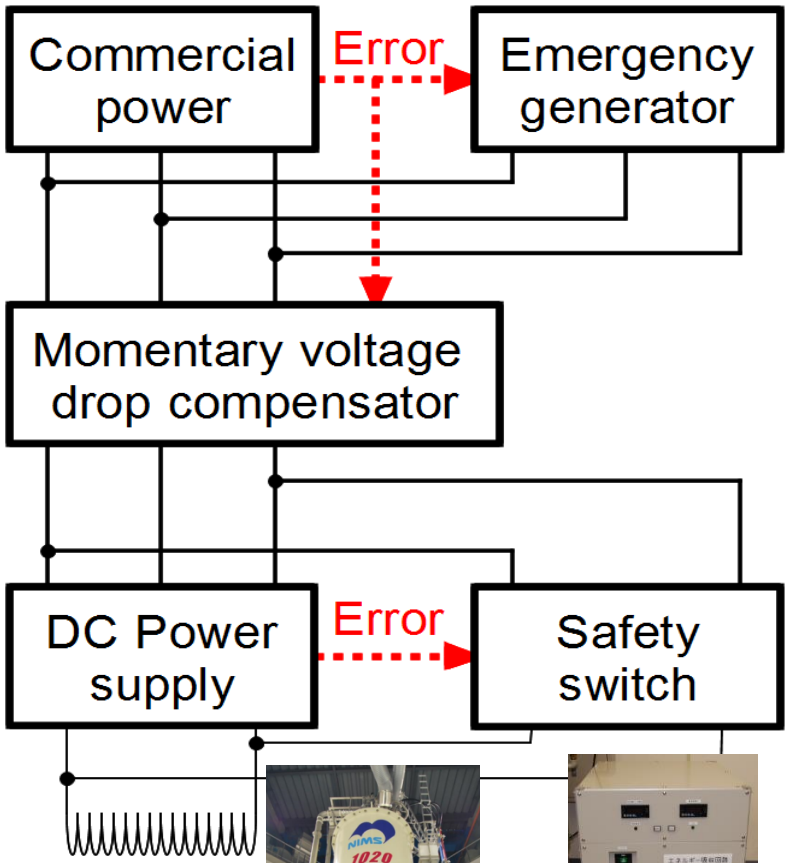
Diagram of power supply system



- Power (commercial power or emergency generator) was supplied to a DC power supply (3 KVA input, 1.2ppm/8hr) through a momentary voltage drop compensator.
- Emergency generator takes 10 seconds to run after the power outage.
- Momentary voltage drop compensator (a kind of UPS) supplies 4 KVA for 15 seconds.
- In case of power supply shuts down, safety switch closes.

S. Matsumoto, to be published

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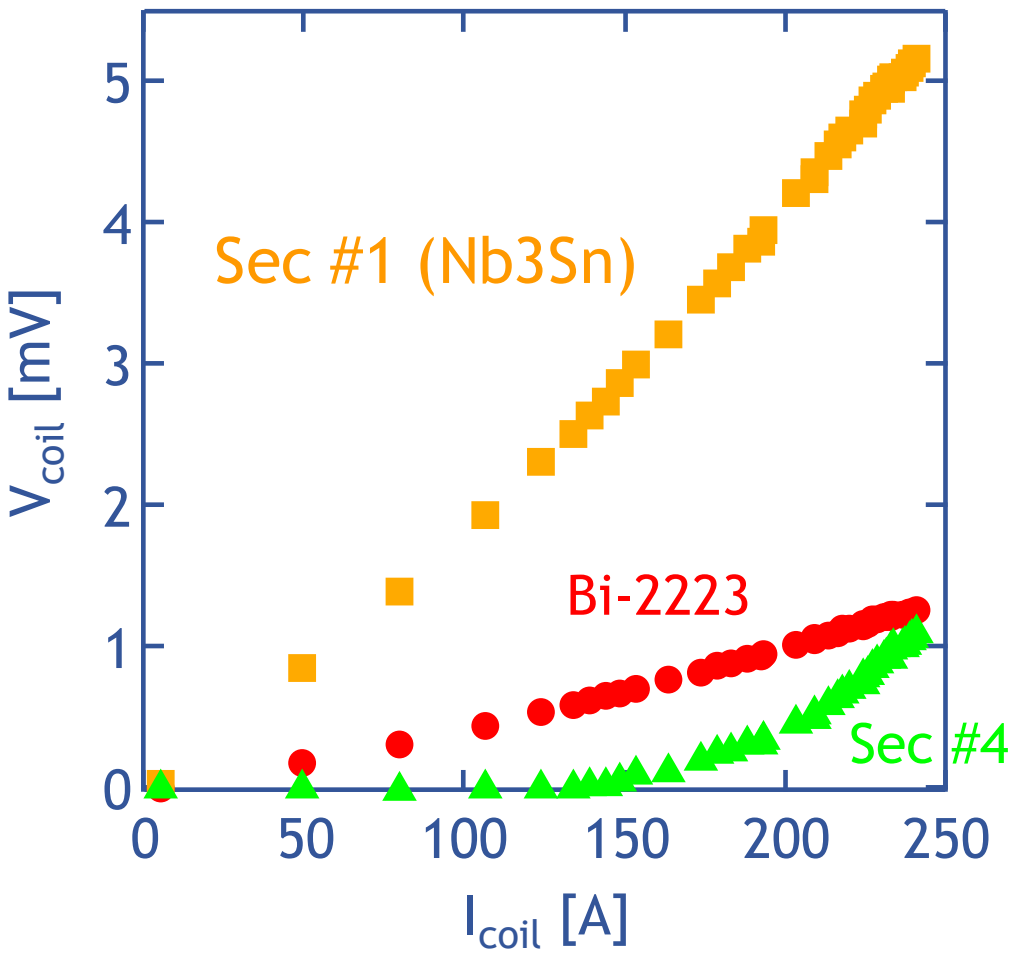
Coils Resistance



Bi-2223 innermost coil
- 5.28 μΩ at 240.5 A (305 mW)
- 1.6 times larger than the design confirmation test
- 5 joints including the coil

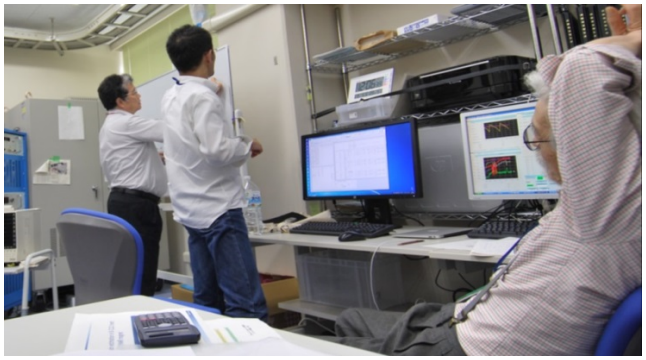
Section #1 (Nb_3Sn)
- 21.5 μΩ at 240.5 A (1.24 W)
- Possible degradation in superconducting joint(s)

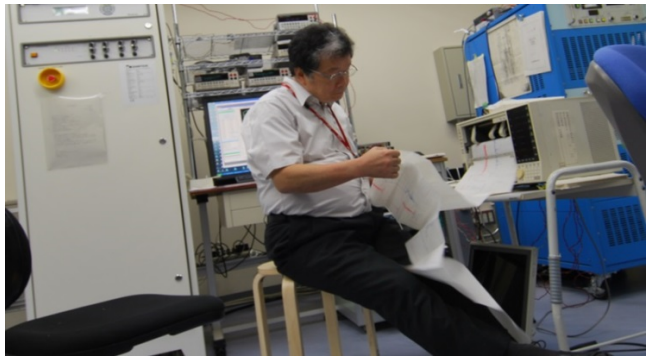
Section #4 (Nb3Sn)
- 4.16 μΩ at 240.5 A (267 mW)
- Degraded joint generated voltage at I>140 A

Heat load from coil= 1.8 W

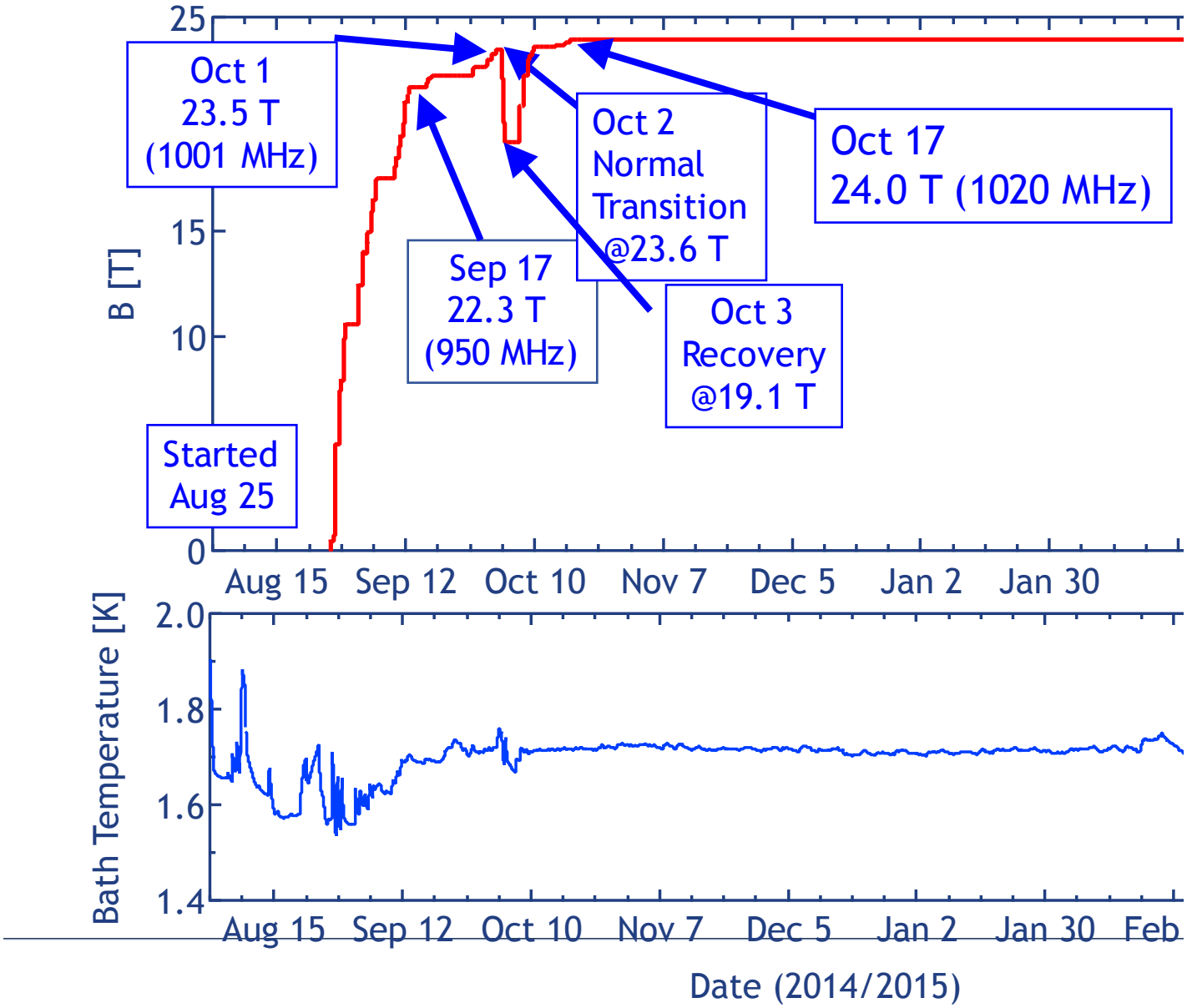
Final decision: Target field 1030 MHz → 1020 MHz

During ramp up





Ramp up and operation



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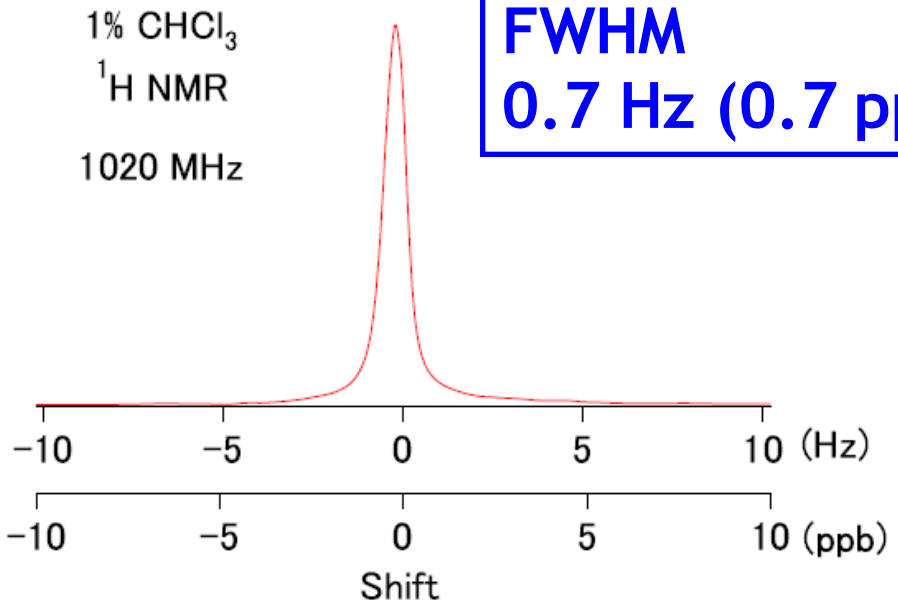
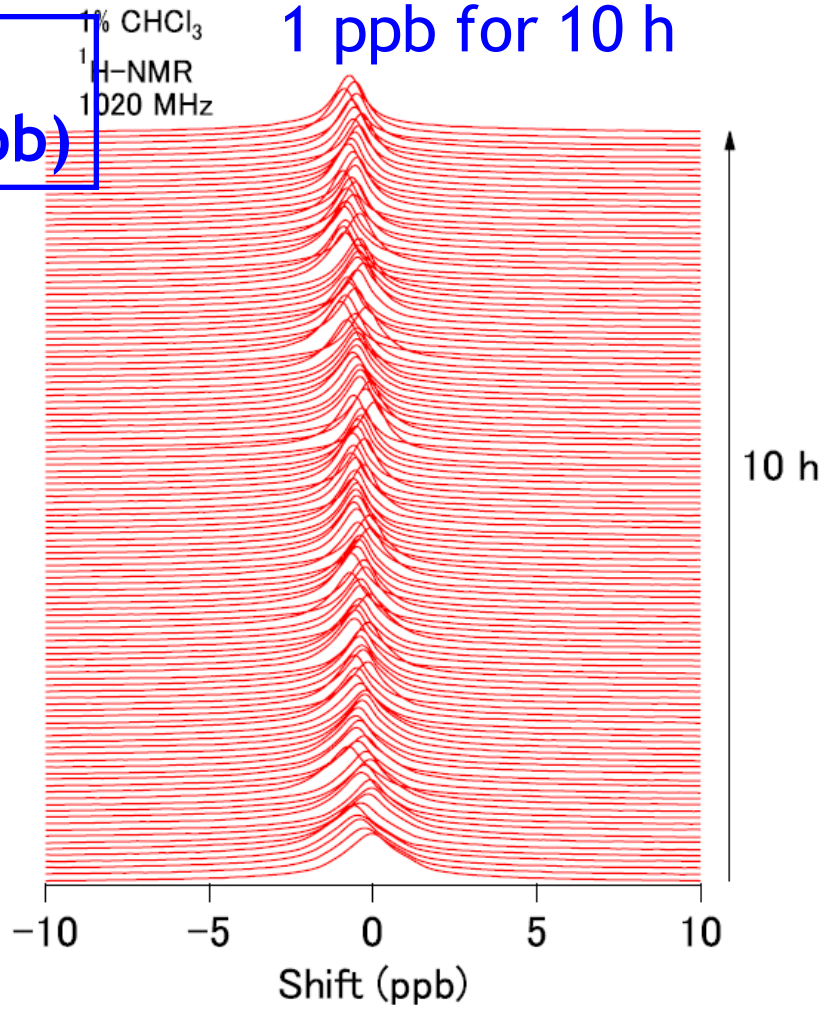
Stability and homogeneity evaluation by NMR



FWHM
0.7 Hz (0.7 ppb)

1 ppb for 10 h



Internal ^{2}H lock operation

K. Hashi et al., JMR, 256 (2015) 30

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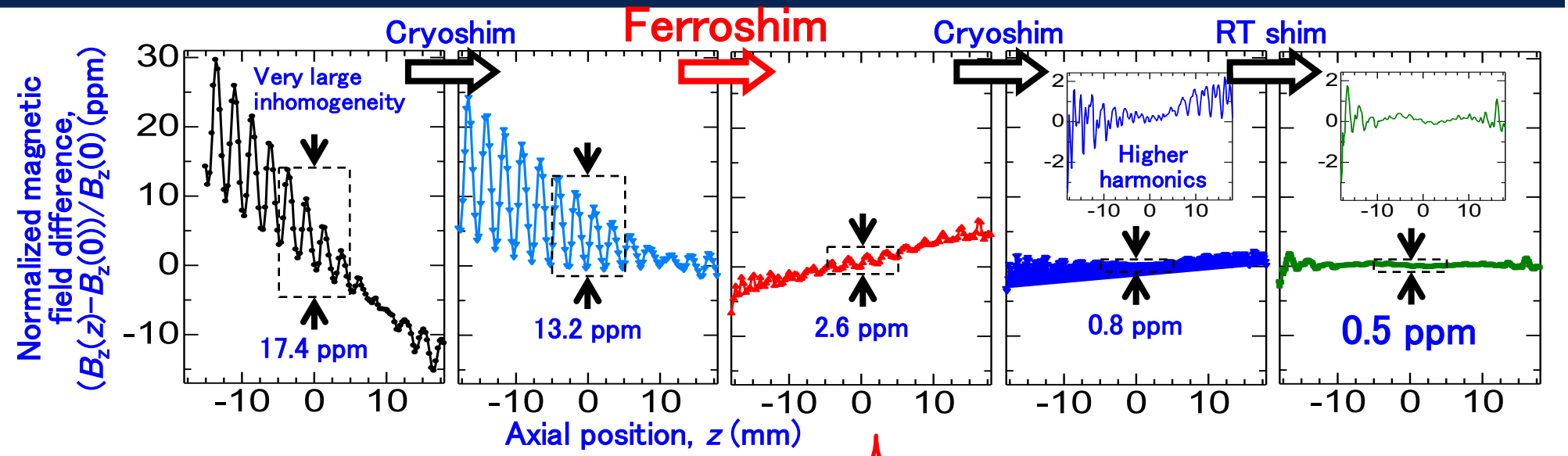
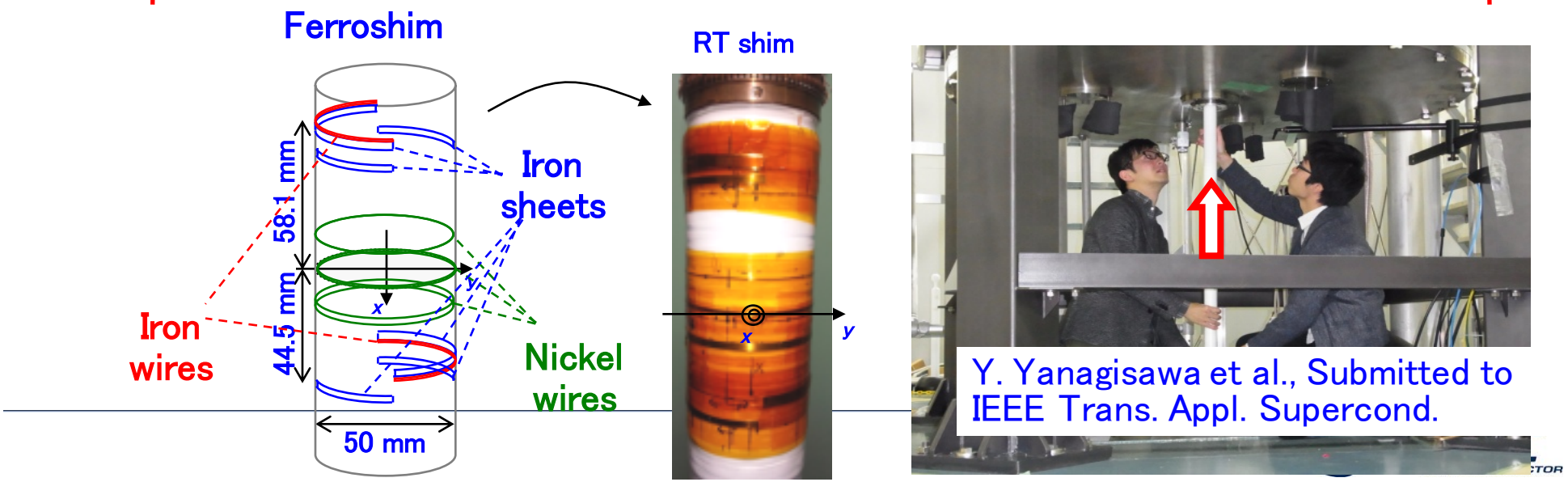
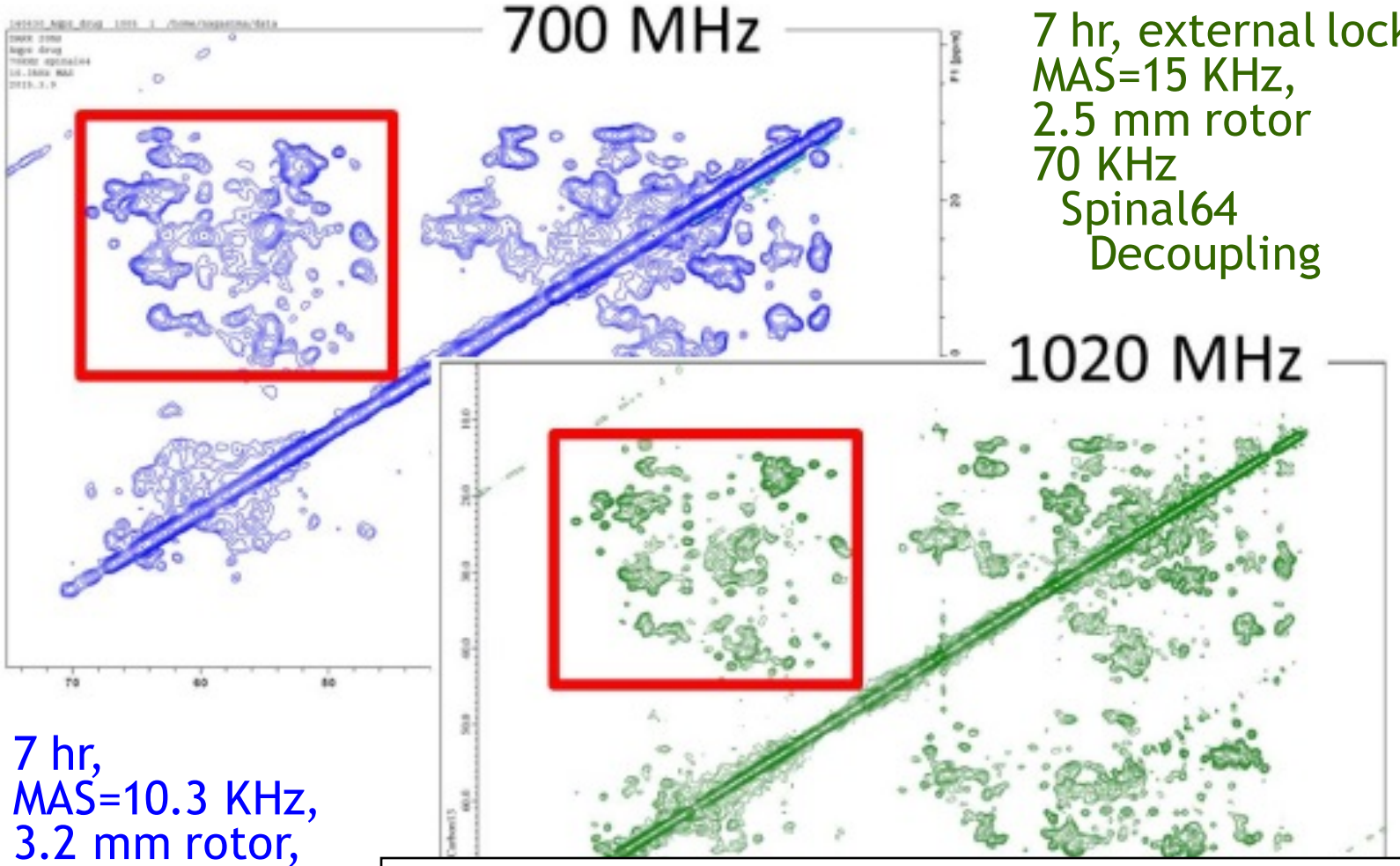
Shimming





Y. Yanagisawa et al., Submitted to IEEE Trans. Appl. Supercond.

NMR data (2D-DARR AquaporinZ)



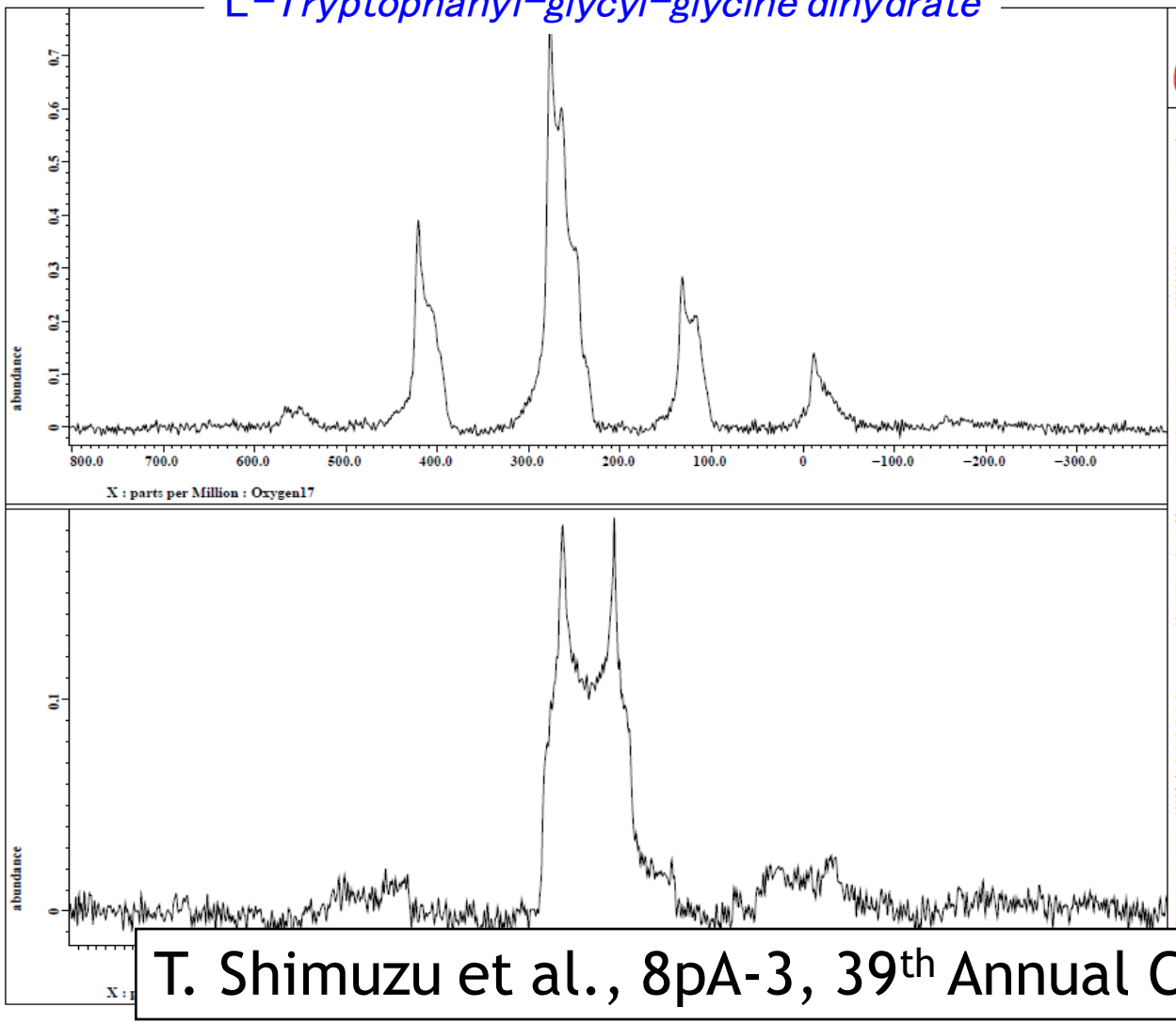
7 hr, external lock
MAS=15 KHz,
2.5 mm rotor
70 KHz
Spinal64
Decoupling

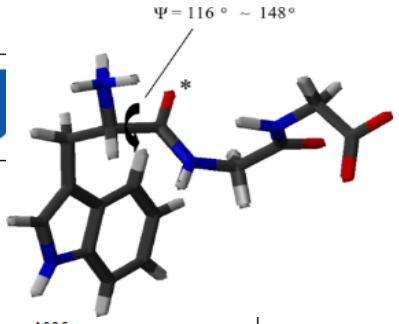
7 hr,
MAS=10.3 KHz,
3.2 mm rotor,
70 KHz
Spinal64
Decoupling

1) H. Maeda et al., Submitted to eMagRes.
2) K. Hashi et al., Submitted to Chemistry Letters

NMR data (^{17}O WGG peptide)

L−*Tryptophanyl−glycyl−glycine dihydrate*






```
Filename
Author
Experiment
Sample_id
Solvent
Creation_time
Revision_time
Current_time

Comment
Data_format
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Dim_units             = [kHz]
Dimensions            = X
Spectrometer          = JNM-ECA600II

Field_strength        = 14.09636928[T] (600[
X_acq_duration        = 5.12[ms]
X_domain              = 17O
X_freq                = 81.36233625[MHz]
X_offset              = 220.0[ppm]
X_points              = 1024
X_prescans            = 0
X_resolution          = 195.3125[Hz]
X_sweep               = 200[kHz]
X_sweep_clipped       = 200[kHz]
Irr_domain            = Proton
Irr_freq              = 600.1723046[MHz]
Irr_offset            = 5[ppm]
Clipped               = FALSE
Mod_return            = 1
Probe_recovery        = 0[s]
Scans                 = 80000
Total_scans           = 80000

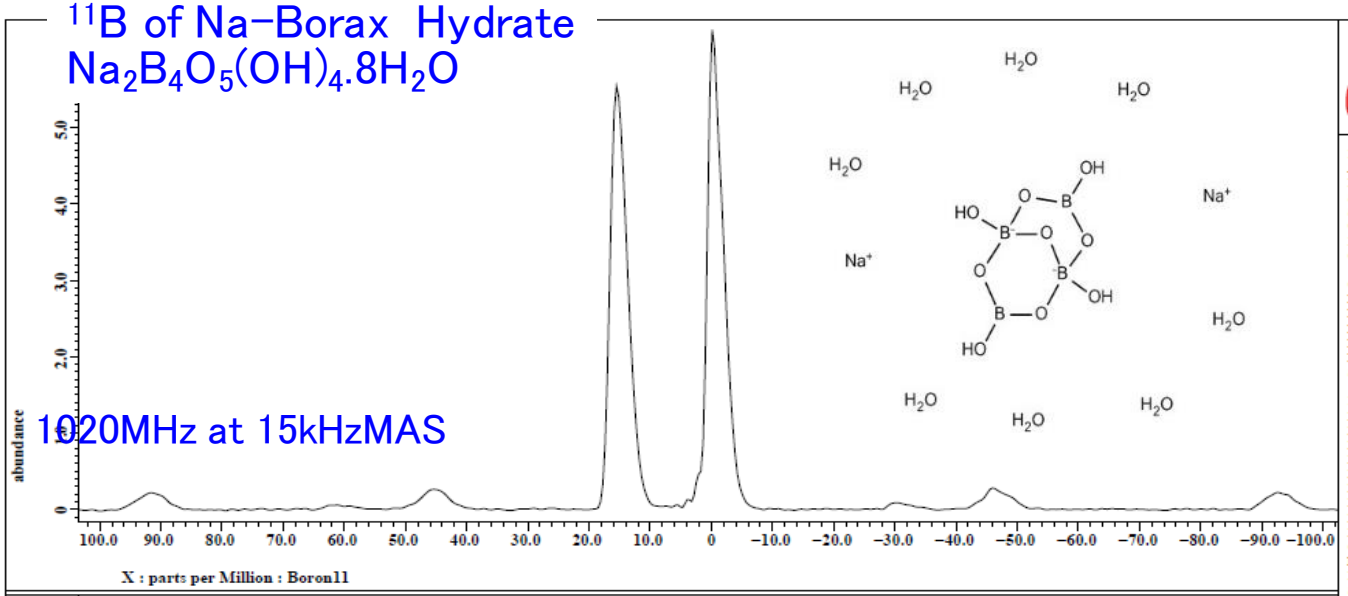
X_acq_time            = 5.12[ms]
X_dwell               = 5[us]
X_pulse               = 0.1[us]
Dec_setup             = #Setup Decouplin
Irr_amp_dec           = 0[m%]
Irr_atn               = 79[dB]
Irr_noise             = CW
Irr_pwidth            = 0[us]
Irr_setup             = #Setup Irradiati
Obs_amp_pulse         = 100[%]
Obs_angle_prep        = 90[deg]
Obs_atn               = 0[dB]
Obs_setup             = #Setup Observe P
Obs_width             = 1[us]
Obs_width_90          = 1[us]
Atn_setup             = #Experiment Atte
Autoshim_track        = AUTOSHIM OFF
Depth2                = FALSE
Depth2_setup          = #Setup DEPTH2 ba
Initial_wait          = 10[ms]
Presat_setup          = #Setup Presatura
Presaturation         = FALSE
Recvr_gain            = 50
Recycle_setup         = #Setup Recycle T
Relaxation_delay      = 1[s]
Relaxation_delay2     = 30[ms]
Repetition_time       = 1.00512[s]
Temp_set              = 450.0[dC]
```

T. Shimuzu et al., 8pA-3, 39th Annual Conf. on Mag. 2015

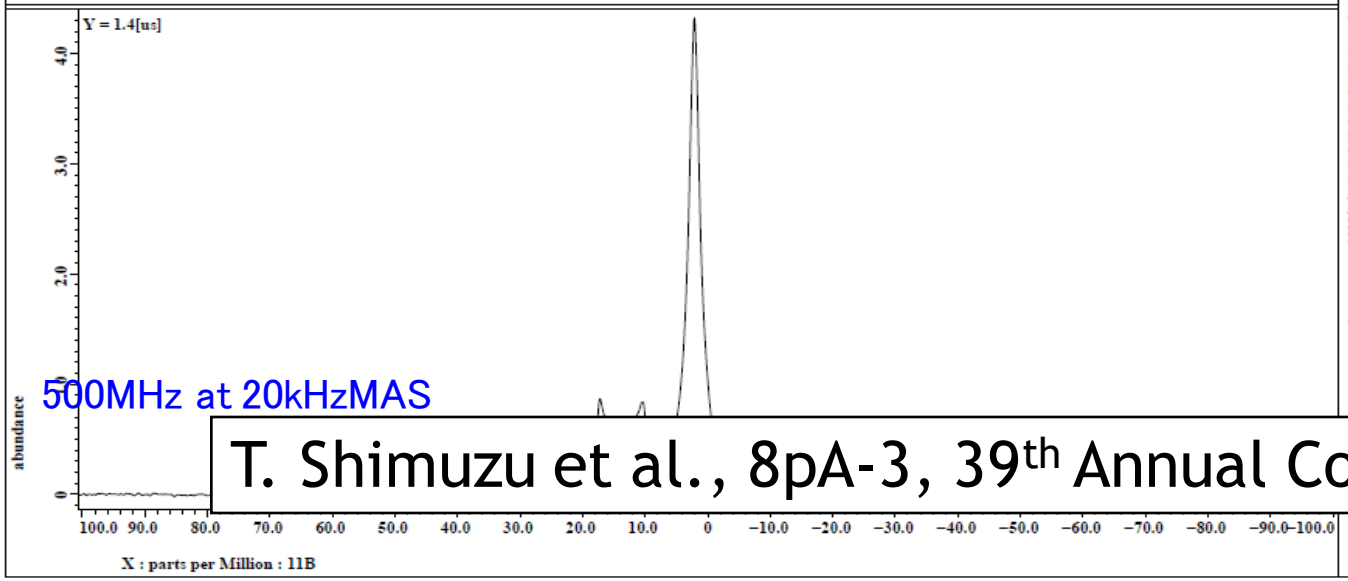
JASTEC SUPERCONDUCTOR

NMR data (^{11}B in borax)

^{11}B of Na-Borax Hydrate
$Na_2B_4O_5(OH)_4.8H_2O$



1020MHz at 15kHzMAS



500MHz at 20kHzMAS

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Revision_time     = 18-NOV-2014 14:42:35
Current_time      = 18-NOV-2014 14:45:29

Comment           = single pulse
Data_format       = 1D REAL
Dim_size          = 4096
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Dim_units         = [kHz]
Dimensions        = X
Spectrometer      = DELTA2_NMR

Field_strength    = 23.97906969[T] (1020[
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X_domain          = 11B
X_freq            = 327.55901306[MHz]
X_offset          = -31[ppm]
X_points          = 1024
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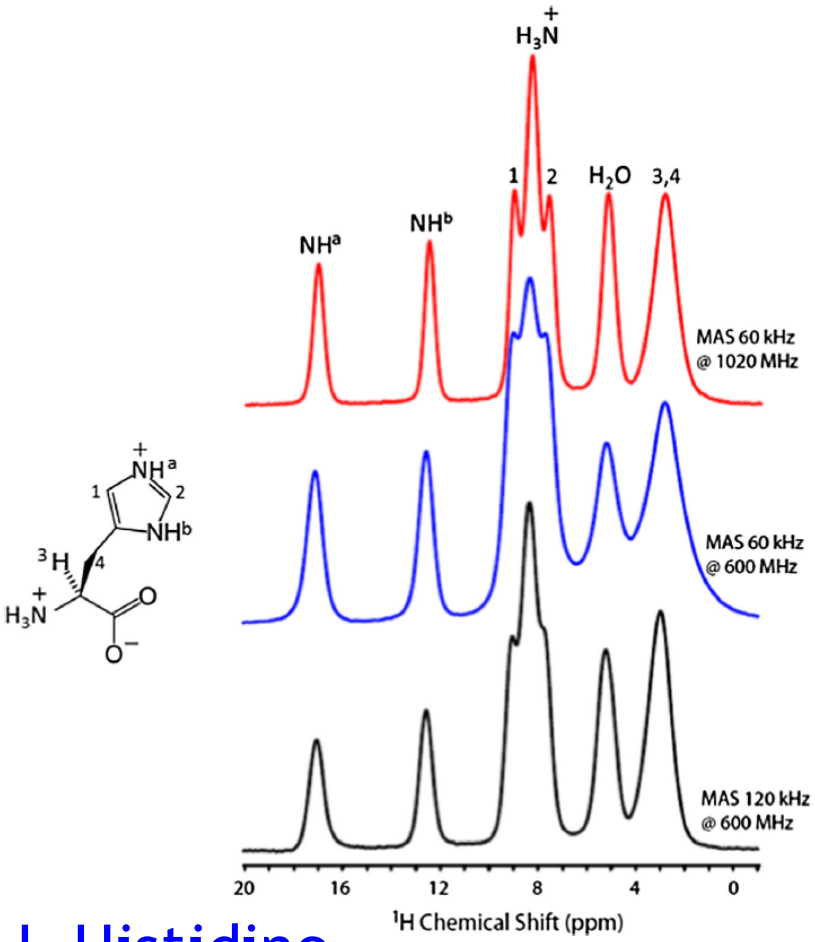
X_sweep           = 500[kHz]
X_sweep_clipped   = 500[kHz]
Clipped           = FALSE
Mod_return        = 1
Probe_recovery    = 12[us]
Scans             = 32
Total_scans       = 32

X_acq_time        = 2.049[ms]
X_dwell           = 2[us]
X_pulse           = 0.1[us]
Obs_amp_pulse     = 100[%]
Obs_angle_prep    = 90[deg]
Obs_atn           = 9[dB]
Obs_setup         = #Setup Observe P
Obs_width         = 3[us]
Obs_width_90      = 3[us]
Obs_xmtr          = 7
Atn_setup         = #Experiment Atte
Autoshim_track    = AUTOSHIM OFF
Depth2            = FALSE
Depth2_setup      = #Setup DEPTH2 ba
Initial_wait      = 10[ms]
Recvr_gain        = 30
Recycle_setup     = #Setup Recycle T
Relaxation_delay  = 4[s]
Repetition_time   = 4.002049[s]
Temp_get          = 21.1[dC]
```

T. Shimuzu et al., 8pA-3, 39th Annual Conf. on Mag. 2015

SUPERCONDUCTOR

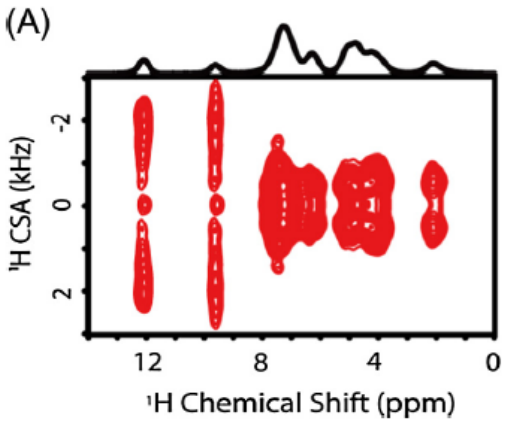
NMR data（L-Histidine･HCl･H_2O, L-Tyrosine）



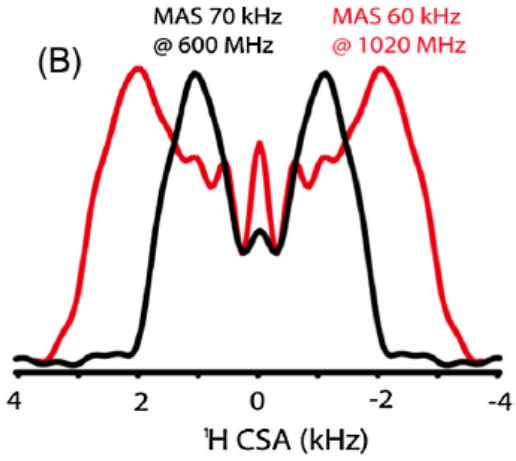
L-Histidine,
1D ultrafast MAS proton spectra

L-Tyrosine

70KHz 600MHz　60KHz 1020MHz



2D CSA/CS correlation at 60 KHz



1H CSA lineshapes

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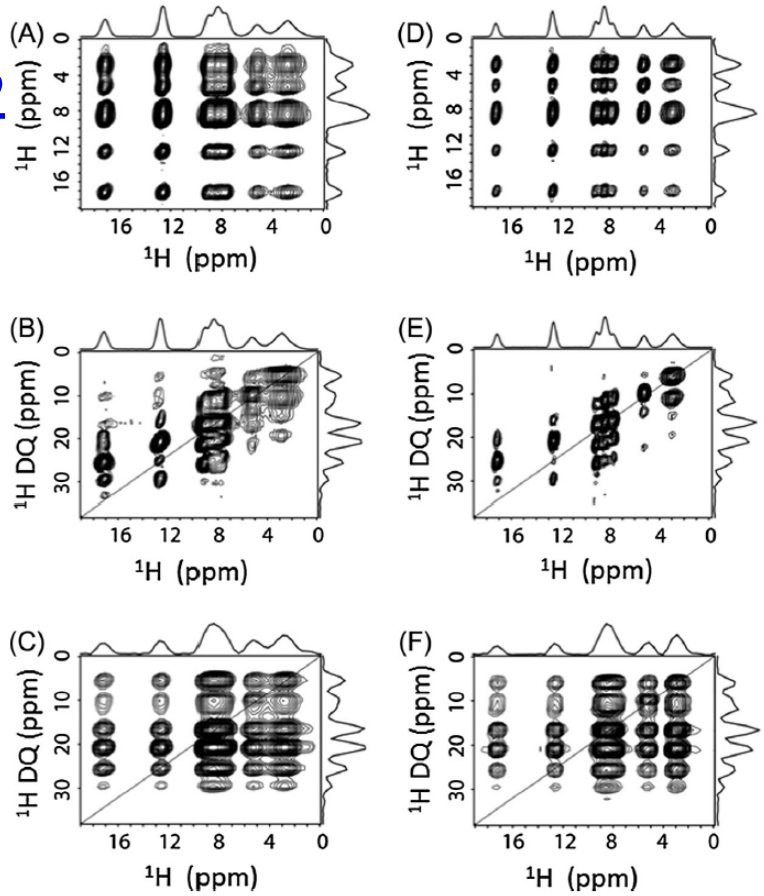
NMR data（L–Histidine・HCl・H_2O）

SQ1/SQ2

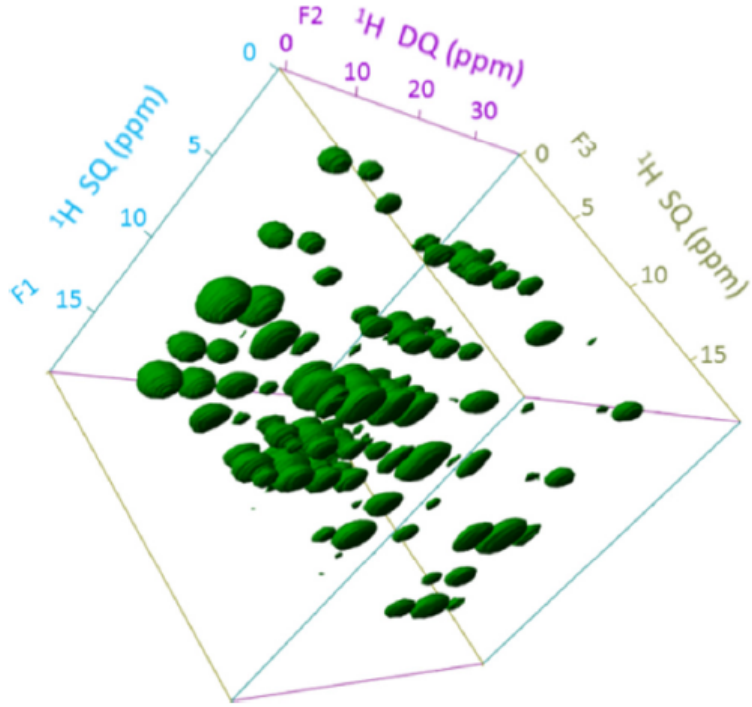
DQ/SQ2

DQ/SQ1



600 MHz　　1020 MHz

Spectral projection



3D SQ/DQ/SQ spectrum
60 KHz, 16t_1, t_2, 8 scans,
6s recycle delay,
BABA-XY16 sequence
with 133.3μs excitation

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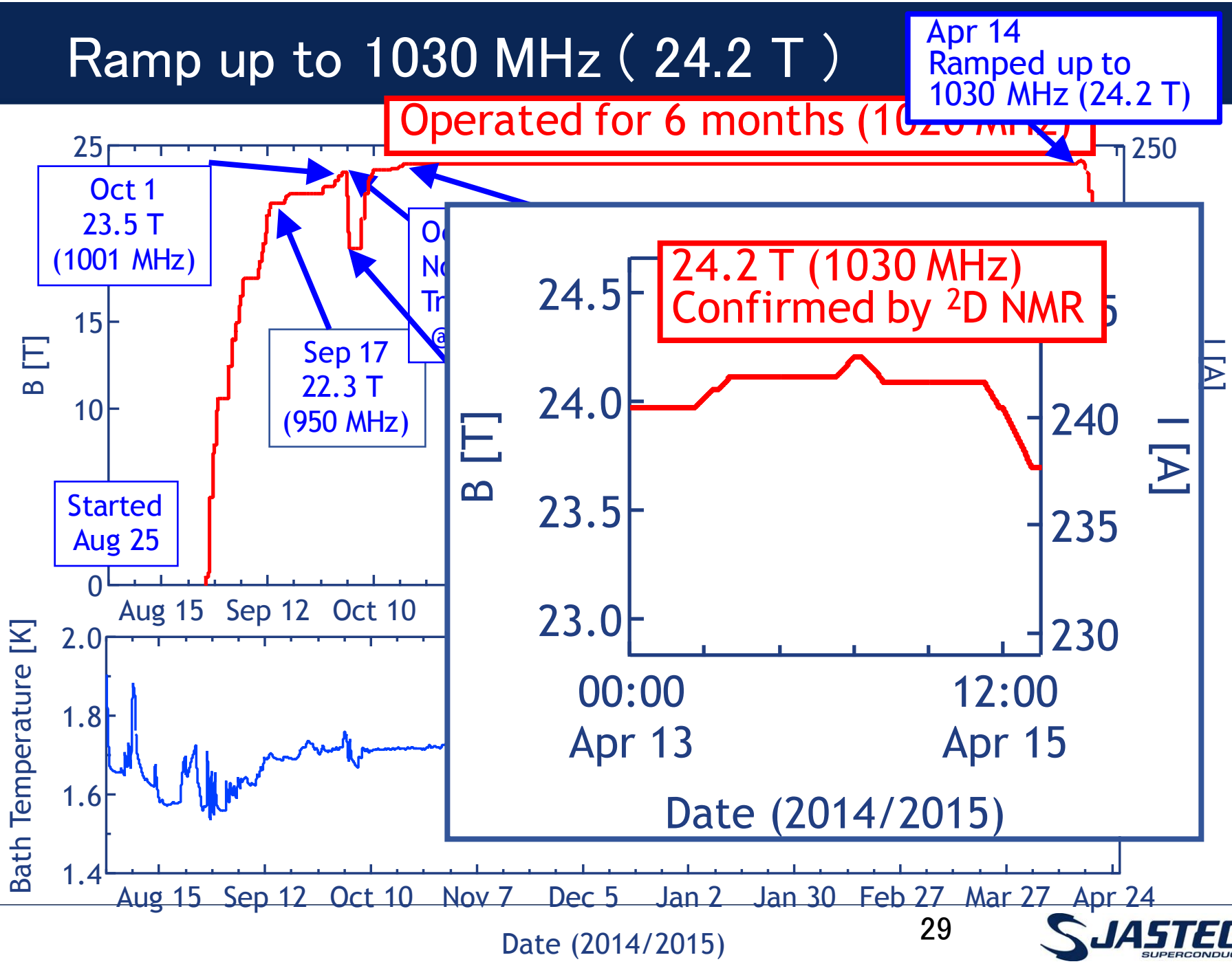
Ramp up to 1030 MHz (24.2 T)



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Summary

We succeeded in upgrading 920 MHz NMR superconducting magnet to 1020 MHz using Bi-2223 innermost coil.

- Bi-2223 innermost coil was developed.
- Seriously damaged by the huge earthquake.
- After >2 year restoration, cooled down to ~1.6 K and operated at ~1.7 K.
- Generated 1020 MHz.
- Operated at 1020 MHz NMR for 6 months by power supply driven mode.
- Stability : 1 ppb/10 hr
- Homogeneity : 0.7 ppb
  with internal ^{2}H lock system operation
- Generated 1030 MHz (24.2 T).

Reference

[1] K. Hashi et al., "Achievement of 1020 MHz NMR," *J. Mag. Res.*, 256, 30, 2015.
[2] G. Nishijima et al., to be published.; MT24 2PoBD-08.
[3] Y. Nishiyama et al., J. Mag. Res., 261, 1, 2015.
[4] G. Nishijima et al., EUCAS 2015 1A-LS-O2.1.
[5] T. Kiyoshi et al., "NMR upgrading project towards 1.05 GHz," *IEEE TAS*, 18, 860, 2008.
[6] T. Kiyoshi et al., "Bi-2223 Innermost Coil for 1.03 GHz NMR Magnet," *IEEE TAS*, 21, 2110, 2011.
[7] T. Shimizu et al., The 39th Annual Conference on Magnetics in Japan, 2015.

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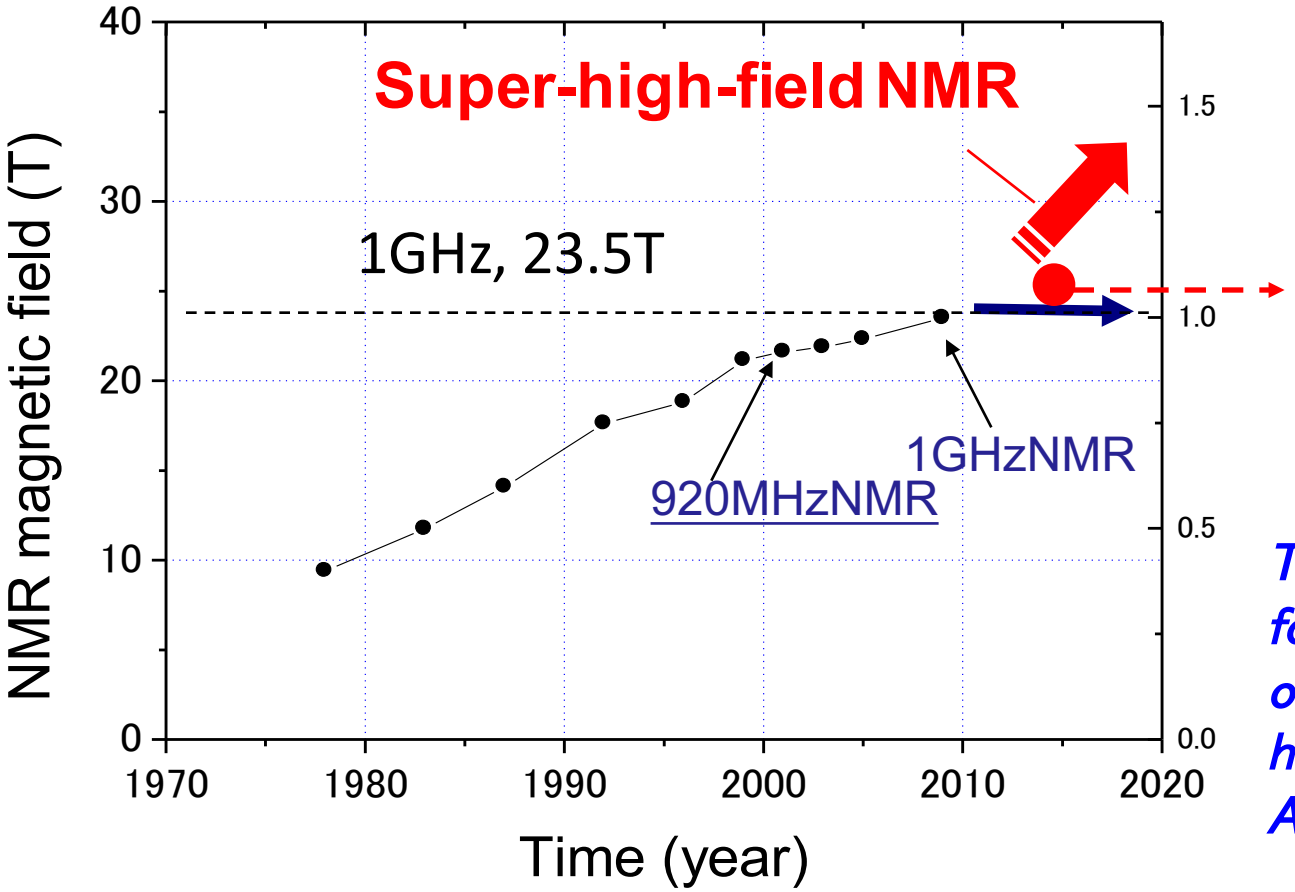
Short Summary



This is one small step for an NMR magnet, but one great leap for super high field NMR (N. A. Armstrong of Apollo 11)

JASTEC SUPERCONDUCTOR

Thank you for your attention