



Workshop "Ultrahigh field NMR and  
MRI: Science at the cross roads @ NIH  
Nov. 12, 2015 8:50-9:10

# Toward Super-High Field and Ultra- Compact Size NMR Magnets Operated Beyond 1 GHz (review)

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and Technology Agency(JST)



# Outline

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## Introduction

- 1. Real characteristics of medium-field LTS/HTS NMR magnets**
- 2. 1.02 GHz LTS/Bi-2223 NMR magnet**
- 3. New perspective on the super-high-field NMR magnets**

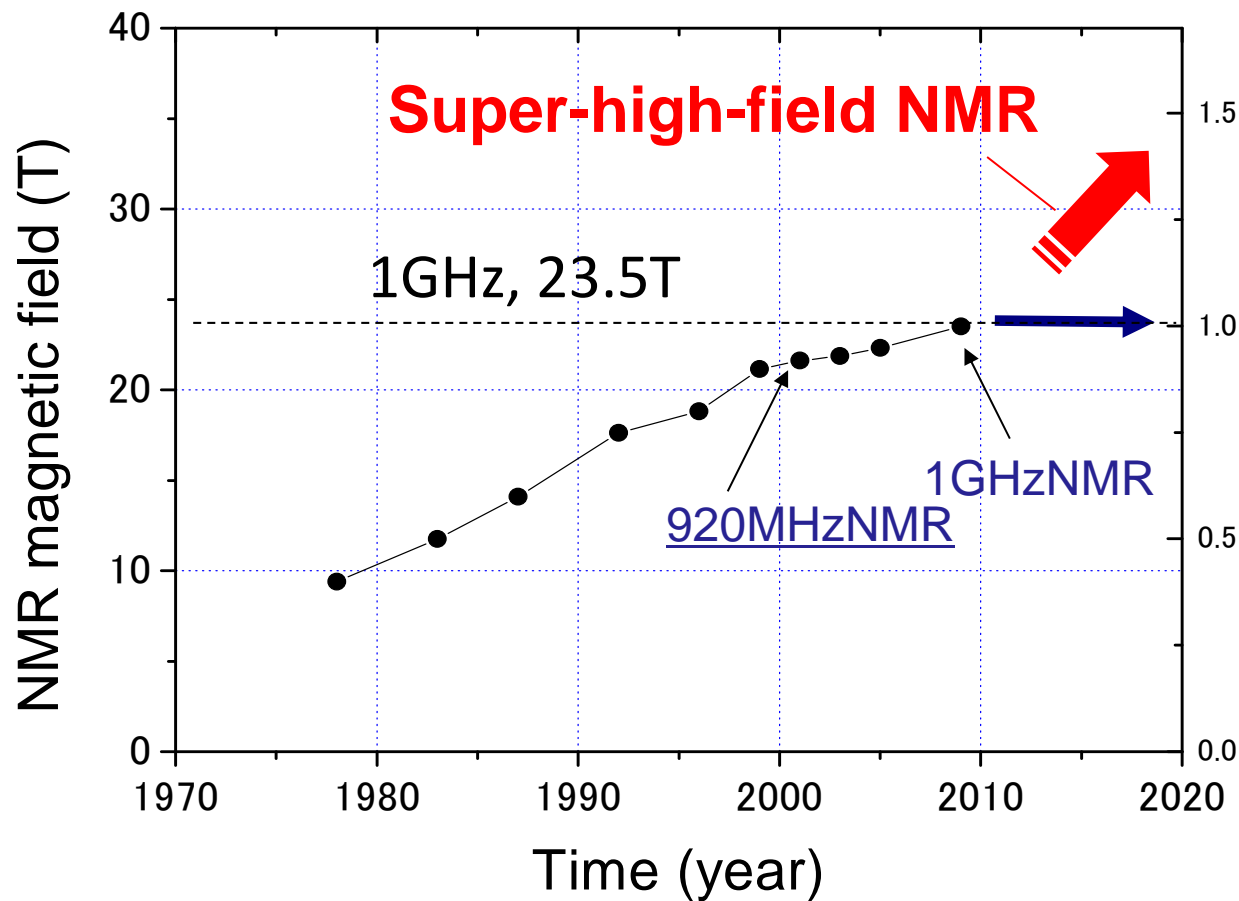


# Introduction

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# History of the NMR magnetic field

Our target is to develop super-high field NMRs



**High Temperature Superconducting (HTS) Magnets**

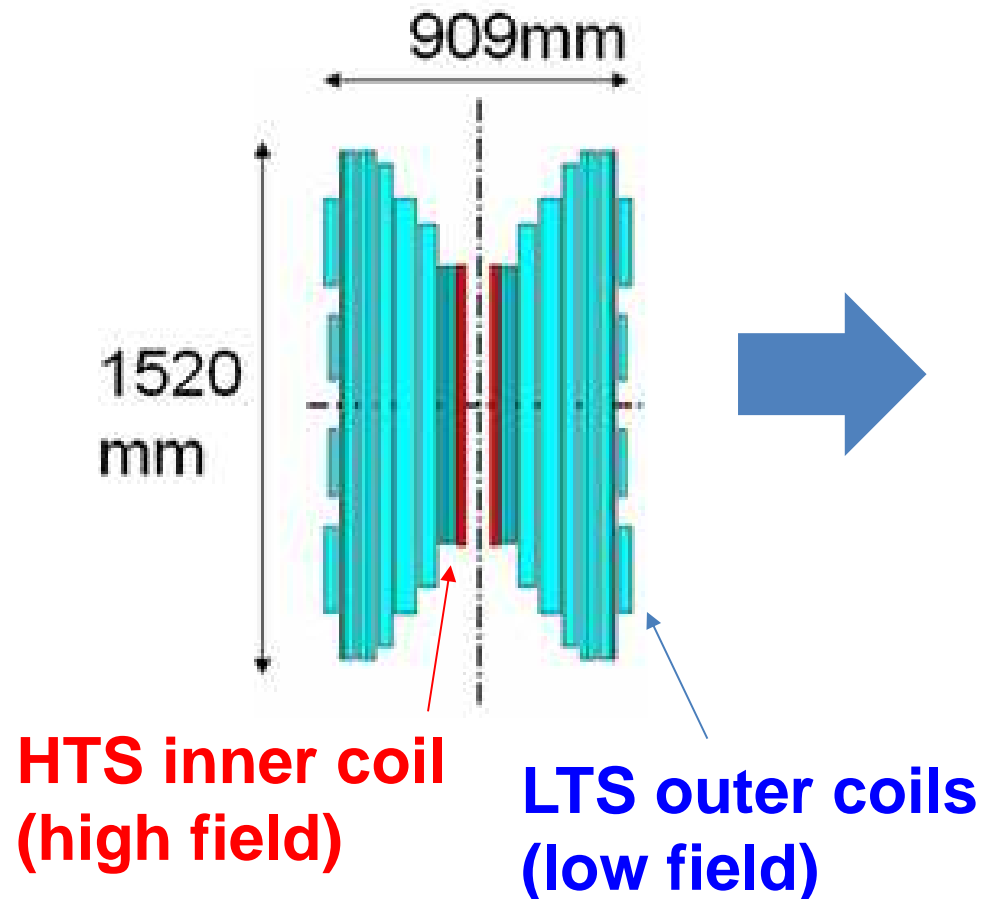
•  $H_{c2} \gg 30T$

**Low Temperature Superconducting (LTS) Magnets**

•  $H_{c2} < 25-30T$

Critical magnetic field

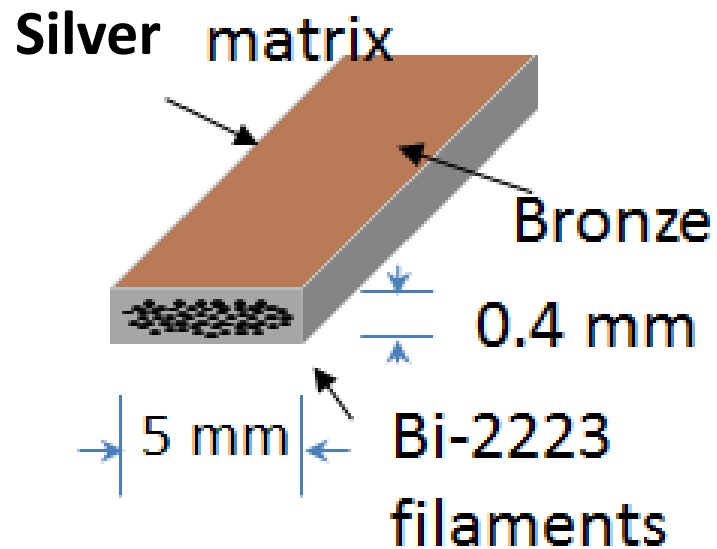
# Basic configuration of a super-high field NMR



**LTS/HTS NMR magnet**  
**Cross-sectional view**

# Two commercialized HTS tape conductors

## 1st generation HTS

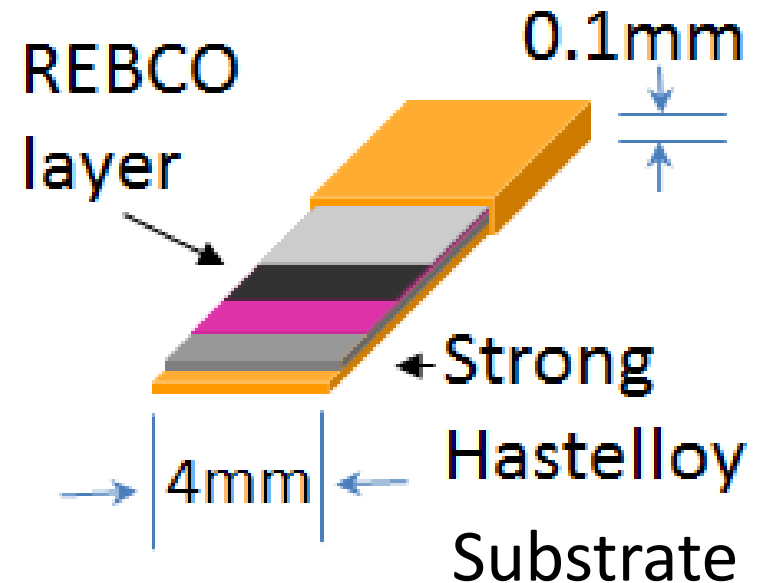


### Bi-2223 conductors



**200MPa (insufficient)**

## 2nd generation HTS



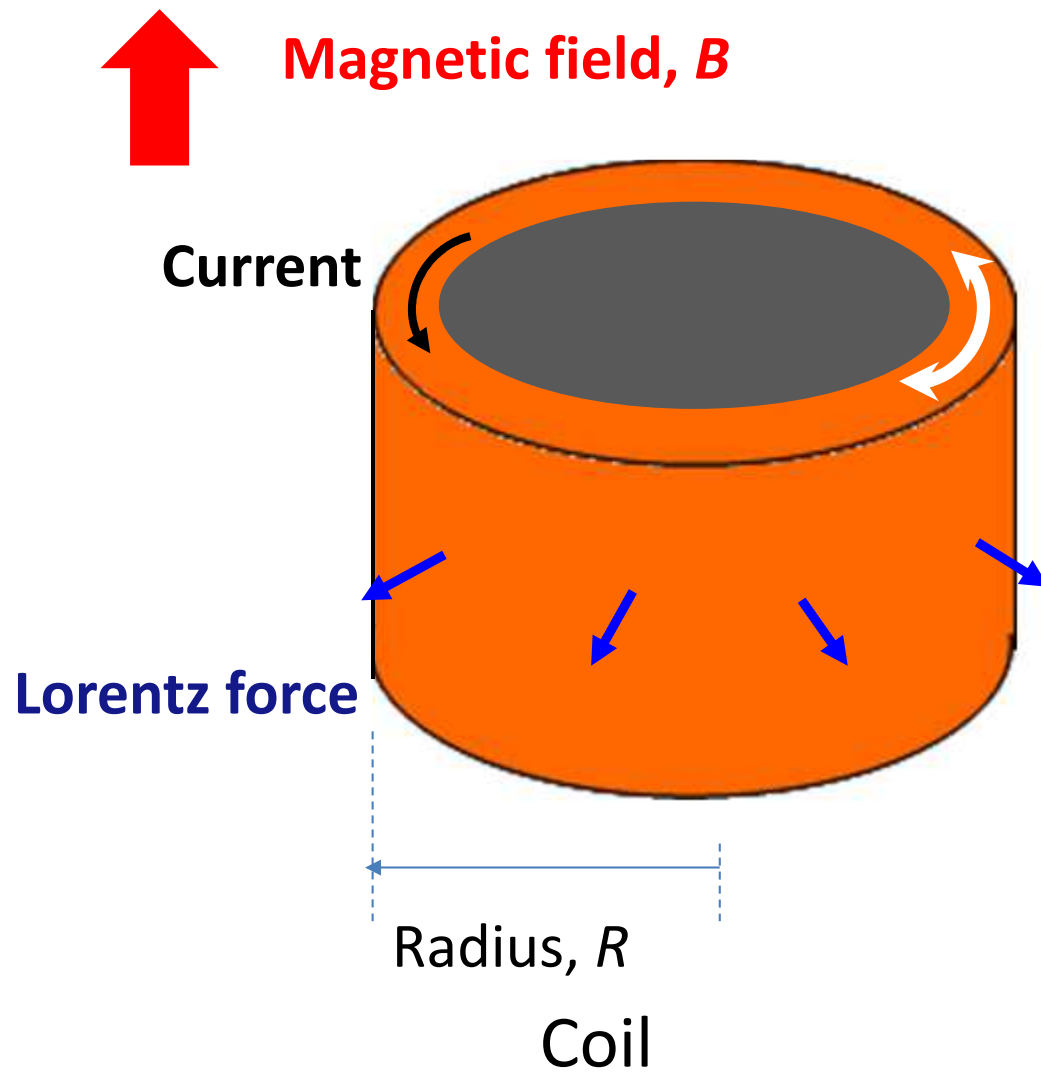
### REBCO conductors



YBCO

**700MPa (strong)**

# High conductor strength reduces the size of the magnet



$$\text{Tensile stress} = BJR$$

$B$ : Field strength

$J$ : Current density

$R$ : Coil radius



If the HTS conductor is strong, a higher current density is available, resulting in compact size magnet



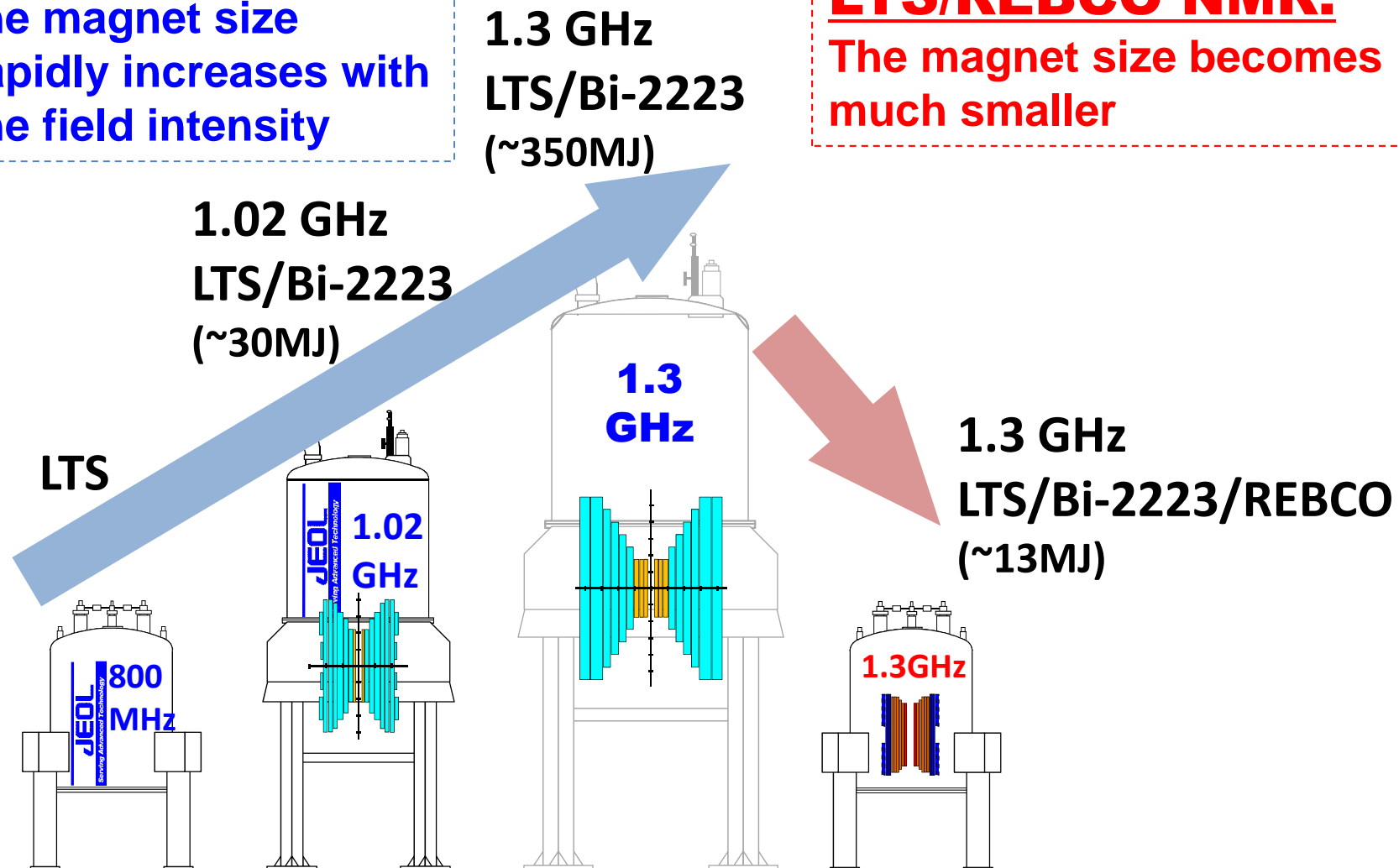
# Current perspective on the super-high-field NMR magnet

## LTS/Bi-2223 NMR:

the magnet size rapidly increases with the field intensity

## LTS/REBCO NMR:

The magnet size becomes much smaller



**My question:  
is this true or is this false?**





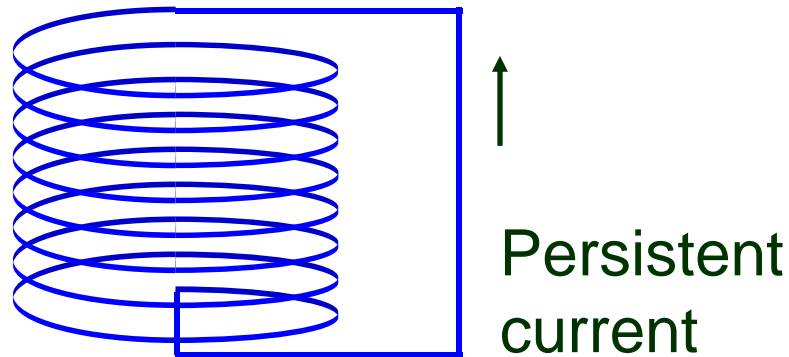
# 1. Real characteristics of medium-field LTS/HTS NMR magnets

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*To check this out, a medium-field LTS/Bi-2223 NMR magnet and an LTS/REBCO NMR magnet were made, tested and compared so that we could achieve the basic knowledge on the performance of LTS/HTS NMRs*

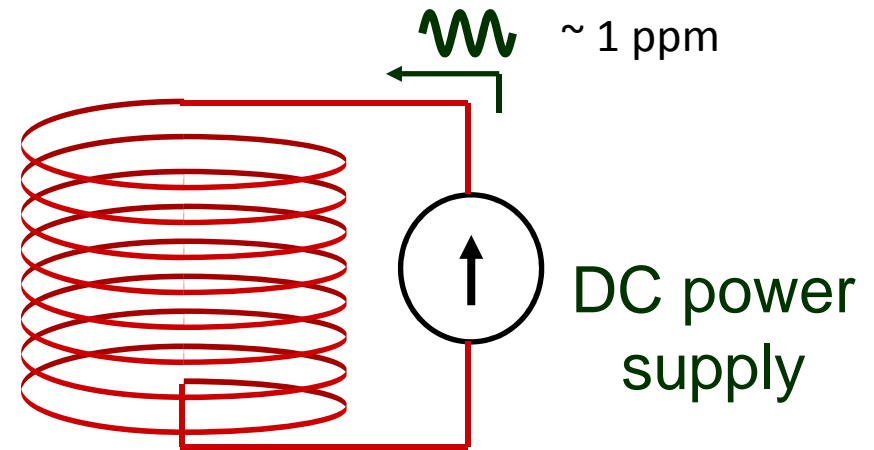
# Operation mode of NMR magnets

## LTS NMR



**Persistent current mode**

## HTS NMR



~~Superconducting joint~~

**Driven mode**

**Field stabilization is difficult**

# A 500MHz-class LTS/ HTS NMR magnet operated in driven mode

**Bi-2223  
inner coil**



(2009)

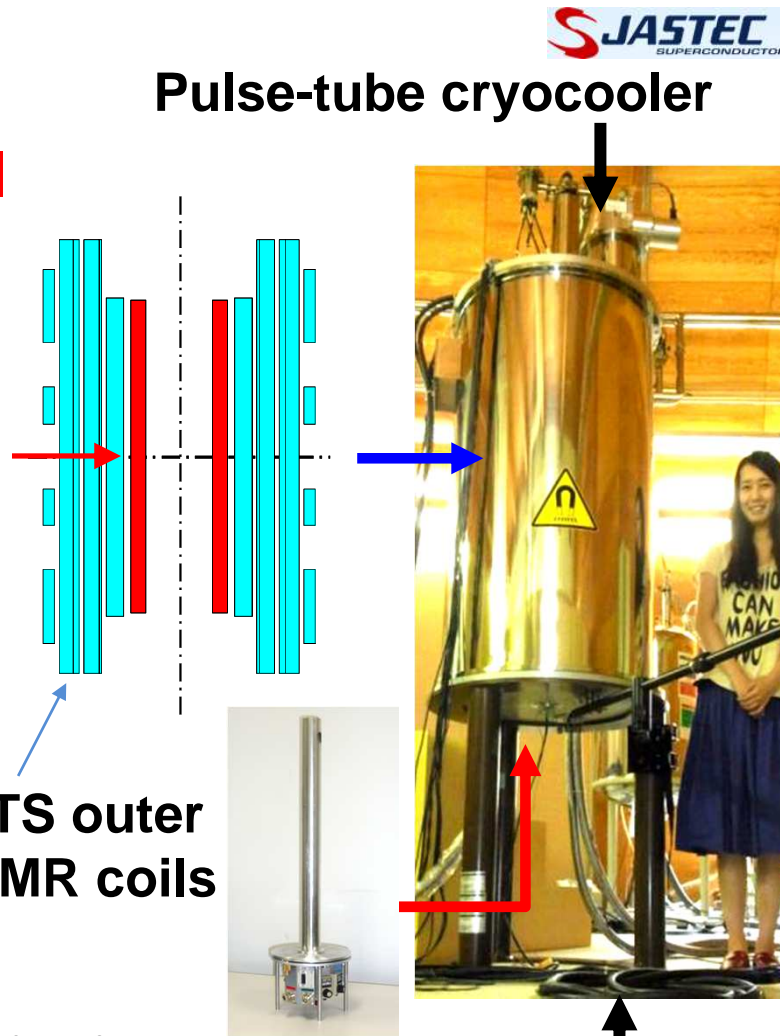
Id 81mm  
Od 121mm  
L 375mm

**REBCO  
inner coil**



(2013)

Id 81mm  
Od 119mm  
L 400mm



**NMR probe**

**Ultra-stabilized  
DC-power supply**

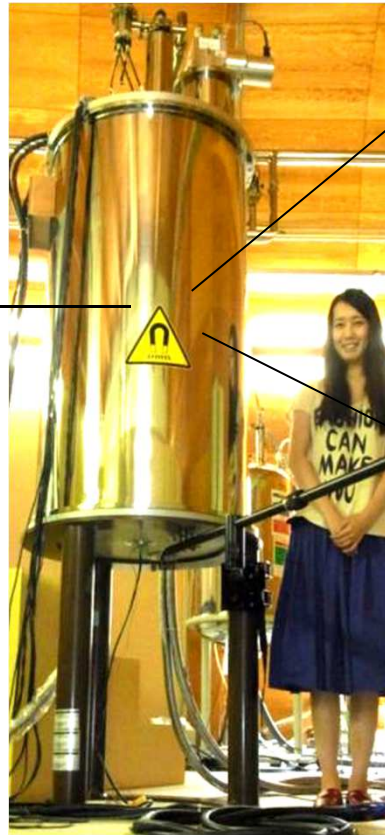


- Y. Yanagisawa et al, *JMR* 203, 274-282 (2010)
- T. Kiyoshi et al, *IEEE TAS* 20, 714-717(2010)
- Y. Yanagisawa et al., *JMR*, 249, 38-48(2014)

# Problems of the LTS/HTS NMR magnet

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**(1) Temporal  
magnetic field  
stability  
: 0.1ppb**

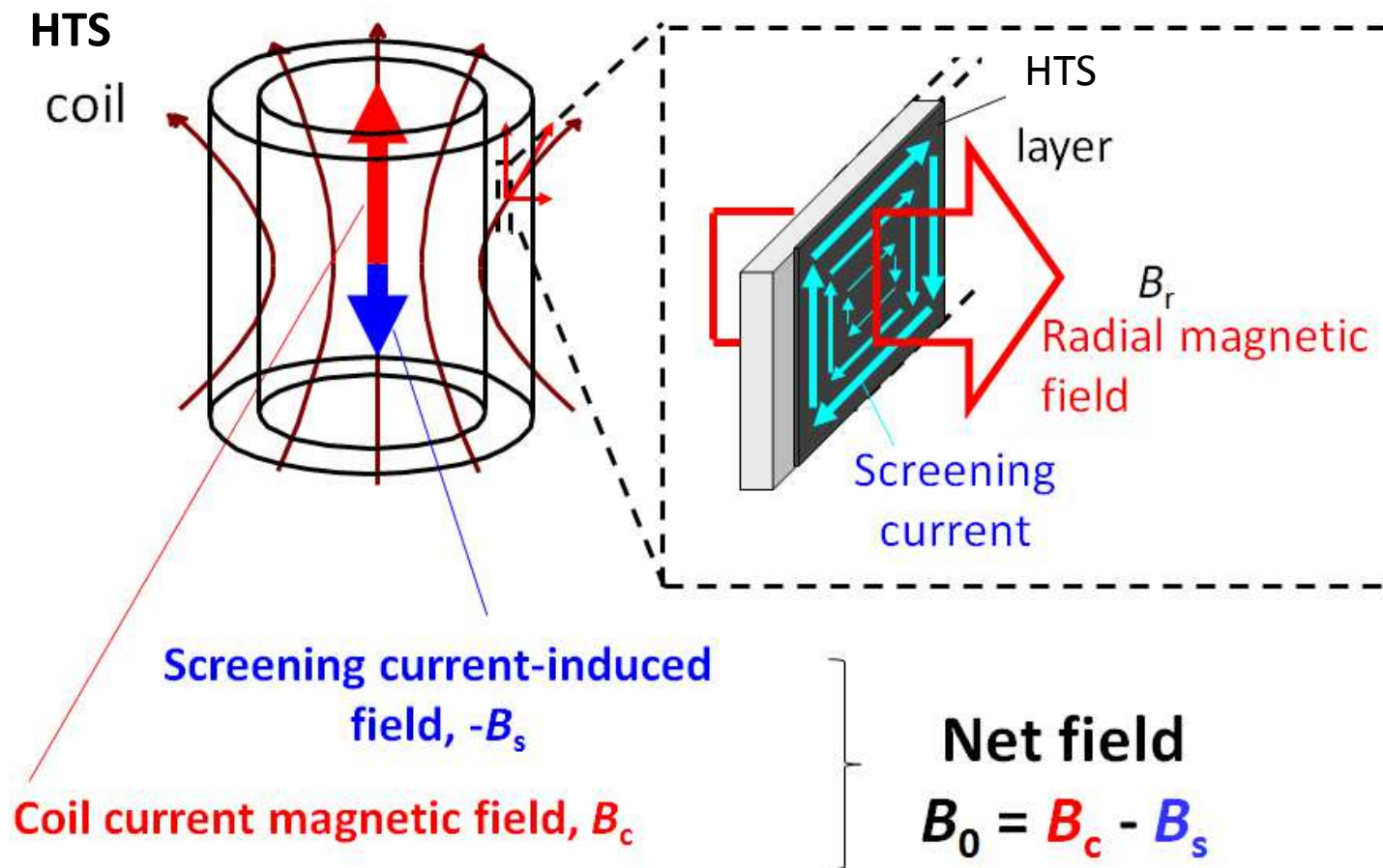


**Field  
intensity**

**(2) Spatial  
magnetic field  
homogeneity  
: 1 ppb**

# **(1) Temporal magnetic field stability**

# Screening current induced magnetic field generated during the magnet charge

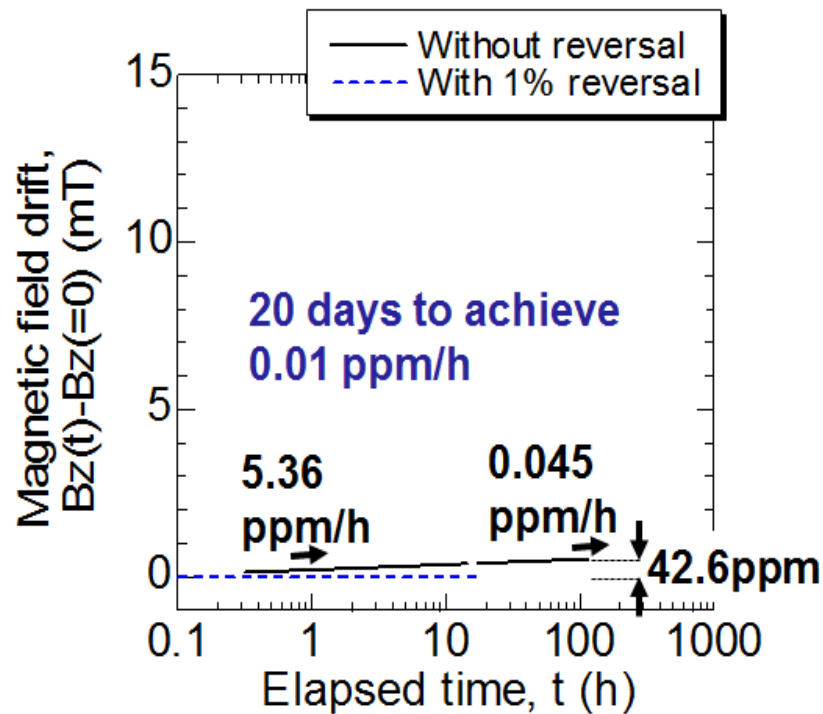


If  $B_s$  is relaxed with time,  $B_0$  shows a positive drift (increase)

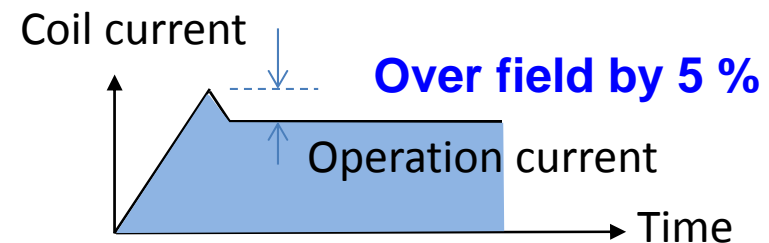
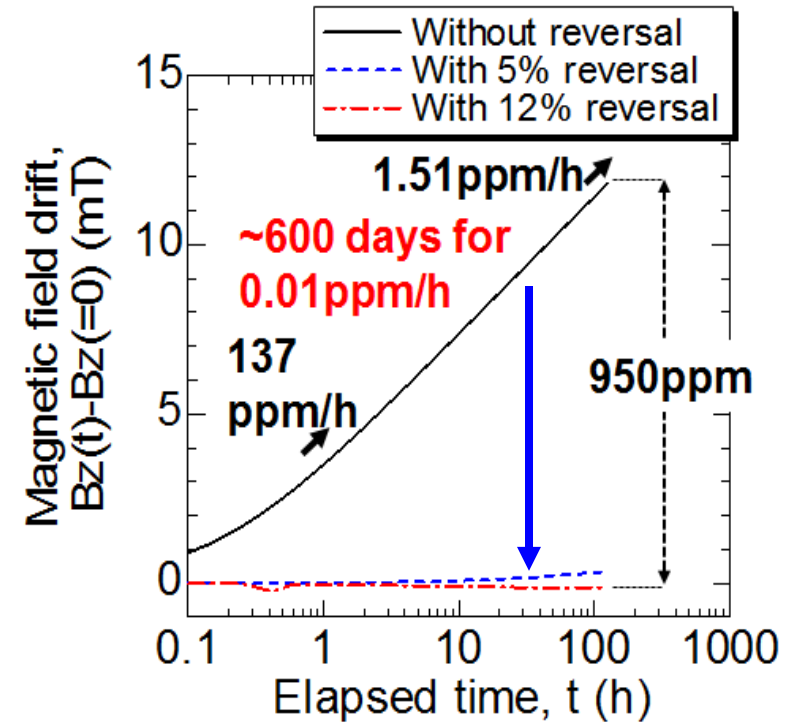


# Temporal field drift after the magnet charge, due to the relaxation of the screening-current

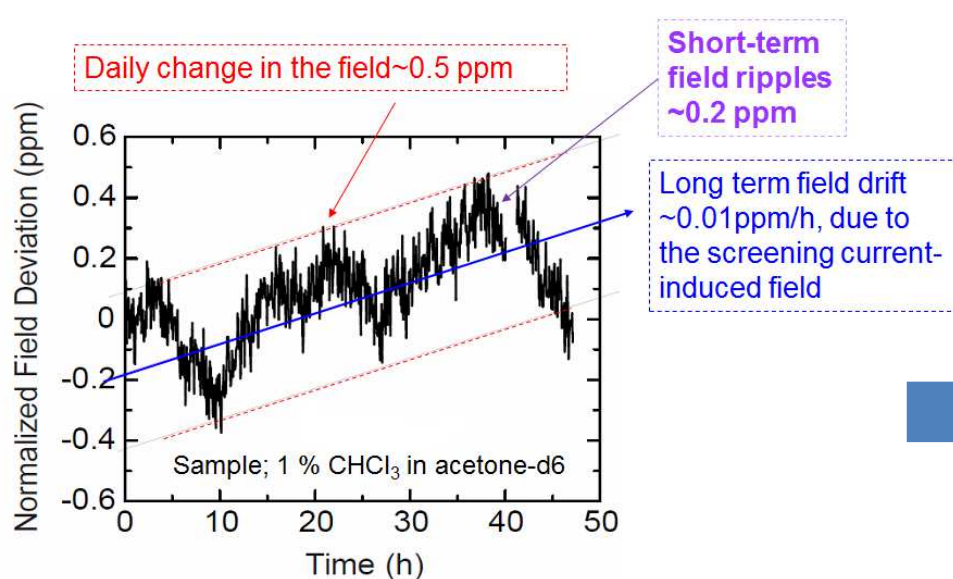
**LTS/Bi-2223 NMR**  
(500MHz, 11.7T)



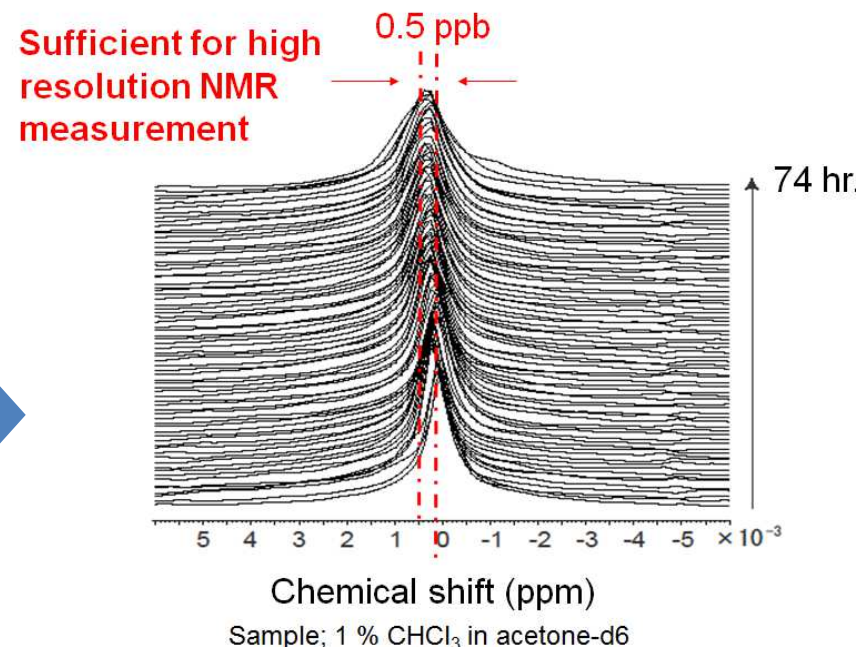
**LTS/REBCO NMR**  
(500MHz, 11.7T)



# Stabilization of the magnetic field fluctuations(LTS/Bi-2223 NMR)



Temporal field fluctuation in driven mode at the 20<sup>th</sup> day



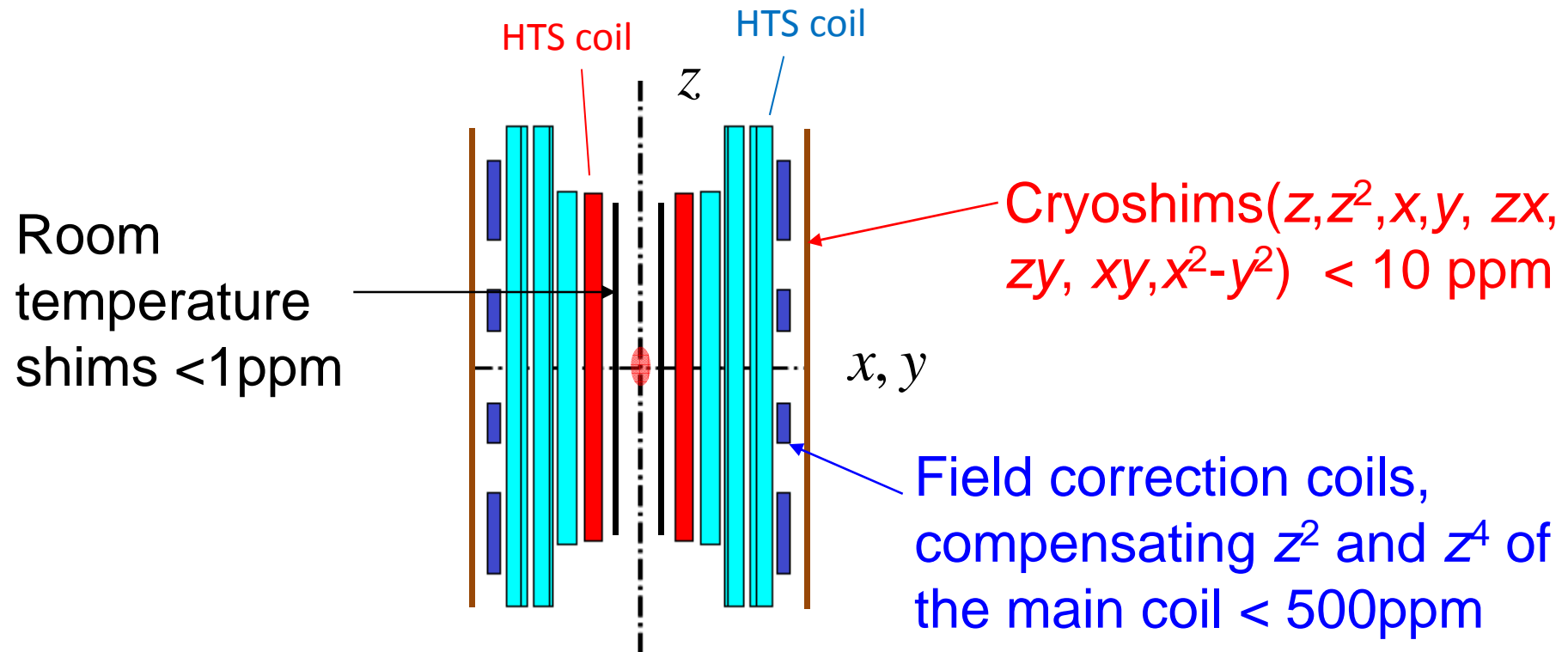
Stabilized magnetic field

**Stabilized by using**

- **Internal <sup>2</sup>H lock for solution NMR**
- **External lock for solid state NMR**

## **(2) Spatial field homogeneity**

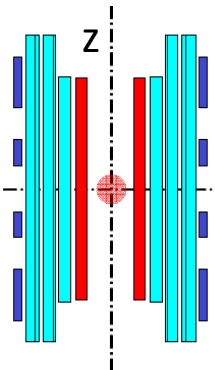
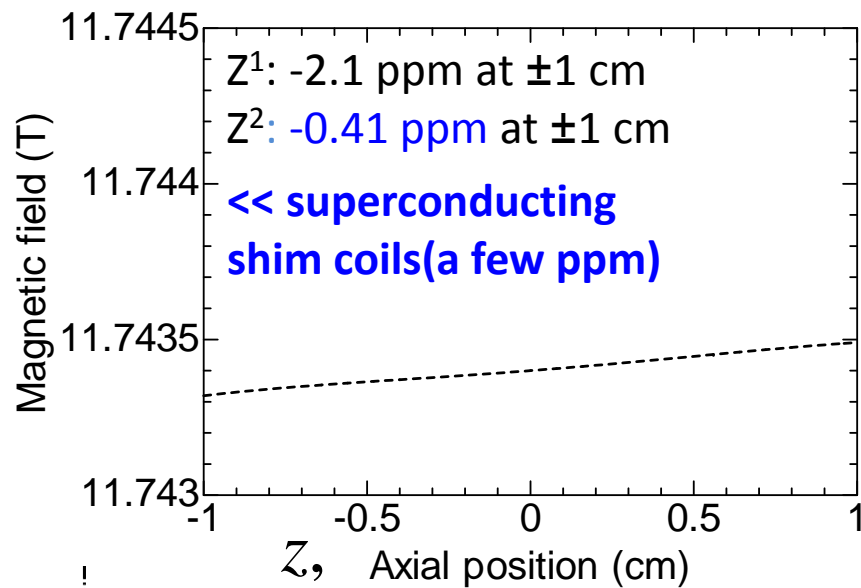
# Magnetic field correction system for the NMR magnet



The magnetic fields generated by the field correction coil and cryoshims are shielded by the screening current induced in the HTS inner coil

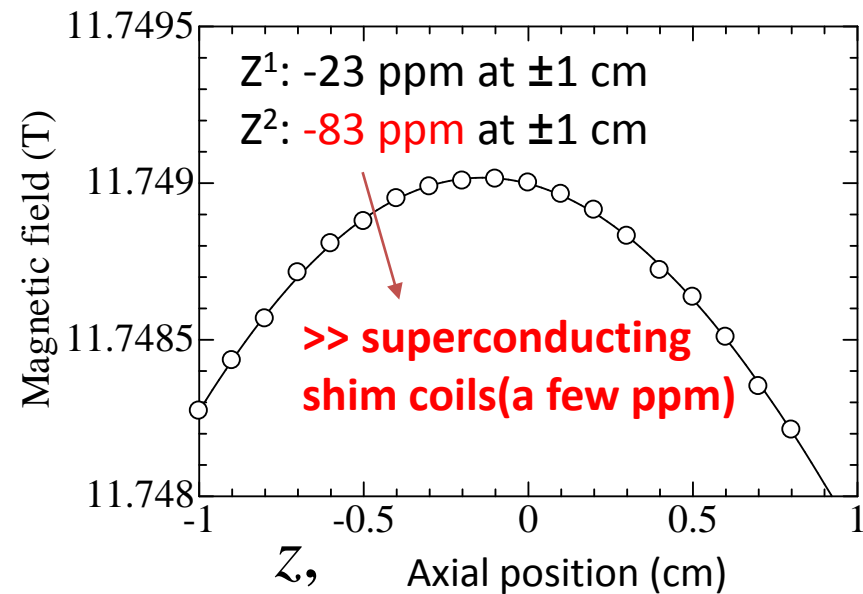
# (a) Field inhomogeneity

## LTS/ Bi-2223 NMR (500 MHz)



Shielding effect is negligible for the Bi-2223 NMR coil

## LTS/ REBCO NMR (500 MHz)



Shielding effect on the correction coil is enormous; the  $z^2$  component generated by the main coil is uncompensated and remains >>  $z^2$  cryoshim

## (b) Performance reduction of cryoshims

	LTS/Bi-2223 NMR	LTS/REBCO NMR
Axial cryoshims (z and z <sup>2</sup> )	→40%	→20%
Radial cryoshims (x, y, zx, zy, xy, x <sup>2</sup> -y <sup>2</sup> )	→20-40%	→5%



**The cryoshim performance is reduced,  
especially for LTS/REBCO NMRs**

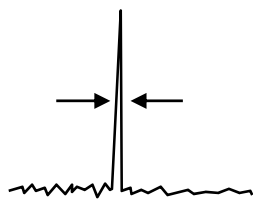
## **(3) NMR spectrum**

# Spectral resolution and sensitivity for three types of NMR spectrometer

	LTS NMR	LTS/Bi2223 NMR	LTS/REBCO NMR
NMR resolution	<1 Hz(2 ppb)	0.7 Hz(1.4 ppb)	15 Hz(38 ppb)
NMR sensitivity S/N	>600	512	28 (400 MHz)

NMR resolution

Chloroform



Nearly the same

NMR sensitivity

Ethyl-benzene

10-times worse than the LTS NMR

<sup>1</sup>H solution NMR spectrum



# The #2<sup>nd</sup> LTS/REBCO NMR magnet

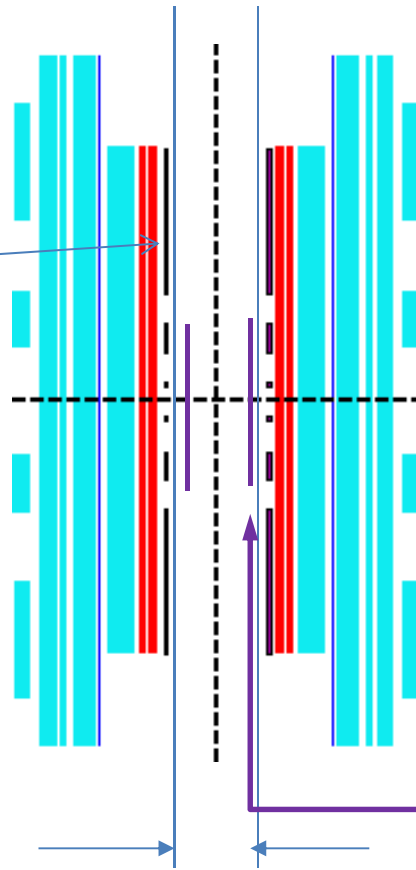
## (2) Ferromagnetic-shims against radial error field harmonics

*x, y, zx, zy, xy, and  $x^2 - y^2$*

### (1) Inner-cryoshims



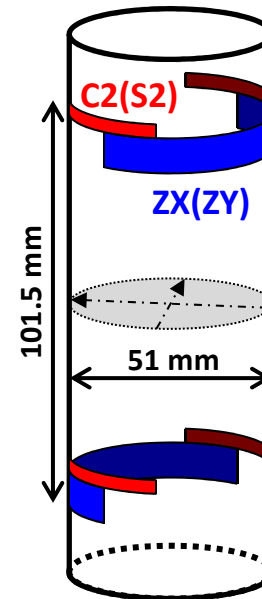
JASTE



RT bore



Steel plates



2014 (RIKEN)



R. Piao et al., to be published in JMR(2016)

# Spectral resolution and sensitivity for three types of NMR spectrometer

	LTS NMR (persistent)	LTS/Bi-2223 NMR (driven mode)	LTS/REBCO NMR (driven mode)
NMR resolution	<1 Hz(2 ppb)	0.7 Hz(1.4 ppb)	0.5Hz(1.3 ppb)
NMR sensitivity	>600	512	<b>318@400MHz</b> <b>(444@500MHz)</b>

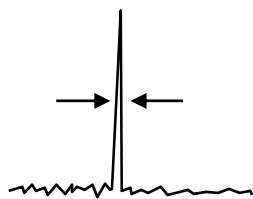


**Nearly the same**

**The field correction was very difficult and required 2months**

NMR resolution

Chloroform

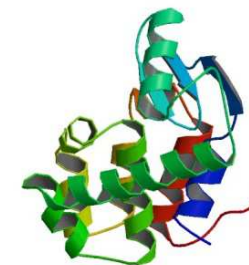


NMR sensitivity

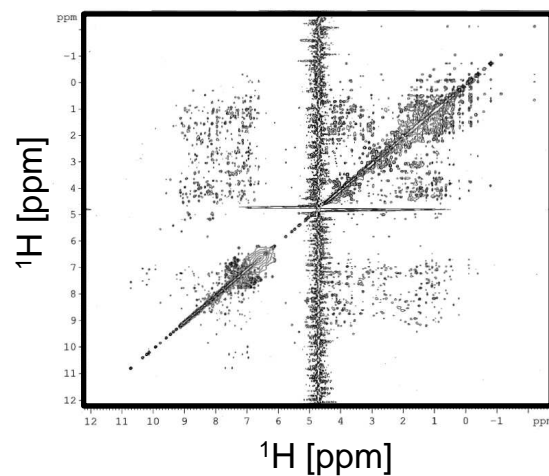
Ethyl-benzene

**<sup>1</sup>H solution NMR spectrum**

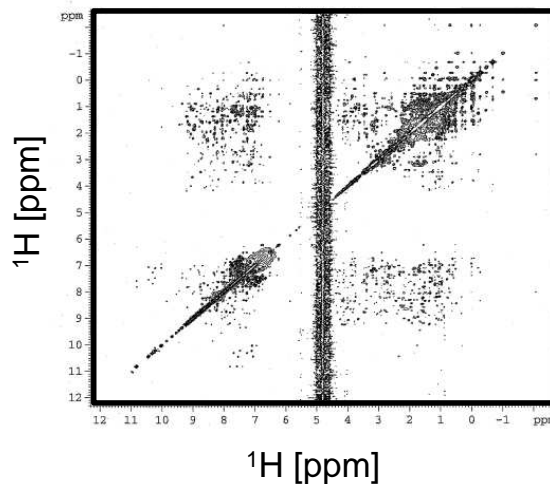
# 2-D NOESY spectra for Lysozyme



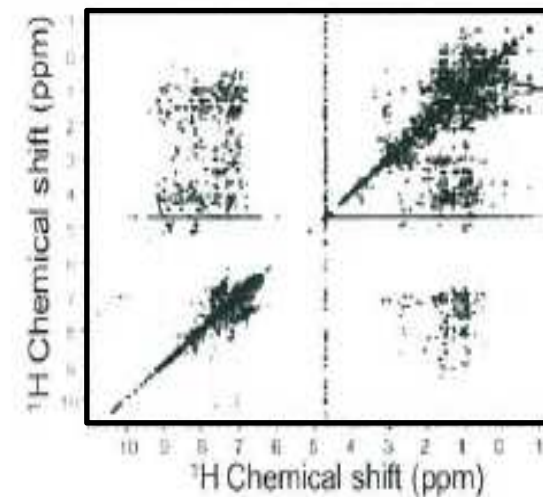
Low  $T_1$  noise, water suppression



**(a) 500MHz LTS  
NMR**



**(b) 500MHz LTS/  
Bi-2223 NMR**



**(c) 400MHz LTS/  
REBCO NMR**



KOBELCO



## 2. 1.02 GHz LTS/Bi-2223 NMR

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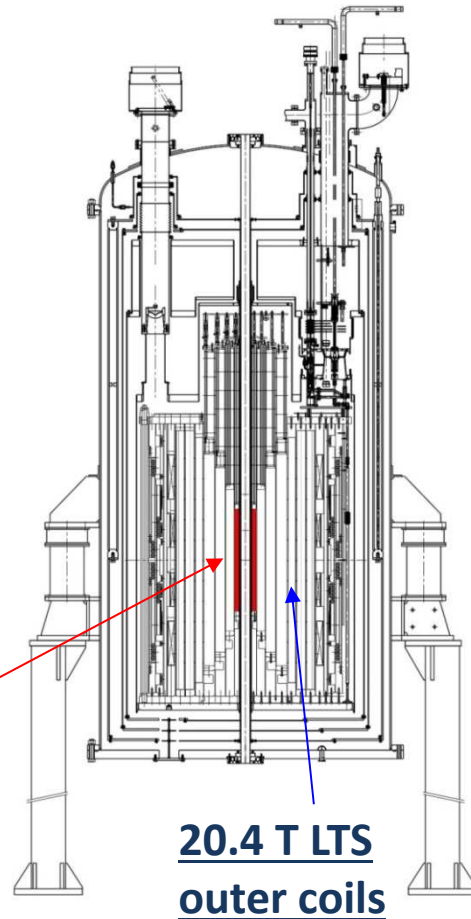
*Based on the method so far developed by using medium-field LTS/HTS NMR magnets, we proceeded to develop an LTS/ HTS NMR operated beyond 1 GHz*

*More details will be given by Dr. Hamada in this workshop*

# 1.02 GHz LTS/ Bi-2223 NMR



KOBELCO



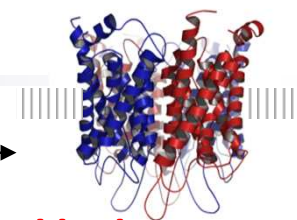
**3.6 T HTS(Bi-2223)**  
**innermost coil**

**20.4 T LTS**  
**outer coils**





# Solid-state 2D-NMR spectra(DARR) for a membrane protein ( Aquaporin Z)



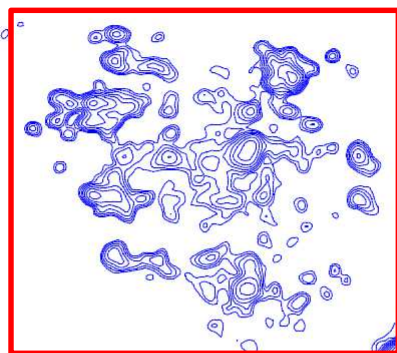
Membrane →

**7 Hours with external lock** Aquaporin  
 MAS=15 kHz; 2.5 mm rotor  
 70 kHz; spinal64 decoupling

140630\_Aqpz\_drug\_1005\_1 /home/nagasima/data  
 DARR 20ms  
 Aqpz drug  
 70kHz spinal64  
 10.3kHz MAS  
 2015.3.9

**14 peaks**

**700 MHz  
 Persistent mode**



**36 peaks**

**1.02 GHz  
 Driven mode**



**Collaboration**

Prof. Pervushin

Dr. Yamazaki  
 in RIKEN

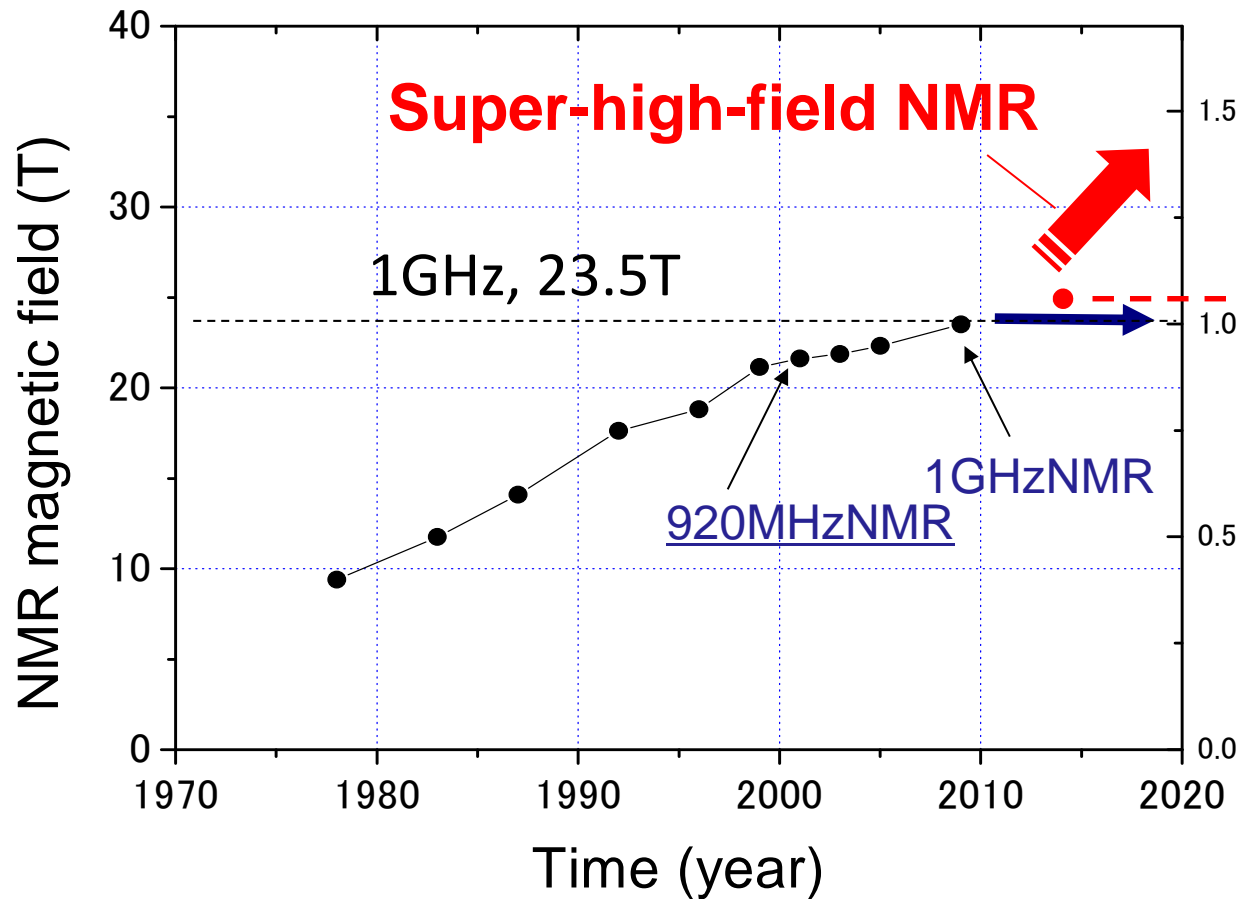


tor;  
 ng

arts per Million : Carbon13

H. Maeda et al., eMagRes (Wiley) to be published (2016)

# One great leap !



*This is one small step for  
an NMR magnet; one  
giant leap for super high  
field NMR*

*- N. A. Armstrong of  
Apollo 11 -*



### 3. New perspective of the super-high field NMR

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# We must reconsider the current perspective based on the screening current effect

## LTS/Bi-2223 NMR:

- The magnet size is large.
- The effect of screening current is small.

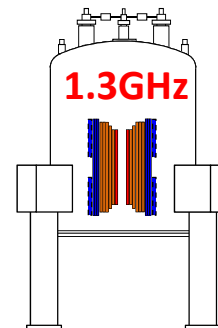
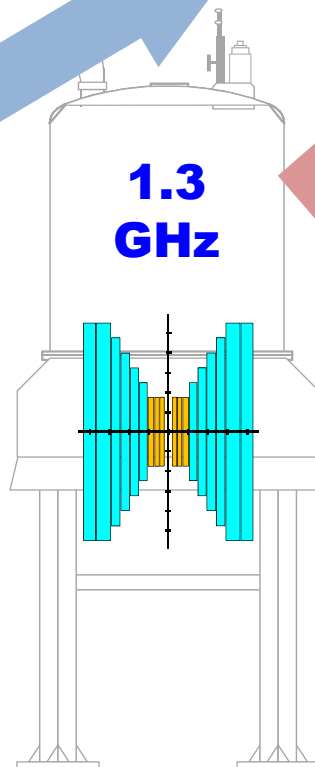
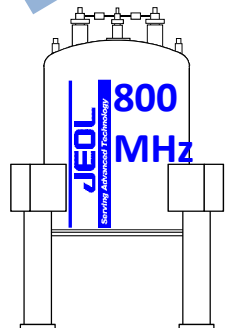
1.3 GHz  
LTS/Bi-2223  
(~350MJ)

## LTS/REBCO NMR:

- The magnet size is small.
- The effect of screening current is large.

1.02 GHz  
LTS/Bi-2223  
(~30MJ)

LTS

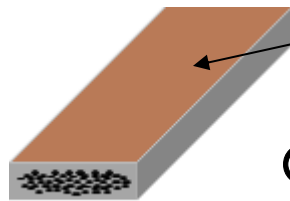


1.3 GHz  
LTS/Bi-2223/REBCO  
(~13MJ)

# (a) New HTS conductors

## Bi-2223 conductor

200  
MPa

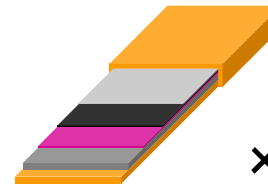


Bronze  
Reinforcement

- Screening current
- △ Mechanical strength

## REBCO conductor

700  
MPa



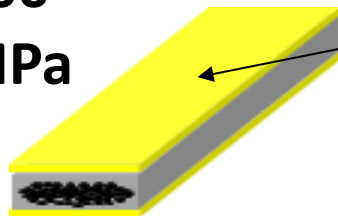
- × Screening current
- Mechanical strength



Both high mechanical strength and low screening current are obtained

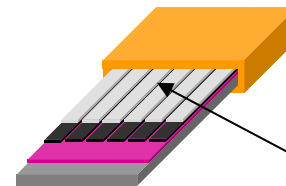
## Stronger Bi-2223 conductor

400  
MPa



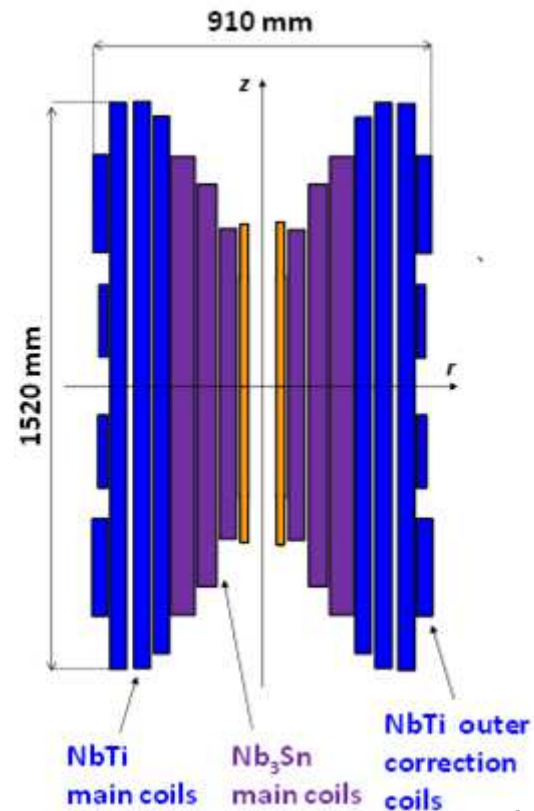
Ni-alloy  
reinforcement

## Multi-filamentary REBCO conductor



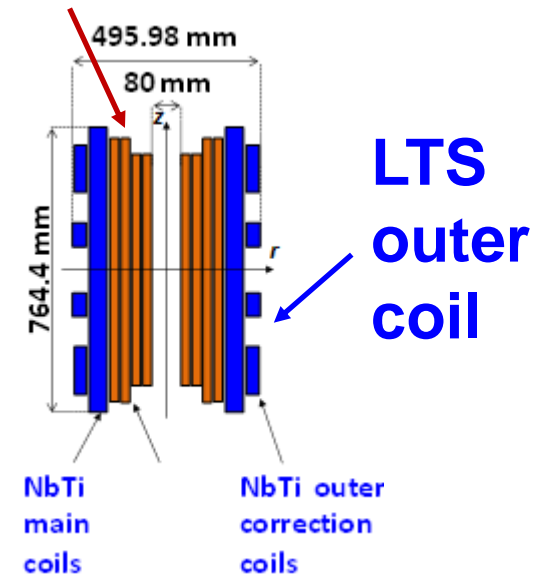
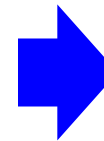
Separated  
into filaments  
(scribed)

## (b) Optimal design



### New 1.2GHz LTS/ HTS NMR

**HTS inner  
coil**



**Thin solenoid of the HTS**

**If we increase HTS coils, we can enhance the current density, reducing the magnet size**

# New perspective on the super-high field NMR

- a. New HTS conductors
- b. Optimal magnet design
- c. Simple cooling system



**The next target!**

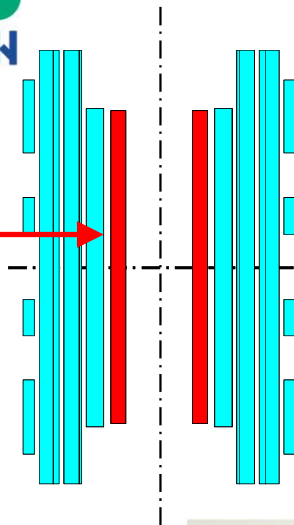
## **Finally --**

*So far we have limited our discussion to the HTS tape conductor, however we have another choice*

# Development of an LTS/ Bi-2212 NMR magnet



**Bi-2223/ Bi-2212/ REBCO**



The screening current effect becomes negligible.

- **D. C. Larbalestier** et al. *Nature materials*(2014)
- **Y. Yanagisawa** et al, *JMR* (2010)
- **T. Kiyoshi** et al, *IEEE TAS* (2010)

**NMR probe**  
( $^1\text{H}$ ,  $^{13}\text{C}$ ,  $^{15}\text{N}$ )

Will be installed in RIKEN in 2015



Sakura  
桜  
22  
Cherry

Thank you very much for your attention !



RIKEN NMR Facility