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Commercial Ultra-High-Field NMR Magnets with HTS Conductors

Robert Herzog on behalf of Bruker's UHF NMR team

16 November 2021

Picture: Transport of the ETH Zurich 1.2 GHz magnet on air cushions to its installation location. From a video by Matthias Ernst / ETH Zurich

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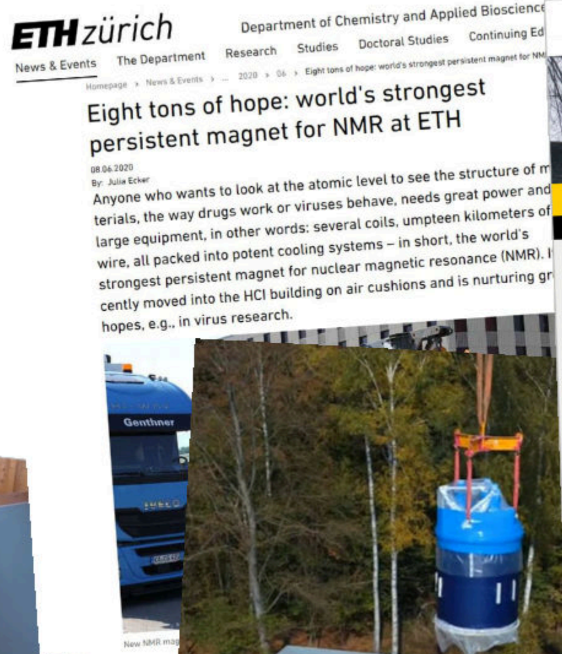
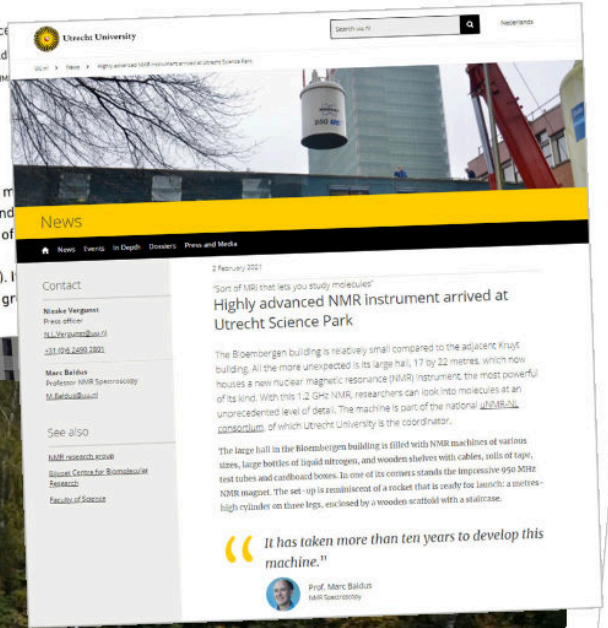
Press echoes to deliveries of Bruker NMR systems with HTS-LTS hybrid magnets

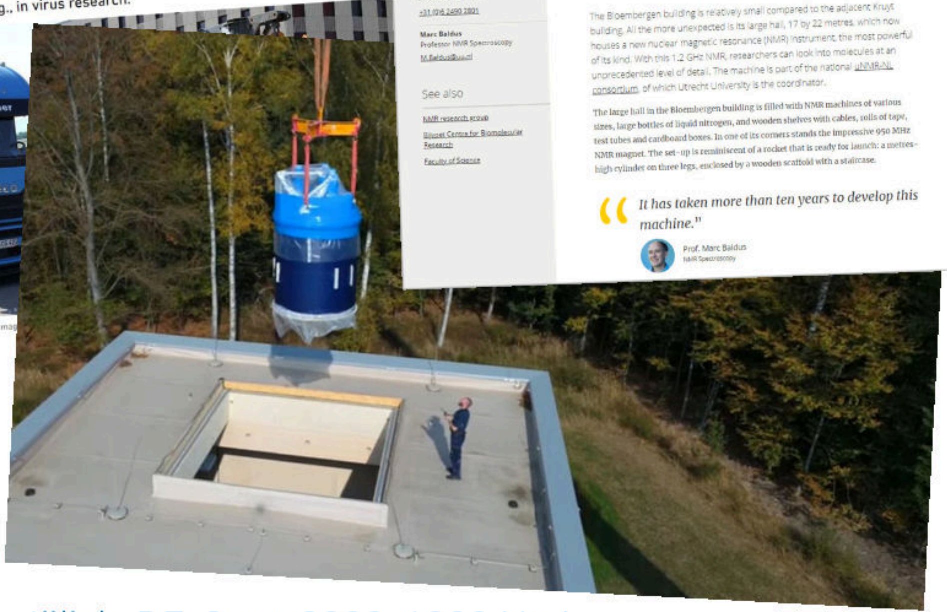
Memphis, Tennessee, US, Sept. 2019, 1100 Nr.1

Zurich, CH, May 2020, 1200 Nr.2



Utrecht, NL Feb. 2021, 1200 Nr.5





Göttingen, DE, July 2020, 1200 Nr.3

Florence, IT, March 2020, 1200 Nr.1

Jülich, DE, Sept. 2020, 1200 Nr.4

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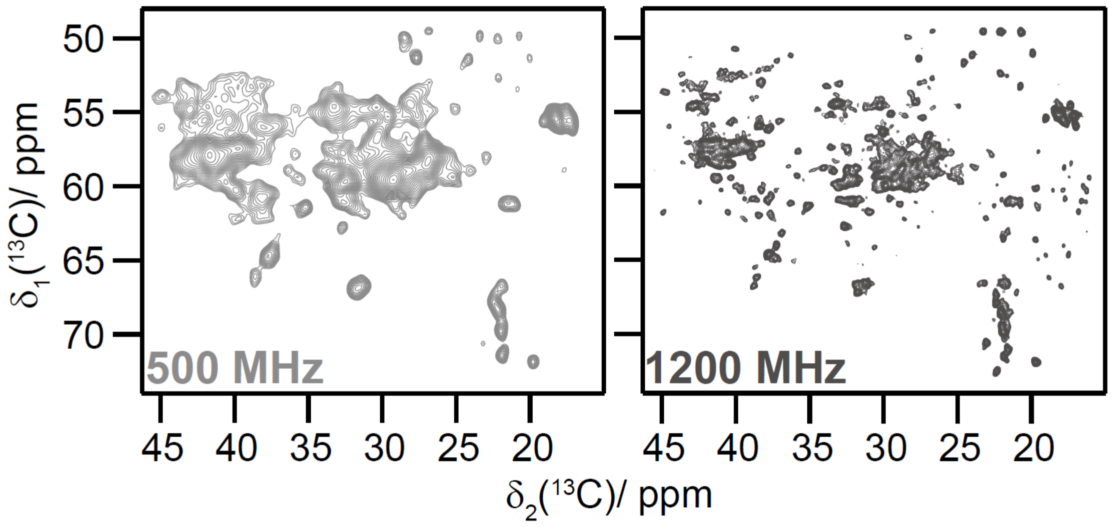
Ever higher fields for high resolution NMR – motivation and benefits

Ever higher fields for NMR spectrometers – why really?

- Generating magnetic fields for NMR spectrometers stronger than attainable with LTS became a vision as soon as the HTS were discovered, because:
  - Higher fields mean higher resolution (dispersion), i.e. better peak separation
    - In higher dimension NMR experiments the resolution increases further with field ($\propto B_0^n$)
  - Stronger fields lead to a better signal to noise ratio (SNR) – the signals are genuinely small
    - The bigger energy split between up and down spin states leads to a stronger occupation difference of these two states → stronger NMR signal from sample



20 ms DARR spectra of the DnaB helicase from Helicobacter pylori, recorded at 500 MHz (11.7 T) and at 1.2 GHz (28.2 T).
Source: doi.org/10.1101/2021.03.31.437892

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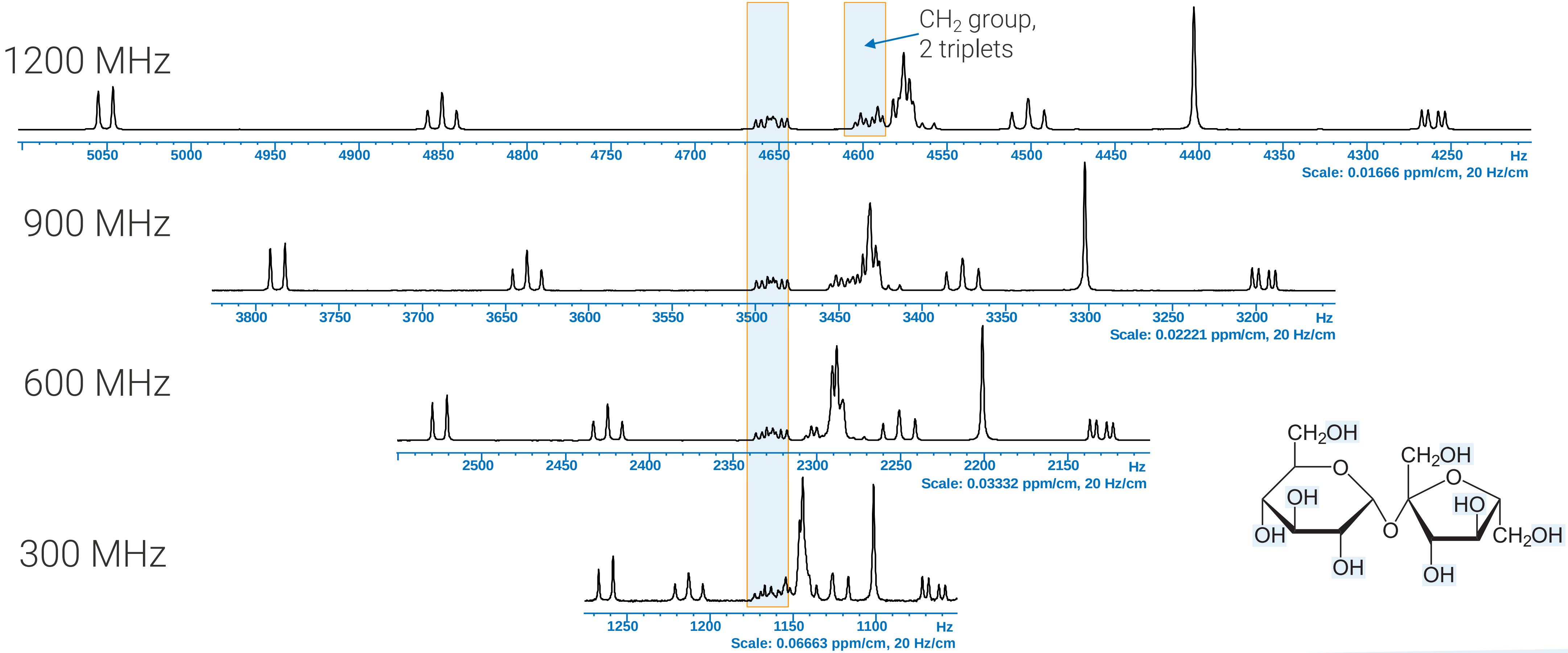
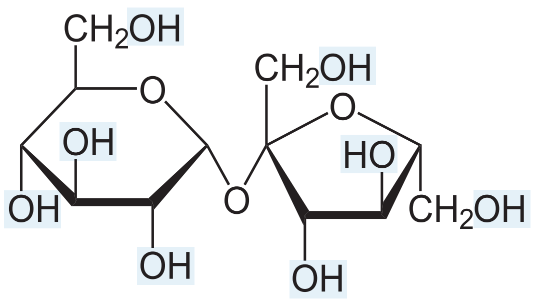
Dispersion & Sensitivity at Ultra High Fields

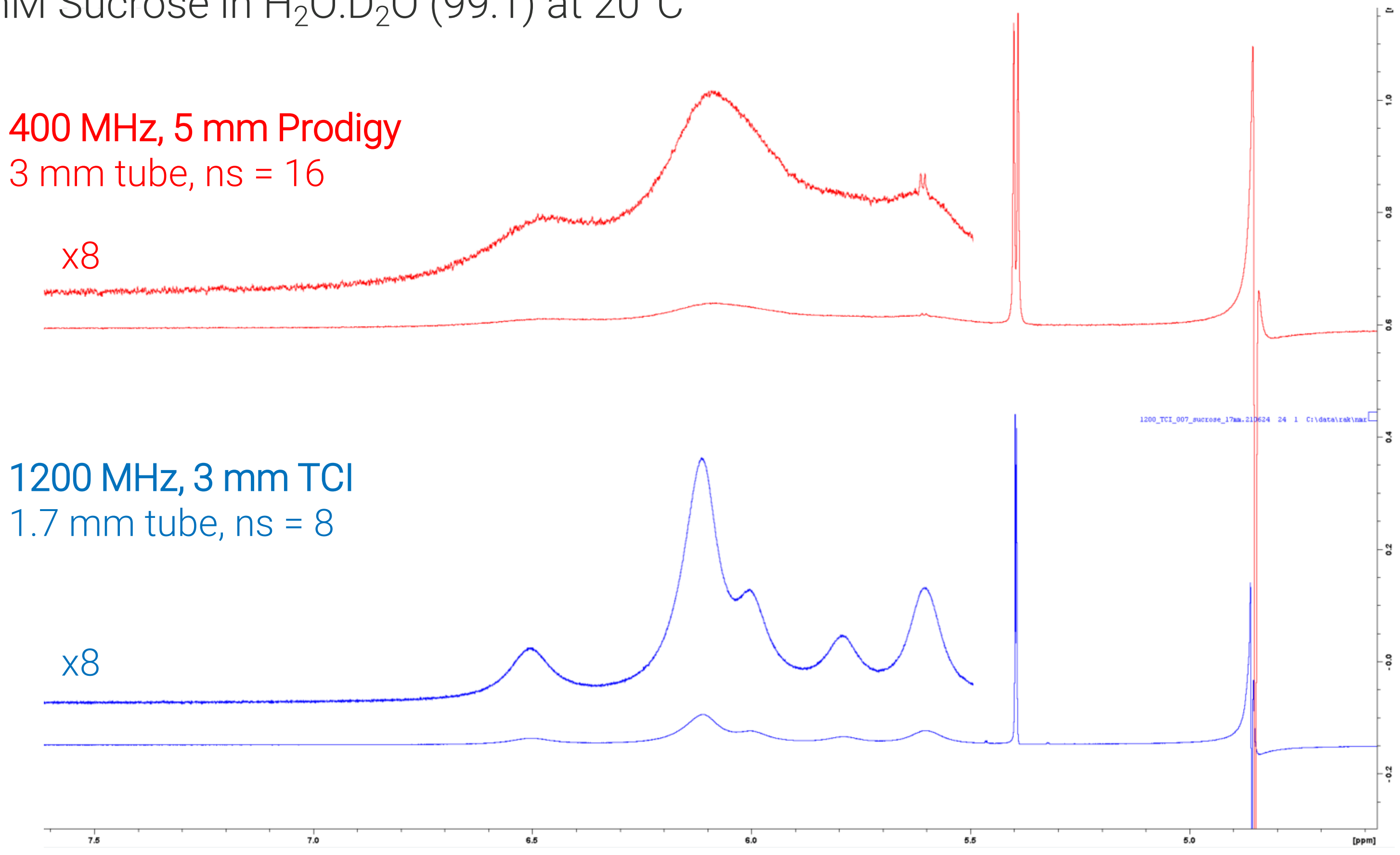
Sugar signals of 2 mM Sucrose in $H_2O:D_2O$ (9:1) illustrate the dispersion gain with increasing field strength



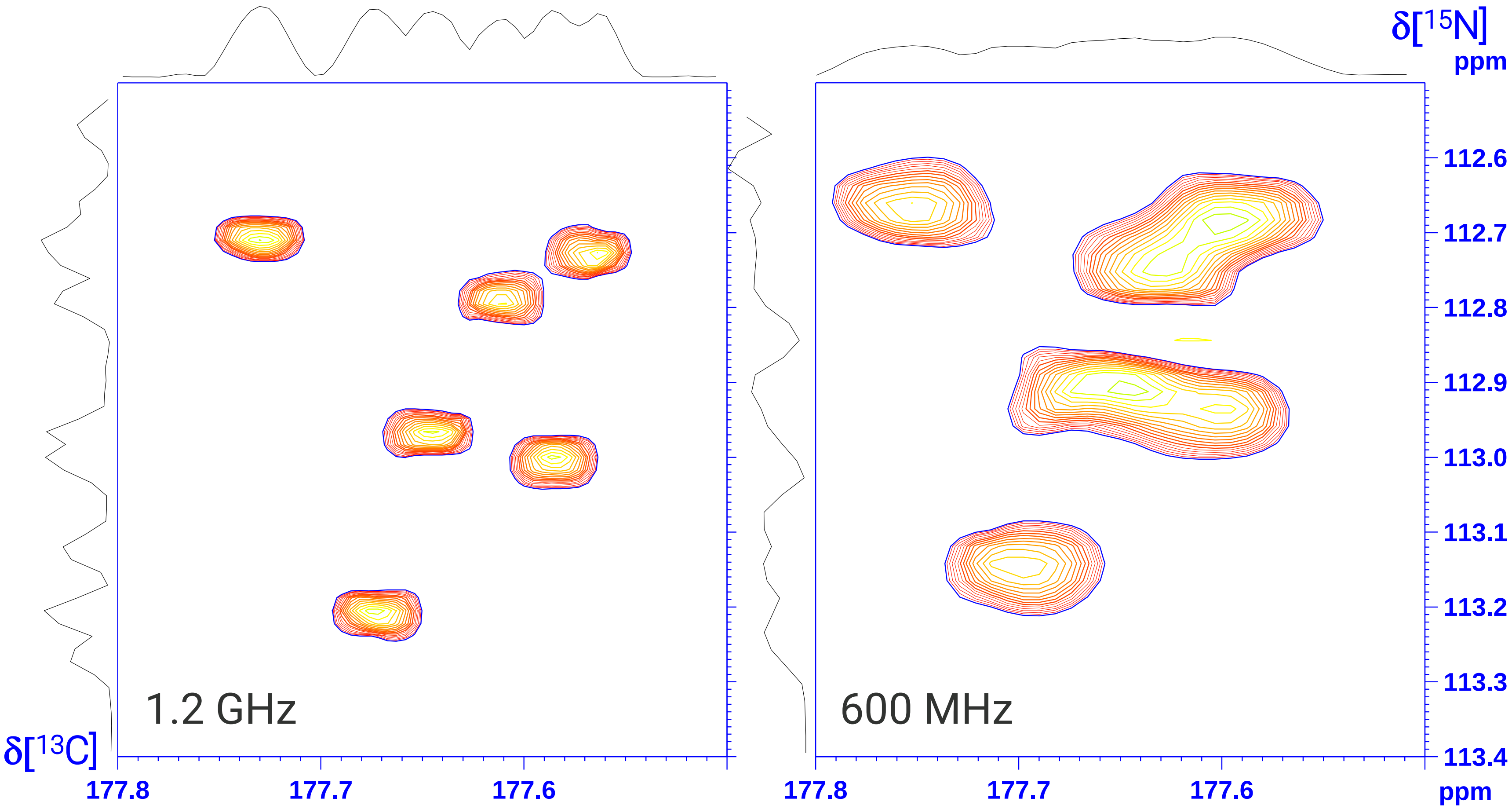
Exchange spectroscopy: Sensitivity at Ultra-High field line sharpening at UHF for slow/medium exchange

- Hydroxy proton signals, 100 mM Sucrose in $H_2O:D_2O$ (99:1) at 20°C





1.2 GHz High-Resolution NMR: α-Synuclein Intrinsically disordered proteins benefit from UHF & ^{13}C detection.



~1 mM α-Synuclein $^{13}C/^{15}N$ in 20 mM KPi, 100 mM NaCl, 50 uM EDTA, pH 6.5

2D ^{13}C-^{15}N CON (IPAP)

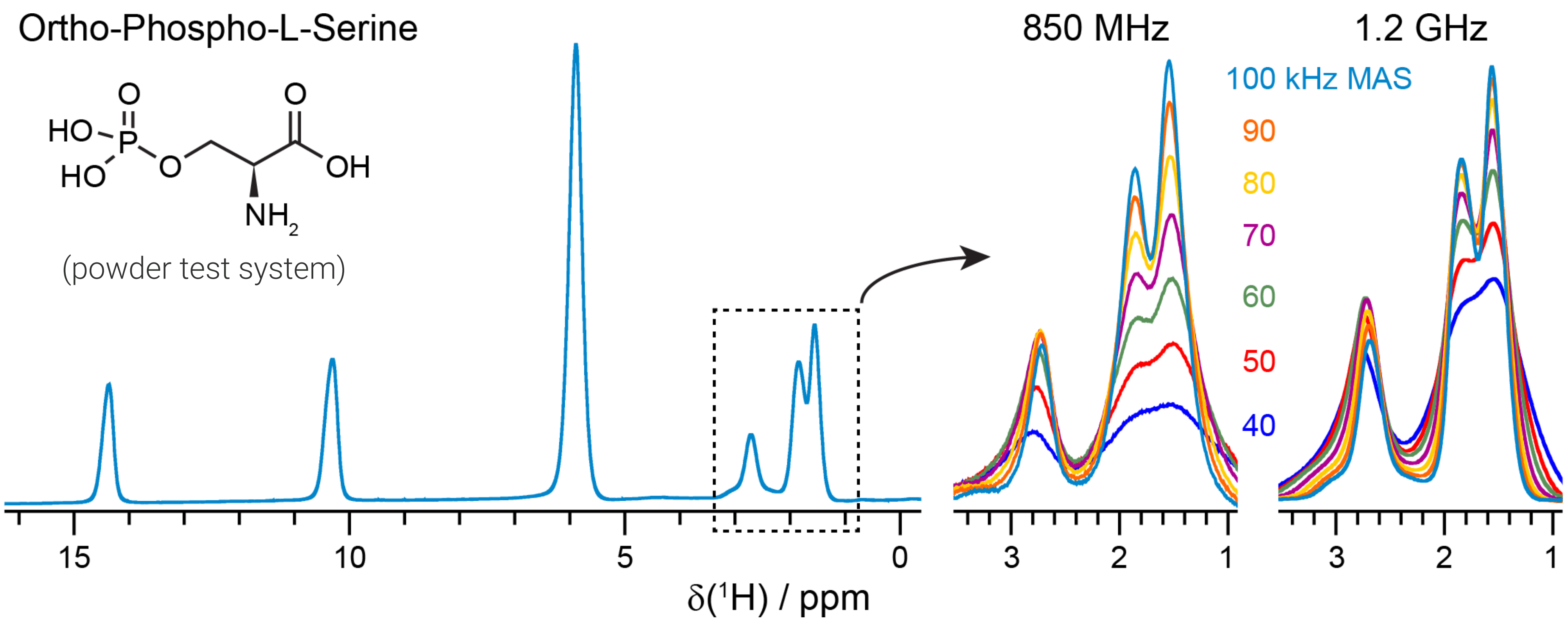
TD = 2k x 800
AQ 184ms x 94ms
NS = 8

Expt. Time: 4h 41 min

1.2 GHz: 3 mm TCI CryoProbe
600 MHz: 5 mm TCI CryoProbe

Sample & data courtesy L. Banci, C. Luchinat, R. Pierattelli; CERM, University of Florence

100 kHz FastMAS at 28.2 T / 1.2 GHz
0.7 mm HCN CP-MAS



Sample & data courtesy of Prof. B. Meier, Dr. T. Wiegand, M. Schledorn, ETH Zurich

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NMR applications benefitting from Ultra-high field

- Dispersion scales linearly with field and the power of dimensionality
- Self-orientation due to paramagnetic centers scales with B_0^2
- RDC, RCSA & Relaxation Dispersion scale with B_0^2
- Detection of otherwise invisible states due to more favorable exchange regimes at higher fields (biologically relevant little populated conformational states)
- Carbohydrate and intrinsically disordered protein research
- TROSY effects in liquid state NMR
- Fast-MAS ^{1}H detected solid-state NMR
- Low-gamma and quadrupolar nuclei in solid-state NMR
- BUT: Only realized with very high-quality magnets!

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Principal requirements on UHF NMR magnets – field and quench, homogeneity and drift

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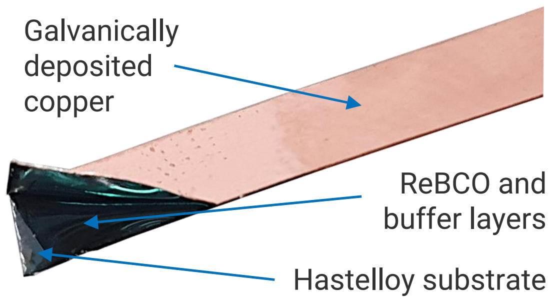
Principal requirements for UHF NMR magnets

- The obvious: It must be possible to reach nominal field and operate the magnet safely.
  - R&D goal Nr.1 for new HTS – LTS hybrid magnets
  - Challenge: Manage the strong Lorentz forces acting on the conductors.
  - Challenge: Prevent structural damage in case of a quench.
- Very high homogeneity of the magnetic field in the sample space for high-resolution NMR
  - Specification: $\delta B/B \leq 10^{-9}$ in the sample volume (~20 mm long cylinder, ø 5 mm)
  - Challenge: Magnet shimming despite strong screening currents in the HTS tapes.
- Very time-stable magnetic field (very small field drift)
  - Specification: 10 ppb/h (loss of 1% of field in ~ 110 years)
  - Challenge: Reduce the joint resistances sufficiently

Reaching field – critical currents, forces and quench protection

Practical conductors with sufficient $J_{c,eng}$ at high fields (> 20 T)

- $J_{c,eng}$ drops sharply in Nb_3Sn conductors approaching 1 GHz (~23.5 T)
- Fortunately HTS conductors, with their high $J_{c,eng}$ at significantly stronger fields, are able to step in now.



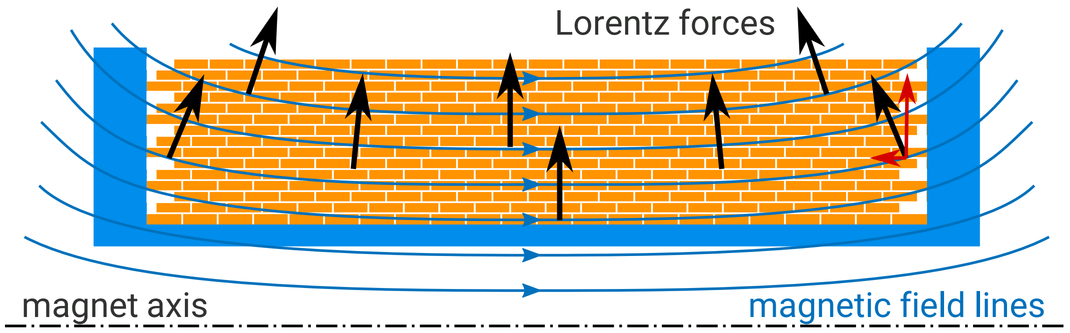
ReBCO coated conductor

Lorentz forces increasing with field must be carefully managed

- Hoop stresses increase in the high-field region of a coil (higher field), but also in the lower field region (larger radii).
- The magnet design must limit the axial pressures to tested values.

Quench protection to prevent structural damage

- During quench events currents induced in the HTS part must be limited.



Lorentz forces (black arrows) act on the conductors (orange) of a winding pack in the magnetic field (blue lines), which the current they carry generates.

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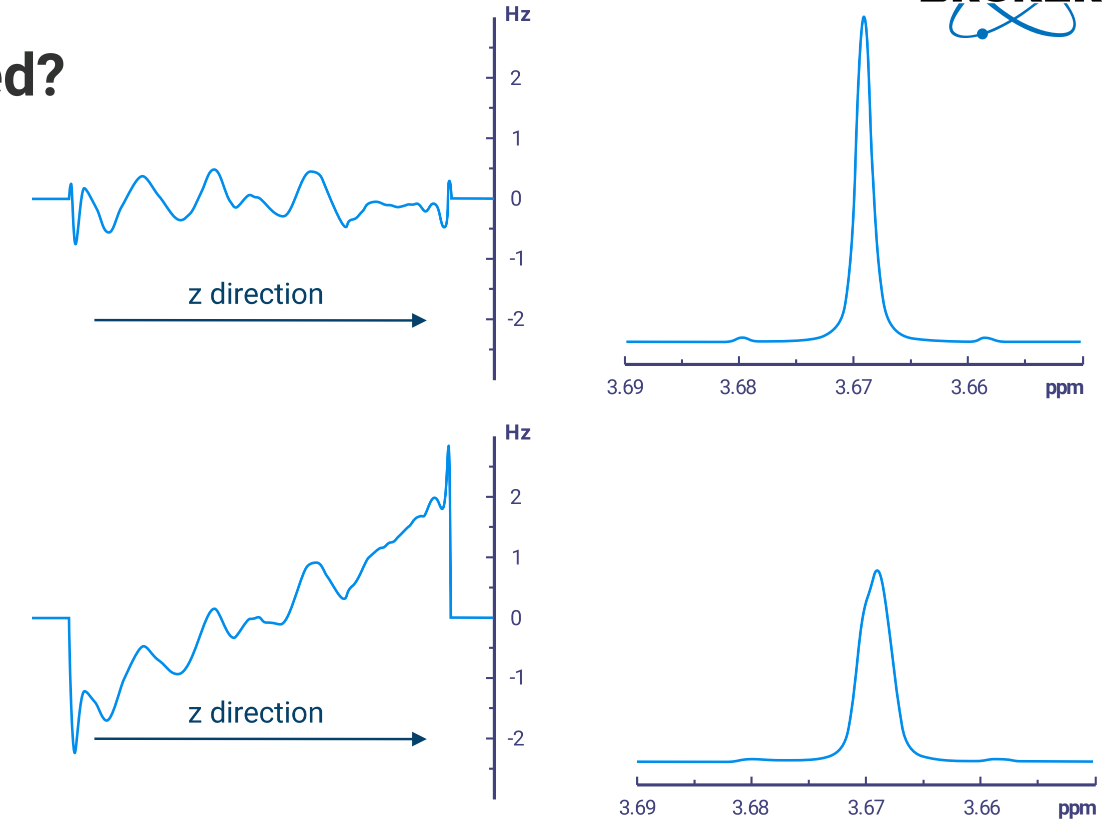
Why is such a high homogeneity required?

The intrinsic requirement

- The spins of nuclei are often only very weakly coupled to surrounding nuclei and electrons.
- This weak coupling makes the nuclear spins interesting, non-interfering sensors of their (chemical) environment.
- It also makes the resonances sharp, i.e. the intrinsic line widths very narrow: ~1 Hz
- To avoid smearing out the peaks, all nuclei in the sample must be at exactly the same field: $\delta B/B \leq 10^{-9}$



Left: Field profiles of a well-shimmed 1.2 GHz magnet (top) and with a z-gradient deliberately added (bottom).

Right: Corresponding line shapes of a sucrose sample. The broadened and less high line at the bottom clearly shows the imperfect summation of signals from spins at different locations in the sample.

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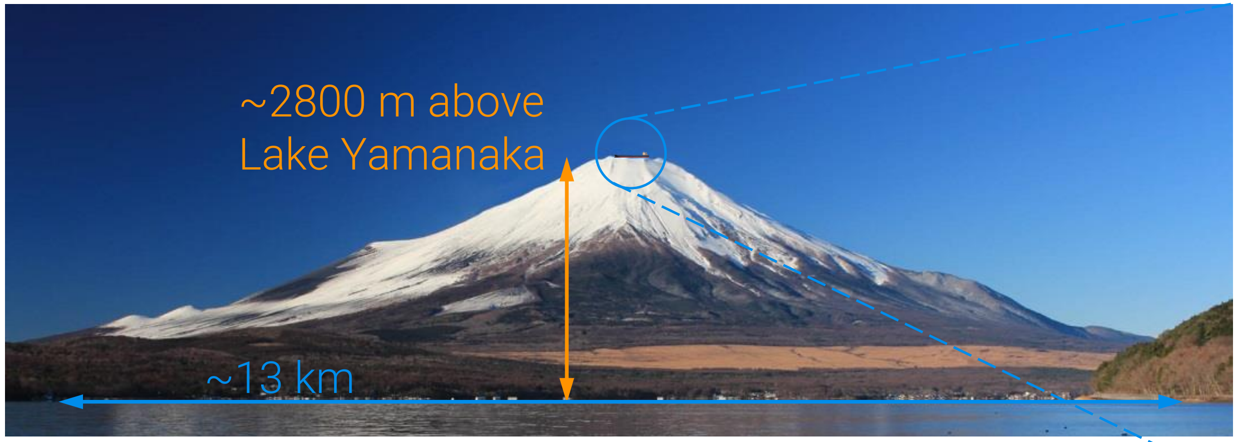
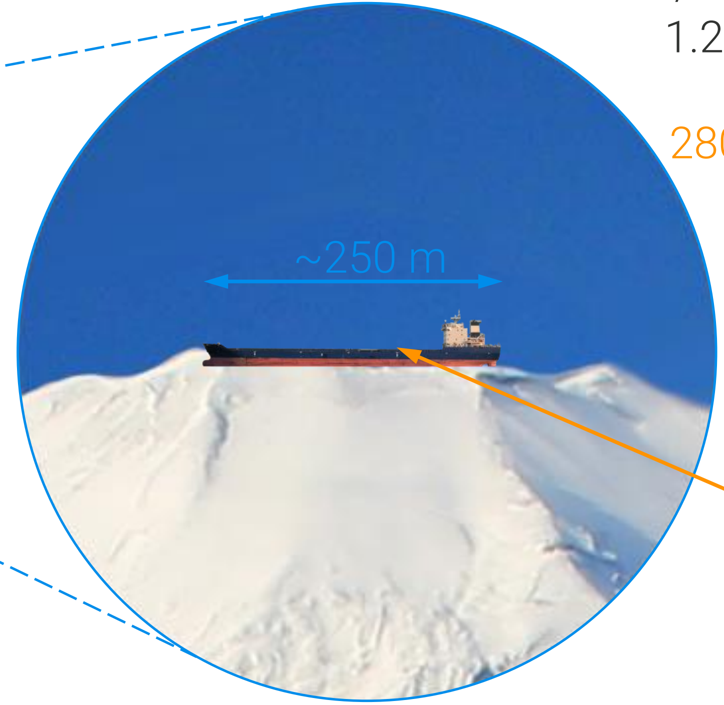
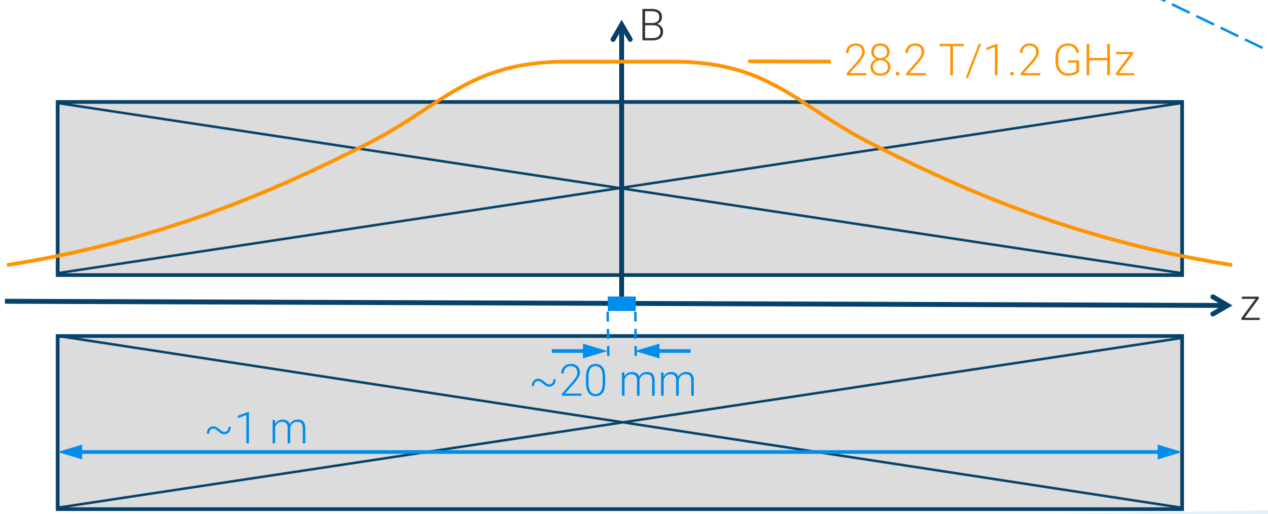
Homogeneity requirements for NMR measurements

$\delta B/B \leq 10^{-9}$:
1.2 GHz → ~1 Hz
28 T → ~25 nT
2800 m → ~2.5 µm

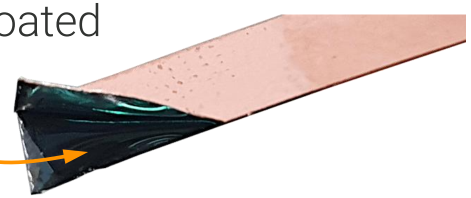




250 m long tanker: deck must be flat to ~2.5 µm !



2.5 µm is about the thickness of a ReBCO layer in a coated conductor tape!!



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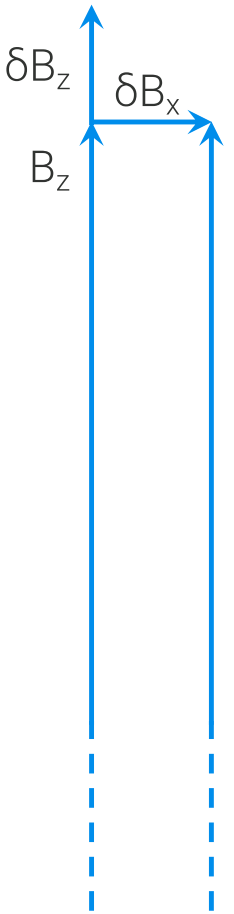
How can such a homogeneity be achieved in practice?

Nature helps: the Laplace equation for B_z:

- The resonance peaks depend on |**B**|, but in a homogeneous solenoidal field only B_z counts.
- In a region without currents and magnetic material (e.g. the magnet bore) the Laplace equation for B_z is valid: $\Delta B_z = 0$ [rot B = 0, div B = 0]
- This is also the equation for a stretched membrane: there are no bumps, the shape is only determined by *forces* (~currents & mag. material) at the rim of or outside the region.
- Any solution of $\Delta B_z = 0$ can be developed around a point into a *special* Taylor series where the coefficients are called gradients and the terms harmonic functions.
- Shim coils pull the membrane really flat!

|**B**| does *not* change when adding δB_x if $\delta B_x \ll B_z$, but it *does* when adding a δB_z !



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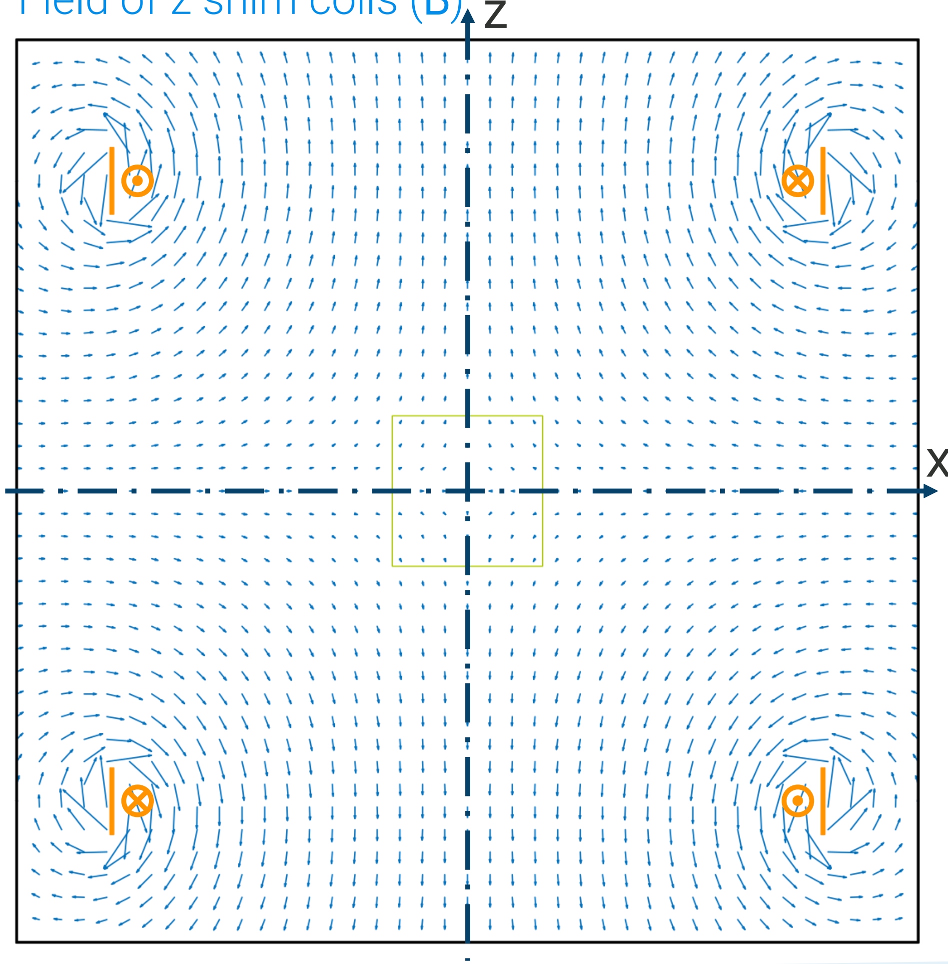
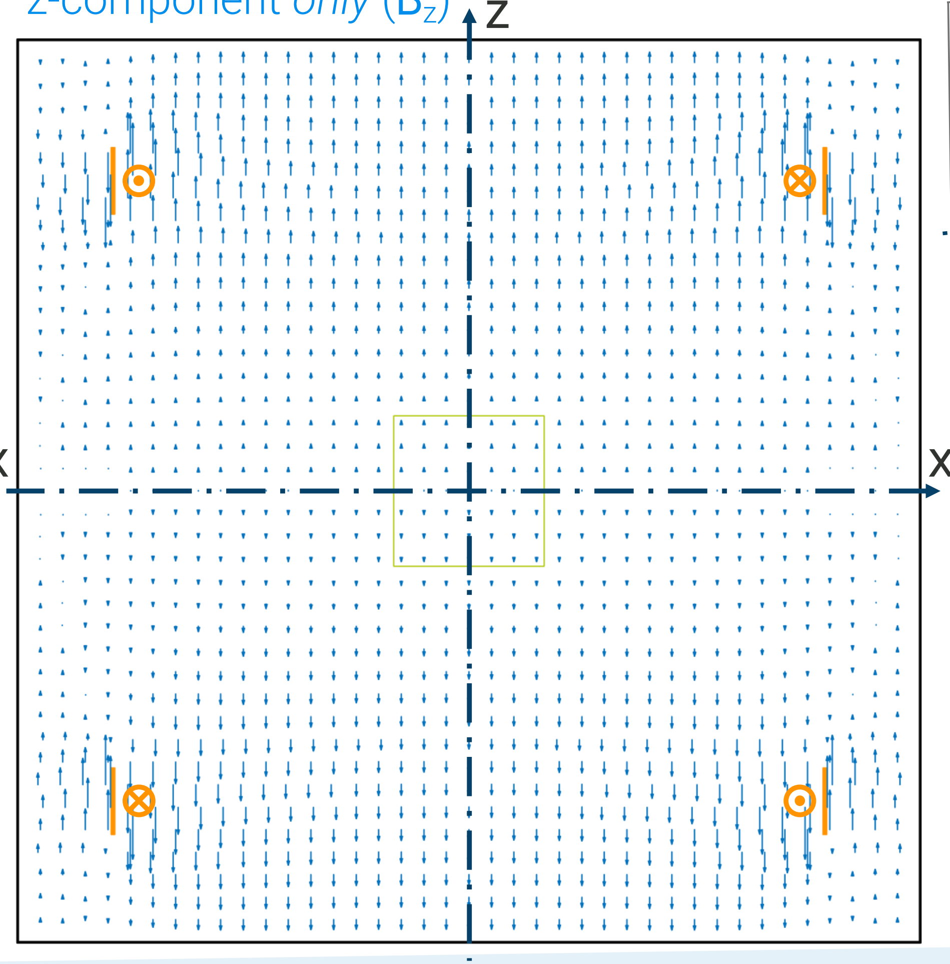
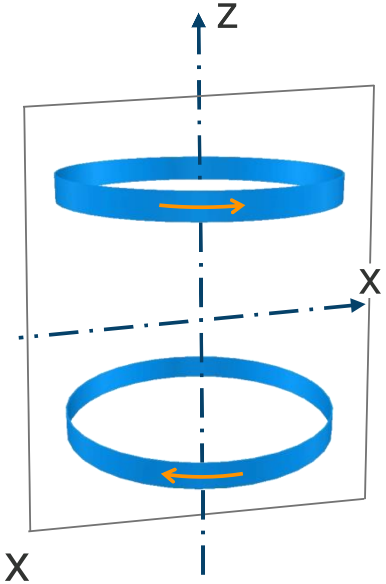
Shim coils generating a z-gradient (superconducting)

Field of z shim coils (B)



z-component *only* (B_z)





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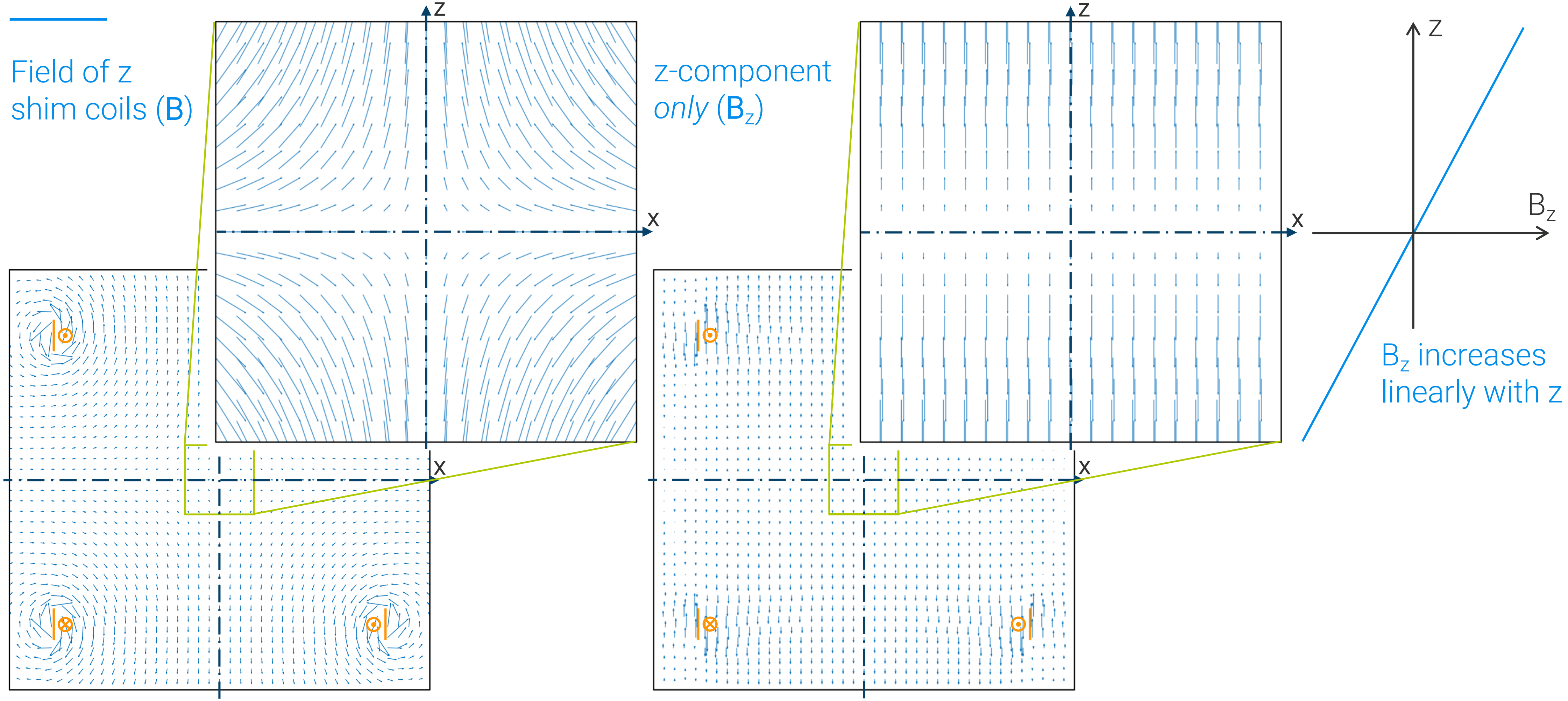

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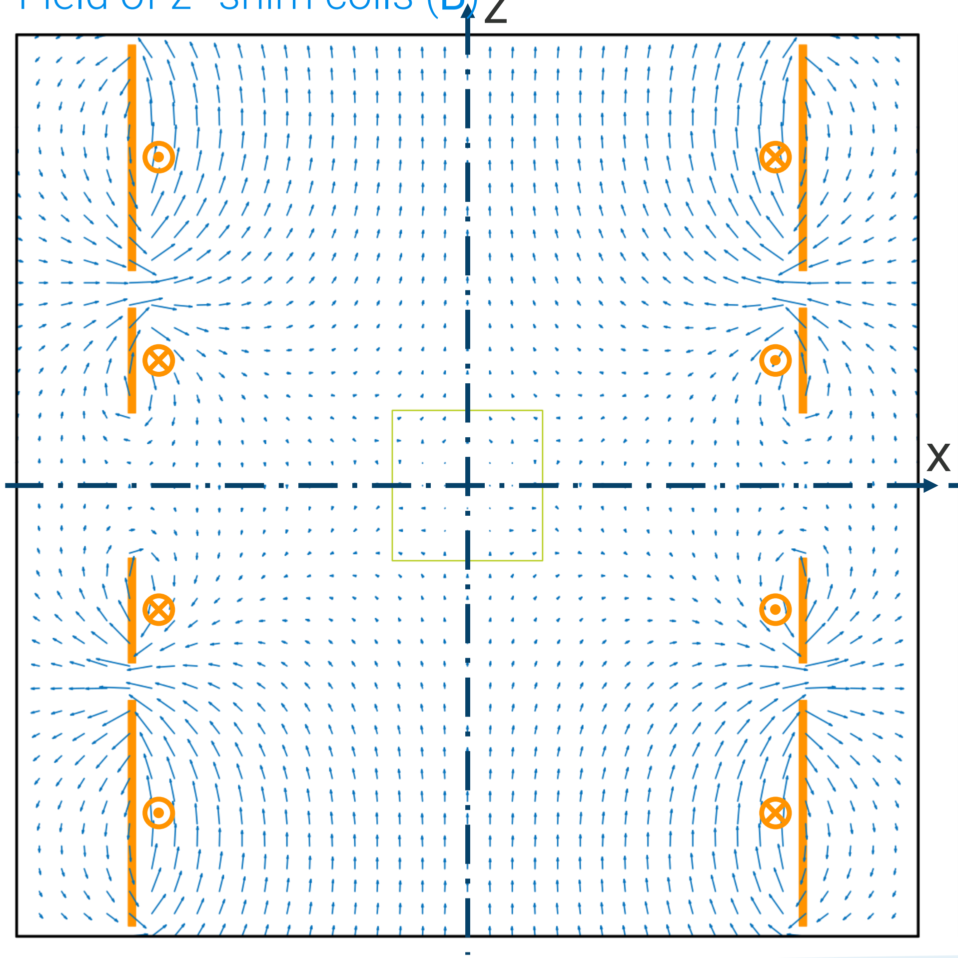
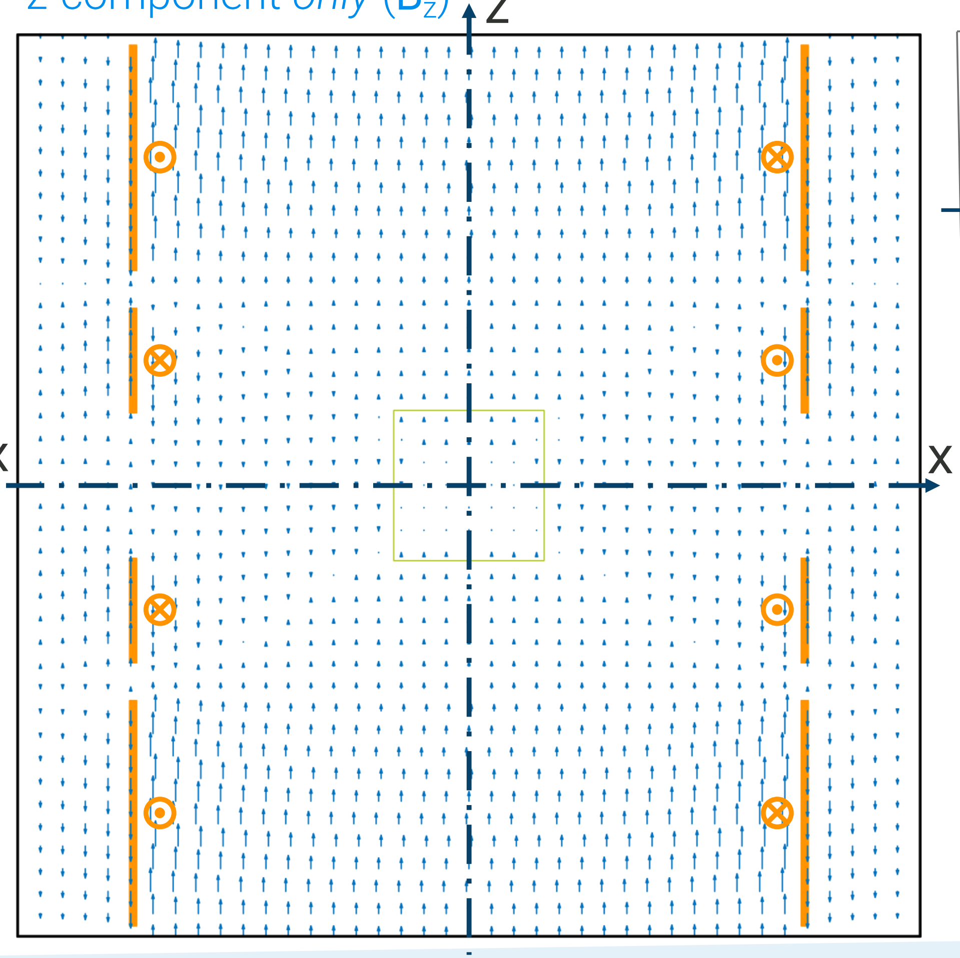
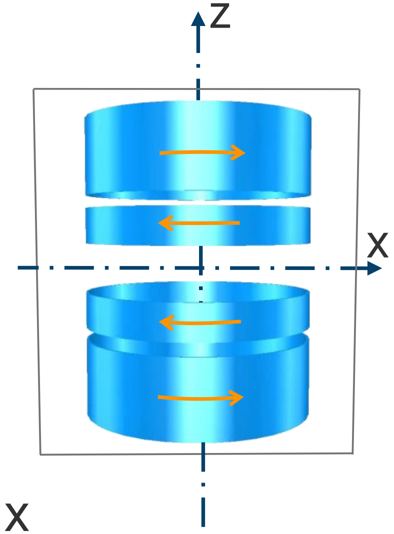
Shim coils generating a z^2-gradient (superconducting)

Field of z^2 shim coils (B)



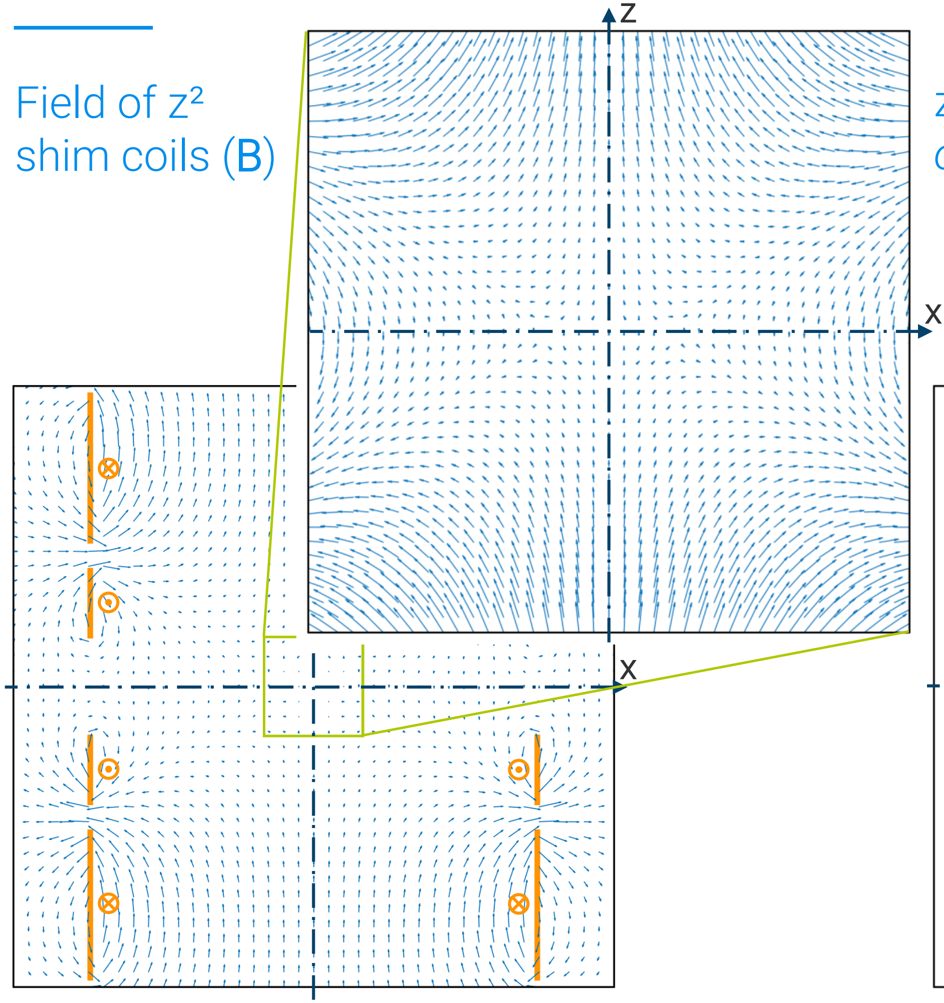
z-component *only* (B_z)



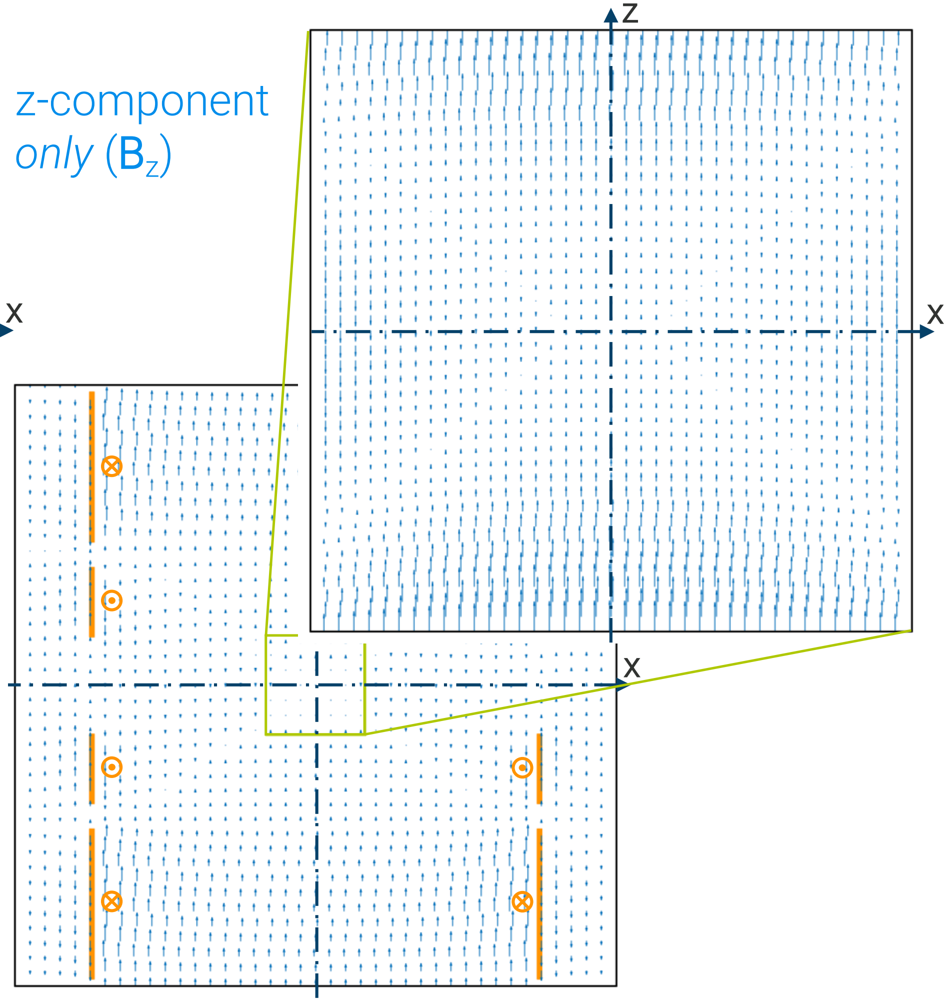


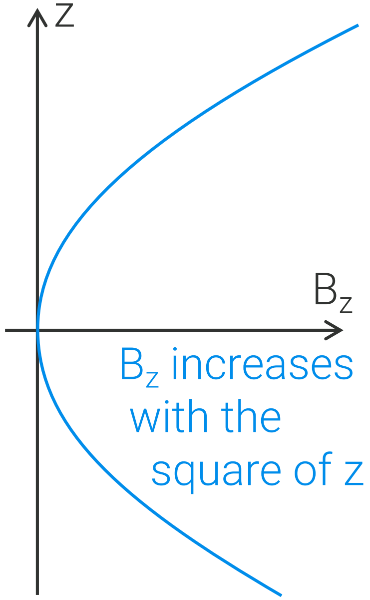
Shim coils generating a z^2-gradient (superconducting)

Field of z^2 shim coils (B)



z-component *only* (B_z)





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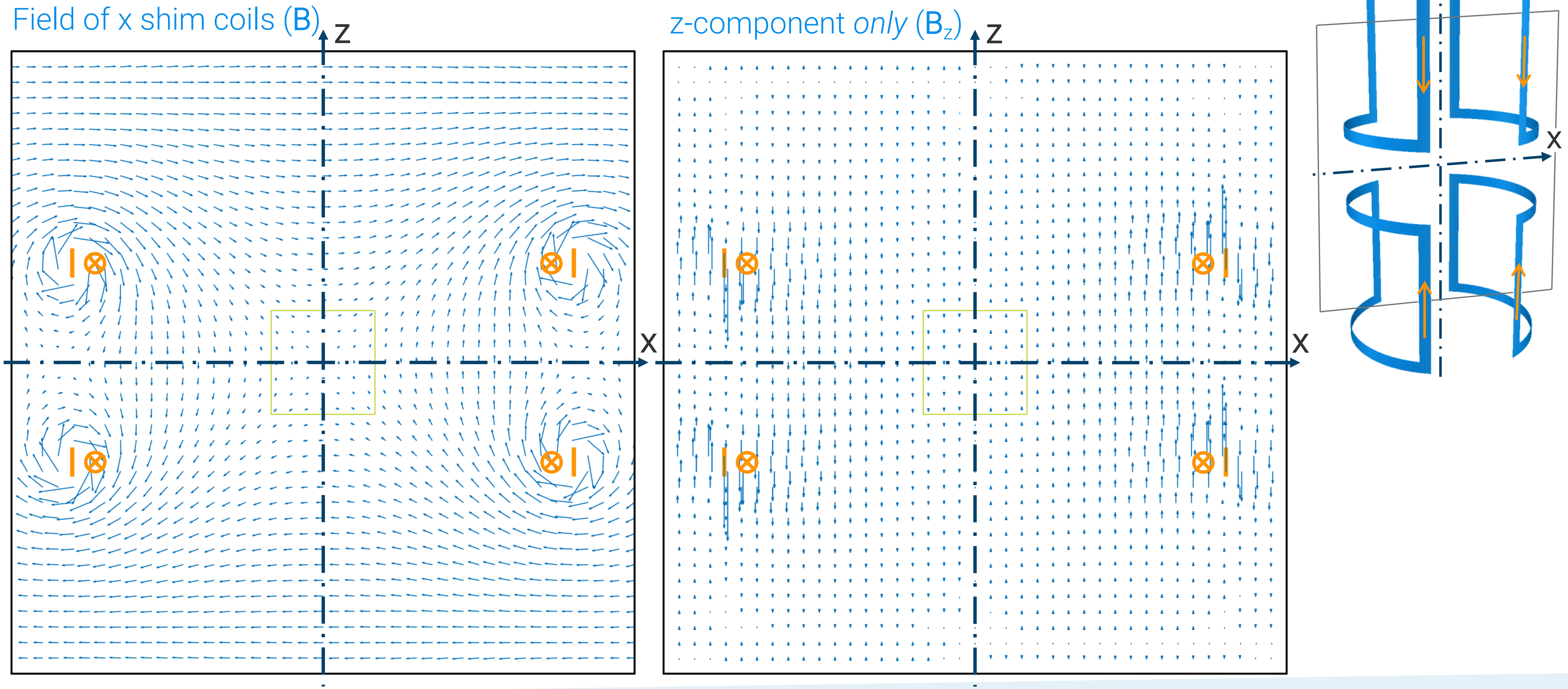
Shim coils generating a x-gradient (superconducting)



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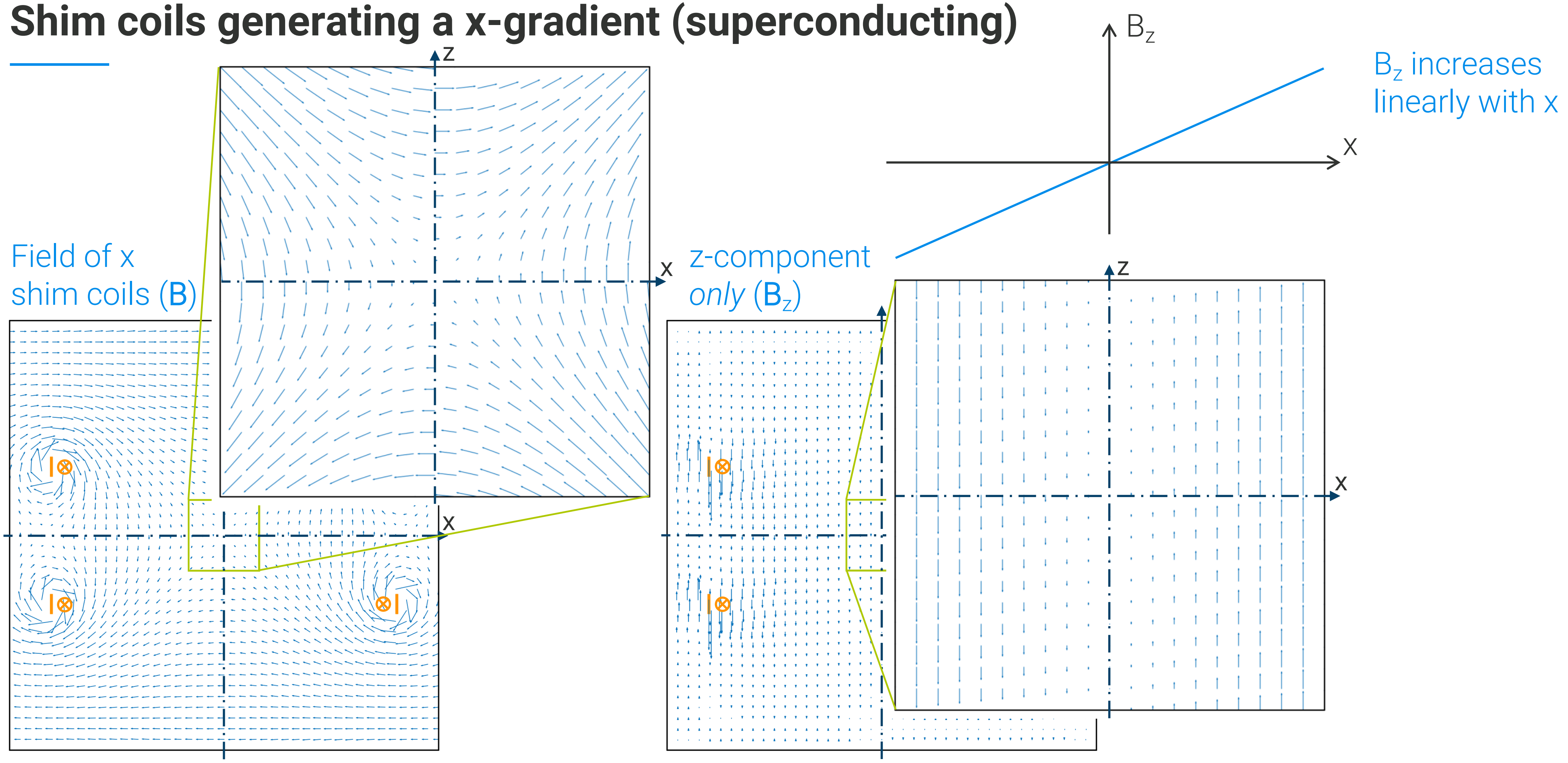
Shim coils generating a x-gradient (superconducting)



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To really get there: the room-temperature shim system

The BOSS3 standard bore RT shim system

- 38 non-orthogonal shim coils, controlled via a shim matrix, provide 36 shim functions, of which 8 are on-axis.
- The on-axis shims do not generate a B_0 shift.
- Usually Bruker software controls the RT shims, TopShim or AutoShim.
- A separate B_0 coil, driven by the lock system, serves to keep the main field accurately constant over days, if needed, compensating for small external field disturbances as well.

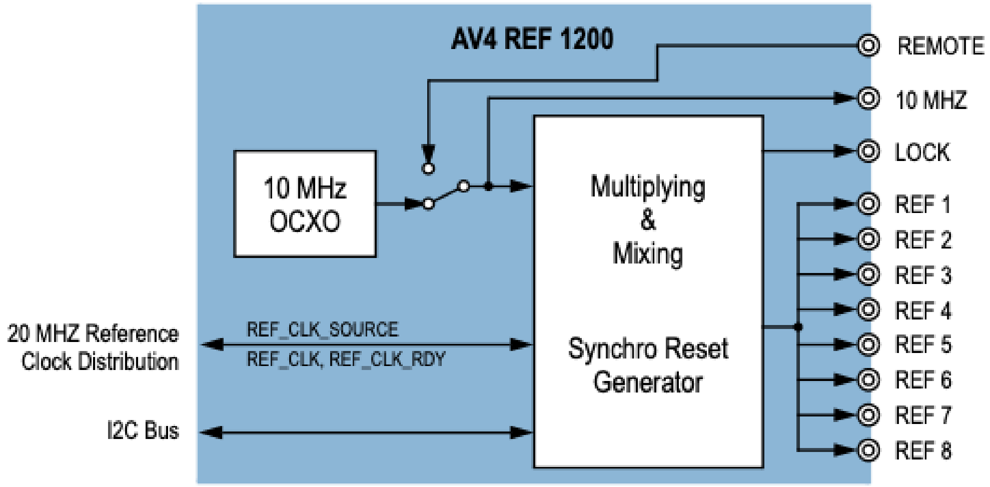
36 RT shim functions
$Z, Z^2, Z^3, Z^4, Z^5, Z^6, Z^7, Z^8$
X, Y XZ, YZ XZ^2, YZ^2 XZ^3, YZ^3 XZ^4, YZ^4 XZ^5, YZ^5
$(X^2 - Y^2), XY$ $(X^2 - Y^2)Z, XYZ$ $(X^2 - Y^2)Z^2, XYZ^2$ $(X^2 - Y^2)Z^3, XYZ^3$ $(X^2 - Y^2)Z^4, XYZ^4$ $(X^2 - Y^2)Z^5, XYZ^5$
X^3, Y^3 X^3Z, Y^3Z
B_0

38 independently powered shim coils

Temporal stability of NMR magnets

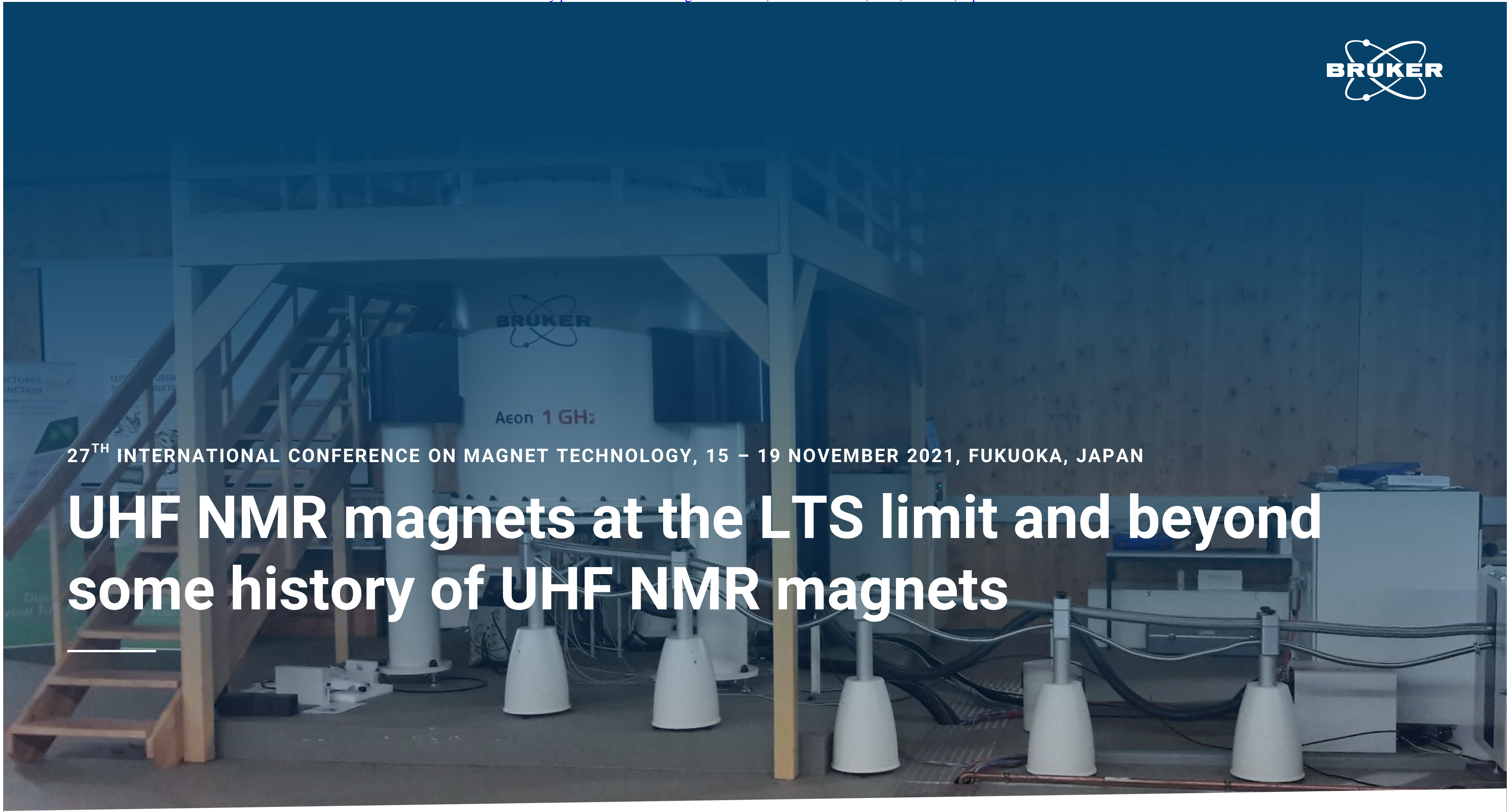
Drift specification for high resolution NMR magnets: 10 ppb/h

- NMR experiments often run for many hours, even days
- The ~1 Hz natural line width imposes a very tight specification in the time domain: 10 ppb/h (10^{-8}/h)



Is the electronic equipment stable enough?

- The oven-controlled quartz of the Avance Neo console has a stability of 10^{-9}/day, during shorter time spans even better.
- All frequency reference signals throughout the spectrometer are derived from this one quartz by phase locked multiplying and mixing. Between participating units delay lines assure correct phase differences.
- Ultimate stability comes from the locking system: a feedback loop, sampling the lock and observation channels at 6.6 kHz, keeps clock deviations below 10 mHz, if disturbances are not too big or fast and the lock signal is strong and sharp.



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UHF NMR magnets at the LTS limit and beyond some history of UHF NMR magnets

Picture: 1 GHz NMR magnet at the University of Bayreuth, Germany

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Approaching the LTS limit – the path to 1.0 GHz (23.4 T)

- 2000: Varian and Oxford Instruments present first 900 MHz NMR data
- 2001: Bruker installs first 900 MHz NMR magnet (unshielded) at a customer site
- 2002: Japanese scientists build a 920 MHz NMR magnet (15% bronze Nb_3Sn)
- 2004: Bruker introduce 900 MHz NMR magnet with active shielding
- 2005: Varian and Oxford Inst. deliver first 950 MHz NMR magnet (unshielded)
- 2006: Bruker presents the first shielded 950 MHz NMR magnet
- 2009: Bruker installs unshielded 1.0 GHz NMR magnet in Lyon, France (15% bronze Nb_3Sn, 4.5 m high, 12 t, 5 Gauss line radius of 12 m)
- 2015: Japanese team upgrades the 920 MHz to a 1020 MHz NMR magnet using HTS (Bi-2223) and operates it in driven mode.
- 2016: Bruker commissions the first actively shielded 1.0 GHz NMR magnet in Germany, further magnets follow to Israel, Canada and the UK.

Ox. Inst. 900 MHz NMR magnet in Jülich

The first 1.0 GHz NMR magnet in Lyon

GHz-class NMR magnet projects in Japan

- First 1.02 GHz (24.0 T) LTS/Bi-2223 NMR magnet at NIMS in 2015
  - Driven mode: First run for more than half a year! Special protection circuit in case of power outages.
  - High resolution NMR in driven mode successfully achieved, but persistent mode magnets still preferable.
  - Ferromagnetic shims in bore in addition to superconducting shim coils.
- 1.3 GHz (30.5 T) persistent mode NMR magnet project pursued in JST-Mirai Program since 2017
  - Superconducting joints with Bi-2223 and ReBCO tapes
  - Innermost ReBCO coil: non-insulated layer wound
  - Completion planned in fiscal year 2024 (24-04 to 25-03)

Achievement of 1020 MHz NMR
J. Mag. Res. **256** 30-33, July 2015
https://doi.org/10.1016/j.jmr.2015.04.009

To 1.3 GHz project:

TUE-PO1-722-05
WED-PO2-613-12
WED-PO2-613-15
WED-OR2-302-03
THU-PO3-710-09
THU-OR4-401-07
FRI-OR6-603-07

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GHz-class NMR magnet projects at MIT

- MIT 1.3 GHz LTS/HTS NMR Magnet project
  - 835 MHz ReBCO insert: 40 stainless-steel-co-wound no-insulation (NI) double pancake (DP) coils with a reinforced cross-over turn (see THU-PO3-405-03)
  - driven mode operation
  - Coil inner ø: 88 mm
  - Planned shimming: HTS inner shims + ferromagnetic shims + RT shims
  - Quench-back heaters to minimize induced current
- Tabletop 1-GHz Microcoil NMR magnet
  - Tabletop liquid-helium-free 1 GHz Microcoil (25 mm RT bore diameter) magnet under development
  - 23.5 T / 12.5 mm cold bore ReBCO magnet prototype (2021, see WED-OR2-302-01)

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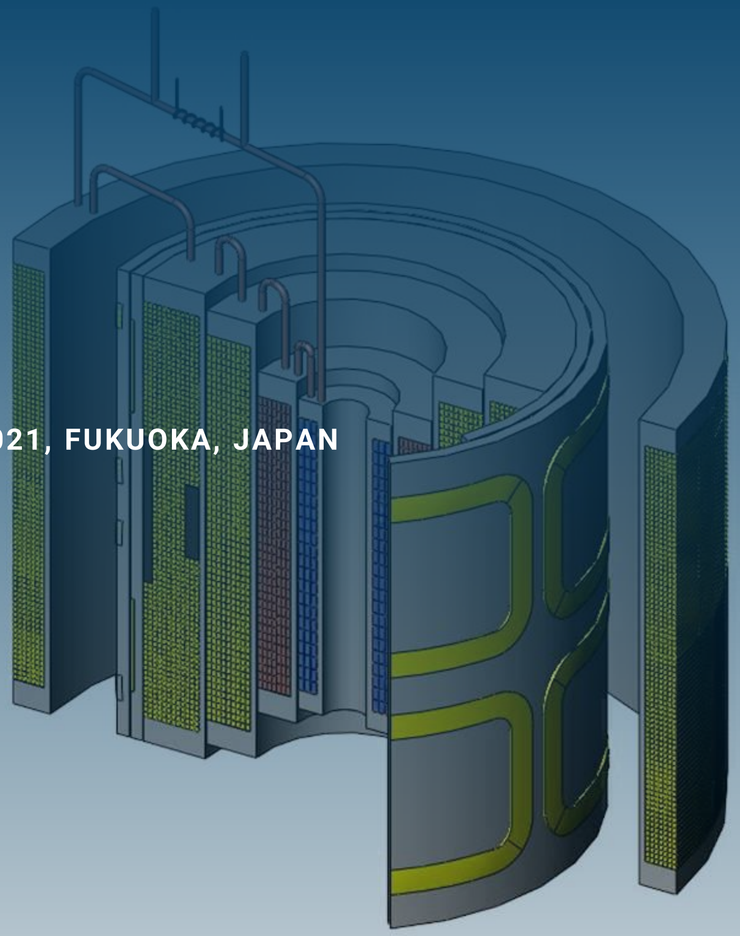
Bruker's 1.x GHz HTS-LTS hybrid NMR magnet program



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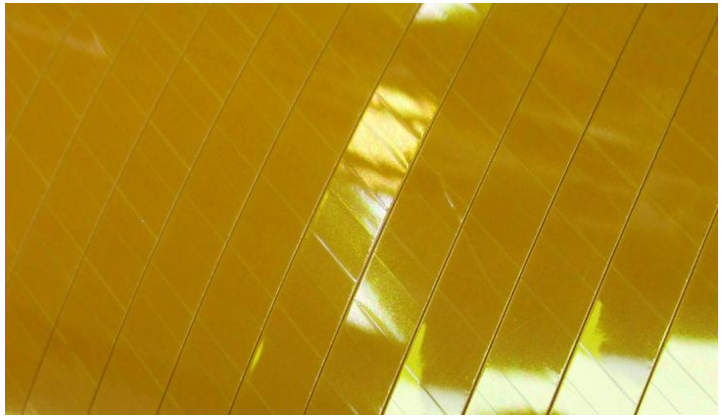
Bruker's R&D program for an UHF HTS-LTS hybrid magnet

- About 10 years ago the availability *forecast* of CCs with high J_e at 4 K and high field in quality and quantity became promising enough to start the development of a UHF HTS-LTS hybrid magnet at Bruker.
- The NMR community showed a clear interest at that time too.
- Using a series of test and prototype coils we tried to answer the following major feasibility questions, partially with academic partners:
  - How to manage the strong hoop stress and axial pressures?
  - How to protect the magnet during a quench?
  - Will the homogeneity and temporal stability be good enough for high-resolution NMR experiments?

28 layers each

Design choices for the UHF HTS-LTS hybrid magnets



- Use a minimum of CC tapes: tape availability; leverage 2 K LTS technology including active shielding.
- Layer-wound HTS coil(s): full utilization of long unit lengths; minimum number of joints; compact and homogeneous winding pack *and* leverage existing winding expertise. Need to develop tape winding and jointing techniques.
- Force management: calculated stresses low enough to remain below the strain limit of the CC substrate; a compact, layer-wound winding pack should withstand axial pressures.
- Insulated CC tapes: defined current path during energization (better homogeneity), de-energization and quenches; less time to settle at reached field.
- All HTS and LTS coils electrically in series: simpler charging procedure than separate circuits (e.g. for HTS and LTS); only one pair of current leads and one power converter.

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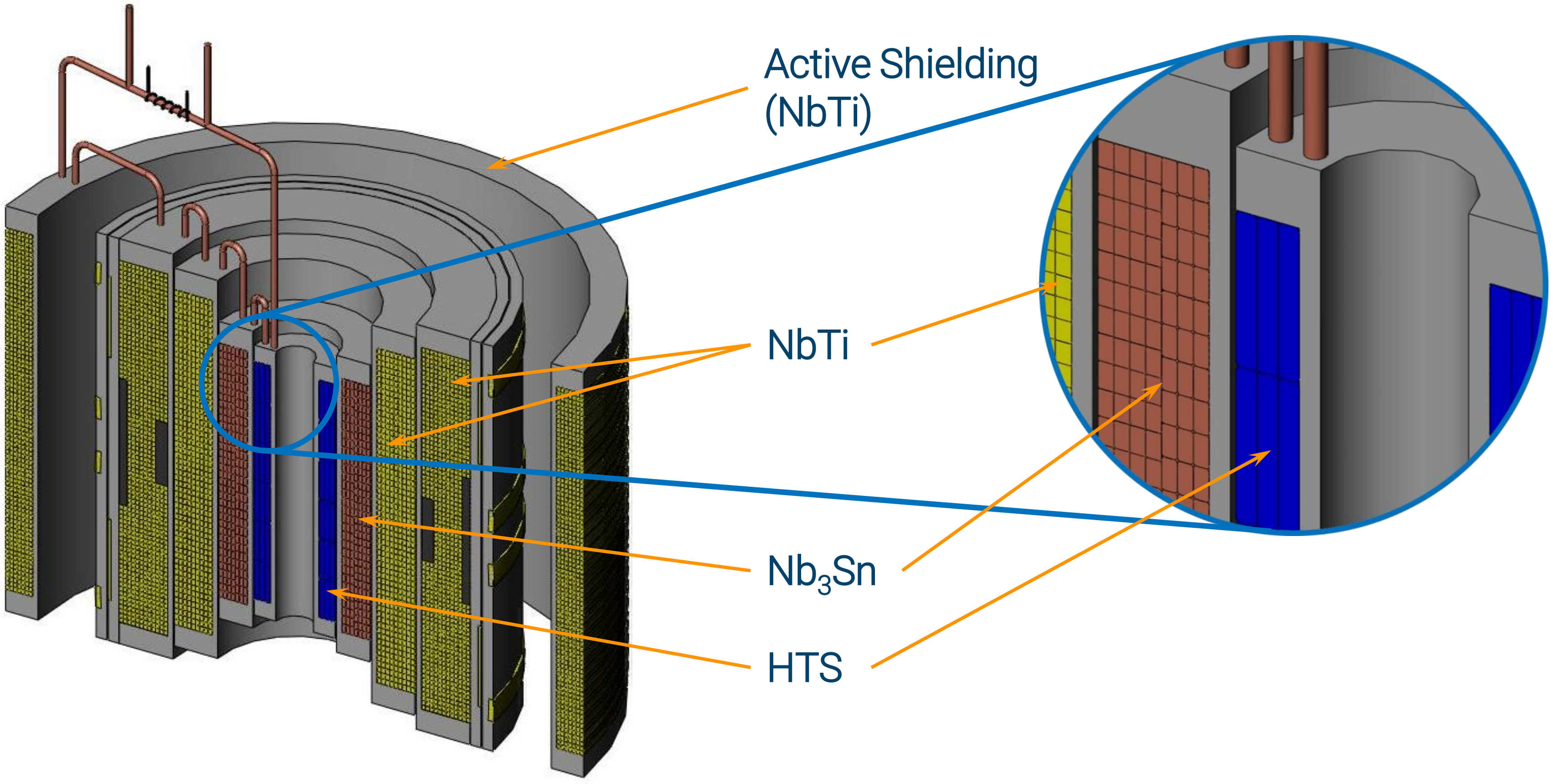
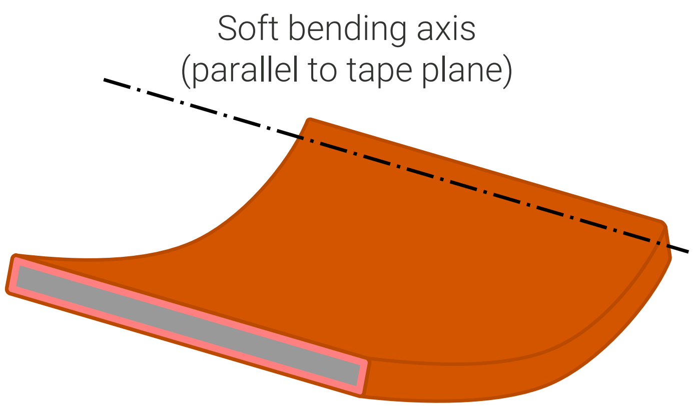
Design of the UHF HTS-LTS hybrid magnets



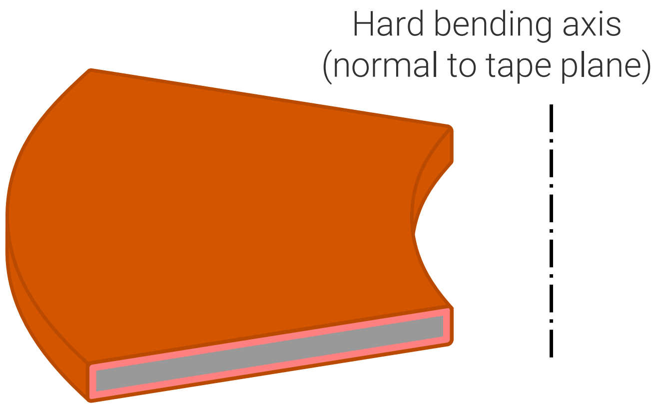
Artistic impression of the 1.2 GHz magnet design

Winding Coated Conductor tapes

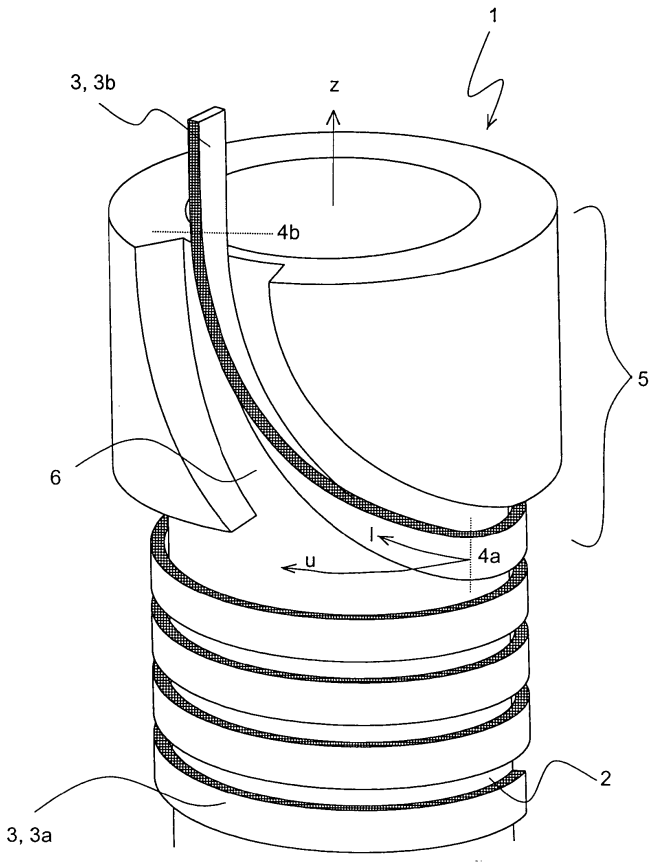
- Attempt to wind with a minimum of hard bending everywhere,
- including the region around the entry to and the exit from the main winding pack.



Soft-bending a CC tape



Hard-bending a CC tape



Bruker Patent US 7,215,230 B2

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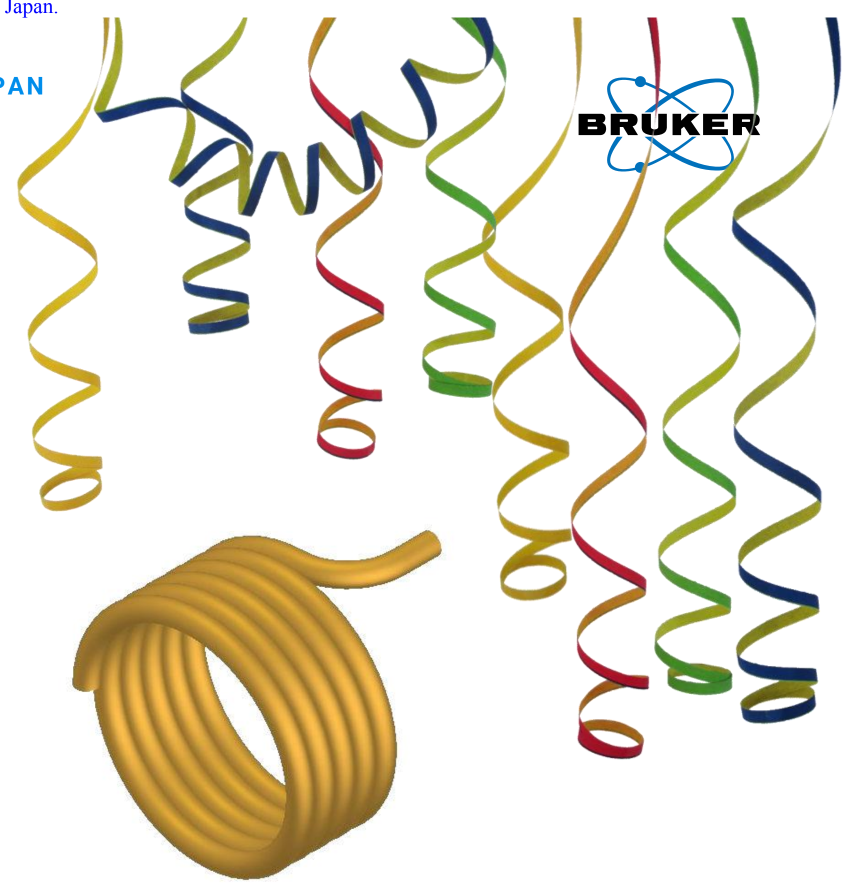
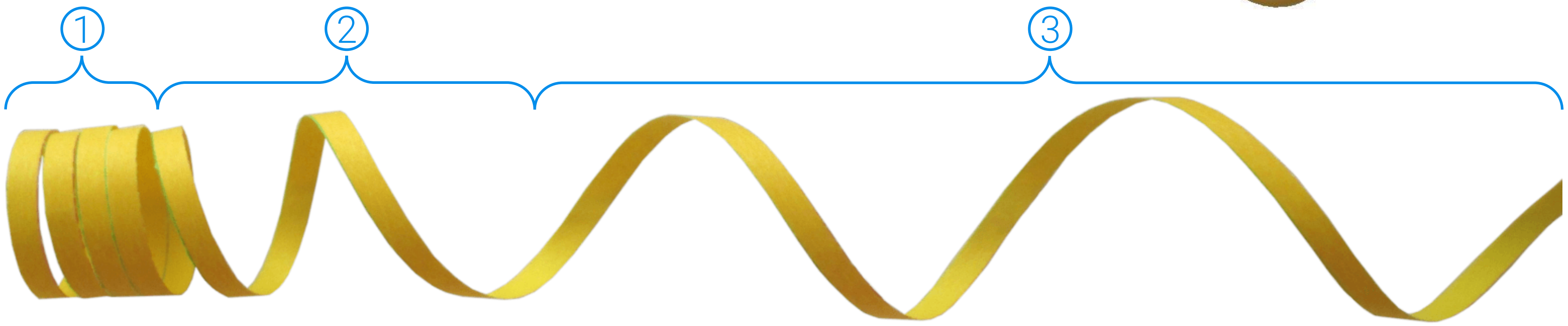
Minimize hard bending everywhere

①
- In the regular winding pack turns touch each other – dense winding
  - Tape wound on a cylinder, a developable surface, zero hard bending

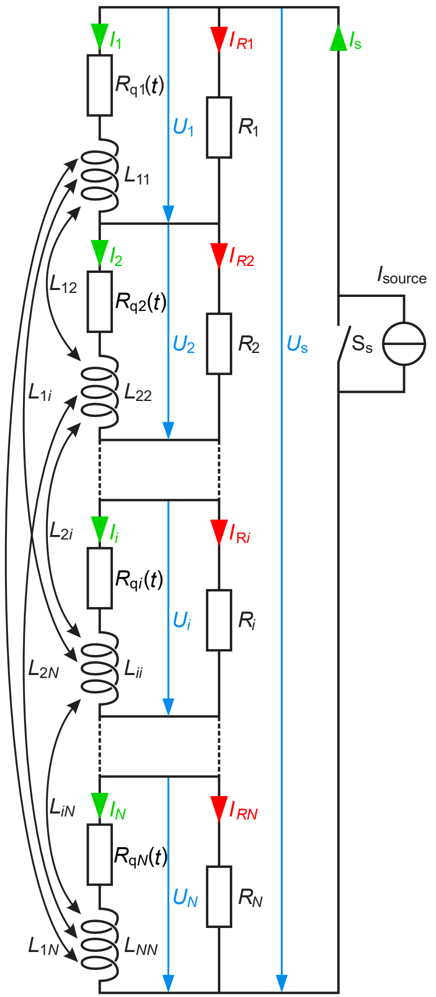
②
- Transition region with an increasing pitch length of the helix
  - It is still possible to find a developable surface, but it is complex. Some differential geometry is required.

③
- Tape exit may be on a cylindrical surface, but with a large, constant pitch length.

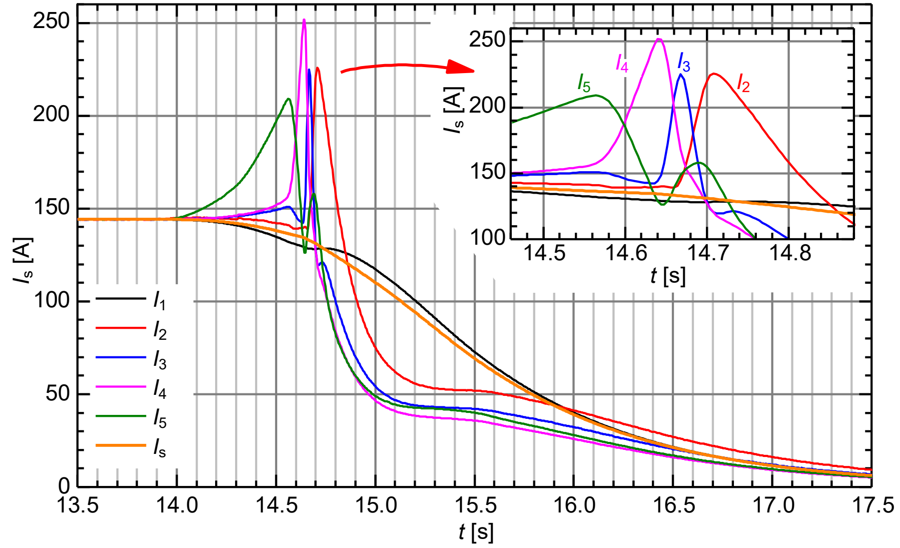




Typical quench protection scheme of LTS NMR magnets



- All coils of an NMR magnet are electrically connected in series (current: green arrows).
- A resistor is connected in parallel to each *protection section* (R_1...R_N).
- If a quench starts in a coil, the resistance there rises rapidly ($R_{qi}(t)$), the current is pushed to the parallel resistor and by inductive coupling the current in the other coils increases, depending on the mutual inductances L_{ij}!
- The next coil reaching $I_c(T)$ quenches and a chain reaction of quenching coils starts.
- The magnetic energy is quickly dissipated without the hot-spot temperature reaching unacceptably high values.



Analysis of a quench in a 600 MHz magnet some years ago

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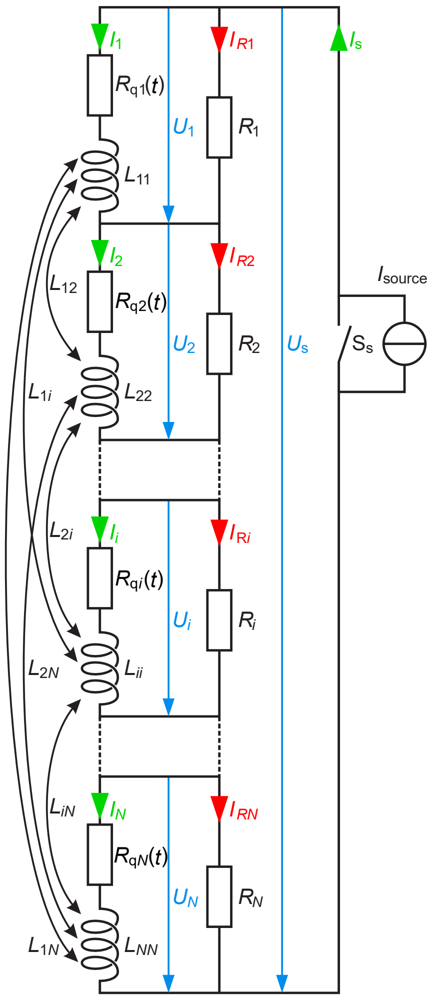
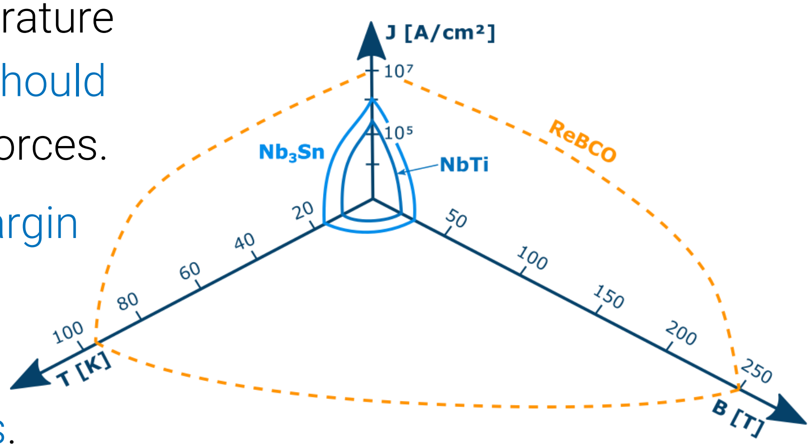
Options for quench protection in LTS-HTS hybrid NMR magnets



- The LTS scheme depends on rather small temperature and/or current margins of the conductors: they should quench with little overcurrent to limit additional forces.
- The HTS tapes have a very large temperature margin when operated in liquid helium.
- In case of a quench, the current through the HTS tapes must be limited by some other means.
- Active or passive quench protection systems are conceivable:
  - Active: An external electronic circuit monitors coil voltages and, upon detecting a quench, triggers the discharge of the HTS coil(s), e.g. via quench heaters.
  - Passive: With a suitable electric circuit (e.g. with diodes) energy from the protection section quenching *first* may be used to power quench heaters attached to the HTS coil(s). Other schemes exist as well.



Simplified critical surfaces of three technical super-conductors

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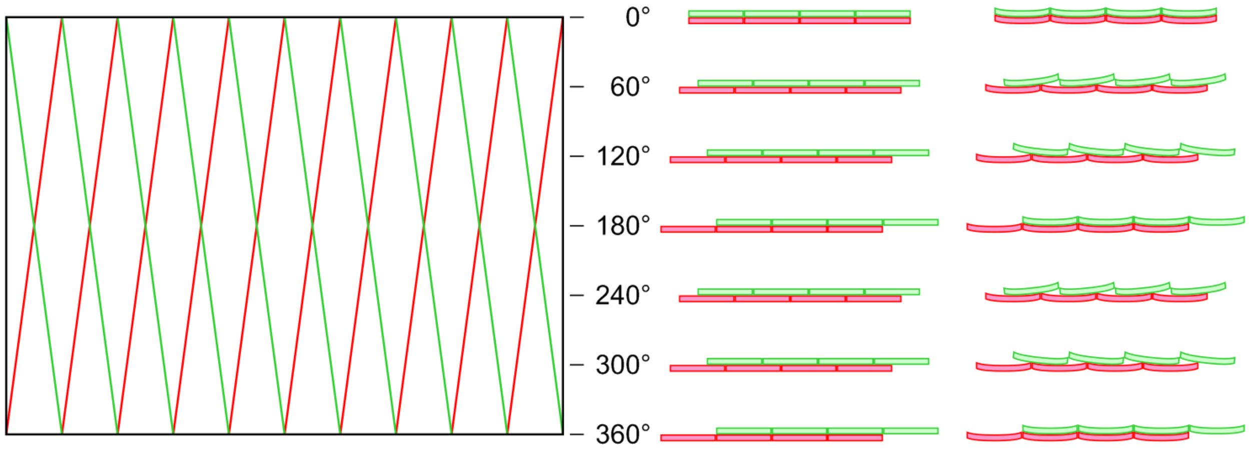
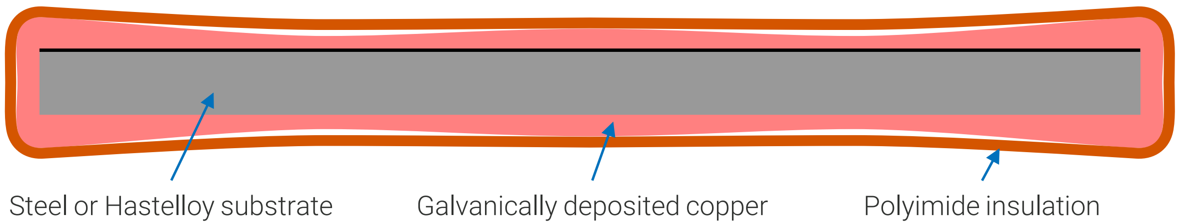
Experience gained so far with Bruker’s HTS-LTS hybrid NMR magnets

Bruker's requirements for Coated Conductors for layer-wound NMR coils (1) – Geometry

- The goal is a compact winding pack with a minimal void fraction.
  - The tape cross section should be as *rectangular* as possible, irregularities lead to voids.
  - Example curved tapes ("C-bow"): difficult to wind, gaps in winding pack.
  - Example dog-boning (non-regular galvanic deposition of copper): voids in winding pack, possibly less copper than specified



Pink: first layer, green: second layer



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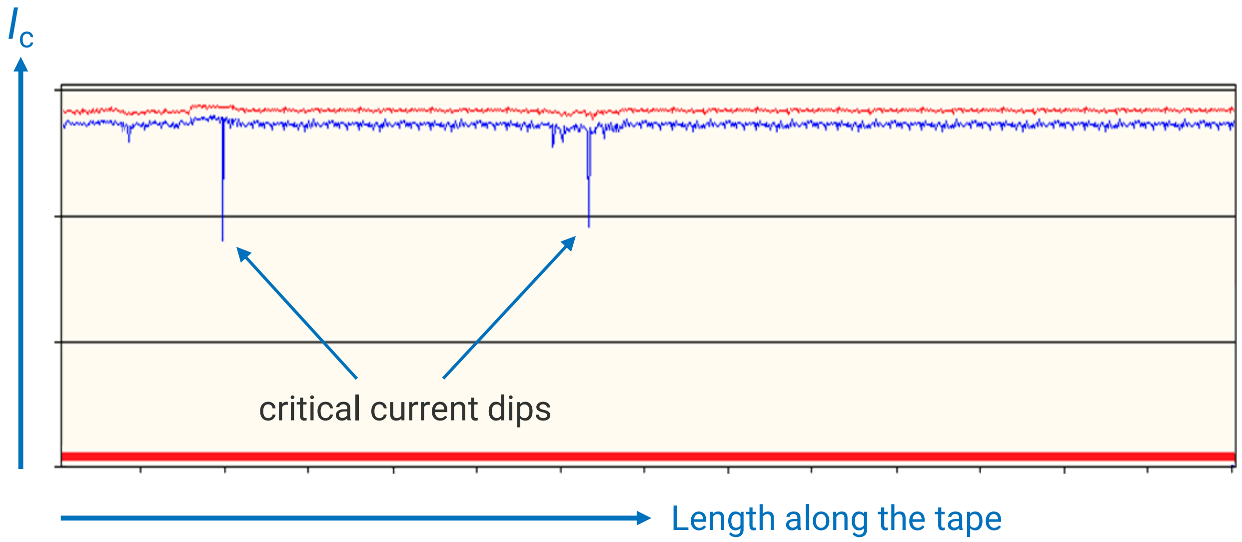
Bruker's requirements for Coated Conductors for layer-wound NMR coils (2) – Critical current

- Several commercial suppliers are now able to manufacture Coated Conductors with good I_C at 4 K and 10 T B||c. (~300 A to >500 A)
- For NMR magnets it is important that there are no drop-outs (I_C dips) along the entire tape length used.
- The available unit lengths, which often result from dips, *could always be longer*.
- Hastelloy substrate



TapeStar measurement (symbolic data)

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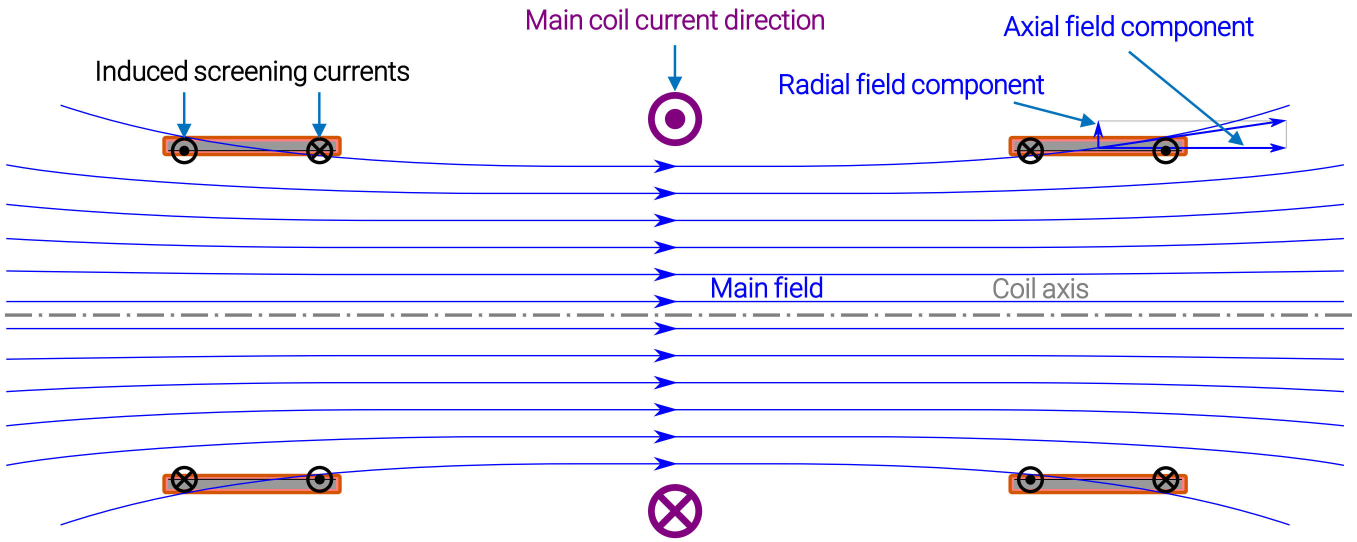
The homogeneity of HTS-LTS hybrid magnets – screening currents

- HTS screening currents strongly influence the magnet's homogeneity
  - The 4 mm wide tapes offer a big area to induce loop currents, which tend to screen the tape centre
  - At the centre of a solenoid the screening currents mainly generate additional field and a z^2 gradient.



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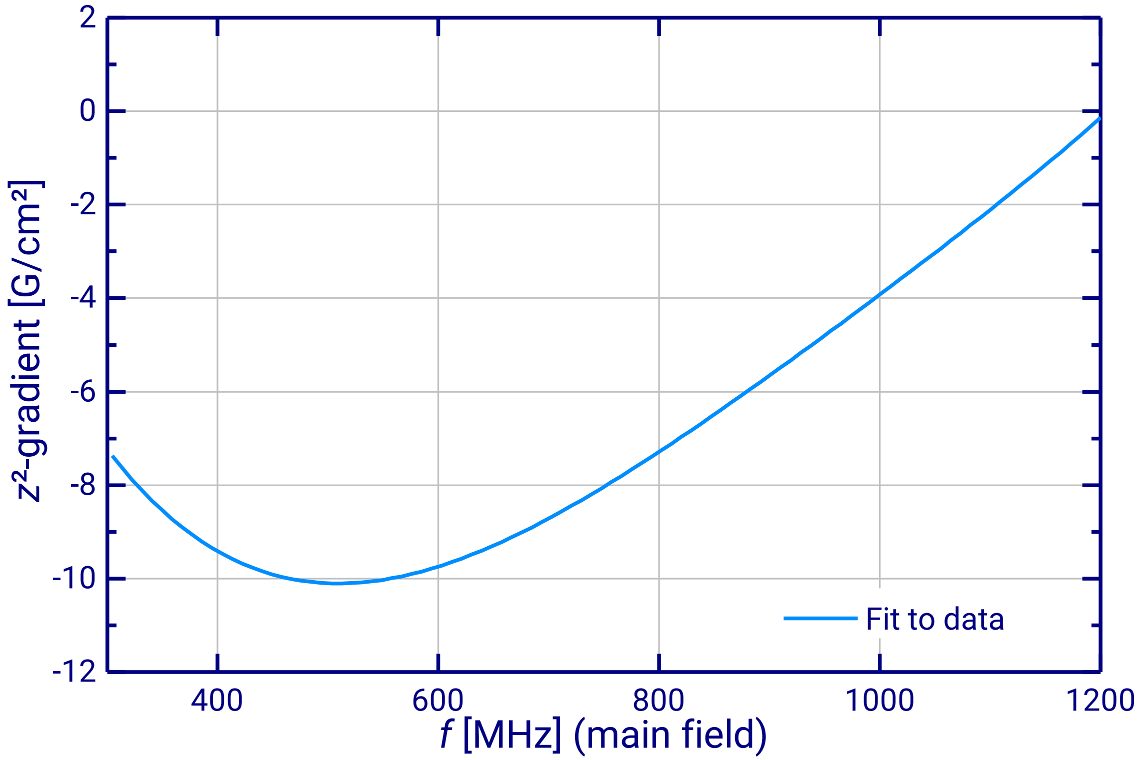
z^2 gradient of HTS-LTS hybrid magnets during energization

- During magnet energization the z^2 gradient varies considerably.
- The goal is to get zero z^2 gradient at the target field.
  - This effect must be considered in the homogenization process.



z^2 gradient of a 1.2 GHz magnet during energization

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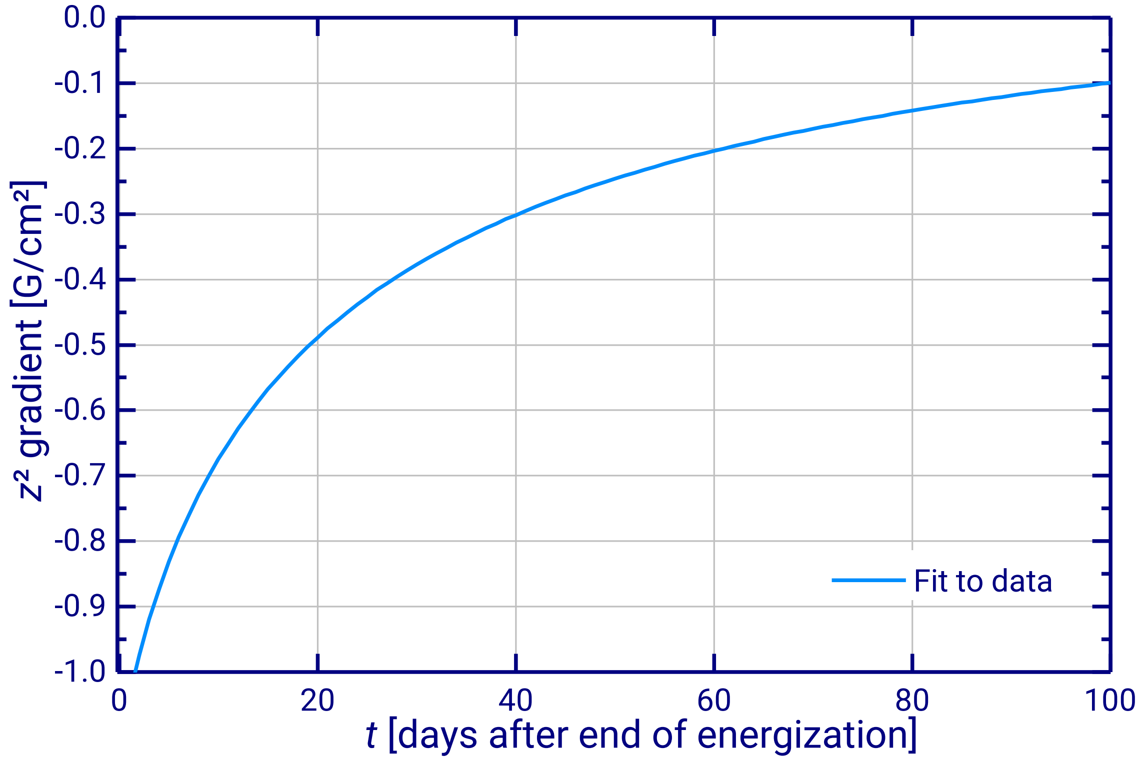
Initial drift of the z² gradient of HTS-LTS hybrid magnets

- The relaxation of the screening currents also leads to an initial increase of the z² gradient.
  - Also, this effect must be considered in the homogenization process.
- The z² gradient stabilizes after several weeks.



Initial drift of the z² gradient of a 1.2 GHz magnet after reaching field (full energization)

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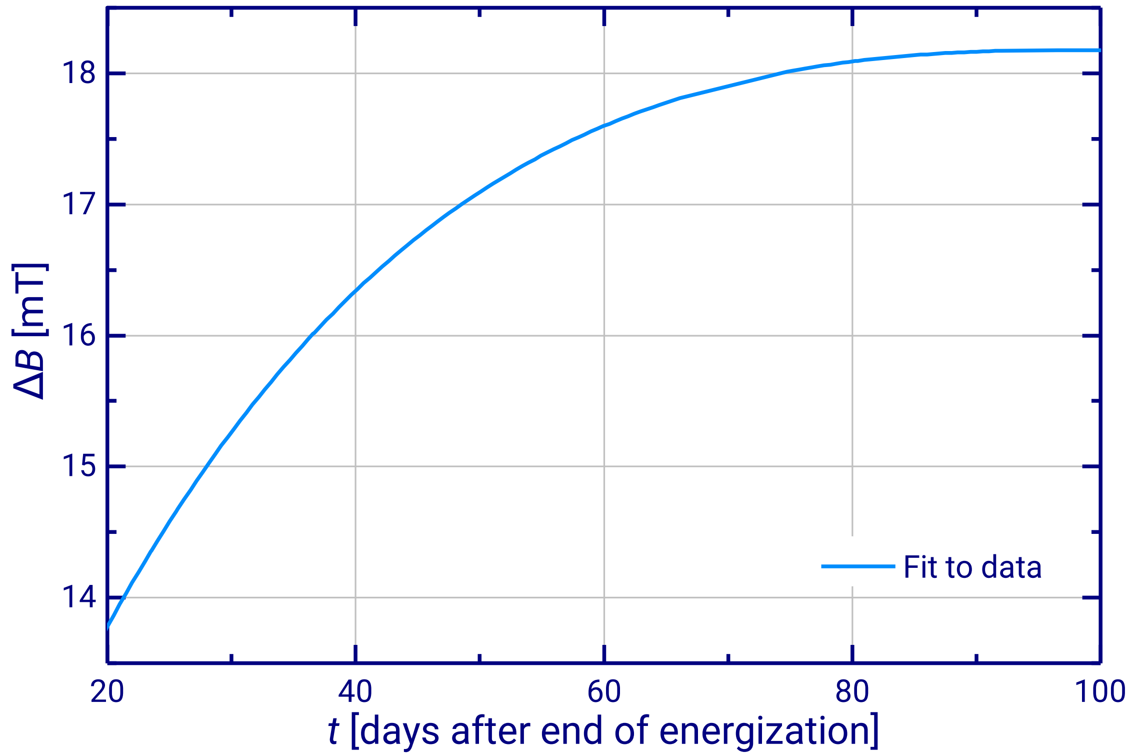
Initial drift of HTS-LTS hybrid magnets

- The relaxation of the screening currents leads to an initial increase of the main field
  - An overshoot after reaching nominal field is therefore not needed.
- The main field stabilizes after several weeks as well.
- Eventually the absolute value of the drift becomes small enough to stay within the specification



Initial drift of a 1.2 GHz magnet after reaching field (full energization)

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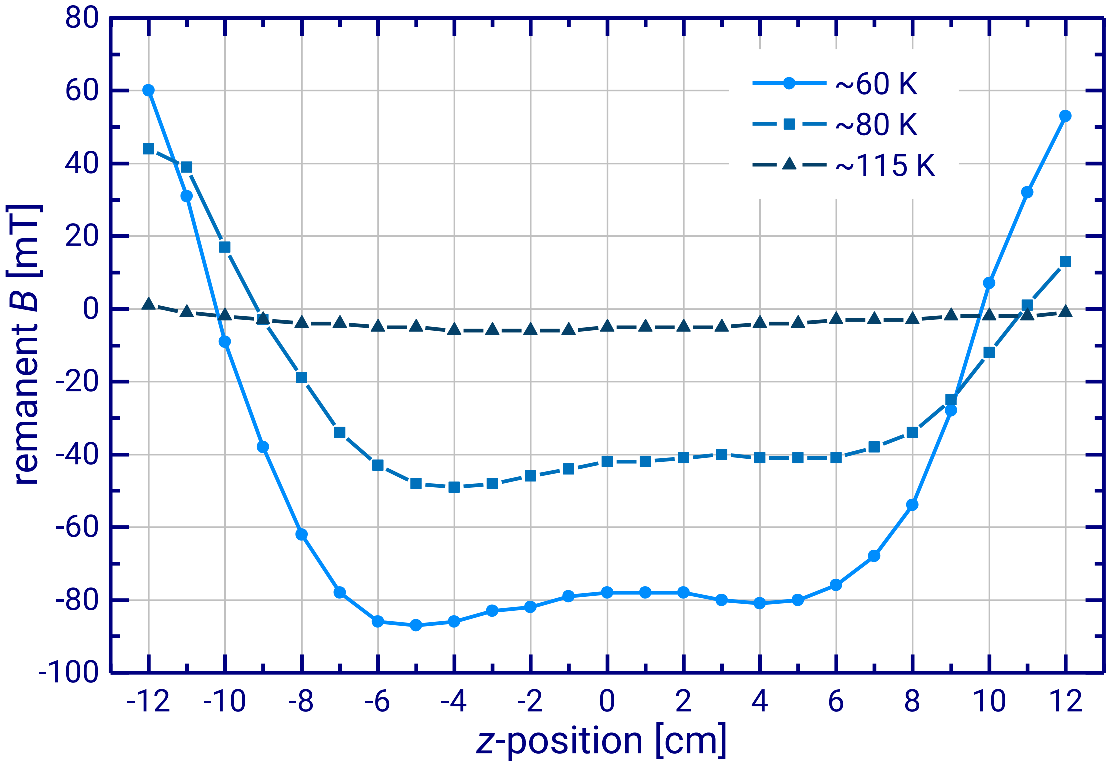
Remanent field of HTS-LTS hybrid magnets

- After complete de-energization of a hybrid magnet the screening currents in the CC tapes generate a remanent magnetic field.
  - To start a new energization sequence at a defined magnetic state after a quench, the screening currents must be removed by warming up the magnet to a sufficiently high temperature.



Remanent field profile of a 1.2 GHz magnet during warm-up after reducing the magnet current to zero

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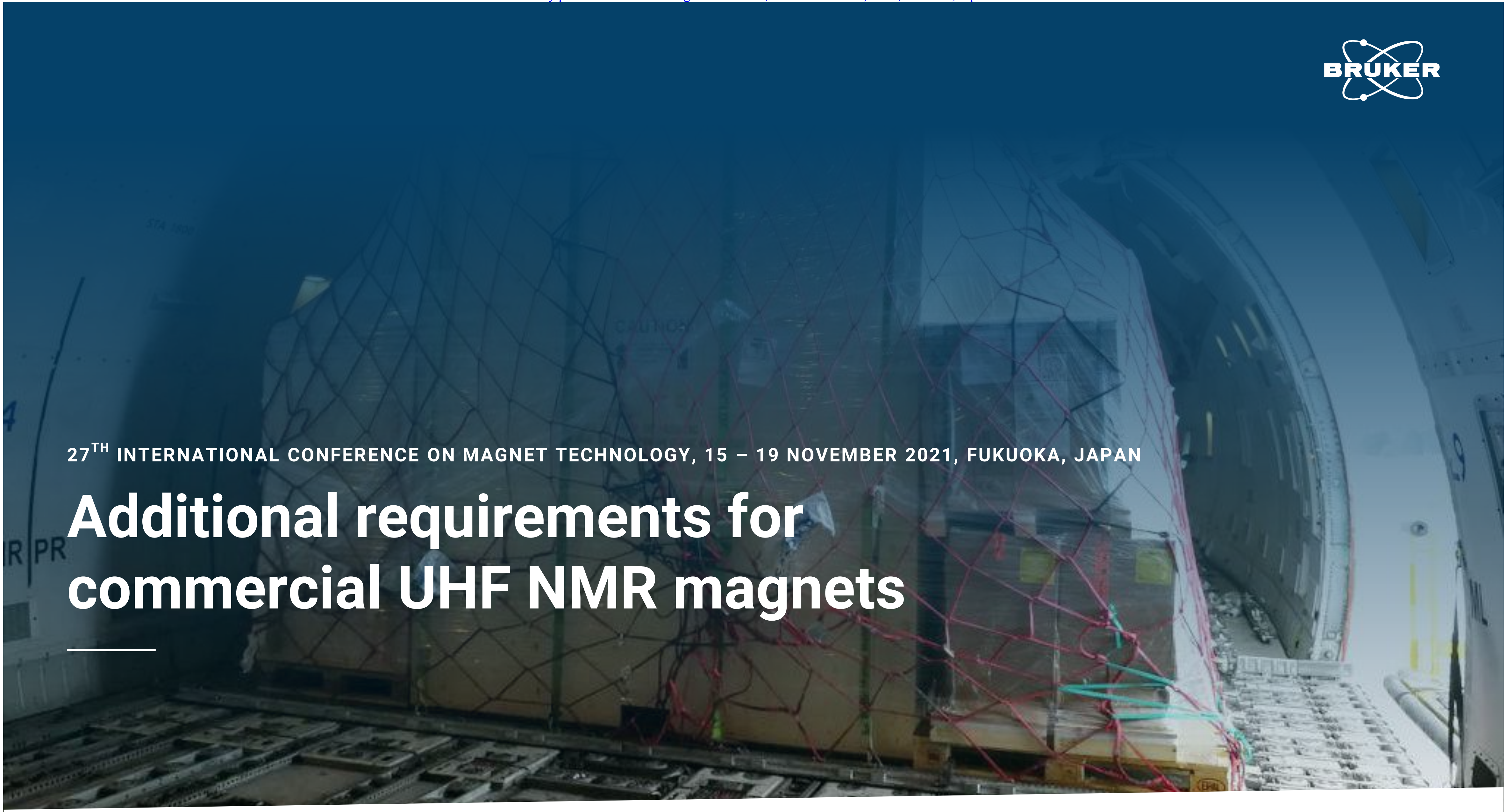

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Additional requirements for commercial UHF NMR magnets

Picture: A 1.2 GHz NMR magnet inside the body of a cargo airplane

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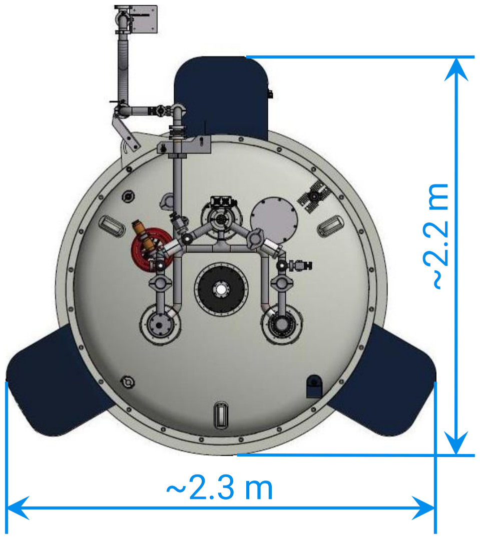
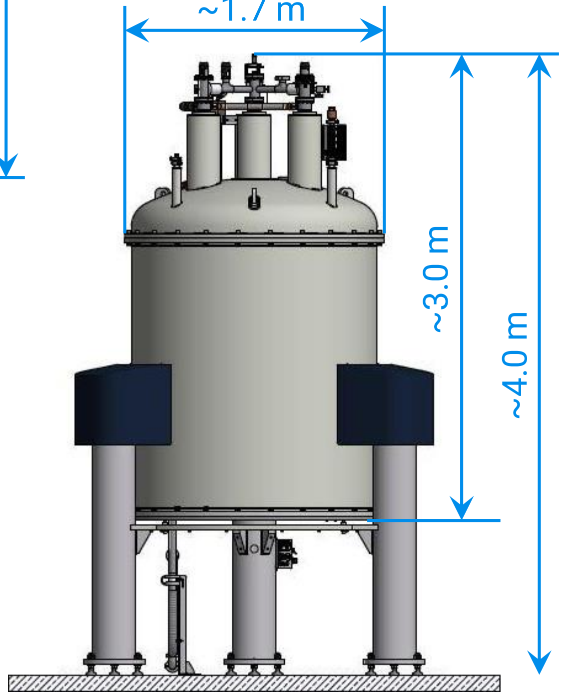
Additional requirements for commercial systems (1)

Direct customer benefits

- Easy siting
  - System height and required ceiling height
  - Active shielding for a minimum stray field
  - Resilience to EM disturbance and vibrations
  - As “light” as possible (floor loading and on-site transport)
- Ease of use
  - High reliability at installation and during operation
  - Highly automated operation and system monitoring, not least for remote assistance
- Cryogen consumption
  - Acceptable consumption requires persistent mode operation
  - Long holding time of cryogens, easy refill process

Magnet type	Ascend 1.2 GHz
Operating field	28.19 T
System height	~4.0 m
System weight	~8000 kg
Actively shielded	





Additional requirements for commercial systems (2)

Indirect customer benefits

- Manufacturing considerations
  - Reliability of design and manufacturing processes
  - Stable and reliably supply chain, in particular superconductors, for product availability even after long funding processes of customers
  - Compatibility with other Bruker systems, e.g. standard RT bore of ø 54 mm to accommodate the standard RT shim system and NMR probes
- Logistical needs
  - Transport
    - Shippable without special equipment or permissions on a truck with regular external dimensions
    - Must fit into airplane for intercontinental deliveries
  - Installation with industry-standard cryogenic and other equipment

Lifting a 1.2 GHz NMR magnet through the roof to its installation place at Jülich, DE

The wooden transport box with a UHF NMR magnet inside the body of a cargo plane – *just* fitting through the door!

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Where the coils are wound

Coil winding hall in Fällanden, Switzerland

- Bruker winds several types of NMR coils (NbTi, Nb_3Sn and CC) in Switzerland, close to Zurich
- The winding machines were developed and optimized in house

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Cryostat assembly

The UHF cryostat assembly hall in Fällanden, Switzerland

- After assembly, the magnets are mounted into the cryostats here
- Bruker 1.2 GHz magnets are operated at 2 K. The associated cryogenics technology, assembled here, has been successfully employed at Bruker for several decades.

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Magnet and system test stands

On this test stand the first 1.2 GHz magnet reached field

More stands to test several magnets in parallel

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HTS-LTS hybrid NMR magnets delivered and ordered summary and outlook

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Installed 1.2 GHz NMR Systems (1)

CERM, University of Florence, IT

ETH Zurich, CH

MPI Göttingen, DE

Installed 1.2 GHz NMR Systems (2)

FZ Jülich, DE

Utrecht University, NL

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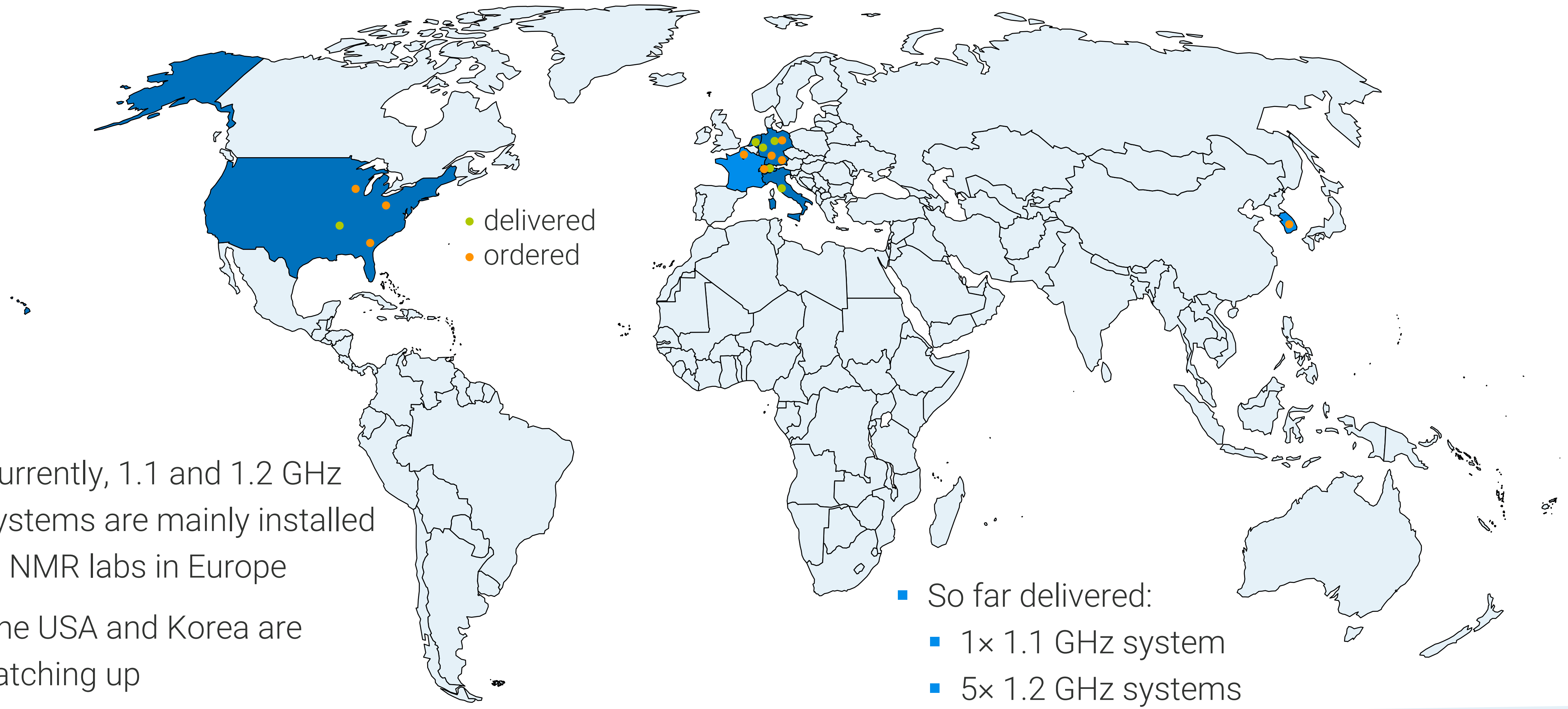
1.1 and 1.2 GHz NMR systems using Coated Conductors ordered or delivered worldwide (November 2021)

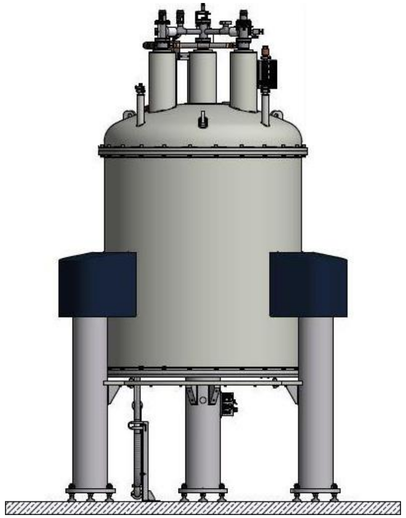


- Currently, 1.1 and 1.2 GHz systems are mainly installed in NMR labs in Europe
- The USA and Korea are catching up

- So far delivered:
  - 1× 1.1 GHz system
  - 5× 1.2 GHz systems

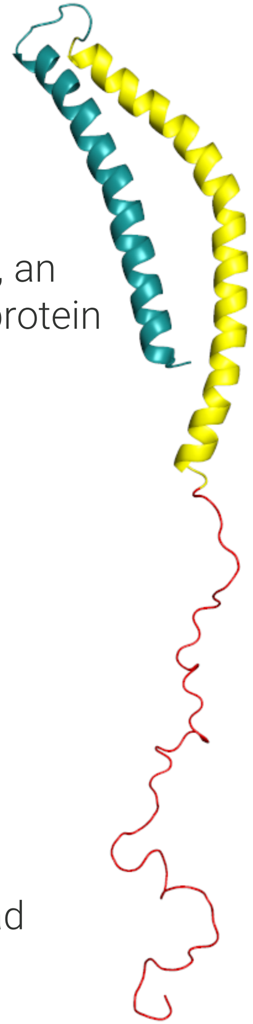
Bruker 1.x GHz NMR magnets in between…

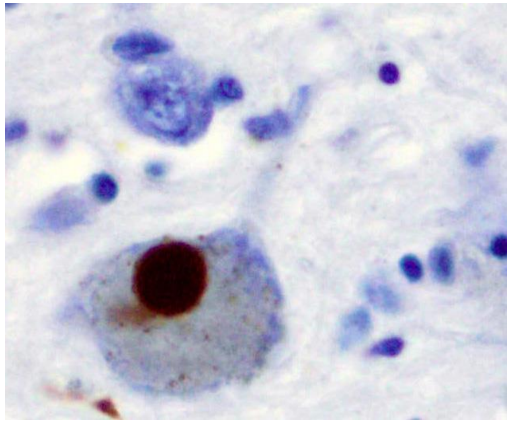
…ReBCO Coated Conductors and…



α-Synuclein, related to Parkinson's disease.

Structure of α-Synuclein, an intrinsically disordered protein





Positive α-Synuclein staining of a Lewy body from a patient who had Parkinson's disease.
(Pictures: Wikipedia)

…our customers, who discover, study and develop complex new substances and materials for the improvement of health and life!

Acknowledgements

Many, many thanks to the numerous people who contributed to the success of the UHF HTS-LTS hybrid NMR magnet project, inside and outside Bruker!

Cake at the occasion of reaching 1.2 GHz for the first time.
Hand made by a skilled Bruker technician.

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