Published in final edited form as:

IEEE Trans Appl Supercond. 2014 June ; 24(3): . doi:10.1109/TASC.2013.2285287.

A 1.5-T/75-mm Magic-Angle-Spinning NMR Magnet

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Abstract

We are currently working on a program to complete a 1.5-T/75-mm RT (room temperature) bore MAS (magic-angle-spinning) NMR (nuclear magnetic resonance) magnet. The MAS magnet comprises a z-axis 0.866-T solenoid and an x-axis 1.225-T dipole coil. The combination of the fields creates a 1.5-T field pointed at 54.74 degrees (magic angle) from the rotation (z) axis. During the 2nd year of this 3-year Phase I program, both coils have been wound and testing has begun. Some preliminary field mapping has been performed, and the design of the MAS magnet assembly has been completed. During the final year, the magnet assembly will be integrated into the cryogenic structure and tested at ~5.5 K in a solid nitrogen environment. Each coil will be energized separately, and the magnetic field will be mapped accurately. We expect a bare magnet uniformity of 100 ppm over a 10-mm diameter, 20-mm-long cylindrical volume. Then, using the field data, the uniformity will be improved to < 0.1 ppm with a combination of ferroshims and cryoshims. Final field measurement will be performed as the cryostat-magnet system is spun manually at ~0.1 Hz.

Index Terms

Magic-angle-spinning superconducting magnet; nuclear magnetic resonance (NMR)

I. INTRODUCTION

AROTATING magic-angle field can be created either electrically or mechanically. In the electrical approach, the rotating field is achieved by a combination of a constant field in the rotation axis and a time-varying field normal to the constant field. While this approach has worked in a low-field (< 50 gauss) copper and permanent magnet designs [1]–[5], it is impossible to create a time-varying field approaching 1 T with a superconducting magnet, chiefly because of ac losses. In contrast, the mechanical approach uses a combination of two dc superconducting magnets that are rotated.

Phase I has two specific aims: (1) build a superconducting magnet system comprising a z (axial)-field solenoid (B_z) and an x-y dipole (B_x), whose combined magic-angle field, B_{ma} , of NMR-quality and 1.5 T points at an angle of 54.74° (magic angle) from its spinning (z)

axis; and (2) demonstrate an innovative cryogenic system that houses this superconducting magic-angle-field (MAF) magnet and to be rotated at 6 Hz in Phase II.

II. MAGNET DESIGN AND ANALYSIS

The magnet design configuration is provided in Fig. 1. The design consists of a three-part solenoid coil wound on a central stainless steel support tube onto which two opposing coils each comprised of two double pancake saddle coils are mounted. An iron yoke surrounds the assembly which is held between two end flanges.

Table I summarizes the coil design parameters for this magnet. The magic-angle field is 1.5 T, comprised of a 1.2247-T dipole field and a 0.8660-T solenoid field. The NbTi conductor has been provided by Supercon Inc. (Shrewsbury, MA). The 18-filament conductor has insulated dimensions of 1.6 mm $\times$ 0.8 mm with a copper-to-superconducting ratio of 7 : 1.

We expect the magnet to have an as-wound field homogeneity of < 100 ppm over a $\phi 10$ -mm, 20-mm-long cylindrical volume oriented along the magic-angle axis. Currently, we are developing a technique for active and passive shimming of the MAF by use of superconducting shim coils and ferromagnetic tiles to produce an NMR-field quality of < 1 ppm.

Magnetic field analysis was performed with the 2D FEMM magnetic field program. The field plot for the dipole is provided in Fig. 2, while the solenoid plot is shown in Fig. 3. For the dipole coil, the surrounding iron yoke significantly reduces the required ampere-turns by more than 30%, while creating a peak field of ~ 1.3 T in the iron itself, which is, as required, well below the saturation level of the 1010 steel plates. Although the solenoid coil adds a field to this dipole-induced field, the iron yoke field will still be below the nominal 1.5 T saturation level.

III. STRUCTURAL ANALYSIS

An ANSYS 3D structural model was developed to analyze the stresses created by the solenoid-dipole interaction which generates an internal torque of ~ 1740 N m between the two coils. As shown in Fig. 4, the peak stress of ~ 70 MPa occurs in the solenoid support winding slots. This level of stress is acceptable for the stainless steel mandrel. The model analyzed the thermal contraction and effect of 6-Hz rotation. Table II gives a summary of these data.

IV. COIL FABRICATION

In our previous paper [5], we discussed the fabrication of a short-length version of these coils. Now, the first full-length double pancake coils have been wound. There will be four layers of windings in each dipole coil, each coil comprised of two double-pancake saddle coils wound on top of each other. This topology requires one persistent joint between each double pancake.

A simple winding machine similar to that used to wind quadrupole coils at Jefferson Laboratory [6], designed and constructed, formed this saddle coil into shape. The coils were wet-wound with Shell Epon 815 epoxy and wound at a winding tension of ~40 N. The torsional stiffness of the NbTi wire made it difficult to maintain the shape of the coil after winding. After several winding trials, we resolved this by bonding the underside of the first layer of the double pancake with a 0.5 mm-thick epoxy-glass layer. After this layer was fully cured, the winding guides were adjusted, and the second layer of the double pancake was wound. A photograph of the dipole is provided in Fig. 5.

The solenoid coil shown in Fig. 6 has been completed and is ready for initial testing. This coil consists of three subcoils wound into separate slots. One continuous length of wire was used with crossover slots between each coil. This coil was also wet-wound with Epon 815 epoxy.

During this development, we have repeatedly made successful, persistent NbTi–NbTi joints which can carry over 300 A at 4.2 K in self field, which exceeds the operating current for both the dipole (244.5 A) and solenoid (237.9 A). These joints will be strategically place in low-field regions of the magnet assembly where the magnetic field is < 0.25 T. One joint will be required to connect the two double pancakes for each dipole. Also, one joint will be used between the two dipole coils and two more to connect the whole assembly to a persistent current switch. For the solenoid coil, only two joints will be required to connect the coil to its persistent current switch.

V. COIL TESTING

The dipole coil was tested several times, including a final test up to 275 A at 1 A/s, showing no quench behavior. Fig. 7 shows a circuit used in these tests. Note that in actual operation, the dipole coil will be in persistent mode and its protection will rely on standard protection method for persistent-mode magnets [7].

Next, the second double pancake will be wound for each coil, and the assembly will be tested again, along with the solenoid coil.

Preliminary field mapping results were performed at 5 A in liquid nitrogen. For each test, the Hall sensor was mounted on an axial probe with the Hall sensor radially positioned 10 mm from the coil axis. These data (Fig. 8) show $< 1\%$ error between measurement and calculation. A resolution of $\sim 0.1\%$ is the limit of the Hall sensor in our measurement rig, but it at least provides a preliminary check of the field uniformity.

Once the dipole and solenoid coils with the iron yoke are assembled and operated at 4.2 K, a 3D mapping with a higher field resolution will be performed, in order to determine the shimming requirements. Then, a combination of ferromagnetic and superconducting shims will be employed.

VI. CRYOGENIC DESIGN

The innovative cryogenic design concept discussed in previous literature [8]–[12] developed at MIT will be employed, in which the magnet will be immersed in solid nitrogen (SN_2).

This all-solid cold body ameliorates thermo-fluid issues associated with liquid under rotation. Also, solid nitrogen ensures a uniform temperature throughout the windings and provides a large thermal mass, enabling the magnet to maintain its operating field over a time period even when a flow of liquid helium (LHe), its primarily cooling source, is shut off. The Phase I cryostat, due chiefly to a thermal mass of the 10-liter SN_2 , will have an estimated warm-up time period, from 4.5 K to 5.5 K, of > 1 h.

VII. CONCLUSION

The fabrication and testing of an MAS magnet system are well underway, and the initial results look promising. The final assembly and testing of this magnet will be completed in Phase I, scheduled to end in August 2014. The test includes operation in persistent mode at 4.2 K and in the range 4.2–5.5 K under solid nitrogen cooling.

Accurate field mapping will be conducted with an NMR probe. Then, we will shim the field with a combination of superconducting shim coils and ferro shims. Both shims will be designed and built to enable them to withstand 6-Hz rotation, which takes place in Phase II.

ACKNOWLEDGMENT

The authors would like to thank J. Colque, N. Candelino and P. Allen of the MIT Francis Bitter Magnet Laboratory.

This work was supported by the National Institute of Biomedical Imaging and Bioengineering of the National Institutes of Health under Award Number R01EB013231.

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Fig. 1. MAS magnet design concept. The solenoid tube is wound into the slots in the central solenoid mandrel onto which the dipole coils are mounted. The magnet assembly is surrounded by iron laminations for shielding and also to reduce the magnet operating current.

Fig. 2.
Dipole field plot showing the central 1.225 T uniform region.

Fig. 3.
Solenoid field plot showing the 0.866 T uniform region in black.

Fig. 4. ANSYS structural model showing stresses (MPa) due to solenoid–dipole interaction. The peak stress (black region) is 70 MPa.

Fig. 5.
First double-pancake layer of full-size dipole magnet.

Fig. 6.
Solenoid coil wound and ready for testing at 4.2 K.

Fig. 7.
Circuit diagram for 4.2-K test setup.

Fig. 8.
Field mapping results at 77 K and 5 A showing good agreement between measured values and pure cosine field profile.

TABLE I**MAS MAGNET SUMMARY**

Item	Units	Value
Center magic-angle field (B _{ma})	[T]	1.5
Solenoid (axial, z) field (B _z)	[T]	0.8660
Dipole (x-y) field (B _x)	[T]	1.2247
Center field orientation	[degree]	54.74 from z-axis
Axial (spinning axis) RT bore	[mm]	75
NbTi insulated conductor width; thickness	[mm]	1.60; 0.85
OD of solenoid-dipole structure	[mm]	137.5
Overall coil length	[mm]	486
Overall wire length (dipole/solenoid)	[m]	740 / 260
Operating current (dipole/solenoid)	[A]	244.5/237.0
Rotation frequency about z-axis	[Hz]	~0.1 (Phase 1)
Operating mode w/ LHe transfer		persistent, continuous
Temperature range w/o LHe transfer	[K]	4.5 (nominal) 5.5 (limit)
Operation duration w/o LHe transfer	[hr]	~1.25
Homogeneity @ 100Φ X 20 MM	[ppm]	<1 w/ferro-shimming

TABLE II**STRUCTURAL ANALYSIS SUMMARY**

Case	Item	Value
6-Hz Rotation	Cold Mass Stress (MPa)	0.50
	Radial Displacement (mm)	1e-4
Electromagnetic Torque (1740 N-m)	Stress (MPa)	70
	Radial Displacement (mm)	0.04