Published in final edited form as:

IEEE Trans Appl Supercond. 2017 June ; 27(4): . doi:10.1109/TASC.2017.2672718.

Inductively-coupled Frequency Tuning and Impedance Matching in HTS-based NMR Probes

Vijaykumar Ramaswamy^{1,*} [Member, IEEE], Arthur S. Edison^{2,*}, and William W. Brey¹

¹Florida State University, Tallahassee, FL, 32301, USA

²University of Georgia, Athens, GA, 30602, USA

Abstract

Nuclear Magnetic Resonance (NMR) probes based on High Temperature Superconducting (HTS) resonators have demonstrated significant gains in detection sensitivity. However, the widespread acceptance of this technology has been limited by some unresolved issues including the mechanical unreliability of the moveable inductive loops used to adjust tuning and matching. In order to improve reliability, we propose to implement frequency tuning and impedance matching of HTS resonators using fixed inductively coupled loops and variable capacitors. By analyzing the loss mechanisms associated with inductive loops, we predict that using a superconducting inductive loop for tuning and matching will not only improve the reliability of HTS probes, but also provide improvements in sensitivity.

I. Introduction

Improving the detection sensitivity of Nuclear Magnetic Resonance (NMR) spectroscopy continues to be a challenging problem for probe designers. Cooling the probes to cryogenic temperatures was first implemented more than two decades ago [1]. Shortly thereafter, the high Q-factor of High Temperature Superconducting resonators was exploited to demonstrate significant improvement in NMR sensitivity [2]. Since then, the major commercial vendors of NMR equipment have produced cryogenic probes based on normal-metal conductors, and these cryogenic probes have become a routine tool in chemistry and biochemistry laboratories around the world. However, HTS-based probes have found acceptability only in some niche applications. This work is aimed at improving the tuning and matching mechanism which is currently an important obstacle in the widespread adoption of HTS-based probes.

A. Construction of HTS Probes

The overall construction of HTS based probes has remained relatively unchanged since they were originally introduced. A pair of HTS resonators, patterned out of thin-film YBCO on planar sapphire substrates, straddles the sample [3], [4]. Thermal isolation is provided by evacuating the space surrounding the coils. Sample temperature is regulated by passing dry heated gas over the sample tube. Energy is transferred to the resonators using inductive

*Vijaykumar Ramaswamy and Arthur S. Edison were at University of Florida, Gainesville FL, 32310 when this work was performed.

coupling to a small loop. Impedance matching to the resonators is adjusted by translating this inductive loop as explained in [5]. Similarly, fine frequency tuning is achieved by translating a shorted inductive loop. Other applications of inductive coupling and tuning in NMR are also known [5], [6]. More recently, tuning of HTS resonators using sapphire plates has also been investigated [7].

B. Analysis of Moveable Loops

The moveable loop mechanism has been implemented successfully in several HTS probes [8]–[10]. Moveable loops are relatively easy to implement and provide an adequate tuning and matching range. Since there is no attachment to the HTS resonator, swapping either a resonator or a loop is a simple process. The moveable loops also have some significant drawbacks including an effect on the magnetic field homogeneity when the loop is moved, and the possibility of failure over long term.

The primary drawback associated with the use of moveable loops is their lack of mechanical stability. When used with single-sided HTS coils, the loops can be positioned so they slide against the back of the substrate which reduces motion of the loops. However, with double-sided [9] or double-resonance [11] coils, a sliding loop would tend to scratch the YBCO. Further, the loops and their supports can also bend out of position after repeated adjustment or even due to thermal stress, causing a change in the tuning range which renders the probe unusable. In an extreme case, the loops also present a greater risk of scratching the double-sided HTS resonator.

While the wires used to fabricate the moveable loops are susceptibility-compensated, the compensation is never perfect. As a result, moving a loop close to the sample in a shimmed magnetic field degrades the linewidth of the spectrum. In one example using our ^{13}C -optimized probe, a change in position of the ^{13}C tuning loop broadened the ^1H spectral lines to reduce the signal peak height by up to 15%. While this apparent loss in sensitivity may be quickly regained by reshimming, this is not ideal especially in automated high-throughput NMR spectroscopy systems.

Loss due to the inductive loops is also an important issue. Losses in the tuning loops arise due to two mechanisms, namely losses due to i) the transport current around the loops and ii) eddy currents in the loop. Losses due to transport current in the loops increase proportionally with the series resistance of the wire. On the other hand, eddy current losses increase with the diameter of the wire. For a fixed tuning loop, it is possible to compute a wire diameter that produces a minimum loss. However, for a moveable tuning loop, there is no single optimum wire diameter. Because the coupling loop is normally terminated in a relatively high impedance coax, losses in the coupling loop are mainly due to the eddy currents.

Equivalent circuit models for the tuning mechanism are shown in Fig. 1. The resonator (or pair of resonators) is modeled by a tank circuit comprising L_2 - C_2 , and R_2 is the intrinsic resistance of the resonator, such that the resonance frequency is $\omega_2 = 1/\sqrt{L_2 C_2}$ and quality factor is $Q_2 = \omega_2 L_2 / R_2$. The moveable loop is modeled as inductor L_1 with series resistance R_1 , with a quality factor $Q_1 = \omega_1 L_1 / R_1$. Tuning is adjusted by varying the coupling

coefficient k between 0 and 1. The probe resonance frequency ω and quality factor Q (ignoring eddy current losses) can be expressed as parametric functions of k .

$$\omega = \frac{\omega_2}{\sqrt{(1-k^2)}}, \text{ and} \quad (1)$$

$$Q = \frac{1}{\sqrt{(1-k^2)}} \frac{Q_2}{\left(1 + \frac{k^2}{\sqrt{(1-k^2)}} \frac{Q_2}{Q_1}\right)}. \quad (2)$$

II. Fixed Inductive Loop Tuning and Matching

We propose to improve the inductive tuning and matching mechanism by replacing the two moving loops by a single fixed inductive loop with a network of adjustable capacitors. Similar inductive tuning mechanisms have been used previously in MRI coils [12]–[16]. The proposed circuit for the tuning and matching network is shown in Fig. 2A.

A circuit analysis of the HTS resonator and tuning circuit is useful for better understanding the loss associated with a given frequency shift. Fig. 2B shows the circuit model of an HTS resonator inductively coupled to a resonant tuning circuit. In this figure, the HTS resonator is modeled as a tank circuit L_2 - C_2 with series resistance R_2 , such that the resonance frequency is $\omega_2 = 1/\sqrt{L_2 C_2}$ and quality factor is $Q_2 = \omega_2 L_2 / R_2$. The inductive loop is modeled as L_1 with series resistance R_1 , and C_1 is the tuning capacitor, such that the resonance frequency of the tuning circuit is $\omega_1 = 1/\sqrt{L_1 C_1}$ and quality factor is $Q_1 = \omega_1 L_1 / R_1$. Since the position of the inductive loop is held fixed with respect to the resonator, the coupling coefficient k remains unchanged. Instead, the two shared resonance modes of the coupled circuit can be tuned by varying the capacitor C_1 , which in turn adjusts ω_1 . We are only interested in the mode which is closest to ω_2 since it has most of its energy in the HTS resonator and it will provide better coupling to the NMR sample. Using simple loop equations, the resulting frequency ω and quality factor Q (ignoring eddy current losses) of this mode can be expressed as parametric functions of k and ω_1 as

$$\omega = \frac{\omega_2}{\sqrt{(1-k^2 X)}}, \text{ and} \quad (3)$$

$$Q = \frac{1}{\sqrt{(1-k^2 X)}} \frac{Q_2(1-k^2 X + k^2 X^2)}{\left(1 + \frac{k^2 Q_2}{\sqrt{(1-k^2 X)} Q_1} X_2\right)}, \quad (4)$$

where $X = \left(\frac{1}{1 - \frac{\omega_1^2}{\omega_2^2}} \right)$ is useful for simplification. The term ω appears on both sides (3) and hence this expression is only solved numerically.

Different cases arise depending on the value of X , that is, depending on the tuning circuit frequency relative to the resonance frequency of the system. First, it can be seen that when a shorted moveable tuning loop is used, $\omega_1 = 0$, so $X = 1$, and (3) and (4) reduce to (1) and (2) respectively. As ω_1 is increased toward ω_2 , the value of $X > 1$ and thus $\omega > \omega_2$ which can be thought of as a positive tuning shift of the HTS resonator. In the case when $\omega_1 > \omega_2$, the value of $X < 0$ and $\omega_1 < \omega_2$ which can be thought of as a negative tuning shift. Finally, it can be seen that as $\omega_1 \rightarrow \infty$ for an open loop, the value of $X = 0$ and produces no tuning shift irrespective of k .

Solving for (3) and (4) numerically, the effect of the various levels of coupling on the tuning and quality factor can be analyzed. Fig 3A shows the tuned frequency of the probe $f (= \omega/2\pi)$ as a function of the independent frequency of the tuning circuit $f_l (= \omega_l/2\pi)$ for different levels of coupling k for the case where $\omega_1 > \omega_2$. This case $\omega_1 > \omega_2$ results in negative frequency shifts. The frequency shift achieved increases as the resonance of the tuning loop approaches that of the resonator and as the tuning loop is more closely coupled to the resonator. Loose coupling also reduces the possible tuning range. The end of the $k=0.1$ curve represents the lower limit of the tuning range where the adjustable parameter ω_1 approaches ω_2 . The tuning range for larger values of k extends below the frequency range plotted in Fig. 3A.

Fig. 3B shows the reduction in probe quality factor Q/Q_2 over the tuning range. In all cases, using a normal metal loop to tune the probe reduces the Q . However, a strongly coupled resonant tuning loop retains more of the resonator Q over the tuning range than a weakly coupled loop. We can attribute the reduced Q to increased transport loss in the tuning circuit required to make up for the reduced coupling. For comparison, Q/Q_2 over for the case of a moving shorted loop as predicted by (1) and (2) is also shown. Even though the shorted loop actually produces positive tuning shifts, they are shown here as negative for comparison. We have also considered (not included in this abstract) the case when $\omega_1 < \omega_2$, but the loss associated with a given frequency shift is always worse than the case of a shorted moveable loop. It is clear from Fig. 3B that a fixed resonant loop can provide equal or better Q over the tuning range compared to a moving loop, but only if the coupling is sufficiently strong. However, the loss from eddy currents has been ignored in this analysis so far. As a normal-metal resonant inductive loop is positioned closer to the resonator in order to increase the coupling, eddy current losses in the wire will tend to increase and may offset any gains made in reducing the transport losses using a strong coupling.

III. Experimental Measurements

Tuning and matching of a single spiral HTS resonator using a fixed coupling loop with variable capacitors was performed in order to test the results obtained from the circuit analysis techniques.

A. Simulation

A combination of electromagnetic field simulation and circuit simulation was used to evaluate the range of tuning capacitors required. A 2% tuning range was desired. A coil resonating at 139 MHz was simulated in HyperLynx (Mentor Graphics) to obtain the reflection coefficient S_{11} at a port attached to strongly coupled loop. A second resonance of the spiral was observed at approximately 400 MHz. The S_{11} parameter data were imported into the circuit simulation program Eclipse (Arden Technologies, Inc.). The circuit shown in Figure 2A was evaluated in order to determine the resonance frequency as a function of the tuning capacitor. The resonance of the tuning loop crosses that of the second mode and produces a large shift in the second mode. Nevertheless, the desired fundamental mode varies smoothly and monotonically with the tuning adjustment. Based on this simulation, we chose variable capacitors varying from 1–16 pF for both the tuning and the matching capacitors.

B. Implementation

Tuning and matching using a fixed coupling loop and a capacitive network was tested in a cryogenic test station at 25 K. The cryogenic test station was suitably modified to accommodate tuning rods for variable capacitors. The tuning rods could be adjusted from outside the vacuum space to vary the capacitors inside the test station under vacuum operated at cryogenic temperatures. A printed circuit board was fabricated to accommodate variable capacitors for tuning and matching. Inductive coupling loops were fashioned out of normal-metal wire of both 26 AWG (to produce a coupling of approximately $k = 0.10$) and 20 AWG (for $k = 0.15$) for comparison. In each case, the loop was shaped to approximately match the shape and size of the HTS resonator, and placed touching the substrate on the back side. Variable capacitors from Sprague-Goodman (1–16 pF) were used to adjust tuning and matching.

The HTS resonator used was resonant at a frequency of 139.7 MHz with a Q of 3,800. The Q was then measured as it was tuned using variable capacitors. Fig. 4 shows the frequency shift of the coupled system as a function of the resonance frequency of the tuning circuit. Since the tuning circuit resonance is higher than that of the HTS resonators, the coupled system resonates at a lower frequency than the HTS resonators alone. The minimum frequency shift of approximately 0.3% is seen at the smallest value of the capacitor. As the value of the tuning capacitor is increased, the resonance frequency of the coupled system is shifted down. The tuning circuit with 20 AWG wire loop produces nearly 6% maximum tuning shift, whereas the tuning circuit with a 26 AWG wire loop produces nearly 3.5% maximum tuning shift. As expected, the loop with a larger k produces a larger range of tuning shifts. Fig. 5 shows the measured Q over the tuning range. Three-point Q s are measured using the -3dB points on the S_{11} curve. The measured Q values are normalized to the coil- Q in the absence of the tuning circuit. Using the 20 AWG wire loop, even at the lowest level of tuning (0.3%) the measured Q is only approximately 40% of the original Q of the resonator. Using the thinner 26 AWG wire loop, the Q is approximately 55% of the original Q of the resonator. Since there is very little current in the tuning circuit for this low level of tuning, the Q loss must be mainly attributed to the eddy currents in the loop. As expected, more eddy current loss was seen in the thicker wire loop. The loop position was

adjusted only for good coupling to the HTS resonator, and no consideration of the eddy current loss was made. At the maximum tuning shift, approximately 10% of the original Q of the HTS resonator is retained. Since the position of the loop is unchanged, all additional loss as the coil is tuned must be solely attributed to transport current losses in the loop. As expected, the thinner wire has more loss associated with transport current as the HTS coil is tuned.

Using a normal-metal fixed inductive loop and variable capacitors, an adequate tuning range was easily achieved. However, a significant loss in Q was observed over the tuning range. This observation is consistent with the circuit analysis predictions.

IV. Discussion

Use of a superconducting inductive loop will eliminate losses due to eddy currents, while simultaneously achieving tight coupling to the resonator. This loop may be patterned on the same substrate as the coil to maximize the coupling coefficient. In order to connect capacitors across the inductive loop, normal-metal wire bonds to the superconducting loop will be required. Therefore no improvement will be achieved in the Q_I of the tuning circuit itself. However, the Q of the overall resonance is improved by reducing the transport current in the loop.

It is very important to obtain a sufficient tuning range in HTS-based probes without a decrease in Q , since sensitivity of the probe is proportional to the square root of Q . However if tuning range is limited to optimize the Q , then the usefulness of the probe may be compromised.

Fixed loop tuning such that the resonator is tuned down also simplifies probe construction in another way. In the present construction of HTS probes, while the resonance frequency can be shifted up by trimming away regions of the resonator by laser ablation, there is no means to shift down the resonator frequency. Since the in-probe tuning also shifts up the frequency, a resonator which overshoots the target due to variability in patterning is rendered useless. In order to avoid overshooting, the HTS resonators are designed to have resonance frequency well below the target in the patterning step, and the frequency is brought up in small iterative steps by laser trimming. Since the new fixed loop tuning method allows bringing down the frequency without much reduction in Q , slight overshooting of the target frequency is not very critical. Thus, the resonator can be designed to resonate much closer to the target frequency.

V. Conclusion

A tuning and matching mechanism for HTS probes which uses a single fixed coupling loop along with a variable capacitor network is presented. Using circuit analysis, the different configurations are considered in terms of tuning circuit frequency and level of coupling. We predict that a strongly-coupled superconducting inductive loop with an adjustable capacitor network will not only improve the probe's mechanical reliability, but also provide improvements in sensitivity.

Acknowledgments

This work was supported in part by the National Institutes of Health under grant NIH-NIBIB R01EB009772. A portion of this work was performed at the National High Magnetic Field Laboratory, which is supported by National Science Foundation Cooperative Agreement No. DMR-1157490 and the State of Florida.

References

1. Styles P, et al. A high-resolution NMR probe in which the coil and preamplifier are cooled with liquid helium. *J Magn Reson*, 1969. 1984; (3):397–404.
2. Anderson WA, et al. High-sensitivity NMR spectroscopy probe using superconductive coils. *Bull Magn Reson*. 1995; 17(1):98–102.
3. Anlage SM. High temperature superconducting radio frequency coils for NMR spectroscopy and magnetic resonance imaging. Apr.2000 ArXivcond-Mat0004346.
4. Ramaswamy, V., Hooker, JW., Withers, RS., Nast, RE., Edison, AS., Brey, WW. eMagRes. Vol. 2. John Wiley & Sons, Ltd; 2013. Microsample Cryogenic Probes: Technology and Applications; p. 215-228.
5. Froncisz W, Jesmanowicz A, Hyde JS. Inductive (flux linkage) coupling to local coils in magnetic resonance imaging and spectroscopy. *J Magn Reson* 1969. Jan; 1986 66(1):135–143.
6. Bowyer P, Finnigan J, Marsden B, Taber B, Zens A. Using magnetic coupling to implement ^1H , ^{19}F , ^{13}C experiments in routine high resolution NMR probes. *J Magn Reson*. Dec.2015 261:190–198. [PubMed: 26619074]
7. Koshita K, et al. Development of HTS Pickup Coils for 700-MHz NMR: Resonance Frequency Tuning Using a Sapphire Plate. *IEEE Trans Appl Supercond*. Apr; 2016 26(3):1–4.
8. Brey WW, Edison AS, Nast RE, Rocca JR, Saha S, Withers RS. Design, construction, and validation of a 1-mm tripleresonance high-temperature-superconducting probe for NMR. *J Magn Reson*. Apr; 2006 179(2):290–293. [PubMed: 16423543]
9. Ramaswamy V, Hooker JW, Withers RS, Nast RE, Brey WW, Edison AS. Development of a ^{13}C -optimized 1.5-mm high temperature superconducting NMR probe. *J Magn Reson*. Oct.2013 235:58–65. [PubMed: 23969086]
10. Yamada T, et al. Electromagnetic Evaluation of HTS RF Coils for Nuclear Magnetic Resonance. *IEEE Trans Appl Supercond*. Jun; 2015 25(3):1–4.
11. Ramaswamy V, Hooker J, Withers R, Nast R, Edison A, Brey W. Development of a ^1H - ^{13}C Dual-Optimized NMR Probe Based on Double-Tuned High Temperature Superconducting Resonators. *IEEE Trans Appl Supercond*. Apr; 2016 26(3):1–5.
12. Schnall MD, Barlow C, Subramanian VH, Leigh JS Jr. Wireless implanted magnetic resonance probes for in vivo NMR. *J Magn Reson* 1969. Jun; 1986 68(1):161–167.
13. Silver X, et al. In vivo ^1H magnetic resonance imaging and spectroscopy of the rat spinal cord using an inductively-coupled chronically implanted RF coil. *Magn Reson Med*. Dec; 2001 46(6): 1216–1222. [PubMed: 11746589]
14. Ginefri J-C, et al. Implanted, inductively-coupled, radiofrequency coils fabricated on flexible polymeric material: Application to in vivo rat brain MRI at 7 T. *J Magn Reson*. Nov.2012 224:61–70. [PubMed: 23041797]
15. Decorps M, Blondet P, Reutenauer H, Albrand JP, Remy C. An inductively coupled, series-tuned NMR probe. *J Magn Reson* 1969. Oct; 1985 65(1):100–109.
16. Raad A, Darrasse L. Optimization of NMR receiver bandwidth by inductive coupling. *Magn Reson Imaging*. 1992; 10(1):55–65. [PubMed: 1545682]

Fig 1. Equivalent circuit model for an HTS resonator L_2 - C_2 tuned by a shorted moveable tuning loop L_1 .

Fig 2.

(A) An inductive loop with a L-network of adjustable capacitors used to tune and match to the HTS resonator. (B) Equivalent circuit model for tuning circuit shows HTS resonator L_2 - C_2 tuned by resonant inductive loop L_1 - C_1 .

Fig 3.

(A) Prediction of probe resonance (f) as a function of the tuning circuit resonance (f_t) for various levels of coupling. (B) Normalized Q over the tuning range, when an HTS resonator ($Q_r=10,000$) is tuned with a fixed tuning circuit ($Q_f=200$), for various levels of coupling k . The normalized Q when the resonator is tuned by moving a shorted tuning loop is shown for comparison.

Fig 4.

Plot of tuning achieved with a fixed inductive loop and variable capacitor. Two cases shown above are for when the loop was fashioned out of either (A) 26 AWG or (B) 20 AWG loop wire. The tuned resonance (f) is shown as a function of the isolated resonance (f_1) of the tuning circuit. The 26 AWG wire loop reaches the minimum tuned frequency at 134.5 MHz. As seen, a thicker wire loop produces a greater coupling to the HTS resonator, and achieves a larger tuning range.

Fig 5.

Plot of the Q over the tuning range as an HTS resonator is tuned using a fixed inductive coupling loop and variable capacitors. The measured Q is normalized to the original Q of the resonator (Q_2).