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High-field NMR using resistive and hybrid magnets

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Abstract

Resistive and resistive-superconducting hybrid magnets can generate dc magnetic fields much higher than conventional superconducting NMR magnets but the field spatial homogeneity and temporal stability are usually not sufficient for high-resolution NMR experiments. Hardware and technique development addressing these issues are presented for high-resolution NMR at magnetic fields up to 40 T. Passive ferromagnetic shimming and magic-angle spinning are used effectively to reduce the broadening from inhomogeneous magnetic field. A phase correction technique based on simultaneous heteronuclear detection is developed to compensate magnetic field fluctuations to achieve high spectral resolution.

Keywords: Resistive; Hybrid magnet; High field; High resolution; MAS; Ferromagnetic shim; Field homogeneity; Stability; HENPEC; NHMFL

1. Introduction

NMR spectral resolution and sensitivity benefit from increasing magnetic field strength. High magnetic field is also important for a growing number of line-narrowing experiments both those that improve with increasing fields such as the reduction of second-order broadening in solid state NMR of half integer spins [1,2] and those that require a certain high-field strength for optimal TROSY (*t*ransverse *r*elaxation *o*ptimize *s*pectroscop*y*) effect [3]. The conventional Nb-based superconducting NMR technology is approaching its limit about 22 T [4] whereas resistive and resistive-superconducting hybrid magnets can achieve a much higher magnetic field strength. These powered magnets have been successfully used for low resolution studies [5]. A number of explorative high-resolution NMR experiments have been demonstrated using 25 T and 33 T resistive [6–9], and 45 T hybrid [1,10] magnets including several experiments that are insensitive to magnetic field homogeneity and stability [11,12]. NMR experiments at even higher field up to 70 T have been performed using pulsed magnets [13,14]. Ideally the homogeneity and stability should be improved or compensated such that a wider range of NMR experiments can be performed at very high fields using these powered magnets.

Magnetic field shimming [15] has been proved to be effective for improving field homogeneity of superconducting NMR magnets and it can be, in principle, adapted to resistive magnets. Nevertheless, strong shimming gradients are required for the less homogeneous resistive magnet and dissipating the large amounts of heating takes additional bore space. Ferromagnetic shimming is an alternative approach for correcting the large field gradient found in the powered magnets and requires little additional bore space [9,16]. In addition, residual line broadening can be reduced by sample spinning particularly at the magic-angle at which most solid state NMR achieves high spectral resolution. For solution NMR, sample spinning at the magic-angle eliminates the magnetic susceptibility broadening from all substances within the spinning rotor. The other important field quality factor, temporal stability, poses a more challenging task for powered magnets in order to achieve spectral resolution comparable to

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superconducting NMR magnets. The magnetic field of powered magnets consists of fluctuating components with a wide range of time-constant from milliseconds to seconds. Field-frequency lock [17] compensates only the slow field drifts up to at most the sub-second range. Several methods have been proposed to reduce more rapid fluctuations. Inductive field regulation (flux stabilization) [18], originally developed for permanent and iron core magnets has more recently been used for high-field powered magnets. A cooled, highly conductive aluminum cylinder has been successfully employed to shield a room temperature sample from field fluctuations to improve the efficiency of signal averaging in relaxation experiments [19]. Other methods based on NMR data collection and processing such as reference deconvolution [9,20–24] have also been introduced to compensate for field inhomogeneity and fluctuation. In this paper, we present the efforts of improving NMR spectral resolution using powered magnets at the National High Magnetic Field Laboratory (NHMFL) including the development of a field compensating technique that simultaneously acquires the solvent signal. The combination of ferromagnetic shimming, high-resolution magic-angle spinning (HRMAS), and the *he*tero-*n*uclear *p*has*e*-*c*orrection (HENPEC) [25] method achieves better than 40 ppb line width using the 25 T Keck resistive magnet at the NHMFL.

2. Experimental

This development effort towards high-field and high-resolution NMR has been mostly carried out with a 25 T–52 mm resistive magnet whose development was partially supported by the Keck foundation. The Keck is an electromagnet driven by 38.8 kA DC current through three stacked Bitter coils. A 31.8 mm split of the middle coil generates a Z^2 field gradient that partially cancels the Z^2 magnetic field profile of a finite solenoid for improving the field homogeneity. The magnet bore has 52 mm inner diameter accessible from both the top and the bottom of the magnet. Voltage ripples from the power supplies, 7000 L/min flow of cooling water running through the magnet for dissipating the 19 MW generated heat and small variation of cooling water temperature (−20 ppm/°C) [24] contribute to the overall magnetic field fluctuations. More technical information on the Keck magnet can be found in references [26–28]. The 45 T hybrid magnet holds the world record for the highest dc magnetic field. The magnet consists of an outer superconducting magnet which generates 11.4 T magnetic field and a resistive inserts capable of generating 33.6 T for a maximum combined field of 45 T. The magnet was operated at 40 T for the NMR experiment, corresponding to 1.7 GHz proton Larmor frequency, to prolong the experiment time using less power and cooling water. The field-stability is similar to that of the Keck magnet which uses the same power supply and cooling water system. However, no additional effort such as the coil split was included in its design and construction, therefore the field homogeneity is much worse than that of the Keck magnet at about 1000 ppm over 1 cm DSV.

All NMR experiments were performed using a Tecmag Discovery console with dual-receivers capable of simultaneous acquisition of two different nuclei. A double-resonance HRMAS probe using a 4 mm Bruker MAS stator and susceptibility matched solenoidal rf-coil was built at the NHMFL for experiments on the 25 T Keck magnet. The sample volume was restricted to 5 mm in length by placing two Teflon inserts at the two ends of the 4mm Bruker rotor. The proton and deuteron Larmor frequencies are 1064 MHz and 163.4 MHz, respective, at 25 T. The 31 mm resistive bore of the hybrid magnet is accessible only from the top. Therefore, a single-channel 2 mm MAS probe capable of spinning with the probe loaded upside down from the top was developed for this study. As a safety precaution, operators are not permitted to physically access the bore space above the magnet while the hybrid's resistive insert is at field. A mechanical shaft for hanging the probe was built such that the probe position and orientation can be adjusted remotely.

3. Magnetic field spatial homogeneity

Fig. 1a shows the field map of the Keck magnet soon after its construction. The field map plots the resonance

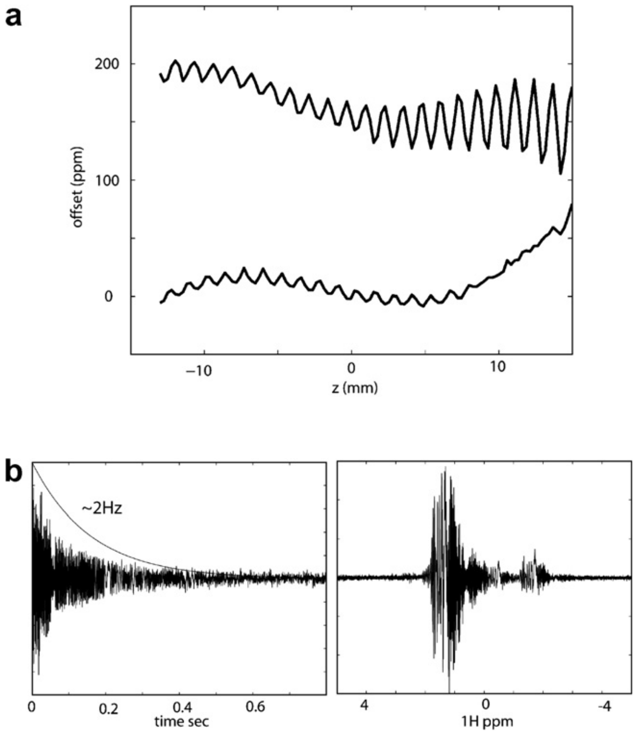

Fig. 1. (a) Magnetic field map before (top) and after (bottom) ferromagnetic shim. (b) ^{1}H free-induction-decay (left) and spectrum (right) of 2% $CHCl_3/CDCl_3$ without any compensation for the fluctuating field. An exponential decay corresponds to 2 Hz line width is plotted along the ^{1}H FID.

frequencies of D_2O in a small spherical capsule (2 mm OD) placed 5 mm off the bore axis as the sample was stepped in a spiral manner across the field center. The map shows a significant amount of first-order transverse inhomogeneity. With the shim coefficients obtained from this map, a ferromagnetic shimming system was designed and constructed by Resonance Research Inc. (Billerica, MA). Numerous pieces of ferromagnetic material were embedded in the glass-epoxy of the outer surface of the bore tube in a distribution calculated from the required shim coefficients. Unfortunately, during the engineering process of the shim system, the outer C-coil of the Keck magnet developed a fault. It was necessary to disassemble, clean and reassemble the coil which introduced significant amount change to the field inhomogeneity. In order to obtain the most homogeneous field, the bore tube was reoriented to be 28 degrees counterclockwise from its original position. The resulting field map in Fig. 1a shows a factor of 4 improvement on field homogeneity from approximately 48 ppm down to 12 ppm over 1 cm DSV.

Fig. 1b shows the ^1H free-induction-decay (FID) of 2% $CHCl_3/CDCl_3$ using HRMAS following installation of the ferromagnetic shim. The long FID was obtained by adjusting the probe's vertical position and horizontal orientation interactively. The length of the FID indicates an equivalent line width of about 6 Hz, which approaches that obtained in shimmed superconducting magnets. At this probe position, the field gradient vector is nearly perpendicular to the spinning axis so that fast sample spinning efficiently averages the dominant first-order inhomogeneity of the Keck magnet. However, the time-domain signal is frequency modulated by the fluctuating magnetic field. A straightforward Fourier transformation yields a spurious spectrum spreading about 4 ppm, more that 100 times as wide as anticipated from the time-domain decay. Thus with ferromagnetic shim and HRMAS, the temporal stability of the Keck magnet becomes the limiting factor for achieving high spectral resolution.

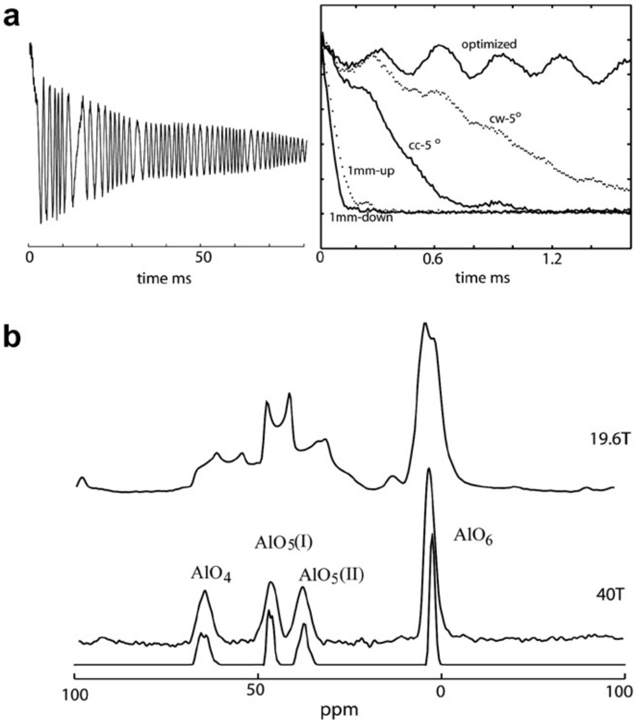

Fig. 2. (a) D_2O free-induction-decay obtained using a 2 mm MAS probe at 40 T hybrid magnet. The signal on the left was obtained by adjusting both the vertical position and probe orientation. The expansion of the first 1.6 ms on the right shows the shortening of the FID with slightly off from the optimized probe position and orientation. (b) ^{27}Al MAS spectrum of $9Al_2O_3{\cdot}2B_2O_3$ obtained with a single scan using hybrid magnet at 40 T in comparisons with a simulated spectrum and a spectrum obtained using a 19.6 T superconducting magnet.

The hybrid magnet is much less homogeneous than the Keck magnet as it was not designed for high-resolution NMR applications. Nevertheless, using a small 2 mm MAS rotor and carefully adjusting the probe position and orientation, it is still possible to obtain a deuterium NMR signal lasting longer than 50 ms (Fig. 2a). As in the Keck magnet, the magnetic inhomogeneity of the hybrid magnet consists of mostly a first-order gradient which can be spun out if spinning axis is adjusted to be perpendicular to the gradient vector. Fig. 2 shows the importance of carefully adjusting the probe position, as only a slight deviation from the optimized position shortens the FID dramatically. This first attempt suggests that this ultra high-field magnet can be useful for high-resolution NMR spectroscopy if the signal is strong enough for to interactively adjust the probe position and a few ppm line broadening from field spatial homogeneity and temporal stability can be tolerated. One of such applications is the reduction of second-order quadrupolar effect of half integer quadrupolar nuclei such as ^{27}Al. Fig. 2b shows a comparison of MAS spectra of $9Al_2O_3{\cdot}2B_2O_3$ acquired at 19.6 T and 40 T. The four alumina sites of the $9Al_2O_3{\cdot}2B_2O_3$ model compound have large quadrupolar couplings and the resulting second-order broadening in the order of tens of ppm makes this sample an ideal case to demonstrate high-field line narrowing [1,29]. The four sites which give rise to overlapped peaks in the 19.6 T superconducting magnet become completely resolved at 40 T. Fig. 2 also shows a simulated 40 T spectrum using the known quadrupolar coupling and chemical shift parameters of this sample. The simulated spectrum shows that even more resolution enhancement might be achieved if the magnetic field of the hybrid magnet was improved to be more stable and homogeneous. For disordered systems, an ultra high-field changes the scale of second-order quadrupolar and chemical shift interactions and can help to extract chemical shift and quadrupolar coupling parameter distribution using one-dimensional MAS spectral acquired at variable field strength. The 1D approach is more quantitative and compliments with popular two-dimensional methods such as multiple-quantum magic-angle spinning (MQMAS) [30] and satellite-transition magic-angle spinning (STMAS) [31] experiments.

4. Magnetic field temporal stability and HENPEC

With the improvement of magnetic field spatial homogeneity of the Keck magnet by ferromagnetic shimming and HRMAS, the field temporal stability becomes the major limiting factor to NMR spectral resolution. Field fluctuations near the time scale of the NMR signal yields a spurious spectrum (Fig. 1b). The random fluctuations and slow field drift also destroy the phase coherence of sequentially acquired signals in multidimensional NMR experiments. Ideally, the problem should be solved or reduced with flux stabilization, field-frequency lock and other techniques. To implement these techniques for resistive and hybrid magnet require an extensive engineering effort. Here we describe an alternative approach based on simultaneous acquisition of a reference signal from the solvent deuteron. The effect of field fluctuations on the signal phase is corrected by data processing following the acquisition similar to the reference deconvolution method [20–24].

The method relies on the assumption that fluctuating field affects the sample uniformly, $B(\vec{r},t) = g(\vec{r})b(t)$. The temporal fluctuation $b(t)$ modulates the NMR signal $S(t)$ only in the form of a phase $\varphi(t)$ while the inhomogeneous spatial field distribution $g(\vec{r})$ causes a signal decay $G(t)$

$$\begin{aligned} S(t) &= S'(t)\cdot G(t)\cdot \mathrm{e}^{-i\varphi(t)} \\ G(t) &= \int_V \mathrm{e}^{-i\gamma g(\vec{r})t}\mathrm{d}\vec{r} \\ \varphi(t) &= \gamma\int_0^t b(t')\mathrm{d}t' \end{aligned} \tag{1}$$

Here $S'(t)$ is the NMR signal assuming a homogeneous and stable magnetic field. It is real for the solvent with a single peak. An expansion of $\mathrm{e}^{-i\gamma g(\vec{r})t}$ in $G(t)$ shows that all even-order terms are real and the first-order expansion term vanishes as $\int_V g(r)\mathrm{d}\vec{r} = 0$. Therefore, only the odd-terms staring from the third-order contributes to the imaginary part. These terms correspond to the non-symmetric lineshape of the solvent signal. For a well shimmed sample, $G(t)$ can be assumed real. Thus, the phase of the deuterium signal reflects directly the fluctuating magnetic field. Fig. 3 shows such measurements of magnetic field fluctuations using the solvent signal. The main component is the 60 Hz AC frequency. High-order harmonics up to 720 Hz are still significant in the Fourier analysis. The high frequency components mostly come from the power supply rectifiers and have less effect on NMR spectra than the low frequency components. It is found that signal modulation from sample spinning has almost no effect on the field fluctuation measurement. The fast (~4 kHz) MAS in the presence of inhomogeneous fields modulates the NMR signal and does cause spinning sidebands. Fortunately, we found that the spatial homogeneity of the Keck magnet does not change over time and the modulation changes only the amplitude but not the phase of the signal.

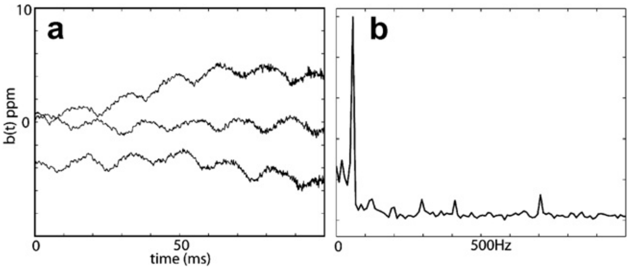

Fig. 3. (a) Magnetic field fluctuations extracted from D_2O solvent signal and (b) a Fourier analysis showing the frequencies of field fluctuations.

The main idea of *he*tero *n*uclear *p*hase *c*orrection (HENPEC) is to determine from the solvent signal the phase compensation $\varphi(t)$ that has distorted the NMR signal. The signal is then corrected by adjusting the phase of each point in the FID by $-\varphi(t)$, scaled by the gyromagnetic ratios of the two nuclei, to remove the effect of the fluctuating magnetic field.

A practical issue arises in the extraction of $\varphi(t)$. The signal phase is an integration of the magnetic field over time. In the cases of DC offsets, slow drift or large fluctuation, the $\varphi(t)$ increases with time and it can exceed the $(-\pi,\pi)$ range. The numerical value of $\varphi(t)$ always folds back to the $(-\pi,\pi)$ range causing a phase continuity problem to the practical implementation of HENPEC. This problem can be solved by reconstructing the phase manually [24]. Here we take an approach by extracting phase increments between adjacent data points. The phase increment is always small due to the short dwell time therefore avoids the phase folding problem.

Following is the procedure of implementing HENPEC with phase increment calculation.

(i) The ^{1}H signal of the sample and the ^{2}H signal of the solvent (it must be a single peak) are acquired simultaneously using the same dwell-clock. The spectral window for both nuclei is sufficiently wide to cover all resonances and modulation sidebands. The ^{2}H solvent is placed roughly at the center. It is important to note that individual scans must be stored separately immediately after the acquisition. Signal average can take place after the HENPEC.

(ii) To reduce noise in the ^{2}H signal, a low-pass frequency filter can be applied. This can be carried out in the frequency-domain using a Fourier transformation. The filter cutoff should be set to preserve all the field fluctuations that are to be corrected, can be simply zero the points outside the filter window. An inverse Fourier transformation of the filtered spectrum leads to a deuterium signal with reduced noise.

(iii) A phase incrementing factor $p_i = phase(d_i^* \cdot d_{i+1})$ is extracted from the deuterium signal d_i. p_i can be set to zero for the first few corrupted points in the cases of receiver dead time. The fluctuating magnetic field is related to this phase increment by $b_i(t) = p_i \cdot sw/(2\pi\upsilon_0)$ where sw and υ_0 are the spectral width and deuterium Larmor frequency, respectively.

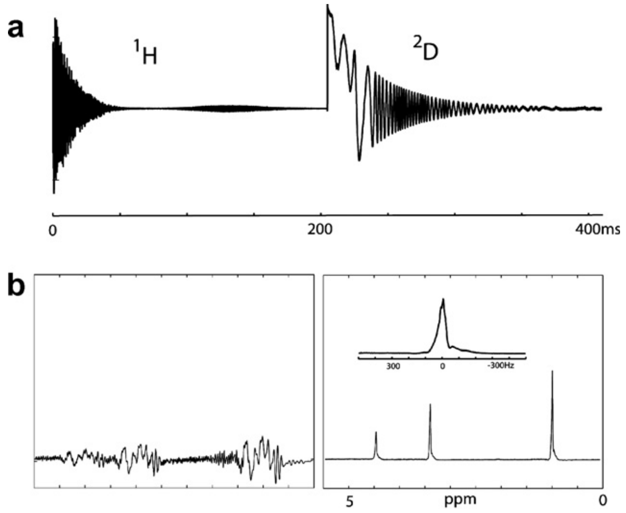

Fig. 4. (a) Simultaneously acquired ethanol proton signal and D_2O solvent signal; (b) proton spectra without (left) and with (right) HENPEC phase correction. The inset shows an expansion of the peak near 1 ppm with FWHH about 40 ppb.

(iv) For the proton signal, a phase function $\psi_{i+1} = \psi_i + p_i(\gamma_H/\gamma_D)$, $\psi_1 = 0$ is calculated and the signal phase is compensated by $h_i = h_i * e^{-i\psi_i}$.
(v) A Fourier transformation of the phase-corrected proton signal yields the compensated frequency-domain spectrum.

Fig. 4 shows the simultaneously acquired 10% ethanol/ D_2O proton signal and the deuterium solvent signal. The spectra without and with the phase correction clearly shows that the phase modulation from the fluctuating magnetic field is mostly compensated by HENPEC. The resulting line width (full width at half height) is about 40 ppb. A significant portion of the line width comes from the homonuclear *J*-coupling among the neighboring protons. The proton FID actually shows a sign of *J*-modulation. There are some features in the proton lineshape that could be the smeared *J*-multiplets by the remaining inhomogeneous broadening. The limits on spectral resolution that can be obtained by HENPEC remain to be investigated.

5. Conclusions

It has been shown that NMR spectral resolution of resistive and hybrid magnets can be improved with a combination of ferromagnetic shimming, magic-angle spinning and the HENPEC method. The ultra high-field magnets offer a unique opportunity for field-dependent studies and some line-narrowing experiments. In optimal cases, the achieved line width is approaching to that of superconductive magnet. The resolution and stability is expected to improve further with active shimming and damping the field fluctuation with magnetic flux stabilization. These efforts are currently underway at the NHMFL with the construction of a new generation of series-connected hybrid magnets. The field homogeneity and stability are expected be much improved for high-resolution NMR at fields much higher than conventional superconductive magnets.

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