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High-Resolution NMR Above 1 GHz in a Resistive Magnet

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Abstract

Resistive magnets generate magnetic fields much higher than superconducting NMR magnets and offer unique opportunities for studying NMR phenomena. However, the field homogeneity and temporal field fluctuations are often insufficient for high spectral resolution. We report here the recent progress of our collaborative effort to use high-resolution magic-angle spinning (HRMAS) and feedback compensation to achieve high resolution NMR above 1 GHz using resistive magnets at the NHMFL.

Experimental

All measurements were performed in the Keck 25 T, 52 mm bore resistive magnet at the NHMFL. Figure 1 shows a double resonance HRMAS probe built from a 4 mm Bruker HRMAS assembly and a magnetic flux pick-up coil (1600 turns) placed on the top of the MAS stator.

Feedback Compensation

Temporal fluctuations in the magnetic field were detected by the pickup coil and reduced by means of the feedback system in Figure 2 that includes a custom-programmed digital signal processor. The correction field $B_c(t)$, ideally cancels $B_f(t)$, is produced by means of a low inductance compensation coil wound on a tube outside of the probe. The compensator transfer function contains a phase lead–lag component to stabilize the closed-loop system and a series of complex conjugate pole-zero pairs to attenuate harmonic field fluctuations caused by power supply ripple. Figure 3 shows the spectra of the magnetic fluctuations observed by the pick-up coil with and without feedback. A 55 to 35 dB improvement is achieved at frequencies from 10 Hz to 200 Hz due to the phase-lead-lag component. The complex conjugate pole-zero pairs provide further disturbance rejection at the largest power supply harmonics located at 60 Hz, 120 Hz, 180 Hz, and 720 Hz.

NMR Measurements

Figure 4 shows the results of HRMAS experiments with and without feedback compensation. The 50% H_2O, 50% D_2O sample was spun at 3.5 kHz. Without feedback compensation, the magnetic field of the Keck resistive magnet has ~4 ppm peak-to-peak temporal fluctuations at 25 T within a time scale of <100 ms. Such fluctuations yields spurious spectra despite that the magnetic field homogeneity is minimized by HRMAS and probe position optimization (no active shim was used). The spectrum obtained using feedback compensation shows a sharp resonance and two spinning sidebands with a FWHM of about 70 ppb. This result is nearly comparable to the line width obtained using the HEteroNuclear PhasE Corrected (HENPEC) method shown in figure 5.

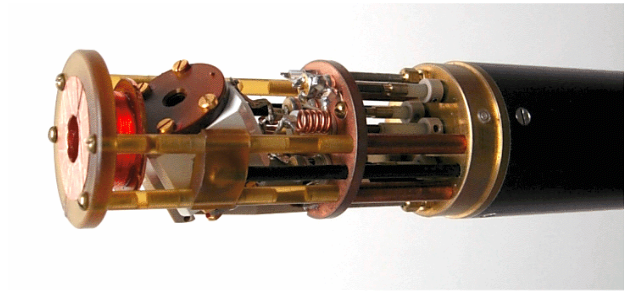

Figure 1: HRMAS Probe.

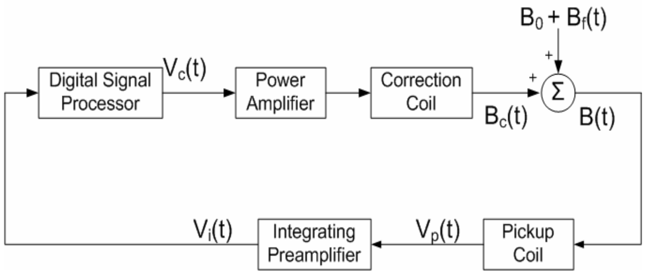

Figure 2: Block diagram of the inductive feedback system.

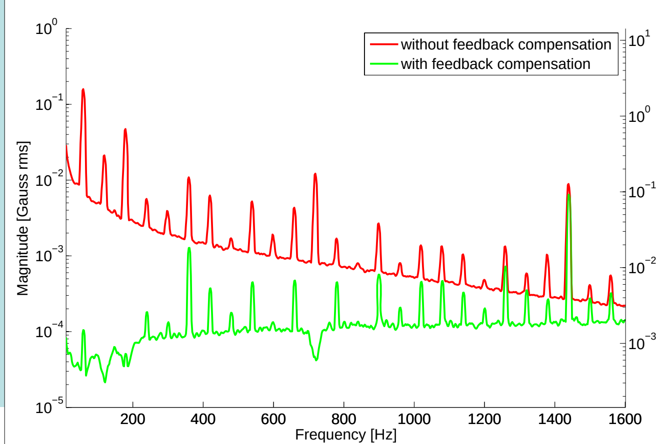

Figure 3. Temporal magnetic field fluctuation spectra for the Keck magnet at 7 T.

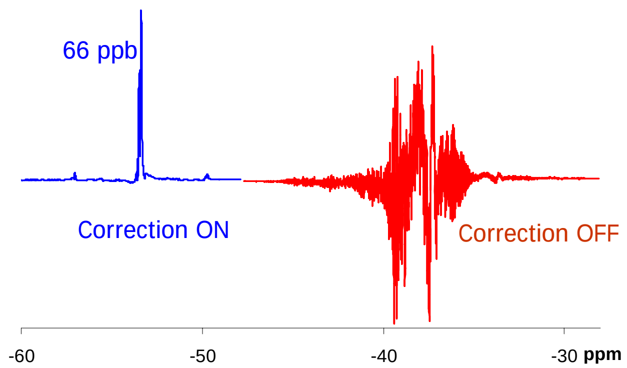

Figure 4. Water peak spectra with and without feedback compensation.

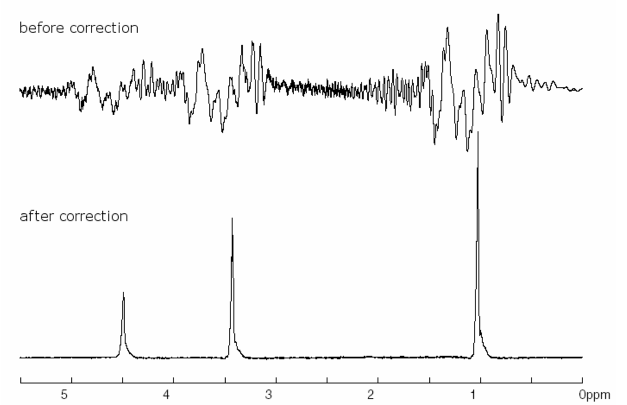

Figure 5. ^{1}H HRMAS spectra of ethanol at 1.06 GHz with and without before HENPEC correction.

References

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