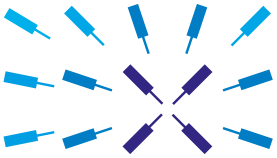
Application note



Zurich Instruments

Magnetic Resonance Force Microscopy

Products: HF2LI, UHFLI Lock-in Amplifiers
Options: MF, PID/PLL, MOD

Release date: April 2015

Introduction to MRFM

Magnetic Resonance Force Microscopy (MRFM) is a recent imaging method at the intersection between Magnetic Resonance Imaging (MRI) and Scanning Probe Microscopy (SPM). Using an SPM approach, it aims at surpassing MRI in terms of spatial resolution and sensitivity, but it still faces some of the challenges of MRI in terms of signal extraction [1].
Today MRI is a mature and powerful technique routinely used in biochemistry, pharmacology and medicine. MRI utilises a strong magnetic field B to force the nuclear magnetic moments μ of the sample to precess around the field direction according to:

$$\frac{d\mu}{dt} = \gamma\mu \times B,$$

where γ is the magnetic moment's gyromagnetic ratio. The precession frequency of the magnetic moment is called Larmor frequency ω_L and is proportional to the B field and to the gyromagnetic ratio, $\omega_L = \gamma B$. For typical experiments ω_L lies in the radio-frequency (RF) range.
Applying an oscillating magnetic field with a frequency ω_L perpendicular to the external magnetic field B induces a resonance in the magnetic moment. After removing this RF field, the free precession of the magnetic moments around the direction of the static field generates a time-dependent magnetic signal that can be measured by pick-up coils and analysed by a lock-in amplifier.
Since the gyromagnetic ratio is different for the magnetic moments of electrons, protons or isotopes with nonzero spins, the application of a uniform magnetic field to a sample composed of different atomic species results in a spectrum with distinct resonance frequencies. Conversely, if the sample is placed in a non-spatially-uniform magnetic field, identical isotopes will have different resonance frequencies depending on the local field strength. Therefore, MRI can be both spatially and chemically sensitive.
Despite the success of conventional MRI, the technique is limited in sensitivity because the macroscopic pick-up coils used to detect the spin resonance signals need to be at least a few centimetres from the sample under test. Therefore, a large number of spins, typically more than 10^{12}, are required to generate a detectable signal which, in turn, limits its spatial resolution [1].

In the early 1990s, this limitation motivated the development of MRFM, in which the pick-up coils are replaced by nanometer-scale force sensors [2]; it uses the MRI principles but employs an SPM-like detection. Among other advantages, the size of these sensors allows them to be much closer to the sample, immediately giving an improvement in sensitivity and resolution.
Despite this higher sensitivity, the magnetic force generated by one nuclear spin is extremely small and even state-of-the-art force sensors require lock-in techniques and long integration times to extract few-spins signals from the background noise. For this reason, a high resolution 3D MRFM image, in which each volumetric pixel contains a small number of spins, currently require several days or even weeks to be acquired. This issue is compounded if more than one isotopic species in a specimen need to be imaged sequentially.
In order to bring MRFM's potential to fruition, its imaging time limitation also needs to be a major development focus. This can be accomplished by increasing the mechanical resonator sensitivity or by employing more advanced signal acquisition schemes. For the latter, Zurich Instruments Lock-in Amplifiers can offer important benefits.

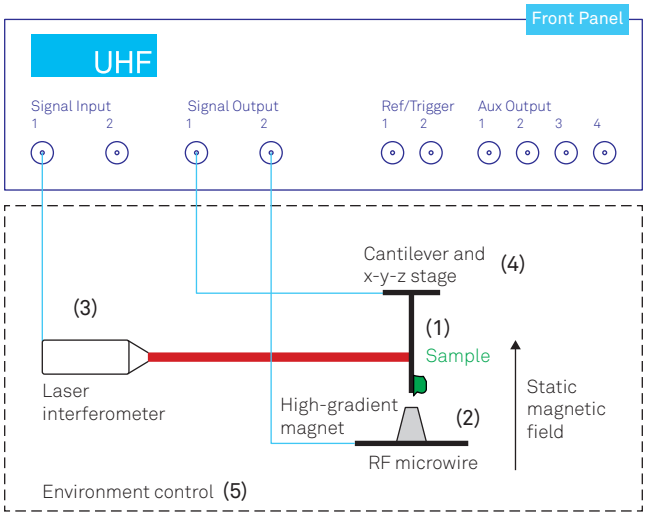


Figure 1. Schematic of basic wiring of a UHFLI within an MRFM setup. Signal Output 1 drives the cantilever and provides active feedback, e.g. for Q control, Signal Output 2 is used to generate the RF signal. Signal Input 1 reads the the signal from the interferometer.

Examples of MRFM measurements

Since its early days, MRFM has produced some very impressive results. Let's consider three of the most recent ones that show the technique's potential.

In 2009, Degen and coworkers [3] demonstrated the potential of the technique for 3D imaging of biological samples, by performing the first 3D reconstruction of a tobacco mosaic virus specimen with resolution in the nm range. The detection was performed on hydrogen nuclei and required an integration time of 1 minute per point, for a total image acquisition time of approximately five days.

In the same year, Mamin and coworkers [4] imaged a carbon nanotube attached to a cantilever. In this case, while the resolution was still in the nm range, the experimenters performed an imaging measurement sensitive to three different isotope species: ^{1}H, ^{31}P and ^{13}C.

More recently, in 2013, Nichol and coworkers demonstrated for the first time a pulsed Fourier-transform approach to MRFM which promises to enhance the technique's sensitivity and reduce acquisition time [5]. Although the authors only imaged a 2D slice, the technique can be readily extended to 3D reconstruction.

MRFM setup and signal processing

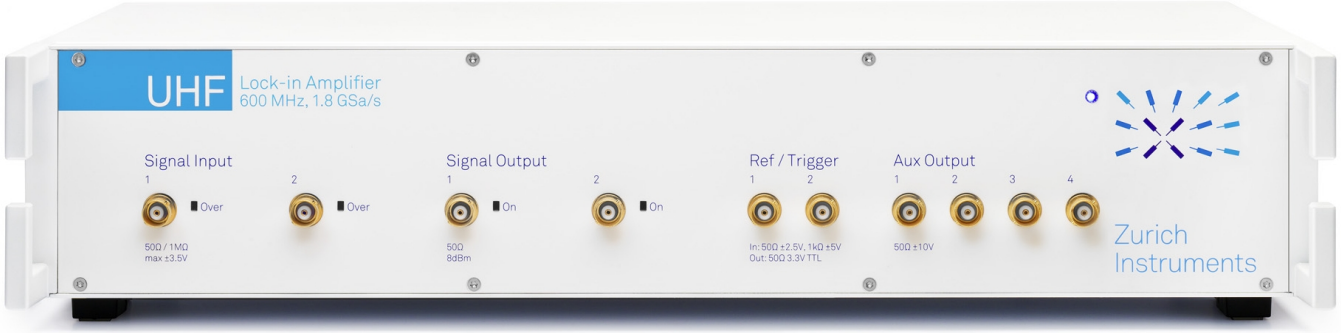
A typical MRFM consists of various components, with signal processing electronics playing a key role in the overall system performance.

MRFM setup components

The main components of an MRFM system, displayed in Figure 1, can be grouped in five major subsystems:

1. force detector and sample holder: usually a highly compliant cantilever with a resonance frequency of a few kHz, a spring constant of 100 μN m^{-1} or less and a quality factor Q of 10^4-10^6;
2. magnetic field generation: the large uniform DC component is generated with a superconducting magnet, to which a high field gradient of a permanent magnet is added; finally, a micro-coil or microwire is used to generate the RF field;
3. readout: a laser interferometer or beam deflection setup in combination with a lock-in amplifier;
4. sample positioning: an x-y-z high precision stage;
5. environment control: to isolate the measurement head from the environment through high vacuum and cryogenic temperatures, sometimes down to the mK range.

The large uniform magnetic field produced by the superconducting magnet broadly defines the spin resonance frequencies, while the small permanent magnet high-gradient field is superimposed to the larger one and generates the spatial non-uniformity necessary for 3D imaging: only spins located within a thin slice of space around the magnet are resonant with the RF field at a specific frequency. A crucial element for the measurement success is the precise phase synchronisation between the RF field and the cantilever motion. The mechanical resonator provides the means to sense the tiny forces – as small as a few attonewton – produced by the spin resonance. Resonators currently used for this purpose range from single crystal silicon cantilevers to nano wires; other materials such as single crystal diamond are also being investigated.



Advanced spin excitation and signal processing techniques

Many approaches are currently being investigated to enhance the sensitivity and thus reduce the image acquisition time. Most of these are based on multiplexing the measurements in the frequency domain. One of such techniques was demonstrated by Oosterkamp and coworkers [6]. The idea is to concurrently excite spins possessing different resonant frequencies – either because they belong to different isotopic species or because they are located at different positions inside the magnetic gradient – and read out their signals simultaneously, reducing the acquisition time by a factor equal to the number of frequencies considered. Each of these populations of spins are excited using a method called Adiabatic Rapid Passage (ARP): the RF field is frequency modulated using sweeps centred around the spin resonance. Every two full sweeps, the spin population goes through a full inversion, which is detected by the mechanical resonator.

In order to implement Oosterkamp's protocol, the RF field needs to be composed of several of these frequency modulated signals, each addressing a specific spin population. Each signal is characterised by a centre frequency equal to the population Larmor frequency and by a modulation repetition rate. The force sensor picks up the spins response at half the modulation repetition rate, and this frequency needs to fall within the sensor's bandwidth. For this reason, the bandwidth of the mechanical resonator needs to be sufficiently broad. Given the inverse relationship between quality factor Q, and bandwidth B, expressed by the formula $B = f_0/Q$, where f_0 is the centre frequency, Q needs to be relatively low. Unfortunately the Q factors of nano resonators typically used in such experiments is on the order of several hundred thousands, so that an active damping scheme – ofter referred to as Q-control – has to be used to tune Q to a suitable level.

Applying this measurement scheme, Oosterkamp has demonstrated detection of three separate resonance signals within a cantilever bandwidth of approximately 50 Hz, thereby reducing imaging time by a factor 3.

Benefits using the Zurich Instruments UHFLI and HF2LI for MRFM

Frequency multiplexing schemes, like the one described in the previous section, need electronic instrumentation capable of generating the complex signals required for the excitation and the simultaneous measurement of multiple independent frequencies. Furthermore, the implementation of various feedback loops to drive the cantilever, as well as for Q-control, demands additional resources.

One possible option for the experimentalist is stacking together off-the-shelf electronic components such as waveform generators, lock-in amplifiers, PID controllers, frequency response analysers, etc. from various vendors. This usually results in a rather complex and sub-optimal wiring scheme with many potential pitfalls and the need to integrate different programming approaches for the various instruments. Moreover, the careful synchronisation between the numerous instruments needs additional attention.

Alternatively, Zurich Instruments offers measurement solutions in which multiple frequency generators, lock-in units, PLLs and PID controllers are well integrated into a single box. The state-of-the-art LabOne user interface allows the user to tap into various points of the signal pathway and conduct both time domain and frequency domain analysis using the on-board oscilloscope, spectrum analyser, plotter, software trigger, etc. The Sweeper tool, for example, makes the identification and characterisation of the resonance frequencies of the cantilevers simple and convenient. Just like a frequency response analyser it can sweep the frequency of the signal driving the cantilever and then record the amplitude and the phase relative to the drive, i.e. plot the transfer function in form of a Bode plot. Once the resonance frequency and the Q factor are determined, the PLL can be set up quickly in order to ensure a drive always on the cantilever's resonance. The PID controllers can help to keep the measured amplitude constant – known as automatic gain control – by adjusting the output amplitude driving the cantilever. In addition the PID Advisor will guide the user quickly towards a set of parameters in order to establish Q-control to lower the effective Q as required by some frequency multiplexing schemes. All this comes without further need of synchronisation and with an API interface offering a high amount of flexibility, ease of integration with C, LabVIEW, MATALB and Python libraries – essential for automating lengthy measurement protocols.

At present, Zurich Instruments offers two measurement platforms suitable for MRFM:

1. the UHFLI Lock-in amplifier offers a signal input bandwidth of 600 MHz and a control loop bandwidth of more than 200 kHz;
2. the HF2LI Lock-in amplifier comes with a signal input bandwidth of 50 MHz and a control loop bandwidth of up to 50 kHz for the PLLs and up to 10 kHz for general PID applications.

Both instrument platforms exploit an all digital approach in which the signals are digitised at high speed and with high amplitude resolution right at the input. FPGA-based electronics allow for accurate data processing, providing plenty of numerical resolution in order not to limit your measurements. With 2 Signal Outputs, 2 Signal Inputs and 4 Auxiliary Outputs both instrument platforms offer a high degree of flexibility for analog interfacing to cater to even the most sophisticated measurement schemes or to serve two simpler experiments at the same time.

Conclusion

Over the recent years MRFM has demonstrated a number of great results and has already a huge potential as a 3D imaging technique at the atomic scale. With further improvements on spin detection sensitivity and acquisition time being made, scientific fields as diverse as structural biology, pharmacology, biotechnology, material science will substantially benefit from this imaging technique.
In this spirit, Zurich Instruments Lock-in Amplifiers can help researchers to achieve best possible performance regarding signal generation, signal acquisition and feedback control allowing them to fully focus on the development of the fundamental measurement schemes and the physics behind them.

References

[1] M. Poggio and C. Degen. Force-detected nuclear magnetic resonance: recent advances and future challenges. Nanotechnology, 21:342001, July 2010.

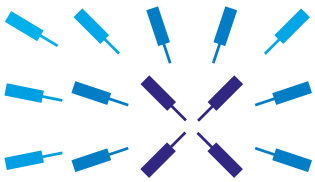
[2] J. A. Sidles. Noninductive detection of single-proton magnetic resonance. Applied Physics Letters, 58(24):2854, 1991.

[3] C. L. Degen, M. Poggio, H. J. Mamin, C. T. Rettner, and D. Rugar. Nanoscale magnetic resonance imaging. PNAS, 106(5):1313--1317, February 2009.

[4] H. J. Mamin, T. H. Oosterkamp, M. Poggio, C. L. Degen, C. T. Rettner, and D. Rugar. Isotope-selective detection and imaging of organic nanolayers. Nano Lett., 9(8):3020--3024, July 2009.

[5] J. M. Nichol, T. R. Naibert, E. R. Hemesath, L. J. Lauhon, and R. Budakian. Nanoscale fourier-transform mri. arXiv, 1302.2977v2, 2013.

[6] T. H. Oosterkamp, M. Poggio, C. L. Degen, H. J. Mamin, and D. Rugar. Frequency domain multiplexing of force signals with application to magnetic resonance force microscopy. Applied Physics Letters, 96:083107, February 2010.



Zurich Instruments

Technoparkstrasse 1
CH-8005 Zurich
Switzerland
zhinst.com

Phone +41 44 5150 410
Fax +41 44 5150 419
Email info@zhinst.com

About Zurich Instruments
Zurich Instruments makes lock-in amplifiers, phase-locked loops, and impedance spectroscopes that have revolutionized instrumentation in the high-frequency (HF) and ultra-high-frequency (UHF) ranges by combining frequency-domain tools and time-domain tools within each product. This reduces the complexity of laboratory setups, removes sources of problems and provides new measurement approaches that support the progress of research.