Status and trends in high performance magnetic imaging using Scanning Probe Microscopy(SPM)

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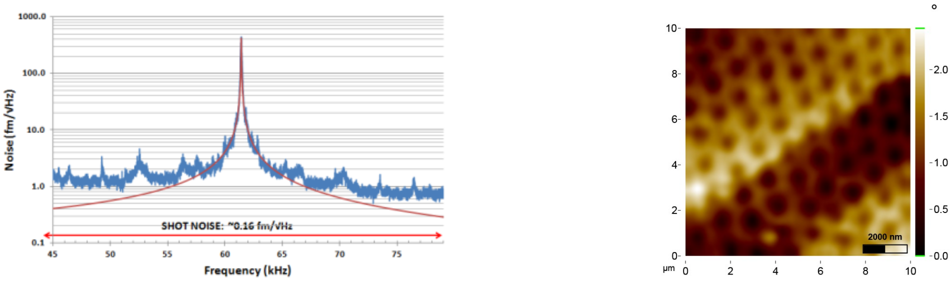
We will give an overview of the state of the art Scanning Probe Microscopes (SPM) for magnetic imaging in the 20mK to 300K temperature range. Recent developments in cryofree cryostats and dilution refrigerators (DR) have opened a new avenue for scientists suffering from heavy Helium costs.

We shall first describe the design of High Resolution MFM which can achieve 10nm magnetic resolution. Such high resolution is possible with unprecedented ~12fm/√Hz noise floor of the cantilever deflection electronics.

We shall also describe a mK-Scanning Probe Microscopes (mK-SPM) operating in Scanning Tunnelling Microscope (STM), Scanning Hall Probe Microscope (SHPM) and Atomic/Magnetic Force Microscope (AFM/MFM) mode in a wide temperature range of 20mK-300K. SHPM images of magnetic materials at 20mK will be presented.

An Oxford Instrument cryogen-free DR (Triton DR400) with 400uW cooling power and 7mK base temperature is used for the experiments. A 1W Pulse Tube cryo-cooler is integrated into the DR. After wiring and attaching the microscope we achieved 20mK base temperature. Piezo driven Stick slip coarse approach mechanism is used to bring the sample in to close proximity of the sample.

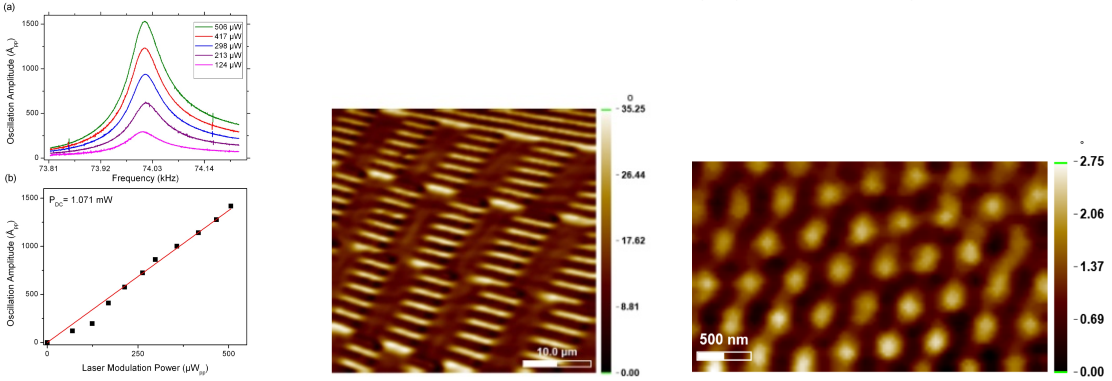
We have also designed a Fabry-Perot interferometer for our mK-AFM which has a measured ~1fm/√Hz noise level @ 4K as shown in Fig.1.(a), while the shot noise limit was ~0.2fm/√Hz. The system uses a dielectric multilayer coating at the end of the fiber to achieve this unprecedented noise level. We tested the microscope in MFM mode with a harddisk sample and imaging Abrikosov vortices in BSCCO as shown in Fig.1.(b)-(c). We hope to improve the noise levels further and achieve better than 5-6nm resolution for mK-MFM.



(a)　　(b)

Fig1.(a) Noise floor of our interferometer (b) MFM image of Abrikosov Vortex Lattice in BSCCO at 4K.

A novel method for excitation of Atomic Force Microscope(AFM) cantilevers by means of radiation pressure for imaging has been developed for the first time. Piezo excitation is the most common method for cantilever excitation. However, it has quite a few drawbacks like causing spurious resonance peaks and non-ideal Lorentzian curves. The force exerted by the radiation pressure is quite weak but sufficient to excite the cantilever to tens of nanometers for imaging in vacuum, as the Q increases to few thousands. An amplitude modulated fiber coupled 1.31μm laser is used to excite the cantilever at its resonance and detect the position for MFM imaging as shown in Fig.2.



(a)　　(b)　　(c)

Fig.2.(a) Calibration of AFM cantilevers using radiation pressure (b) MFM image of Hard disk at 4K (c) MFM image of Abrikosov Vortex Lattice in BSCCO at 4K.

Status and trends in high performance magnetic sensors and their applications

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Planar Hall resistance (PHR) sensors has many advantages (such as high signal-to-noise, small offset voltage and very linear response at low field range) compared to other magnetoresistive sensor [1]. Until now, multilayer sensor structures have been studied to improve the sensitivity of PHR sensor using cross-shaped sensor junction. At this time, the sensitivities of the PHR approximately 3, 7 and 12 µV/Oe respectively [2]. Among these structures, the sensitivity of trilayer structure was higher than others because its interlayer (Cu) reduced the exchange bias field and shunt current. In order to obtain more improvement of sensitivity, we have designed a new geometry (ring-shaped) of the sensor with Wheatstone bridge configuration over existing cross-shaped. For a constant ring width, both the sensitivity and the output voltage is proportional to the ring radius.

We have integrated multiple rings in a one ring junction of the sensor called multi-ring sensor. In using this magnetic sensor, we have made the on-chip magnetometer [3]. The on-chip magnetometer has been made by integrating a planar Hall magnetoresistive (PHR) sensor with microfluidic channels. In order to make this on-ship magnetometer, we uses successive hard and soft photolithography method. The in-plane field sensitivities of the integrated PHR sensor with trilayer structure was approximately 8 µV/Oe. The monitored PHR signals during the oscillation of magnetic nanoparticles droplet of 40 pL showed the reversed profiles for positive and negative z-fields, and their magnitudes increased with the applied z-field strength. The measured PHR signals versus applied z-fields are well fitted with the magnetization curve by vibrating sample magnetometer (VSM) for 3 µL volume; herein the PHR voltage of 1 µV is calibrated to be 0.309 emu/cc volume magnetization. In addition, I will introduce the biochip system based on spintronic devices.

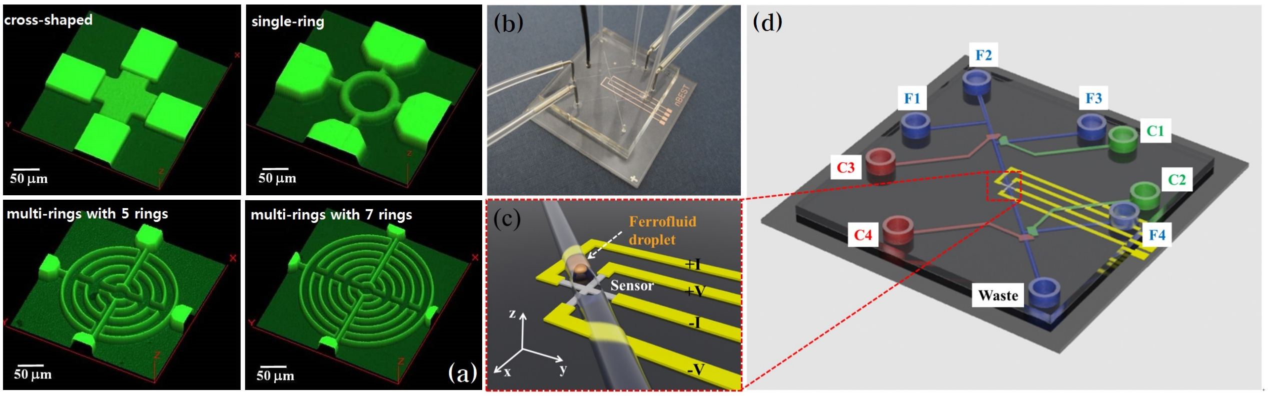

Figure: (a) 3D-microscopic images of various PHR sensor, (b) on chip magnetometer chip, (c) Schematic drawing of ferrofluid droplet coming towards the PHR sensor, (d) Schematic drawing of on chip magnetometer in which the channels (F1, F2, F3 and F4) represented in blue color are flow channels for generation of ferrofluid droplets and the channels (C1,C2,C3 and C4) represented in red color are control channels (valves) for operation of ferrofluid droplets oscillation.

Reference

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[2] T.Q.Hung, S.Oh, B.Sinha, J.R.Jeong, D.Y.Kim and C.G. Kim, J. Appl. Phys. 107, 09E715 (2010).
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Achievement of 1020 MHz NMR

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We have successfully developed a 1020 MHz (24.0 T) NMR magnet shown in Figure 1, establishing the world's highest magnetic field in high resolution NMR superconducting magnets.1)　The magnet is a series connection of LTS (low-Tc superconductors NbTi and Nb_3Sn) outer coils and an HTS (high-Tc superconductor, Bi-2223) innermost coil, being operated at superfluid liquid helium temperature such as around 1.8 K and in a driven-mode by an external DC power supply. The drift of the magnetic field was initially ±0.8 ppm/10 h without the ^{2}H lock operation; it was then stabilized to be less than 1 ppb/10 hr by using an NMR internal lock operation. The full-width at half maximum of a ^{1}H spectrum taken for 1 % $CHCl_3$ in acetone-d_6 was as low as 0.7 Hz (0.7 ppb), which was sufficient for solution NMR. On the contrary, the temporal field stability under the external lock operation for solid-state NMR was 170 ppb/10 hr, sufficient for NMR measurements for quadrupolar nuclei such as ^{17}O; a ^{17}O NMR measurement for labeled tri-peptide clearly demonstrated the effect of high magnetic field on solid-state NMR spectra, as can be seen in Figure 2.

Fig.1 1020 MHz-NMR magnet. It weighs 15 tons, 5 meters in hights and high temerature superconducting coil inside.

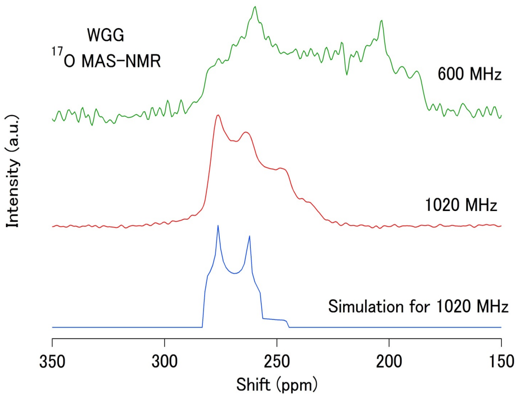

Fig.2 ^{17}O MAS NMR spectra of a labeled peptide L-tryptophanyl-glycyl-glycine dihydrate (WGG) taken at 600 MHz(14.1 T)NMR and 1020 MHz (24.0 T)NMR.　Both resolution and sensitivity can be seen much improved in 1020 MHz compared with 600 MHz.　Simulation for 1020 MHz is also plotted.

Reference

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Microcapillary capsule for nanoscale and real time observation of materials in liquid by transmission electron microscopy

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Nanometer-scale resolution and real-time observation of materials in liquid environments is required for drug discovery, tissue engineering with induced pluripotent stem (iPS) cells, and synthesis of metallic nanoparticles [1–2]. Transmission electron microscopy (TEM) has the potential for such applications because it offers atomic-level resolution and adequate video rates. However, samples must be kept under ultra-high vacuum conditions,, which makes observation of aqueous solutions challenging. Here, we propose an innovative ‘wet-TEM capsule’ consisting of a sample container separated by nanometer thick membranes that enable an electron beam to be transmitted without liquid leaking when the capsule is inserted into a TEM vacuum chamber.

Fig. 1 is scanning electron microscope image and a schematic of our wet-TEM capsule, which consists of external tubes, silicon support substrate, window region, and silicon nitride membranes. First the silicon support silicon substrate with nanometer silicon nitride membranes was etched to create window regions. Subsequently, capillary tubes were integrated in between two chips. The gap between the membranes contains the sample solution that is injected via the external tubes. We will describe recent experimental results on the observation of magnetic nanoparticles using the wet-TEM capsule system.

Reference
1) Niels de Jonge and Frances M. Ross: Nat. Nanotechnol., 6, 11, 695 (2011)
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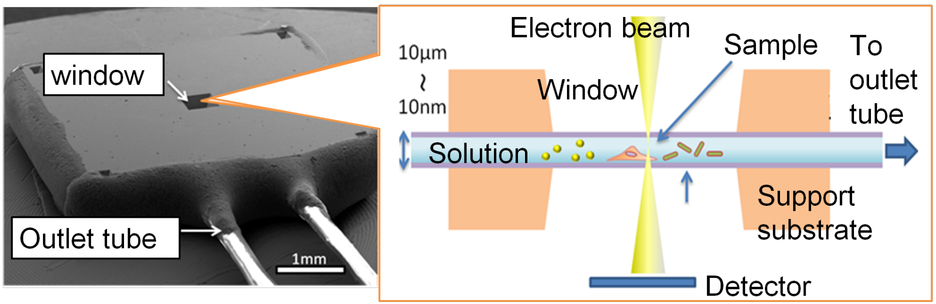

SEM Image

Fig.1 SEM and a schematic image of our capsule

Magnetic nanoparticles for biomedical applications

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Superparamagnetic iron oxides (SPIOs) including γ-Fe_2O_3 and Fe_3O_4 nanoparticles are biocompatible and relatively easy to synthesize; these properties make them the most used magnetic nanoparticles (MNPs) in biomedicine to date. They have been studied for several decades and have contributed to both diagnostics such as MRI contrast agents and therapeutics such as magnetic hyperthermia. However, the relatively low saturation magnetization (M_S) of SPIOs limits their potential in these applications.

Enhancement of the magnetic moment of MNPs is key for improvement of many applications in biomedicine. Considering the characteristic size of biological systems, MNPs with smaller dimensions than normally used SPIOs are preferred as they would increase the spatial resolution. Using MNPs, which have higher M_S and higher magnetocrystalline anisotropy energy than SPIOs, one can significantly improve efficiency in various biomedical applications. Moreover, these magnetically superior ultrasmall MNPs could lead to revolutionary and novel clinical applications.

Recently, mono- and bimetallic superparamagnetic MNPs have become readily available thanks to the development of a range of synthetic techniques. In general, the metallic MNPs exhibit higher magnetic properties than oxide MNPs, and thus those MNPs increasingly attract attention in various biomedical fields. In addition, various heterostructured multi-functional MNPs including magnetic-plasmonic core/shell/shell MNPs (Figs. 1 and 2)[1] have been recently developed for bioimaging, magnetic separation, magnetic immunoassay, etc. We review the progress of research on MNPs for biomedical applications.

References

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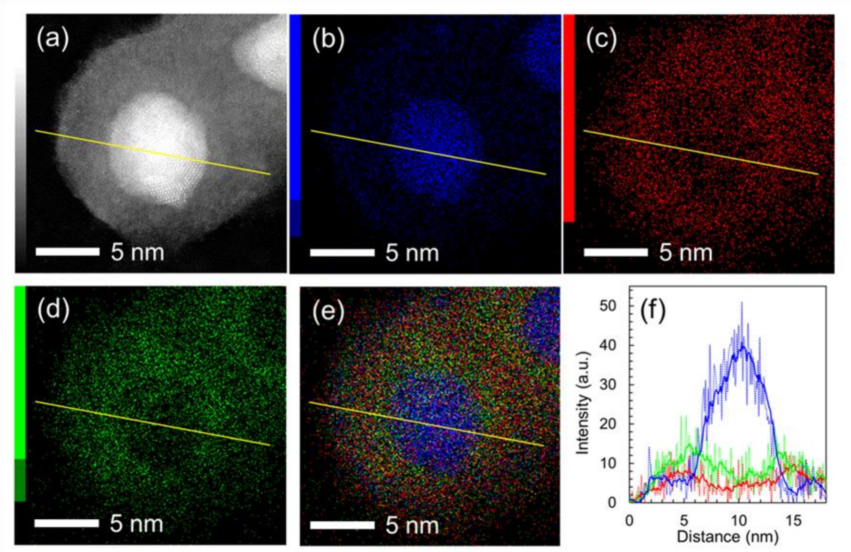

Fig. 1. (a) STEM-HAADF image of a single Ag/FeCo/Ag core/shell/shell MNP. (b–e) EDS elemental mapping images of the single Ag/FeCo/Ag MNP: (b) Ag L edge, (c) Fe K edge, (d) Co K edge, and (e) overlaid image. (f) The EDS line profile at the center of the MNP indicated by a yellow line in (a–e). Blue, green and red lines correspond to Ag L, Co K and Fe K edge intensities, respectively.

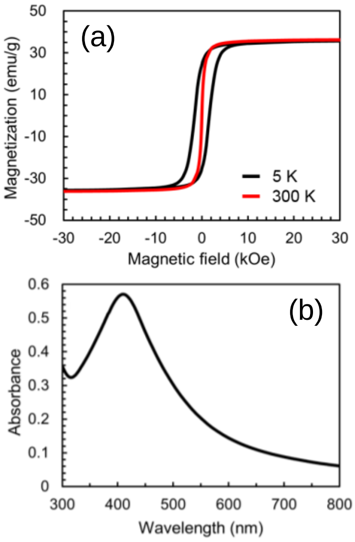

Fig. 2. (a) Magnetization curves of Ag/FeCo/Ag MNPs measured at 5 (black) and 300 K (red). (b) UV-vis spectrum of a hexane dispersion of as-synthesized Ag/FeCo/Ag MNPs.