Highlights MRI: Development of Full Vector Vibrating Sample Magnetometry for Materials Research and Education (NSF: 2216440), Wilhelmus J. Geerts

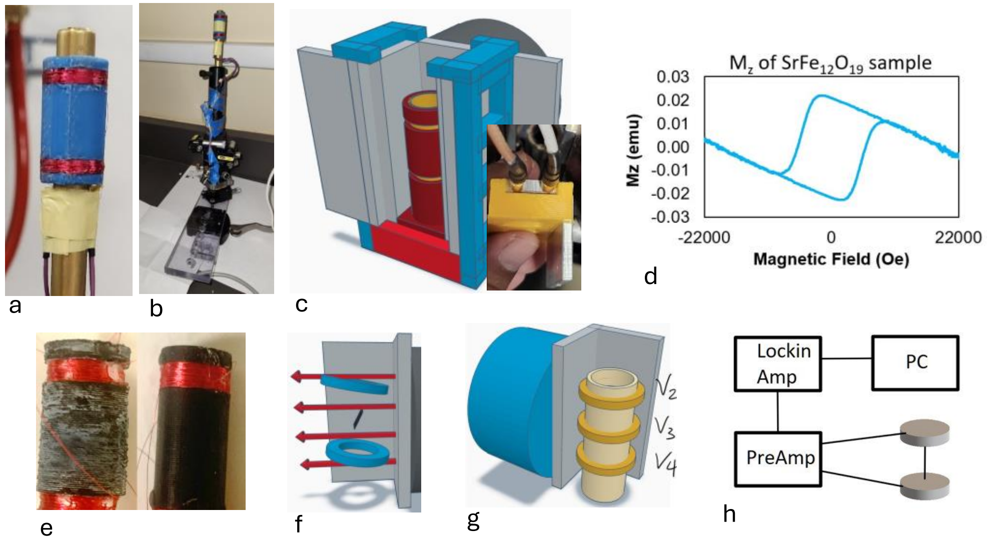

Fig. 1: (a) Initial coil set on FDM printed spool; (b) xyzθ coil alignment system; (c) Rigid Design; (d) z-signal of initial coil with cross-talk and enhanced field noise; (e) FDM and SLA printed spools; (f) effect of θ-coil misalignment on noise; (g) Design of field noise cancelation coil; (h) Use of transformer in preamplifier.

Instrumentation development for EZ9 coil sets:

Initial design: FDM printed spool and optimized coil dimensions to allow for similar sensitivity as y-coil set.

Problems: higher field noise then x and y-coils that are attached to pole pieces (Fig. 1f), significant cross-talk because of xyθ misalignment and coil asymmetry (Fig. 1d), large roughness of FDM printed spools resulting in asymmetric coilset, not possible to align z-coilset and reduce cross-talk because of tight dimensions.

Accomplishments: (1) Mechanical stage improvements: (a) increased rigidity and mass of z-coil fixture; (b) mechanical coupling of z-coil-set to xy-coil set (Fig. 1c)
(2) switched to thermoset SLA printed spool (surface treated) or CNC spool on brass tubing to improve coil symmetry (Rt/Rb=1.01) (Fig. 1e).
(3) Redesigned and manufactured coilset to 15 mm (SLA printed) and to 14 mm (CNC).
(4) Implemented Field Noise cancelation with centered coil (Fig. 1g).
(5) Used transformer preamplifier to avoid impact of Lockin-amplifier noise (Fig. 1h).

Plan for the coming year:

(1) Full test of redesigned coils.
(2) Use of existing coil set attached to pole pieces by inserting a buffer amplifier between separate coils and lockin amplifiers. Realization of a low noise buffer preamplifier.

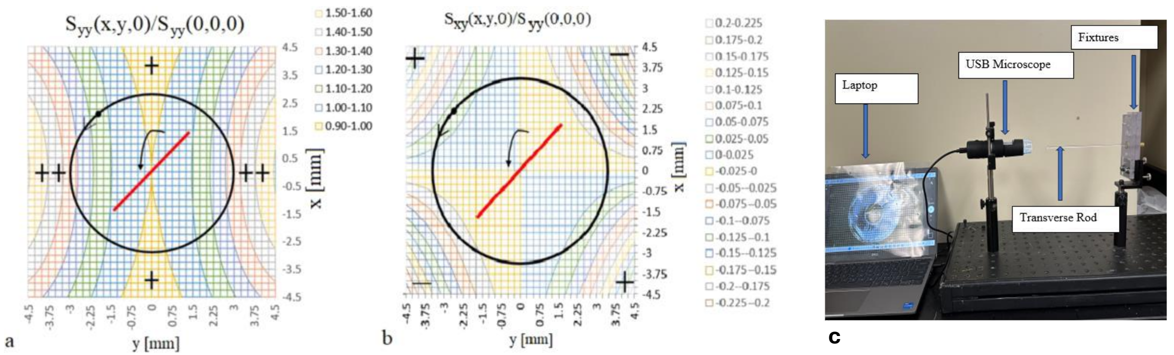

Fig 2: Effect of wobble (circular trajectory) and sample shape on (a) sensitivity and (b) cross-talk of Mallison coil-set; (c) Sample alignment system with USB camera.

Measurement Method development of vector torque magnetometry using a triaxial VSM:

a. Effect of sample misalignment (wobble: sample moves in circle with field angle) and non-azimuthal sample symmetry on biaxial VSM torque curves (Fig. 2a and 2b).
b. Comparison of biaxial torque magnetometry with true torque magnetometry.

Accomplishments:

(I) A significant and large 2θ background was observed in biaxial VSM torque curves resulting in an extra 2θ signal and a slope at high fields in the torque curves. Both effects are correlated.

Solutions: (1) The effect of sample misalignment can be mitigated by (a) Realignment of the sample-holder at each field angle; (b) Use of azimuthal symmetric sample-holders; (c) centering of the sample on the sampleholder using a USB microscope (Fig. 2c); (d) Use of keyed sampleholders. (2) The effect of the azimuthal shape of the sample can be mitigated by: (a) calibrating the VSM with a field angle sensitivity function; (b) using samples that have azimuthal symmetry; (c) For samples that fully saturate one can correct for the shape effect using the high field slope of the torque curves. Although this method works for a 8 mm nickel wire, the method fails for a 6 mm 3D printed strontium-ferrite/PA12 filament that has an easy axis perpendicular to its cylindrical axis.

(II) A small 2θ background was observed in the true torque curves of a strontium-ferrite/PA12 3D printed sample. Through a Jackson-calculation it was shown that the images of the sample in the soft magnetic pole pieces exert an additional net torque on the sample. Although we were able to show that a cylindrical magnet magnetized perpendicular to its cylindrical axis experiences a torque when placed near a soft magnetic boundary, we were not able to directly confirm this torque image effect by using a small permanent magnet as sample in the true torque magnetometer.

Plan for the coming year:

(1) Data analysis package of full vector torque measurements.
(2) Further study of how to correct for sample shape effects in the torque curves measured with a vector VSM.
(3) Collect direct experimental evidence for the torque exerted by the sample's images on the sample.

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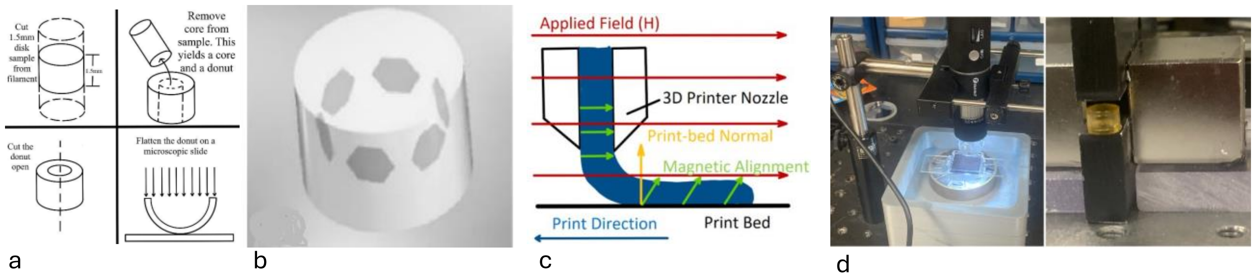

Fig. 4: (a) Design of experiment for magnetic anisotropy study of SF/PA12 filament; (b) alignment of SF platelets in extruded PA12 filament; (c) MFAAM sample printed in longitudinal field; (d) curing setups.

Use of vector VSM, accomplishments:

(a) Magnetic Anisotropy studies on Strontiumferrite/nylon (SF/PA12) 3D printer filaments. The filaments were manufactured using a twin screw extruder. VSM measurements were made on samples taken from different parts of the filament using a dissection tool. The material in the center of the filaments had a lower magnetic anisotropy then the material near the outside of the filament confirming the alignment of the magnetic SF platelets in the shear flow near the die-walls during the extrusion process.

(b) Magnetic Anisotropy study of 3D printed SF/PA12 composites in zero field and in a magnetic field (Magnetic Field Assisted Additive Manufacturing). Single filament samples were printed at 260 ^{o}C using an FDM printer furnished with a magnetic field unit that allows us to apply a homogeneous 0.5 tesla magnetic field to the material during the printing process. Filaments printed in a transverse field show an S=0.95 when measured parallel to the print field direction. Filaments printed in a longitudinal field show a complex anisotropy with an easy axis that is rotated away from the print field direction towards the print-bed normal. These observations confirm that alignment of the particles happens in the FDM-nozzle but that the molten suspension rapidly cools down upon deposition on the print bed, freezing in the orientation of the SF particles;

(c) Two setups to cure PDMS composites in a magnetic field were constructed (0.13 Tesla Halbach setup with small field gradients and a 0.78T NdFeB cube setup that has a smaller field volume (Fig. 4d)). Effect of field gradient and gravitational field on packing fraction and anisotropy is studied with VSM and optical microscope.

(d) Magnetic Anisotropy studies of Fe-doped Ga_2O_3. Electrical characterization has shown that the observed low-temperature magnetic transition in these MBE thin films is not accompanied by an electric transition suggesting that this transition is different from a Verwey transition observed in magnetite.

Plans for the coming year year:

1. Effect of flow parallel to the print bed on magnetic anisotropy of 3D printed magnetic composites.
2. Characterization of magnetic anisotropy of Ni(OH)/PDMS and $Ni_dFe_{1-d}O$/PDMS composites.
3. Characterization of magnetic anisotropy of Fe-doped Ga_2O_3 MBE films over the full temperature range of both instruments

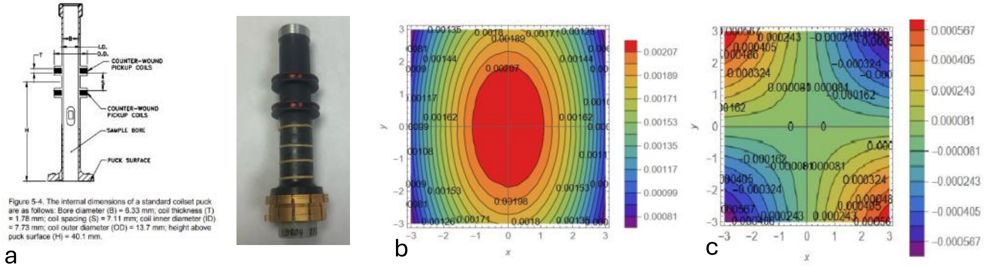

Fig. 3: (a) PPMS Z-Coilset; (b) Sensitivity X-coils; (c) M_y cross-talk X-coils.

Instrumentation development for Quantum Design PPMS coilsets:

Accomplishments: Less space is available, so x-coils are at (+/-8.4,+/-14) mm from sample position with dimensions C_l=3.5mm, C_b=2 mm, C_d=12 mm. Sensitivity and y-cross-talk are approximately 10 times smaller than those of the EZ9 baseline (Fig. 3b and 3c). Calculation is just an estimate as efficient coils have no longer azimuthal symmetry and Legendre Polynomial expansion has to be replaced by spherical harmonic expansion.

Plans for the coming year:

1. Test of Spool materials that is compatible with large temperature range.
2. Finish construction of xy-coil set.
3. Test of xy-coil set.
4. Dissemination of coil-set designs, measurement methods and demonstration studies.

Importance NSF grant 2023-2024:

1. Interdisciplinary training opportunities for 8 students in Physics and Engineering through thesis and dissertation projects. Integration of research and education through Research-driven projects in polymer nanocomposites course in Fall 2023.
2. Development of triaxial VSM magnetometry capabilities on two commercial VSMs. No commercial instruments exist.
3. Development of vector torque magnetometry methods to study magnetic materials with complex anisotropy energy surfaces.
4. Demonstrations magnetic anisotropy studies as the proof of the pudding is in the eating.

Refereed Publications in 2024:

1. https://pubs.aip.org/aip/adv/article/14/1/015048/3061600
2. https://pubs.aip.org/aip/adv/article/14/1/015048/3061600
3. https://pubs.aip.org/aip/adv/article/14/2/025022/3262881
4. https://pubs.aip.org/aip/adv/article/14/2/025245/3266855
5. https://digitallibrarynasampe.org/data/webpages/s2024_webpages/TP24-0000000224.html
6. https://digitallibrarynasampe.org/data/webpages/s2024_webpages/TP24-0000000225.html