Magnetic characterization of FePt:Fe$_3$O$_4$ nanocomposites with quartz tuning forks

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ABSTRACT

Recent capabilities to produce nanocomposite materials with unique properties require characterization for mass production and effectiveness. Although expensive equipment accomplishes this feat in time-consuming scans, this experiment introduces a faster and cheaper method to reveal not only known magnetic properties but also new phenomenon. The nanocomposite of FePt:Fe$_3$O$_4$ possesses a large coercive field, which needs to be characterized. Inexpensive quartz tuning forks are employed to detect small changes in resonance peaks after the application of FePt:Fe$_3$O$_4$ nanoparticles for fast, reproducible scans. The experiment used a small 0.3 T magnet with a hollow bore that permits the passage of loaded tuning forks though varying field strengths to determine corresponding shifts. To date, the effects of magnetic field on the quality factor and resonance frequency of the tuning forks are not systematically reproducible. Although nanoparticles alter the tuning fork response in regions of higher magnetic field, the hysteresis and the coercive field remain indistinguishable. Stronger applied magnetic fields such as a 9 T magnet are necessary to amplify the effects of magnetic field on the loaded tuning forks; however, future work must ensure an increased force to the fragile fork does not introduce overdamping and loss of signal.

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INTRODUCTION

The combination of FePt and Fe_3O_4 nanoparticles into a high-ordered superlattice network that possesses distinct magnetic properties at room temperature may provide a new material for use in a wide variety of disciplines [1]. As technology progresses, shifting from the mesoscopic to nanoscopic scale, demand increases for products with new properties. In particular, the magnetic hysteresis and coercivity of the FePt nanocomposites are of interest to facilitate applications ranging from nanoelectronics to biomedical research. FePt nanoparticle assemblies are chemically and mechanically strong, able to support magnetic transitions and reversals [2]. Sizes of available samples vary in diameter and thickness from 3 nm to 10 nm and as much as 140 nm respectively, which can be readily regulated by mass ratios and methods of preparing the material. Figure 1a is an image of typical FePt nanoparticles much like the samples used in our work. The distinct range of sizes and reproducibility makes FePt assemblies excellent candidates for mass production in various fields. Properties of annealed FePt assemblies include high stability nanoscale ferromagnets with large coercivity [2]. In fact, FePt with mass ratios of more iron exhibit much larger coercive fields.

In addition to the properties of FePt assemblies, characteristics of Fe_3O_4 present advantages for FePt and Fe_3O_4 composites leading to the discovery of new phenomenon. Recently methods of synthesizing Fe_3O_4 nanoparticles produced "nearly perfect superlattice structure" ideal for applications of further assembly [1]. Similar to the FePt nanoparticles, sizes of Fe_3O_4 thickness and diameter can also be controlled by proprietary methods. Figure 1b is an example of the control of producing uniform nanoparticle dimensions. Previous studies also indicate the ease of assembling many Fe_3O_4 nanoparticles into a structure with the capability to multilayer with other materials such as FePt [1]. The nanoparticles are superparamagnetic, may

orient in favorable structures with an applied magnetic field, further enhancing the possibilities of creating FePt and Fe_3O_4 composites with new properties.
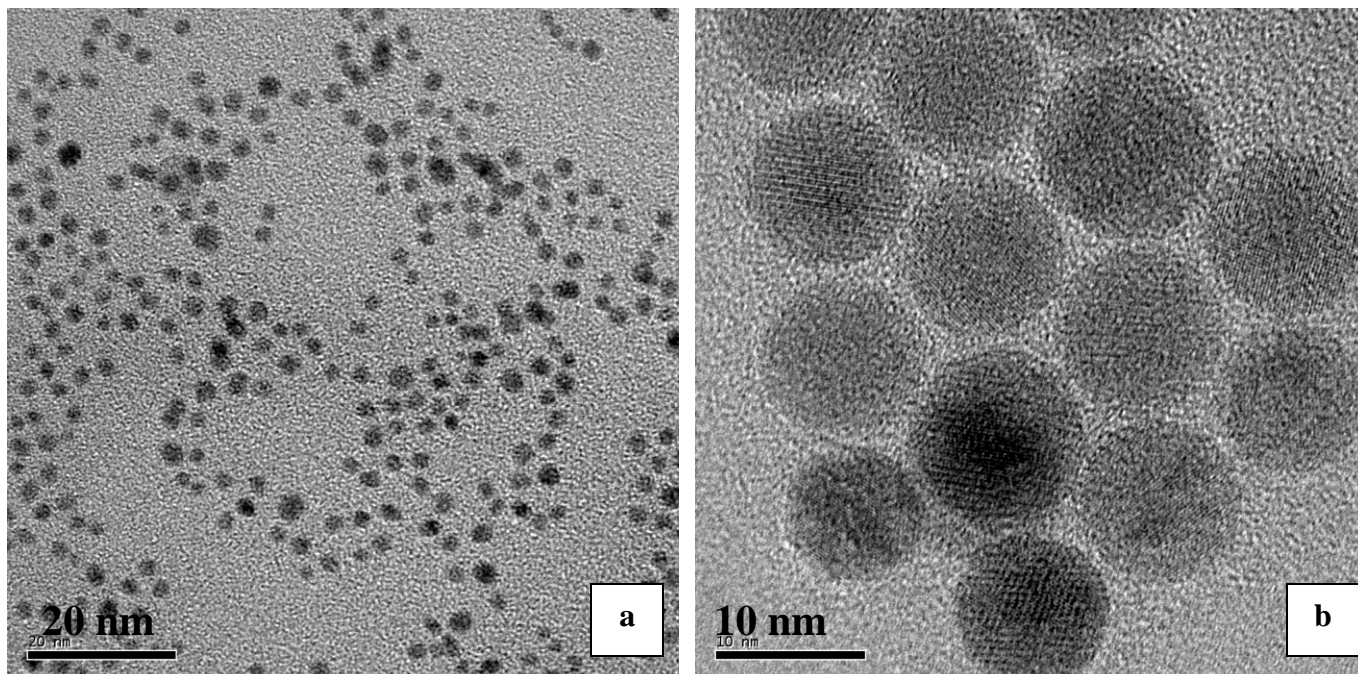

Figure 1 : **a**) FePt **b**) Fe_3O_4 nanoparticles TEM image provided by the Cao Group, UF Department of Chemistry.

Though the two components possess distinguishable properties identifying each material, the composite of FePt: Fe_3O_4 creates new properties physically and interactively. The sample we will test is a 20:1 ratio of FePt: Fe_3O_4 assembly with Fe_3O_4 nanoparticles deposited on the outside of an FePt core. Characterizations with a SQUID (Superconducting Quantum Interference Device) magnetometer indicate magnetic hysteresis and coercivity when ramped from 0 to 7 T and then down to −1.5 T in a controlled temperature environment. Figure 2 represents data from a SQUID magnetometer scanning in the above manner of a 20:1 assembly of FePt: Fe_3O_4. The material is driven to magnetic saturation near 2 T and hysteresis is observed when the applied magnetic field drops from 7 T to −1.5 T with a magnetic moment higher than the initial scan from 0 to 7 T. This particular sample has a coercive field of around 0.5 T; however, coercive field strengths vary with different mass ratios of FePt and Fe_3O_4. Anisotropy

of this composite recognizes the usefulness of such a material in many applications for the
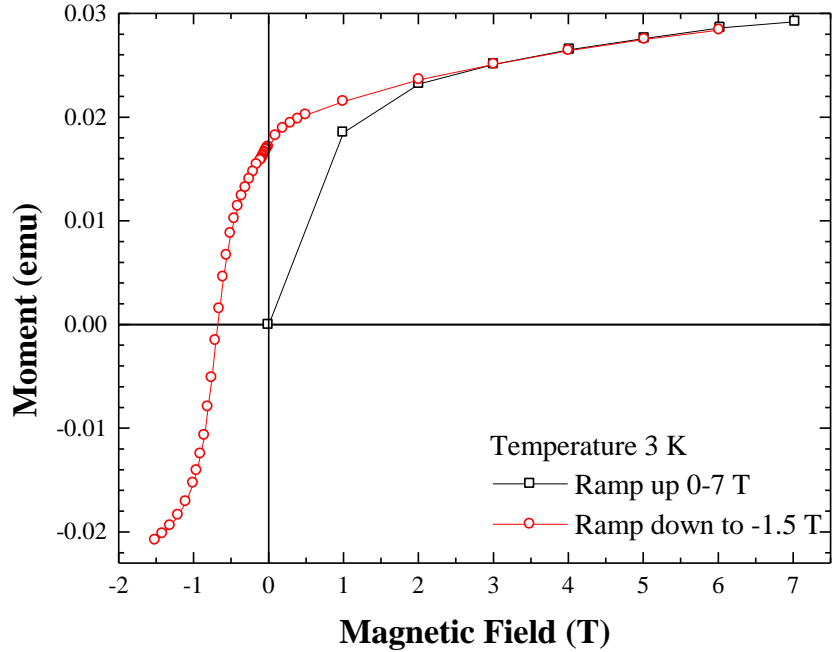
magnetic properties.

Figure 2 : SQUID characterization of magnetic moment
versus field for an $FePt:Fe_3O_4$ assembly.

Although previous characterizations with a SQUID magnetometer clearly depict large

coercitivity and anisotropy, these instruments are expensive and require time-consuming scans

[2]. In the interest of cost reduction as well as quicker results, this work explores possible use of

sensitive quartz tuning forks as a less expensive method to detect the magnetic properties of

nanoparticles in a shorter amount of time. Figure 3 represents the variations of the quality factor

(Q) due to an open or closed fork where Q is calculated by dividing the resonance frequency (f_o)

by the full width half maximum (Δf). Inexpensive quartz tuning forks with high quality factors,

$Q \sim 10^3$-10^5, offer precision frequency measurements capable of detecting small deviations from

a standard resonance frequency [3]. A closed fork, capped inside a vacuum shell, exhibits a much

higher Q than a fork open to ambient air. This difference arises because frictional forces from the

surrounding air damp the tuning fork, widening the resonance peak. Also, f_o is slightly shifted

from the removal of the vacuum cap. These small deviations select tuning forks as prime

candidates to reveal magnetic properties of $FePt:Fe_3O_4$ when nanoparticles are loaded onto the

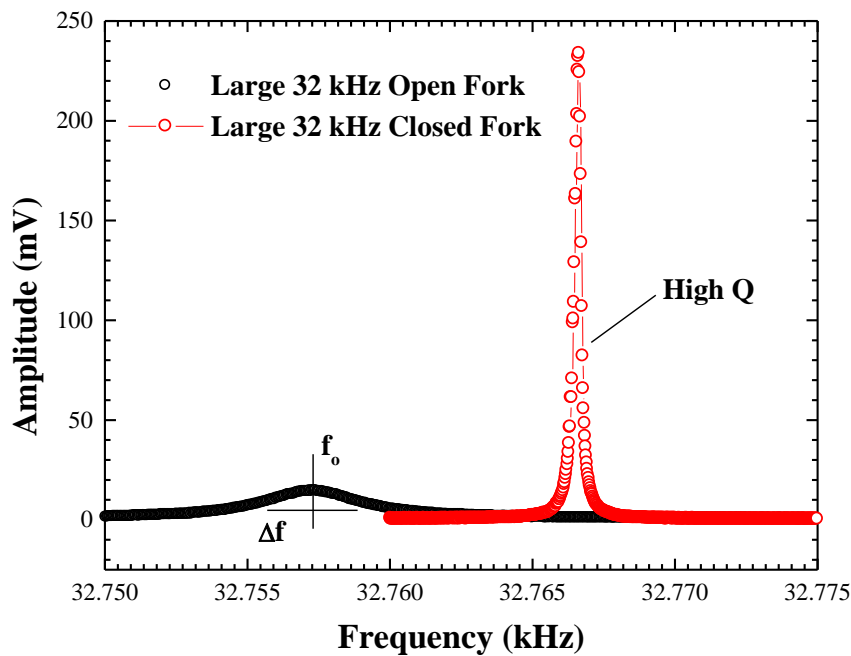ends of a tuning fork and subjected to varying magnetic field strengths at room temperature.

Figure 3: Amplitude versus frequency for large 32 kHz open
and closed tuning forks.

DATACOLLECTION FROM TUNING FORKS

Before beginning the collection of data, large 32 kHz quartz tuning forks (Epson

Toyocom C-TYPE) were carefully opened to expose the two fragile tines. The remaining fork

was integrating into the circuit diagramed in Figure 4. Our experimental method employs the use

of a lock-in amplifier (Standford Research Systems SR530) to measure the real and imaginary

components of voltage across a quartz tuning fork. This technique is incorporated in lieu of a digital multimeter not only because of the ability to measure two components of voltage but also for sensitivity. In our procedure, a lock-in amplifier is synced to a function generator (Agilent 33220A) for a reference signal of $\pm$ 5 V. Both the function generator and lock-in amplifier are connected to a computer to run a LabVIEW program that sweeps an inputted frequency range at intervals of 900ms, recording the voltage from the lock-in amplifier. Two voltage versus frequency graphs representing the real and imaginary components of the response are generated by LabVIEW scripts to visually verify if the resonance was spanned. Measurements are recorded in the absence of an applied magnetic field and also in the presence of various applied fields up to 9 T.
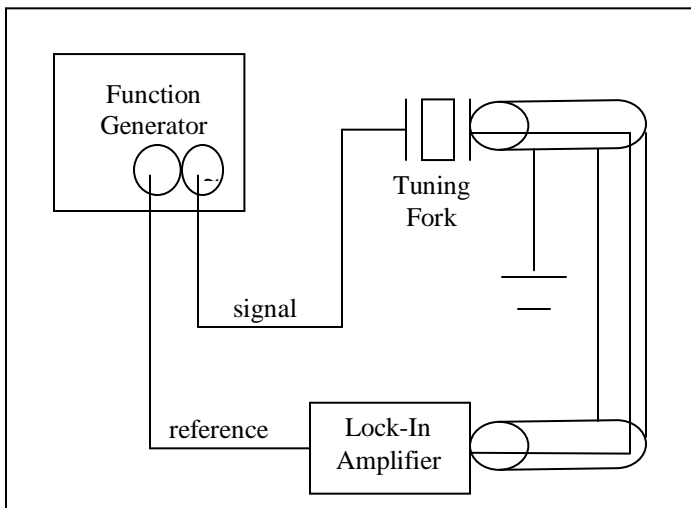

Figure 4: Circuit diagram of resonance peaks data collection on quartz tuning forks.

CALIBRATING MAGNETIC FIELD STRENGTHS

In addition to measuring the resonance frequency of quartz tuning forks, our project is also mapped the magnetic field as a function of distance for several magnets with the use of a Hall probe. The Toshiba THS118 Hall sensor is a small unit, about 2 mm in length and width,

with two voltage and two current terminals. When the Hall sensor is connected and biased with a 5 mA current supply, diagramed in Figure 5, Hall voltages are measured and vary linearly with the magnetic flux the sensor is detecting. In our experiment, the Hall sensor was mounted to a
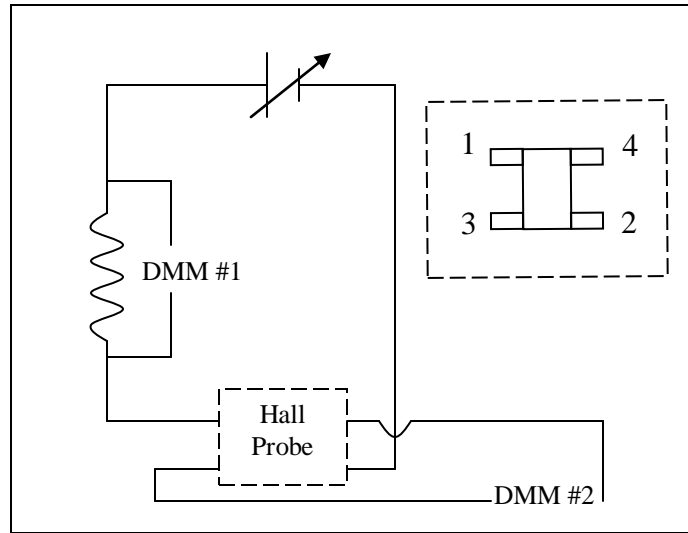

Figure 5: Circuit diagram of Hall probe data collection.

wooden stick with epoxy to detect the magnetic field at various distances through a 0.3 T permanent magnet and 9 T superconducting solenoid (Oxford Instruments). The 9 T solenoid persistent at 1 T was used to calibrate the Hall sensor at two reference points. Hall voltages at zero field and 1 T were recorded to determine the slope of voltage versus magnetic field for the Hall sensor. Results were compared to the specifications sheet, which stated a value of ±10% for the residual voltage [4]. Our calibration for the 0.3 T magnet differed by 13.6% from the values on the specifications sheet to the actual values we calculated, with a standard deviation of 1.69%. Figure 6a depicts the magnetic profile of the 0.3 T magnet used for our experiment. Note the field strength varies the most at the edge of the magnet and also the saddle shape inside the magnet indicative of a cylinder magnet with center bore. The graph is expected to be
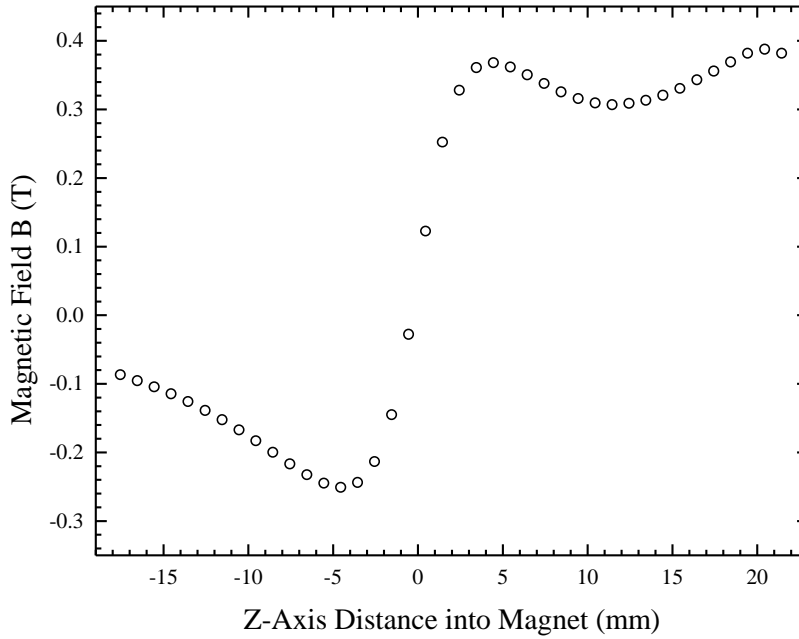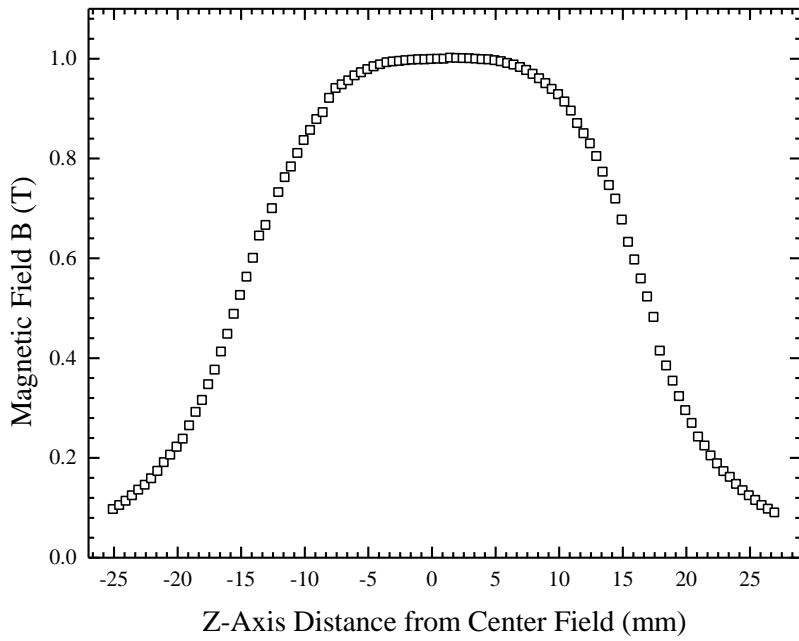
Figure 6: Magnetic field versus distance a) into 0.3 T magnet b) from center of 9 T superconducting solenoid persistent at 1 T.

symmetrical around the center field point at 12 mm if the measurements continued further than

22 mm. Figure 6b similarly depicts the magnetic profile of the 9 T magnet held at 1 T. The shape

of the curve is similar with symmetry around center field at 1 T.

STANDARD OF TUNING FORKS WITH PARTICLES

Empty forks tested at various distances into the magnet indicate little or no fluctuations in resonance frequency or half width maximum. After testing open forks without nanoparticles, we then applied small amounts of material to the ends of the fork. A desirable sample size is deposited into a 2 ml plastic vial with four drops of clear nailpolish (Sally Hansen Hard as Nails). The resulting mixture is vortexed for one to three minutes or until visible dispersion. A myriad of application techniques failed because too much applied force broke the tuning fork; however, a small sewing needle successfully applied the mixture without harming the fragile prongs of the fork. Each fork was left for a minimum of 24 hours before testing for resonance in a magnetic field.

One standard for the experiment was a sample of Fe_3O_4 loaded onto similar forks and examined for shifts associated for added weight and magnetism. Figure 7 is an example of a scan from inside the magnet moving to the outer edge. Both scans into and out of the magnet revealed the same graph as shown, thus anisotropy was not observed. Although a few stray points suggest resonance frequency shifts, we believe capacitive coupling of wires as well as noise around the system produced the false data. Most of the data reflect the anticipated zero shift in resonance frequency with slight visible changes to Q and Δf.
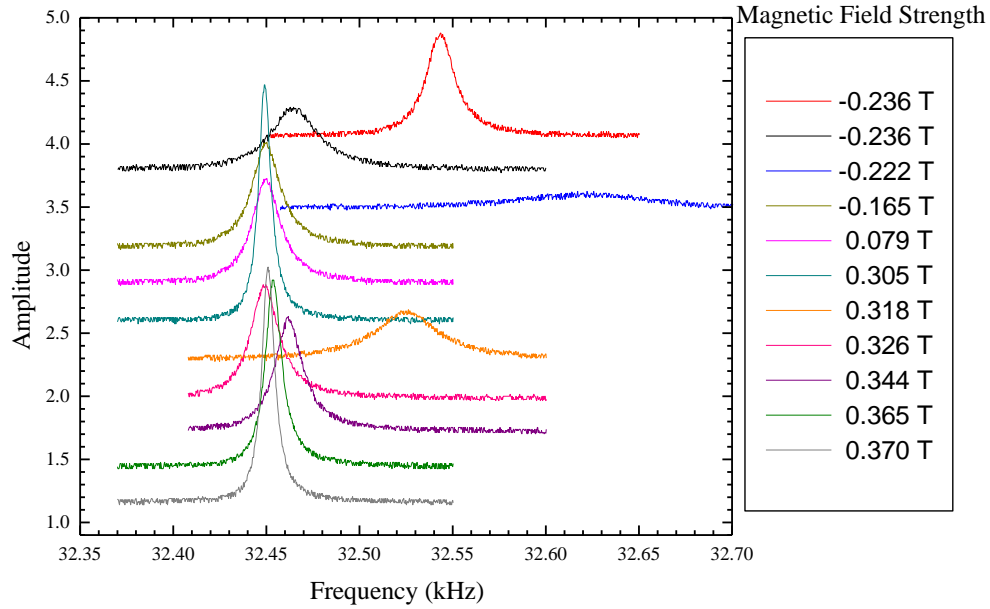
Figure 7: Amplitude versus frequency of Fe_3O_4 particles on tuning fork, arranged according to points of increasing magnetic field.

FePt:Fe$_3$O$_4$ IN 0.3T MAGNET

After sufficient amounts, in the microgram range, of FePt:Fe$_3$O$_4$ nanoparticles were verified on the tuning fork via a microscope, scans at the same point as the standard were performed. Figure 8 indicates the changes in Q and resonance frequency relative to magnetic flux. At points of higher magnetic field more deviation in both Q and resonance frequency occurs with significant shifts in resonance frequency. An indirect relationship of resonance frequency and magnetic field strength is apparent. Overall, more data in the lower magnetic field region are necessary to validate direct effects. Coercive field and hysteresis are not clear in Figure 8, so further analysis of Q and fo was preformed. We explored the relationship of Q and f_o relative to B∇B. Random scattering and shifting is apparent both into and out of the magnet with concentrated action near the zero of B∇B. We expected to see more points in this region because

the zero of B∇B corresponds to higher magnetic field; however, results still remain unclear

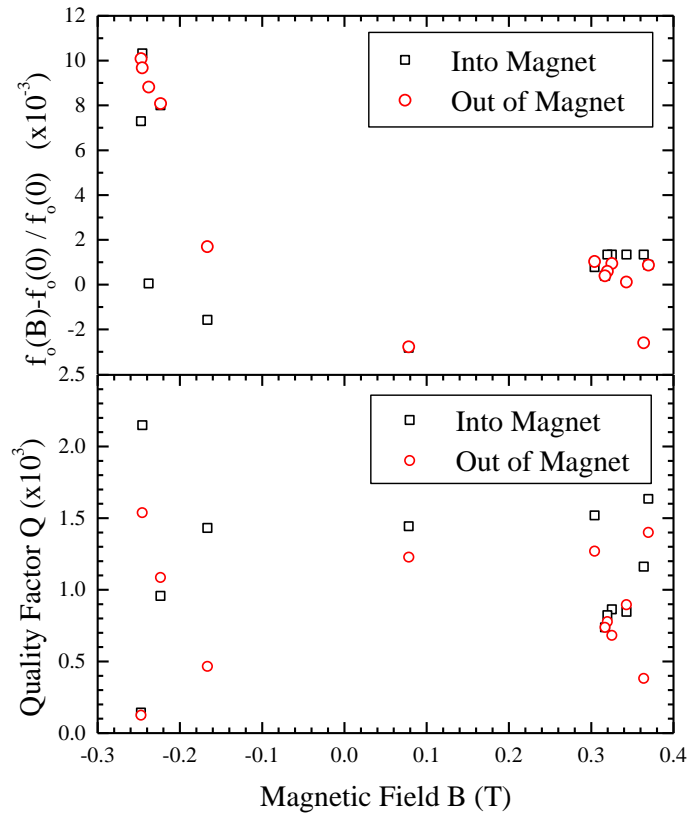regarding of the effects of magnetic field on the loaded tuning fork.

Figure 8 : Resonance frequency and quality factor versus magnetic field for 20:1 FePt:Fe$_3$O$_4$ assembly in 0.3 T magnet.

FePt:Fe$_3$O$_4$ IN 9T MAGNET

Further tests are currently underway in the 9 T magnet persistent at 1 T in efforts to

reduce noise and amplify the effects of a higher magnetic field on the nanoparticles. A new

circuit with the tuning fork, using coaxial cable instead of twisted wire pairs, reduces the

capacitive coupling added to the circuit. One of the main concerns of the coaxial cable is the

added length to accommodate moving into a larger magnet. At this point limitations such as too

much material on the tuning fork resulted in overdamping of the oscillator and loss of signal.

Added capacitance with coaxial cables and noise is still an issue because we cannot resolve the resonance peak with a small Q. Possible solutions to this problem are currently being developed such as shortening the wires, increasing the peak-peak voltage of the circuit, and adding an operational amplifier (op-amp) to filter out the noise and amplify the weak signal.

FUTURE WORK

In this experiment we surpassed the hurdle of creating a method to characterize the magnetic field for magnets of varying strengths with great accuracy. Sensitive, inexpensive quartz tuning forks were employed to detect changes in resonance peaks while moving through the profiled magnets. Nanoparticles of $FePt:Fe_3O_4$ were successfully mounted to the ends of tuning forks and obvious shifts of resonance frequency and quality factor resulted. Although the tuning forks were chosen for sensitivity, the analysis of data was complicated by the inability to isolate the sensitive system and overdamping of the fragile fork. Possible solutions for future work include using a stronger magnet along with reduced added capacitance and an operational amplifier to amplify the effects without overdamping the fork. Also, applying smaller amounts of nanoparticles to smaller tuning forks increases the quality factor, providing a larger resonance peak. Replacing the vacuum seal after the addition of nanoparticles also offers a much higher quality factor and less chance of overdamping the oscillator.

ACKNOWLEDGMENTS

REFERENCES

[1] J. Zhuang, H. Wu, Y. Yang, and C. Cao, "Supercrystalline colloidal particles from artificial atoms," J. Am. Chem. Soc. **129**, 14166-14167 (2007).

[2] S. Sun, C. B. Murray, D. Weller, L. Folks, and A. Moser, "Monodisperse FePt nanoparticles and ferromagnetic FePt nanocrystal superlattices," Science. **287**, 1989-1991 (2000).

[3] J. M. Friedt and E. Carry, "Introduction to the quartz tuning fork," Am. J. Phys. **75**, 415-421 (2007).

[4] <http://www.datasheetcatalog.org/datasheet/toshiba/506.pdf>. 18 July 2009.