Optimization of a Vibrating Sample Magnetometer for a laboratory physics course

Optimización de un magnetómetro de muestra vibrante para un curso de física de laboratorio

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RESUMEN

Este artículo describe la implementación y una optimización detallada de un magnetómetro de muestra vibrante (VSM) para un laboratorio de licenciatura en física. Los parámetros de operación de VSM se discutieron ampliamente usando la configuración de bobinas de Foner y Mallison. Se discutió la influencia de los parámetros implicados (por ejemplo, frecuencia de oscilación, amplitud de oscilación, cambio de velocidad del campo magnético externo, configuración de bobinas, etc.) sobre la tensión inducida en las bobinas de captación. Se utilizó un disco de níquel de 6 mm de diámetro para la calibración del magnetómetro, comparando el bucle de histéresis medido con nuestro magnetómetro con el obtenido utilizando un VSM comercial. Se obtuvieron curvas de magnetización de dos muestras diferentes para probar la sensibilidad del magnetómetro. El magnetómetro de muestra vibrante implementado en el presente trabajo es capaz de detectar cambios en el momento magnético total hasta 10^{-3} emu. La optimización detallada del VSM descrita en el presente trabajo es un ejemplo de cómo resolver un problema real en materia condensada, relacionado con la determinación del valor de magnetización de una muestra magnética.

Keywords: Magnetómetro de muestra vibrante, Magnetometría, Instrumentación,

ABSTRACT

This paper describes the implementation and a detailed optimization of a Vibrating Sample Magnetometer (VSM) for an undergraduate physics course laboratory. The VSM operation parameters were extensively discussed using Foner and Mallison coils configuration. The influence of the involved parameters (e.g. oscillation frequency, oscillation amplitude, rate change of the external magnetic field, coils configuration, etc.) on the induced voltage in the pick-up coils were discussed. A disk of nickel of 6-mm diameter was used for the calibration of the magnetometer, comparing the hysteresis loop measured with our magnetometer with the one obtained using a commercial VSM. Magnetization curves of two different samples were obtained in order to test the sensitivity of the magnetometer. The vibrating sample magnetometer implemented in the present work is able to detect changes in the total magnetic moment down to 10^{-3} emu. The detailed optimization of the VSM described in the present work is an example of how to solve a real problem in condensed matter, related to the determination of the magnetization value of a magnetic sample.

Palabras clave: Vibrating sample magnetometer, Magnetometry, Instrumentation.

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1 INTRODUCTION

Experimental physics courses offered at undergraduate level provides a strong background for experimentation in physics. Traditional experiments performed in these courses could be classified in three different groups: Universal constants determination (e.g. mass-to-charge ratio, Millikan's experiment, etc.), demonstrative physics experiments (e.g. light interference, photoelectric effect, etc.) and techniques related to materials characterization (e.g. Kerr effect, Hall effect in semiconductors, etc.). Particularly, experiments related to materials characterization techniques provide different tools to solve real problems in condensed matter such as the charge-carrier density of a semiconductor, the crystalline structure and the lattice parameter of a thin film or the magnetization value in a magnetic sample. The development of an experiment which consists of a detailed optimization of a simple technique during an

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experimental physics course allows students to propose new ideas and solutions to the problem of extract relevant information in a material of interest. In this case we introduced the Vibrating Sample Magnetometry technique as a solution for the determination of the magnetization value of a sample.

Vibrating sample magnetometry has become one of the most common techniques for the characterization of magnetic materials. This versatile technique provides information related to the magnetic features of different systems studied in material science, such as thin films or magnetic nanoparticles. The first Vibrating Sample Magnetometer (VSM) was developed by Simon Foner in 1956 [1]. VSM operating principle is based on Faraday's law, and consists of measuring the induced voltage in an array of coils due to the change of magnetic flux inside them. The magnetic flux change is caused by the relative motion between an oscillating magnetic sample and the coils' arrangement.

The induced voltage V(t) in the coils of the magnetometer is proportional to the magnetic moment of the oscillating sample, the oscillation amplitude and frequency and to a geometrical factor S(r) as shown in Eq. 1 [2, 3], for a sinusoidal motion of the sample, according to:

$$V(t) = S(\bar{r}) M A \omega \cos \omega t \,, \qquad (1)$$

where M is the magnetization of the sample, A and ω correspond to the oscillation amplitude and frequency respectively and S(r) is known as sensitivity function of the pick-up coils configuration. According to Eq. 1, the sensitivity function and the oscillation parameters dependence (amplitude and frequency) on the induced voltage are two determinant factors of the output signal of the VSM. The sensitivity function, which represents the spatial distribution of the coils' sensitivity, determines the saddle point of the sample – coils configuration [2]. Placing the sample in the saddle point, it is possible to obtain an output signal insensitive to small position variations. An adequate optimization of the amplitude and frequency, allow us to obtain reliable magnetic measurements as will be discussed in the present work. Even though, this paper discuss the optimization of the induced signal in the pick-up coils, it is important to notice that Eq. 1 relates the induced voltage V with the magnetization M of the sample and allow us to perform a magnetic characterization of the samples through the M(H) curve.

Although, some authors have proposed vibrating sample magnetometry as a tool for a materials physics course [3], relevant information related to the optimization of this technique was not discussed. This paper will be focused on the detailed optimization of a Vibrating Sample Magnetometer for the characterization of magnetic materials and its use as a tool for strengthen the concepts of instrumentation, electromagnetism and basics of magnetism acquired during undergraduate formation. On the other hand, the implementation and optimization of this type of magnetometer in an experimental physics course is an important introduction to the basics of instrumentation and magnetic materials characterization. Moreover, the characterization of known samples as Nickel or magnetic recording media will allow students to become familiar with the concepts of coercive field, saturation and remanence obtained from hysteresis loops. These concepts are relevant for the study of interesting effects such as interlayer exchange coupling in thin films [4], exchange bias [5] or superparamagnetism in magnetic nanoparticles [6].

2 EXPERIMENTAL DETAILS

Fig. 1 shows a schematic of the implemented VSM, indicating the electromechanical transducer control system, the lock-in amplifier, the detection coils and the Hall probe configuration.

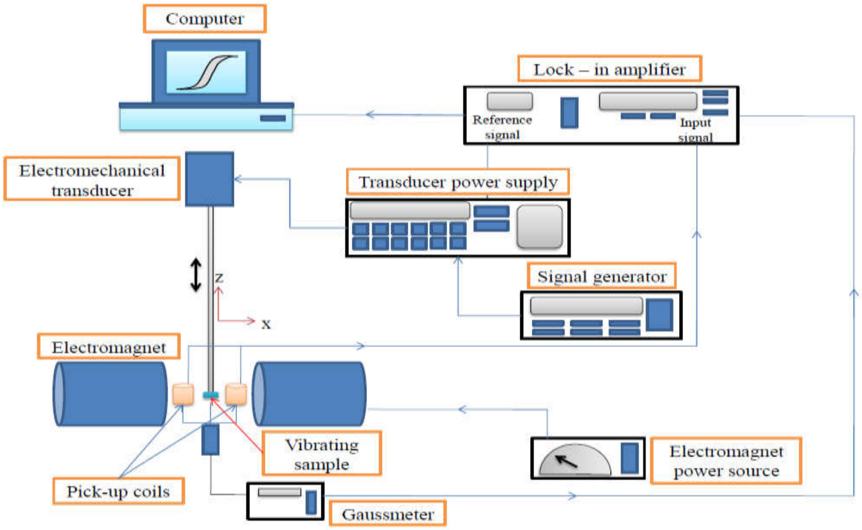

FIGURE 1. Schematics of the implemented vibrating sample magnetometer.

The VSM implemented in the present work uses an electromechanical transducer of linear displacement to generate the oscillation of the sample. The transducer was adapted from an ELSCINT Mössbauer spectrometer model MD - 3. Commercial Mössbauer spectrometers are designed to operate at low velocities in the range 0 - 500 mm/s approximately, with a linear response between the input signal amplitude and the oscillatory displacement in most cases for low frequencies. Due to conventional VSM usually operates at a higher frequency (~ 70 – 90 Hz), the displacement - frequency response will be discussed. The transducer power supply was adapted from the Mössbauer spectrometer using a sinusoidal signal generator as input signal. An AMETEK lock-in amplifier model 5209 was used to detect the oscillatory signal induced in the pick-up coils. An axial Hall probe and a PHYWE teslameter were used to measure the external magnetic field. For the detection of the induced magnetic signal in the pick-up coils, the main configuration used was the proposed by Foner [1]: two coils (3000 turns per coil) with their axis directed along z axis (Fig. 1). Magnetic samples were mounted on an aluminum bar attached to the electromechanical transducer.

a. Optimization parameters

In order to optimize the different parameters related to the sensibility of the induce voltage, we used a disk of nickel with a diameter d = 6 mm and a thickness (tNi) = 400 μm as a sample pattern. Magnetization changes are detected by the Foner coils arrangement placed between the magnet poles as shown in Fig. 1. The operation parameters of the VSM were optimized through the signal-to-noise ratio (S/N) of the magnetic signal induced in the pick-up coils. The optimization of the VSM started with the determination of an adequate value for the oscillation frequency. Fig. 2 shows the dependence of the oscillation frequencies (in a range of 10 - 110 Hz) on the induced voltage in the pick-up coils.

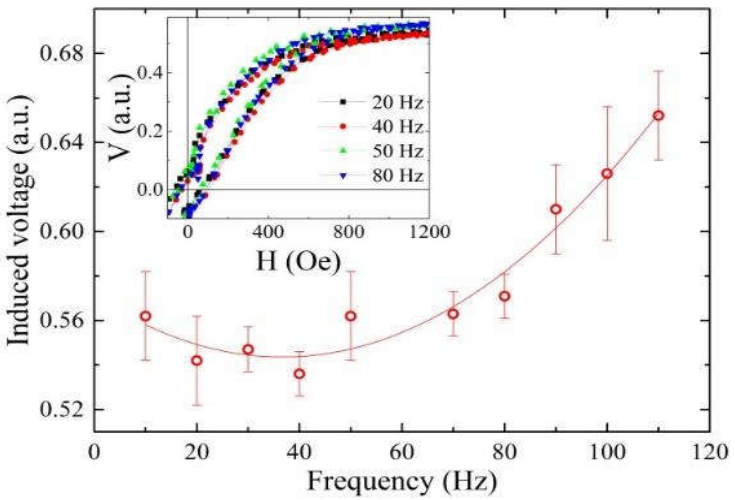

FIGURE 2. Induced voltage in the coils as a function of the oscillation frequency. The values of the induced voltage were taken from the saturation region. The line is a guide to the eyes. Inset: First quadrant of the hysteresis loops of nickel disk at different frequencies.

Induced signal as a function of the oscillation frequency of the sample takes its lowest value around 40 Hz. Inset of Fig. 2 shows some of the measured hysteresis loops in the frequency range mentioned. According to the M(H) curves shown in the inset of Fig. 2, the VSM is able to measure a magnetization loop at different frequencies. However, S/N ratio decreases considerably around 60 and 120 Hz, as shown in Fig. 2. Due to a high S/N ratio and a relatively high value of the induced signal, our magnetometer could operate at 40 or 80 Hz. Our electromechanical transducer was originally design to operate at low frequencies; therefore the selected operation frequency is 40 Hz. According to Eq. 1 the induced signal should be proportional to the oscillation frequency. This is not the case for our magnetometer, probably due to a non-linear response of the oscillation displacement with the frequency in our electromechanical transducer. It is expected that for some value of the oscillation frequency, there is a maximum value of the oscillation displacement, as a result of the mechanical coupling between the electromechanical transducer and the aluminum rod.

Induced magnetic signal as a function of the transducer oscillation amplitude supplied by the waveform generator was also optimized. Fig. 3 shows the linear response of the induced signal in the pick-up coils as a function of the oscillation amplitude. This dependence verifies the linearity between the amplitude of the supplied voltage to the transducer with the displacement of the sample. Inset of Fig. 3 shows the magnetization loops for different values of

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the supplied voltage amplitude of the oscillation in a range 0.2 - 1.8 V.

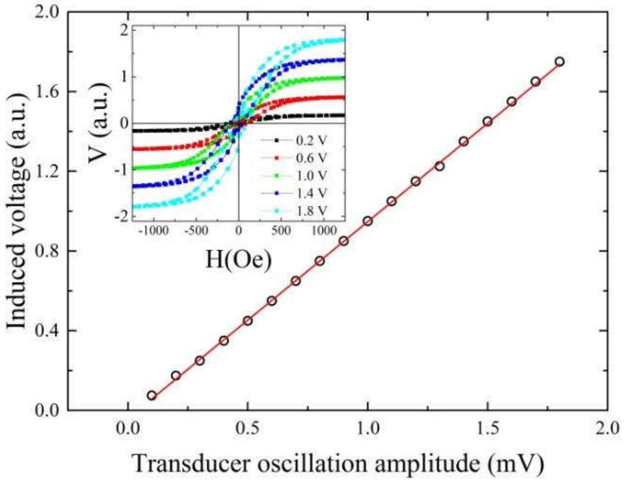

FIGURE 3. Induced magnetic signal as a function of the oscillation amplitude. Inset: Hysteresis loops of a 6-mm diameter nickel disk at different amplitudes of the supplied voltage to the transducer input.

Induced voltage as a function of the lock-in time constant was also performed. As known, lock-in amplifiers uses a technique known as phase sensitive detection, which allow us to filter unwanted signal by a low pass filter. The bandwidth of this filter is controlled by the time constant value (τ) of the lock-in. Lower values of the time constant increase the bandwidth of the signal filter. Lock-in's time constant influence on the induced signal is shown in Fig. 4. Measurements varying the time constant were performed using the same change rate of the external magnetic field.

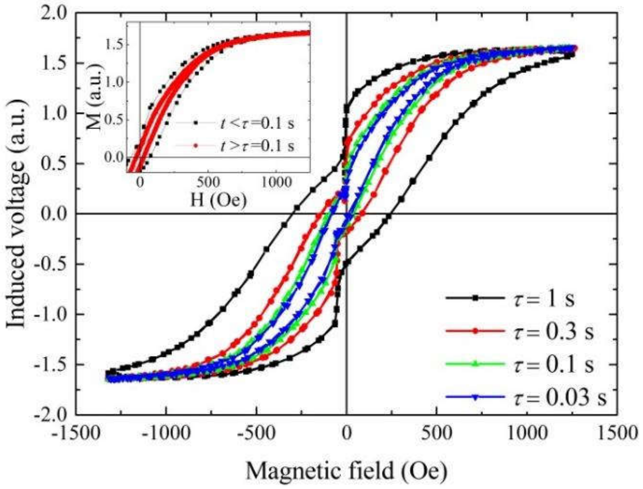

FIGURE 4. Magnetization loops as a function of the lock-in's time constant values τ. Inset: First quadrant of the hysteresis loops of a 6-mm diameter nickel disk at different change rates of the external magnetic field.

The characterization of the samples using τ above 0.1 s showed a distorted hysteresis loop. In this case the change rate of the external magnetic field influences the measurements. In simple words, the Lock-in amplifier operates statistically, and requires a certain number of input values for the average of the data. When reducing the bandwidth of the low pass filter (by increasing τ), the amplifier rejects a greater number of data, therefore it is necessary a longer measurement time in order to obtain a reliable output value. If the change rate of the external magnetic field is lower than the required measurement time, the amplifier will show an output voltage different to the real value. On the other hand, a lower S/N ratio was observed in the measurements performed using τ < 0.1 s although it showed a more conventional hysteresis loop shape for nickel samples. Additionally, varying the change rate t of the external magnetic field, we improved the signal-to-noise ratio significatively. Lock-in amplifier averages more data in a slower change rate of the magnetic field (i.e. bigger time intervals between field steps) compared to the lock-in time constant (t > τ). The increasing of the S/N ratio by diminishing the change rate of the external magnetic field is shown in the inset of Fig. 4. Complementary to the oscillation parameters optimization (amplitude and frequency), the correct use of the lock-in amplifier in this type of magnetometer determines its reliability. Additionally, provides an initial training for young researchers as suggested by other authors [7].

A 4-coils arrangement (Mallison coils) with their axis parallel to magnetic field direction [8], allowed us to determine the saddle point of the coils configuration. The distance between the two pairs of coils (2000 turns per coil) was D = 16 mm. Induced signal measured by the coils was plot as a function of the sample position in the x axis as shown schematically in the inset of Fig. 5.

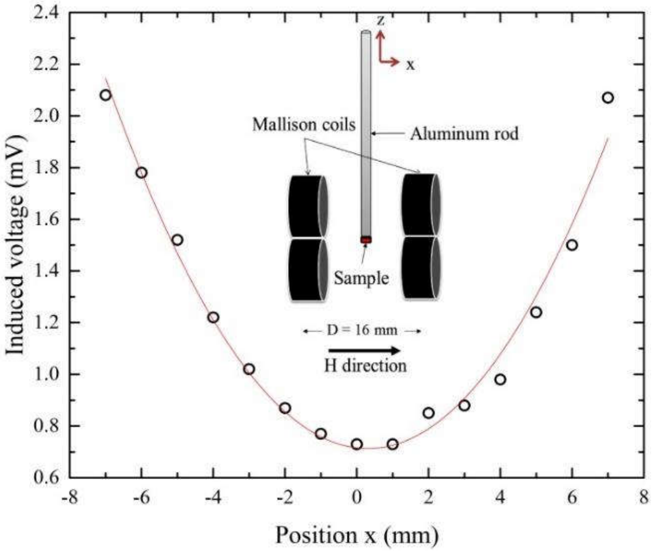

FIGURE 5. Induced signal as a function of the sample position with respect to the pick-up coils. Determination of the saddle point in x-axis of Mallison configuration. The line is a guide to the eyes. Inset of figure 5 indicates a schematic of the sample position for the determination of the saddle point of Mallison configuration.

By measuring the hysteresis loops of nickel disks for larger thicknesses we observed an increase in the signal-to-noise ratio. These results (not shown) give an idea about the spatial region near the saddle point where the induced signal is insensitive to small variations of position. Saddle points of different coils configuration and the influence of shape's samples using theoretical formalism and contrasted with experimental measurements at intermediate physics level will be publish by our group shortly.

The operation parameters obtained from the optimization of the vibrating sample magnetometer are listed in table 1.

TABLE 1 Optimal parameters of the VSM.

Parameter	Value
Oscillation frequency	40 Hz
Oscillation amplitude	1.8 V
Oscillation displacement	~ 1 mm
Lock-in time constant	100 ms
Sample size	~ 6 mm

As it was mentioned previously, a nickel disk of 6 - mm diameter was used as sample pattern for the optimization of the magnetometer. Saturation of nickel samples in bulk state is well determined: Ms ~ 55 emu/g (In CGS units) [9] The hysteresis loop of a Ni disk obtained with our VSM is shown in Fig. 6a). A hysteresis loop obtained with a commercial vibrating sample magnetometer VERSALAB 3T CRYOGEN - FREE VSM is shown in Fig. 6b). Magnetization loops obtained with our magnetometer assure the reliability of the measurements due to a high signal-to-noise ratio in a detection signal range of mV in the pick-up coils.

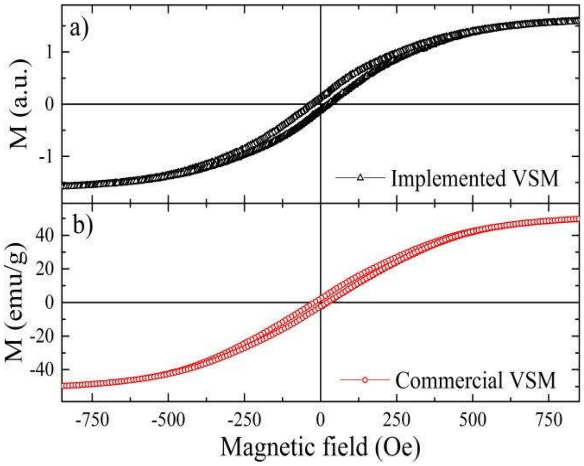

FIGURE 6. a) Hysteresis loop of a nickel disk of 6-mm diameter obtained with the implemented vibrating sample magnetometer. b) Hysteresis loop of a nickel disk of 6-mm diameter obtained with a commercial VSM used for the magnetometer calibration.

Hysteresis loops presented in Fig. 6a) and 6b) allowed us to obtain the proportionality factor K between the induced magnetic signal and the magnetization of the sample. Using the parameters described in table 1 we obtained K = 2.98 ± 0.02 emu/mV. This proportionality factor is directly related to the sensitivity function evaluated at the saddle point of the coils configuration according to Eq. 1.

b. Tested Samples

Two different materials were characterized in order to determine the sensibility of our magnetometer. The first samples were magnetic recording media disks. The magnetization scale of Fig. 7a) indicates that our magnetometer is able to detect magnetic moment changes around 10-3 emu, using the parameters described in table 1.

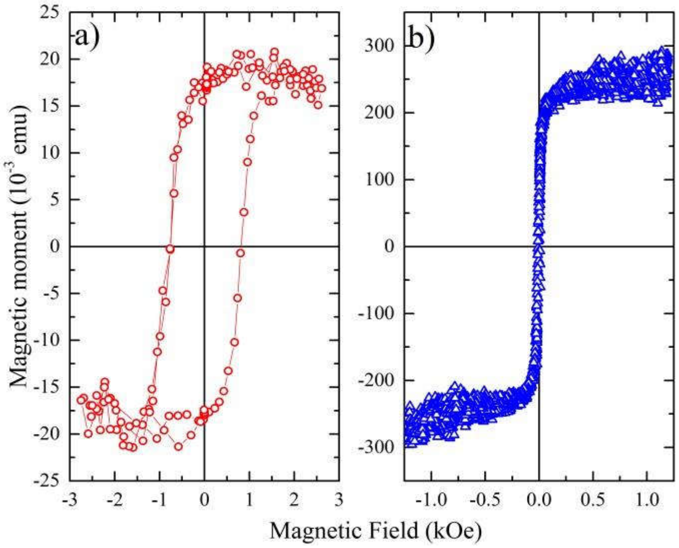

FIGURE 7. Hysteresis loop of a) magnetic recording media disks obtained from a floppy disk. b) 9-mm length amorphous magnetic ribbon of Fe75B15Si10. Both scales are in 10-3 emu.

As it was expected from a magnetic recording media (obtained from floppy disks), M(H) curves shown a coercive field Hc ~ 750 Oe and a MR = 0.7 Ms, where MR is the remanence and Ms is the saturation magnetization respectively. A magnetic material that maintains its saturation state after removing the magnetic field is ideal for magnetic information storage.

On the other hand magnetization loop of amorphous magnetic ribbons of Fe-based alloys were also measured in order to test the sensibility of the implemented VSM. Fig. 7b) shows the hysteresis loop of Fe75B15Si10 magnetic ribbons. In contrast with the hysteresis loop obtained from the floppy, the magnetization curve obtained from the magnetic ribbon shows a high permeability and a very low remanence (MR ~ 0). High magnetic permeability materials are commonly used as a magnetic sensor due to its immediate response to the magnetic field. Pronounced magnetization noise observed at high magnetic field could be due to a slightly bigger sample: 9 mm length compared to 6 mm diameter Ni disk.

The characterization of two samples with different magnetic properties allows the students to discuss the potential applications of those materials through the magnetization curves.

3 CONCLUSIONS

We implemented a Vibrating Sample Magnetometer for the characterization of magnetic samples. The optimization of the operation parameters and the signal calibration were performed through the detection of the induced magnetic signal in a pick-up coils arrangement generated by the oscillation of a nickel disk. The induced voltage as a function of the oscillation frequency, the oscillation amplitude, the lock-in time constant, the sample position and the magnetic field change velocity allowed us to determine the optimal parameters for the operation of the VSM. Comparison between the hysteresis loop of the calibration sample obtained with a commercial VSM and the magnetization curves measured with our magnetometer indicates a high S/N ratio in a detection signal range of mV, being able to detect changes in the magnetic moment around 10^{-3} emu. The used methodology for the optimization of the involved parameters allowed us to exploit the capabilities of the lock-in amplifier and it is an example of how to solve a real problem in condensed matter, related to the determination of relevant information of a sample. On the other hand, the complete optimization of the magnetometer is adequate for the training of young researchers in condensed matter physics and instrumentation fields.

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