NATIONAL UNIVERSITY OF ENGINEERING

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MAGNETOLOGIA DE MUESTRA VIBRANTE COMO HERRAMIENTA PARA VALIDACION DEL PROCESO DE SEPARACION DE MINERALES MAGNETICOS

VIBRANT SAMPLE MAGNETOLOGY AS A TOOL FOR VALIDATION OF THE SEPARATION PROCESS OF MAGNETIC MINERALS

THESIS PROJECT REPORT

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CONTENT

Abstract

This paper presents the design and implementation of a Vibrating Sample Magnetometer (VSM), where we center on the materials magnetic properties such as diamagnetism, paramagnetism, ferromagnetism, anti-ferromagnetism and ferrimagnetism. Where we become acquainted with experimental measurement techniques of hysteresis cycle, and the experimental application of one of these techniques such as Vibrant Sample Magnetometer (VSM),its functioning principle is based on the Faraday induction law, and consists in the voltage measurement in an arrangement of coils, due to the magnetic flux change in its interior. It is described the technique of minerals magnetic separation using Frantz Isodynamic separating tool. Through the VSM magnetically separated samples are characterized, observing the magnetic behavior of the present materials.

Keywords: Vibrating Sample Magnetometer (VSM)

CHAPTER 1: Introduction

1.1 Background

Locally the work done by Luis Aviles [1] describes the design, construction and optimization in the implementation of a Vibrating Sample Magnetometer (VSM) for its use in the characterization of magnetic materials. In this work is given the theoretical fundament which explains the functioning principle of the Vibrating Sample Magnetometer (VSM), carefully describing the design and construction of the system for the properties measurement of a magnetic material that are shown in its magnetization curve or hysteresis curve. Depending on the hysteresis curve shape, the magnetic material can be classified according its application.

1.2 Objectives

The main objective of the present work is to use the VSM technique, with the purpose of certify the magnetic separation of magnetic minerals process.

Design and implementation of a VSM.

Obtain hysteresis curves of dust samples obtained through a Frantz Isodynamic magnetic separator.

CHAPTER 2: Theoretical Fundament

In the present chapter is introduced, in general lines, the theory of magnetism and magnetic materials, classifying materials according to its behavior when they are inside a magnetic field. That's why magnetic materials are classified in: Diamagnetic, paramagnetic, ferromagnetic, anti-ferromagnetic and ferrimagnetic. The magnetic properties of two minerals will be described: Magnetite and Hematite.

2.1. Magnetism

The magnetism is a phenomena which manifests in the materials as an attractive or repulsive force. A materials magnetic properties are the result of the interaction of its atomic structure with the magnetic field. Currently, the applications of the magnetism are in expansion and allow it to be one of the most important pillars in the modern science and technology.

2.2. Magnetic Moment

In an atom each electron has two magnetic moments. The magnetic moment is the effectiveness of the magnetic field associated to one electron. This moments, called Bohr's magneton is:

$$\mu_B = \frac{q.h}{4\pi m_e}.$$

Where q is the electron charge, h the Planck constant and m_e the electron mass. The magnetic moments are generated by the movement of the electron around the nucleus and the rotation of the electron around its own axis is called **spin**.

2.3. Magnetic Susceptibility and Permeability

If the magnetization M is parallel towards an outside magnetic field H.

$$M = \chi H$$

With χ, being the material magnetic susceptibility it is denominated linear material. In this situation, a linear relation between B and H keeps present,

$$B = \mu_o(1+\chi)H,$$

$$B = \mu_o\mu_r H.$$

With $\mu_r = (1+\chi)$, being the relative permeability. Typical values of the magnetic permeability are: [2]

In the void: $\mu_r = 1$

En the matter, generally: $\mu_r \geq 1$

Possible in the matter: $\mu_r \approx 100.00$

2.4. Orbital Angular Momentum and Spin

The magnetism is deeply related to the angular momentum of the elemental particles, so that the quantum theory of magnetism is closely linked to the quantization of angular momentum. Protons, neutrons and electrons have an intrinsic angular momentum 1/2 $\hbar$ known as spin, where $\hbar$ is the Planck constant divided by 2π.

2.4.1. Orbital Moment

The orbital moment can be introduced under conditions of Bohr's atomic model, where electrons spin around a charge nucleus Ze in circular orbits under the influence of Coulomb's potential $\varphi_e = -Ze/4\pi r\epsilon_0$. An electron that is circling in its orbit is equivalent to the current of a revolution where the direction of the current is the opposite of the direction of the circulation due to the negative charge that it presents. If the velocity of electrons is v, its period of rotation is $\tau = 2\pi r/v$ and the equivalent current is $I = -e/\tau$. The magnetic moment associated with turning the current $\mathfrak{m} = \mathrm{I}\mathcal{A}$ is $-\frac{1}{2}e\vec{r}x\vec{v}$, where the vector product shows the direction of $\mathfrak{m}$. In terms of angular momentum, $\ell = m_e\vec{r}x\vec{v}$, the moment is,

$$\mathfrak{m} = -\frac{e}{2m_e}\ell.$$

The proportionality between the magnetic moment and the angular momentum is in general,

$$\mathfrak{m} = \gamma\ell.$$

Where the proportionality of the factor γ is known as the gyromagnetic relationship. For the orbital motion of the electrons, γ is -(e / 2m_e); The minus sign means that $\mathfrak{m}$ and ℓ are directly direct due to the negative charge of the electron.

The orbital angular momentum is quantized in units of $\hbar$, such that the component of m in a particular direction, chosen as the z-direction, is

$$\mathfrak{m}_z = -\frac{e}{2\mathfrak{m}_e}\mathfrak{m}_\ell\hbar, \text{ where } \mathfrak{m}_\ell = 0, \pm 1, \pm 2, \dots\dots$$

Here $\mathfrak{m}_\ell$ is a quantum orbital magnetic number. The natural unit for electronic magnetism is therefore the Bohr magneton, defined as

$$\mu_B = \frac{e\hbar}{2\mathfrak{m}_e},$$

Where $1\mu_B = 9.274 \text{ x}10^{-24} A\mathrm{m}^2$. The component "z" of the quantum magnetic orbital moment is an integer number of the Bohr magneton. The remarkable difference between an electron in a steady state of quantum mechanics and a charged classical particle is that the former can move indefinitely in its orbit as a kind of perpetual motion or electron super-current while the classical particle, or an electron in a non-quantized orbit, it must emit energy because of its continuous centrifugal acceleration.

The classical orbital movement will soon cease as a result of the loss of radiation. The relation (3.5) is expressed alternately in terms of a factor "g", which is defined as the ratio of the magnitude of the magnetic moment in units of μ_B for the magnitude of the angular momentum in units of $\hbar$: $(|m|/\mu_B) = (g|\ell|\hbar)$. Hence g is exactly 1 for the orbital motion.

The derivation of (3.4) can be generalized for non-circular orbits. From (2.3), $\mathfrak{m} = \mathrm{I}\mathcal{A}$ for a planar return of any shape and area $\mathcal{A}$. The angular momentum of an electron moving with a velocity ω, $\ell = m_e r^2 \omega$ is a constant around the orbit. The current $\mathrm{I} = -\mathrm{e}/\tau = -(\mathrm{e}\ell/\mathfrak{m}_e)\langle 1/2\pi r^2\rangle_{av} = -e\ell/2\mathfrak{m}_e\mathcal{A}$, where $\langle\cdots\rangle_{av}$ is the average along the orbit. Therefore $\mathfrak{m} = -(\mathrm{e}/2\mathfrak{m}_e)\ell$. [3]

2.4.2. Atoms in a Magnetic Field

Now consider an atom with Z_{at} electrons, whose Hamiltonian $\widehat{\mathrm{H}}_0$, is given by:

$$\widehat{\mathrm{H}}_0 = \sum_{\mathrm{i}=1}^{Z}\left(\frac{\mathrm{p}_\mathrm{i}^2}{2\mathfrak{m}_e} + \mathrm{V}_\mathrm{i}\right).$$

Where $\frac{\mathrm{p}_\mathrm{i}^2}{2\mathfrak{m}_e}$ is the kinetic energy and V_i is the potential energy of the i-th electron.

When a constant and uniform magnetic field B given by $B = \nabla \mathrm{x} A$ is applied, and considering the gauge,

$$\vec{A}(\vec{r}) = \frac{\vec{B}\mathrm{x}\vec{r}}{2}.$$

The Hamiltonian of the system is modified by forming:

$$\widehat{\mathrm{H}} = \widehat{\mathrm{H}}_0 + \mu_B\left(\hat{L} + g\hat{\mathrm{S}}\right).\hat{B} + \frac{e^2}{8\mathfrak{m}_e}\sum_\mathrm{i}(\hat{B}\mathrm{x}\hat{r}_1)^2$$

$$\widehat{\mathrm{H}} = \widehat{\mathrm{H}}_0 + \widehat{\mathrm{H}}_1$$

Where $\widehat{\mathrm{H}}_1$ represents the modification of the Hamiltonian $\widehat{\mathrm{H}}_0$ due to the external magnetic field $\hat{B}$. The Hamiltonian $\widehat{\mathrm{H}}_1$ can be written as,

$$\widehat{\mathrm{H}}_1 = \mu_B\left(\hat{L} + g\hat{\mathrm{S}}\right).\hat{B} + \frac{e^2}{8m_e}\sum_\mathrm{i}(\hat{B}\mathrm{x}\hat{r}_1)^2 = \widehat{\mathrm{H}}_1^{\mathrm{para}} + \widehat{\mathrm{H}}_1^{\mathrm{dia}}.$$

Where $\widehat{\mathrm{H}}_1^{\mathrm{para}}$, is known as the paramagnetic term and $\widehat{\mathrm{H}}_1^{\mathrm{dia}}$ is known as the diamagnetic term. [2]

2.5. Materials that do not retain any remaining magnetization

2.5.1. Diamagnetism

Behavior of those materials whose atoms spins are oriented parallel but inversely to the external field. Its magnetization is weak and in the opposite direction to the field and therefore its susceptibility, X is negative and of very low values. These materials are called diamagnetic, and we can see that they are repelled very slightly before a magnetic field like that of a magnet. Its fundamental characteristic is that they have the energy levels of their complete electrons (even electrons in their layers) and there are no unpaired magnetic moments. However, when an external field is applied, a moment is induced that tends to compensate the magnetic flux that is exerted on the orbitals, an effect that gives them their distinctive weak magnetization and in the opposite direction to the field. There are numerous examples of them among the main constituents (minerals) of rocks, such as silica (quartz), calcium carbonate (calcite), silico-aluminates (feldspar), carbon and organic materials, as well as Most of the metals in the periodic table: Copper, Zinc, Silver, Cadmium, Gold, Lead and Bismuth.

2.5.2. Paramagnetism

Behavior of materials that when exposed to an external field, the spins of their atoms are oriented parallel and in the same direction as the external field, their magnetization is weak and in the same direction as the field. Its susceptibility, X is positive and of low magnitude but somewhat higher than in the diamagnetic group. These materials are called paramagnetic, and we can distinguish them because they are slightly attracted by a magnetic field like that of a magnet. Its characteristic is that in them the energy levels of their electrons are incomplete (odd electrons in their layers). There are numerous examples of these materials among the rock-forming constituents, mainly iron silicates (ex. biotite, lithite, amphiboles, pyroxenes, olivine), as well as iron and magnesium carbonates and clays such as montomorillonite. Also many rare earths are paramagnetic.

2.6. Materials that do retain remaining magnetization

Materials which retain magnetization, even in the absence of an external field, are generally known as ferromagnetic "S.L." (Sensu Lato: broad direction) or more colloquially as "magnetics" and usually are easily identifiable because they are easily attracted by a magnet. In them the spins of their electrons are spontaneously coupled, parallel aligned by an interaction that remains even in the absence of external field;

Property called spontaneous magnetization. This property is presented in the elements of the first transition series with unpaired electrons in the 3d layer, mitigating the magnetization that arises from the orbital movement of the electrons.

2.6.1. Ferromagnetism

In the simplest cases, as in the metals Fe, Ni, Co, as well as in materials called "ferrites" made of Fe and Boron with Barium, Strontium and Molybdenum; In which the electron spins of adjacent cations are coupled directly in the same direction, since between them operates an exchange force, which occurs because the energy of neighboring dipoles is smaller when the dipoles are aligned than in any other position . These materials are what are strictly termed ferromagnetic S.S. (Sensu Stricto: Strict sense). Fe Curie temperature is 770 ° C.

2.6.2. Anti-ferromagnetism

In more complex compounds, such as the oxidation of elements of the first transition series, the electron spin of the cations is shared with the electron layer of an intermediate anion (Oxygen, in the case of oxides) A force of super-exchange. This implies that the spin direction of the electron of the neighboring cations is inverted, creating forces (lattices or networks) is balanced, there will be no resulting net magnetization. In this case the substances are called anti-ferromagnetic. However, pure anti-ferromagnetism is very rare, since any imperfection causes a net unbalance resulting in a weak parasitic magnetization. Examples of imperfect anti-ferromagnetisms are hematite, goethite, ilmenite and ulvoespinela. The hematite at low temperatures (-10 ° C) is perfect anti-ferromagnetic, its Neel temperature is 675 ° C.

2.6.3. Ferrimagnetism

In complex compounds like the previous case, but in which there is a lack of balance between the lattices and one is greater than the other, there is a resulting net magnetization. Materials with this property are called ferrimagnetics. Examples are iron oxides, with spinel structure, such as magnetite (Curie temperature of 575 ° C) and maghemite, as well as pyrrhotite (monoclinic structure).

A special type of magnetic behavior is the so-called super-paramagnetism that presents extremely small ferrimagnetic grains (0.001 - 0.01 micrometers in diameter), characterized by an induced magnetization under an external field H that are unable to retain once removed the field at room temperature , A phenomenon attributed to the development of thermal vibrations.

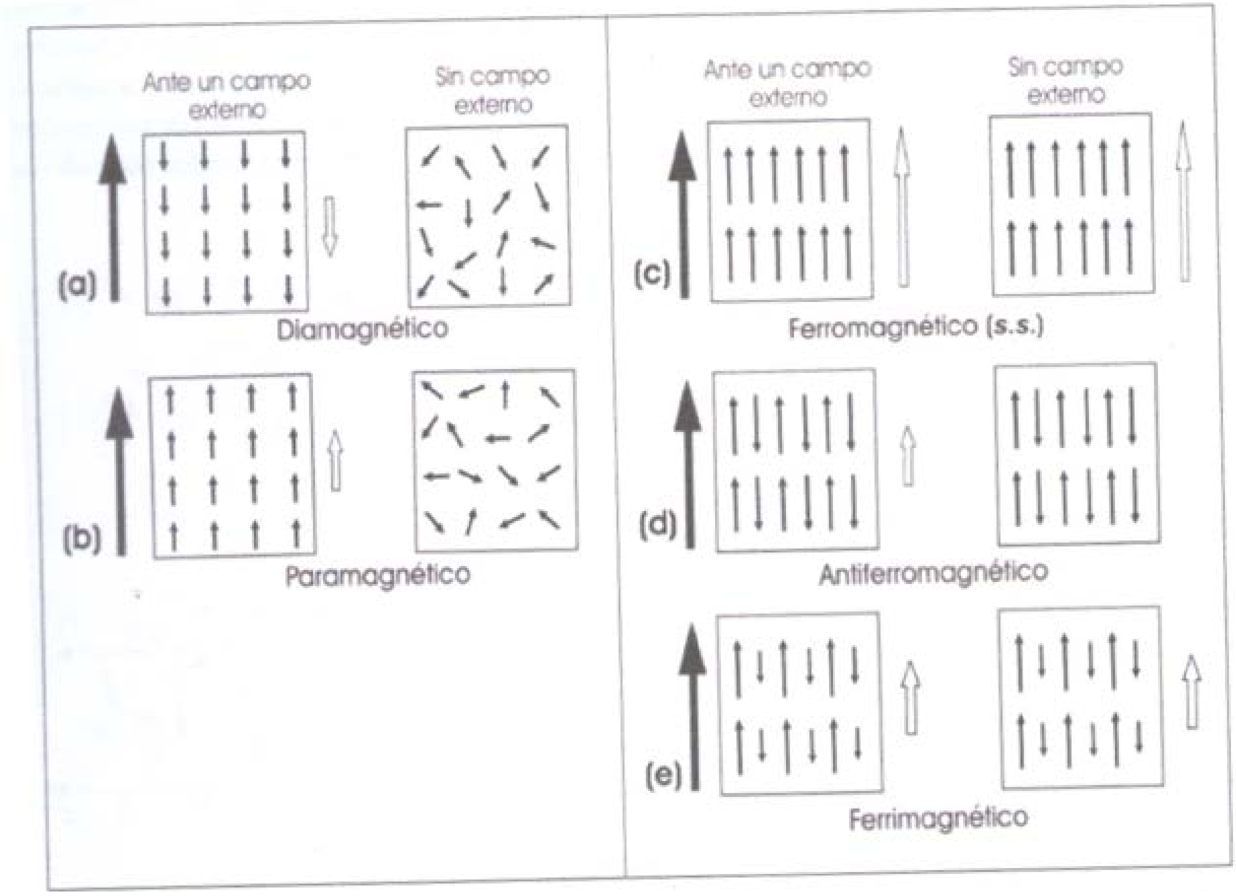

Fig. 1: Different types of magnetic behavior. The filled arrows indicate the applied field and the emptying magnetization

2.7. Cycles of hysteresis and identification of magnetic mineralogy

Hysteresis cycles graph the behavior of the materials by applying the field in one direction until reaching the material M_s (point 2, Fig. 2), then once the field has been removed to where it retains its M_r (point 3, figure 2) And composting from here again apply a field in reverse direction until a M zero is reached (point 4, Fig. 2) and again the M_s of the material (now in the reverse direction, point 5 Fig. 2), followed by the removal of the Field to where the material retains its M_r (point 6) and from here to the beginning of a new cycle of magnetization with a field in the initial sense. The graphs of these cycles allow to identify the type of material by: (1) the values of M_s and the value of the applied field under which it is acquired (H_s in Fig. 2) and (b) the amplitude of the cycle or the value of coercivity (value of the field in point 7). Usually these cycles of hysteresis are plotted using the field of induction B, in teslas, it is applied with laboratory instruments and with the value of normalized magnetization in order to better compare the magnetic behavior between the different materials as seen in the Fig. 3 shows the behavior of diamagnetic, paramagnetic and super-paramagnetic materials in graphs a, b and c respectively and of imperfect ferromagnetic (hematite), ferrimagnetic (SD magnetite) and simple pseudo-domain (PSD) . This is a routine technique of the most used for the identification of magnetic mineralogy not only for AMS works but also for Paleomagnetism in general in all its various applications. [4]

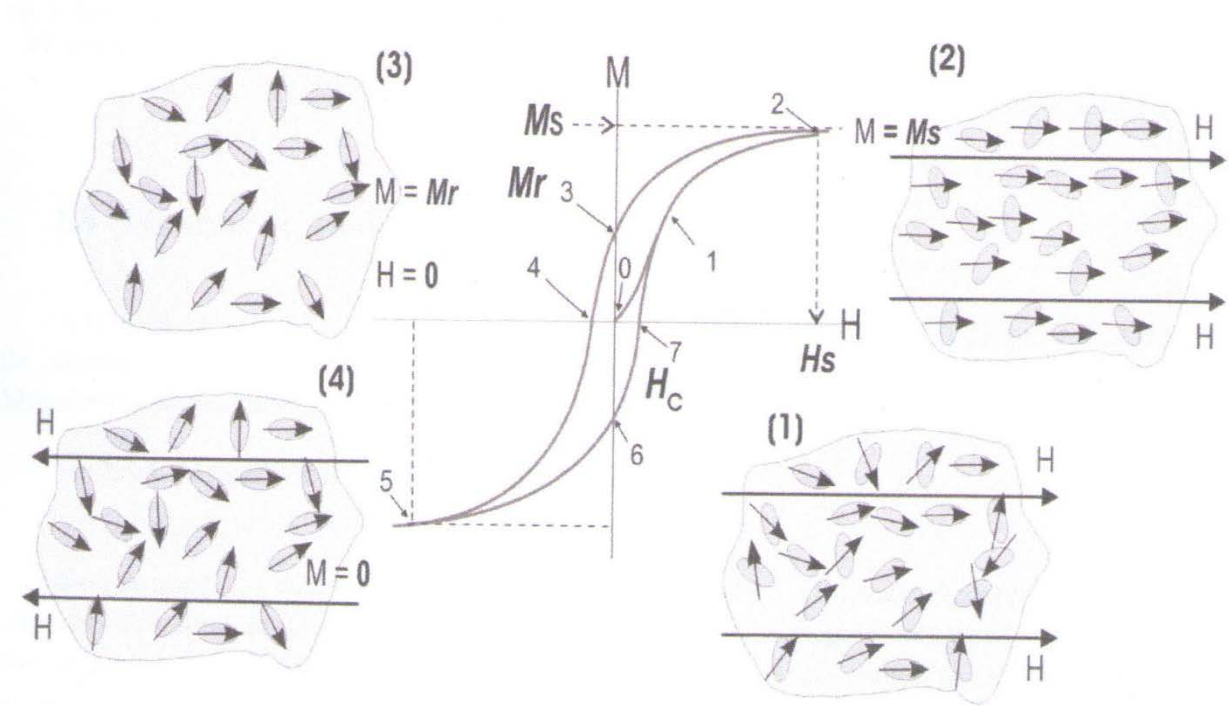

Fig. 2: Graph of the curve of the hysteresis cycle for a material with 5% by volume of elongated single-domain magnetite particles.

M = Magnetization M_S = Saturation Magnetization

H = Magnetic exposure field H_S = Saturation field

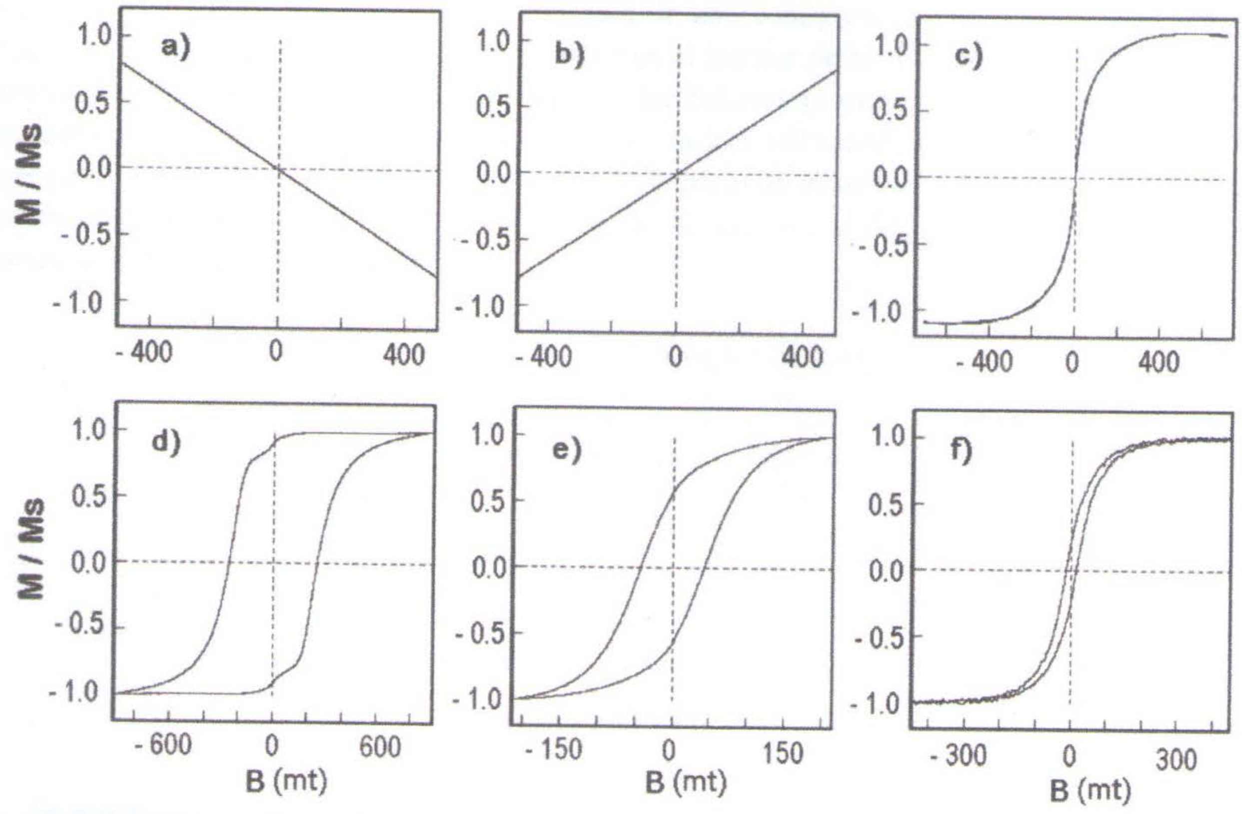

Fig. 3: Graphs of hysteresis cycles illustrating the behavior of diamagnetic (a) materials, (b) paramagnetic, (c) superparamagnetic, (d) imperfect antiferromagnetic: hematite, (e) ferrimagnetic: magnetite SD, (f) simple pseudodominium (PSD).

2.8. Magnetic properties of measurement techniques

The techniques used in the measurement of histesis cycles (M (H) curves) are the vibrating sample magnetometer (VSM), the Superconducting Quantum Interference Device SQUID and the Magnetic-Optical Kerr Effect Magnetometer (MOKE)

2.8.1. Vibrating Sample Magnetometer (VSM)

This technique was developed by Foner, who built the first Vibrating Sample Magnetometer (VSM) in 1959. Vibrating sample magnetometry is a technique that takes advantage of the property of some materials to acquire a net magnetic moment. The way in which the magnetic moments interact produces the appearance of magnetic domains (they are organizers of magnetic moments that have the same direction inside a material, since the vectorial sum of these moments as a result of the internal magnetic field of the material) and the different Classes of magnetic ordinances of matter (diamagnetic, paramagnetic, ferromagnetic, anti-ferromagnetic and ferrimagnetic). This technique gives us a cycle that shows the history of material magnetization, called the curve or cycle of hysteresis, which is a graphical representation of the different states through which the material passes through the work cycle.

The vibrating sample magnetometer performs magnetic induction measurements due to the relative motion between a set of detector coils and a sample. A magnetic sample oscillates (generally in the z-direction) in the vicinity of a detector coil configuration. This oscillation generates a magnetic flux change inside the coils. According to Faraday induction law a voltage is induced in the coils proportional to the magnetization of the sample. The magnetization of the sample can be varied using an external magnetic field H generated by an electromagnet to obtain the hysteresis curve of the sample. In Fig. 4, a simple outline of the location of the sample and the coils in the vibrating sample magnetometry equipment is shown.

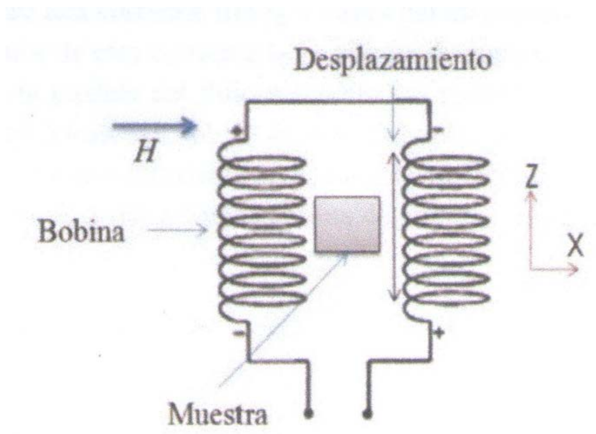

Fig. 4: Schematic layout of the direction of displacement of the sample and the location of the coils in the vibrating sample magnetometer.

The vibrating sample magnetometer (VSM) measures the corporate magnetic behavior of all parts of the sample, it is to say, the sample as a whole.

2.8.2. SQUID Magnetometry

The SQUID magnetometry (Superconducting Quantum Interference Device) is a very precise technique to detect very small magnetic fields. It is even possible to detect the magnetic fields of the brain that are on the order of 10^{-14}T. This experimental technique is one of the most technological applications Usefulness of superconductivity and is based in the so-called Josephson Effect [5]. The SQUID device consists of a superconducting ring with two Josephson junctions, it's to say, two semi-rings joined by an insulating layer. In this way, Cooper pairs, responsible for superconductivity, can cross this union through tunnel effect creating a supercurrent whose value is given by,

$$J = J_O \sin\delta$$

Where δ, is the phase difference of the wave functions that describe the quantum behavior of the pairs on either side of the union. J_O Depends solely on the structure of the union, whereas the phase difference depends on the potential applied between the ends,

$$\frac{d\delta}{dt} = \frac{2eV}{h} = \frac{2\pi V}{\phi_O}$$

In the above expression, $\phi_O = h/2e$ is the flow quantum whose value is $2.5x10^{-5}Wb$. When V = 0 the current is independent of time (Josephson DC effect), while if $V \neq 0$ a periodic modulation of the current occurs. (Josephson AC effect). It can be shown that the correinte through the joints varies periodically with the magnetic flux that crosses the ring, the ring being the quantum of flux,

$$J = J_O \sin\delta \cos\frac{e\phi}{h} + \frac{2V}{R}$$

The I-V characteristics of this device depend therefore on the magnetic flux. In a DC SQUID, normally a fixed current Io is established through the device, collecting the variation of the voltage with the flow. The value of this current Io is chosen so that the amplitude of the voltage is maximum. Usually the measurement of the magnetic flux is made directly with the SQUID device. The field to be measured passes through a sensing coil (superconducting coil), which is part of a flux transformer. The supercurrent created by the variation of flow in the detector coil runs through another coil with more turns called the input coil. The flow created by this coil is that measured by the SQUID.

2.8.3. Magneto-optical Kerr effect magnetometer

The Magneto-Optic Kerr Effect or MOKE consists of rotating the plane of polarization of a linearly polarized light beam after striking a magnetic sample. In a classical view of the physical phenomenon, the electrons on the surface of the sample are subjected to the electric field E of the light that impinges on an oscillatory movement, and additionally, they are subjected to Lorentz force due to the magnetization M of the material itself.

The optical behavior is directly related to the electrical permittivity ε, which incorporates elements directly dependent on the value of the magnetization $\bar{M}$ outside the main diagonal. As a result, a perpendicular oscillating component appears simultaneously with the magnetization M and the field E. This oscillation generates the emission from the surface of the linearly polarized light of less amplitude with a plane of oscillation rotated θ with respect to that of the incident light. In addition, an elliptically polarized light beam may appear from the overlap with the reflected beam without rotating.

Depending on the orientation E of the incident polarization beam, the magnet, and the plane of the surface, 3 types of Kerr effect measurement configuration can be distinguished: polar configuration (P-MOKE), longitudinal configuration (L- MOKE) and transversal configuration (T-MOKE). [6]

2.9. Frantz Isodynamic Separator

Magnetic separation is based on placing a mixture of particles in a magnetic field under ideal conditions, where all are subject to the influence of that field and other forces whose direction is an angle with that of field strength. These accessory forces are: magnetic, gravitational, centrifugal, friction or inertia, and attraction or repulsion.

The physical principle of the magnetic separator is as follows:

In order for the separation of one strongly magnetic particle to the other, a necessary but not sufficient condition must exist, where, being immersed in a magnetic field produced by the separator, the magnetic force (F_1^a) acting on the particle with Greater magnetic susceptibility must be greater than the sum of all opposing forces (F_2^a), likewise the magnetic force (F_1^b) acting on the least magnetically susceptible particle must be less than the sum of all opposing forces (F_2^b), therefore, in a separator the following relationships must be fulfilled:

$$F_1^a \geq \sum F_2^a$$

$$F_1^b \leq \sum F_2^b$$

The entrapment relation (R_a) is given by the following equation:

$$R_a = \frac{F_1^a}{F_2^a}$$

Where F_1^a = magnetic force and F_2^a = gravitational force, gravitational, centrifugal, friction, etc. With this condition if $R_a \geq 1$ the separation of the particles can be obtained.

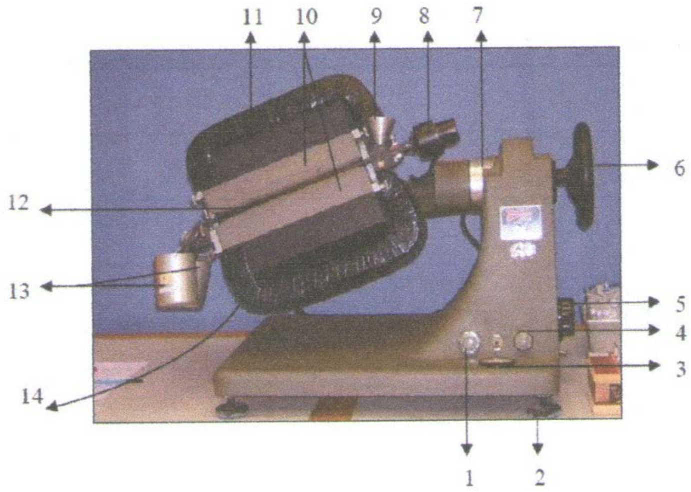

Fig. 5: Schematic of magnetic separator Isodinamico Frantz

Its parts are:

1. Adjustable base vibration control to calibrate equipment
2. Ammeter
3. Power On Indicator
4. Amp control
5. Side slope control
6. Degree indicator for side slope
7. Vibrator
8. Hopper
9. Imam poles
10. Core
11. Separator rail
12. Containers of material
13. Front slope control (located on back of computer)

Magnetic separation is a method of concentration that is used in those cases where different components of a mineral respond differently to an applied magnetic field.

2.9.1. Magnetic separation procedure

A certain number of samples were drawn, in order to obtain a size between 150 and 250 microns. Then, a current value is provided to the separator, which generates a magnetic field proportional to the value of the current, the sample is passed through this field, obtaining two results, each with different mass, the first one has greater magnetic susceptibility That the other, because the magnetic field is inversely proportional to the susceptibility, the second sample is passed again by the separator, but this time to a higher current value, repeating this procedure until there is nothing more to separate magnetically.

CHAPTER 3: Experimental Procedure

In this chapter, a description of the design and assembly of the vibrating sample magnetometer (VSM) will be made, detailing the principle of operation of each instrument used for its implementation.

The implementation of the VSM, in this work followed as a reference the thesis of Luis Aviles [1], gathering results of the work already mentioned, in order to verify and apply them to our interest.

3.1. Assembly Description

In Figure 1 the block diagram of the VSM can be observed, indicating the procedure to follow.

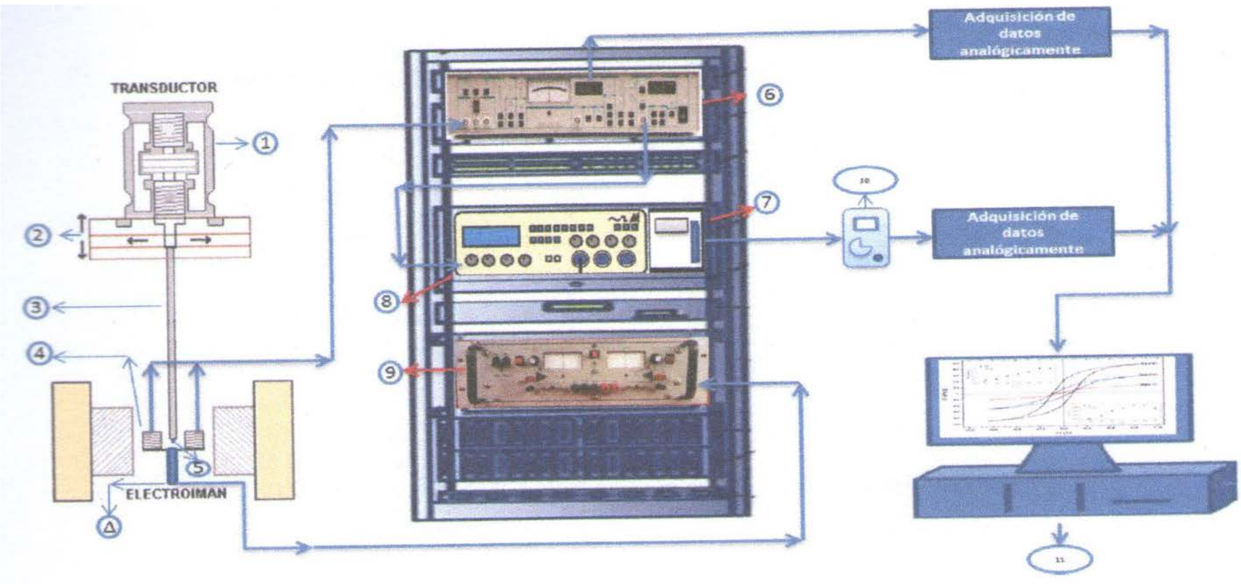

Fig. 6: Schematic diagram of the VSM implemented in the Nanostructured Materials Laboratory.

Where a = Hall sensor, the description and operating principle of each of these instruments will be described below.

3.1.1. Linear displacement electromechanical translator

There are three types of transducers, which are: mechanical, piezoelectric and electromechanical. The used in the present work is the Electromechanical Transducer of a Mossbauer linear displacement spectrometer of the brand ELCINT, model MD - 3. Its principle of operation is simple and resembles that of a double magnetic audio speaker. A rigid axle is mounted horizontally on a suspension to springs that allows it to travel only along the axle. Solidarity to this axis is the drive coil, which moves inside a uniform magnetic field provided by a permanent magnet. When cyclizing current through the coil, the displacement of the same takes place dragging to the axis. Also attached to the shaft is another coil which also moves within a second magnetic field. In this coil induces a voltage proportional to the speed of displacement of the same and therefore proportional to the speed of the source. This voltage is used as speed feedback. It should be noted that this feedback is a voltage proportional to the speed, not existing in the same information regarding the absolute position of the axis. This means that precautions must be taken with regard to the mean value and the speed reference. A schematic of the ASA model K3 electromechanical transducer (discontinuous manufacturing) is shown in Fig.2. The rigidity of the transducer is an important factor in order to avoid mechanical resonances. The linearity is determined by the uniformity of the magnetic fields, the tension of the suspension springs and the friction.

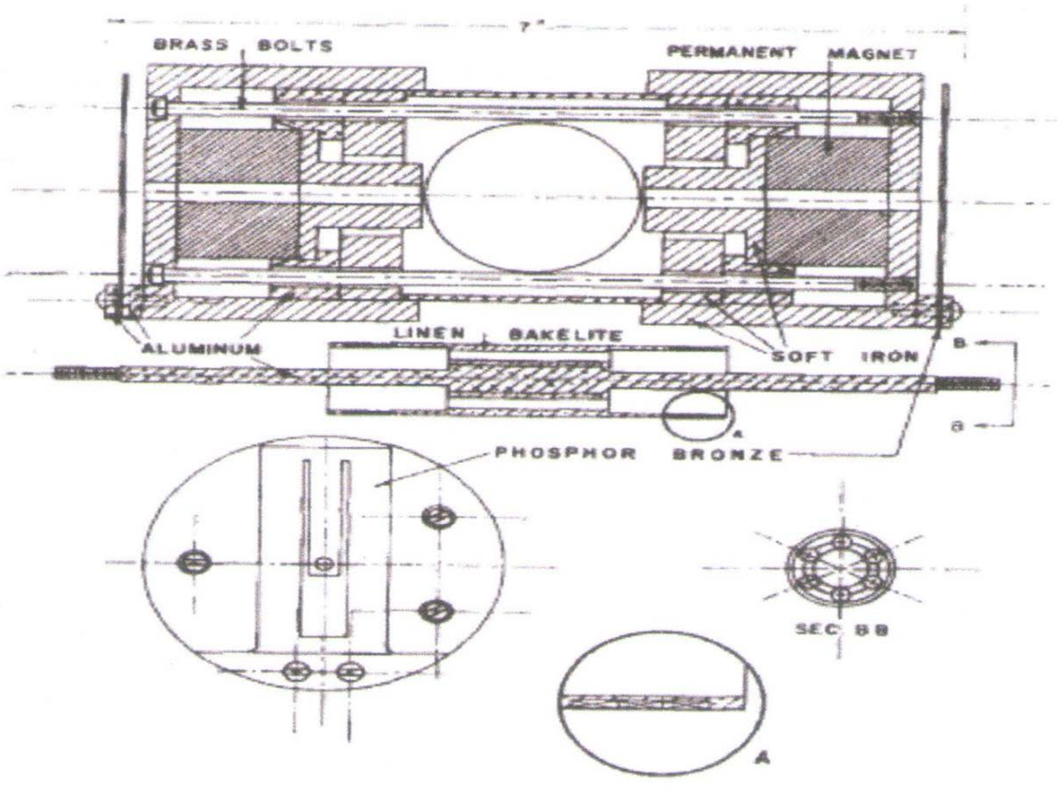

Fig. 7: Details of the electromechanical transducer described by Kankeleit 1964 [7]

Frequencies are supplied between 15 and 22 Hz to the transducer, because a desirable amplitude is obtained in the oscillation of the sample.

3.1.2. Support for electromechanical linear displacement transducer

A rigid table was designed, with the following measures:

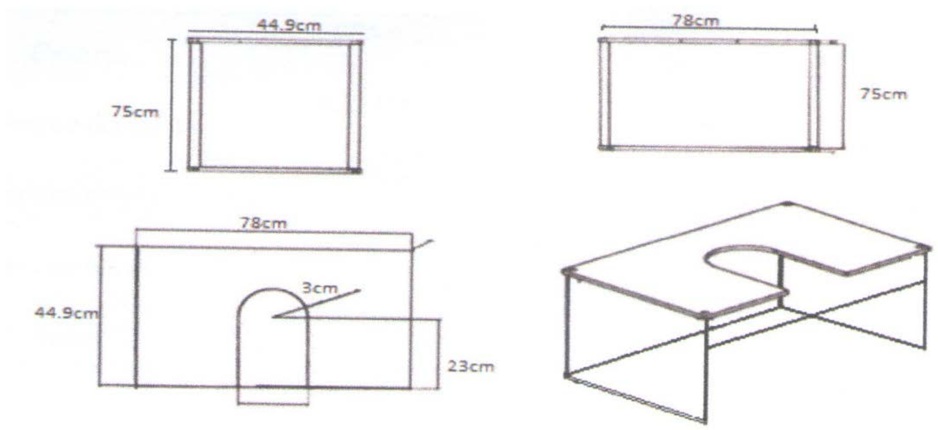

Fig. 8: Projection scheme of the main planes and rigid table dimensions

3.1.3. Aluminum rod

An aluminum rod (Al) of (57 + - 0.5) cm is used, the purpose is to hold the sample at one end and at its other end this is attached to the axis of the transducer, where this rod next to the sample is subjected to a Mechanical oscillation, whose purpose is to induce a voltage in a certain configuration of coils, the signal obtained in the coils has the same phase as the signal obtained from the sample, reason is used as a reference signal to the amplifier lock-in.

3.1.4. Detector coils

The arrangement of detector coils used in the implementation of the VSM, follows the configuration of Mallison, used that configuration of coils, for the effective characteristics that it presents, and are:

- The greater induction of voltage in each of the independent coils, this happens when we approach the sample to a coil, enters more lines of force inside this one and they leave of the other coil next to her.
- Offers greater sensitivity and insensitivity to the position and geometry of the sample.

The arrangement of coils that make up the Mallison configuration were made with 36 AWG copper wire, the coil has been coiled with 2000 turns, in order to obtain a greater induction signal. The Mallison configuration has four coils which are connected in pairs as shown in Fig. 9 each pair is connected in series but wound in opposite directions in order to obtain a net output signal, then these two pairs are connected Of coils obtaining an output signal which is four times the signal generated by a single coil.

Below are two tables of the dimensions of each coil and the characteristics of the Mallison configuration.

Chart 1: Detector coil dimensions

	Dimension
Caliber	36AWG
Wire diameter	0.1270 mm
External diameter	18 ± 5
Internal diameter	16 ± 5
Height	9 ± 5

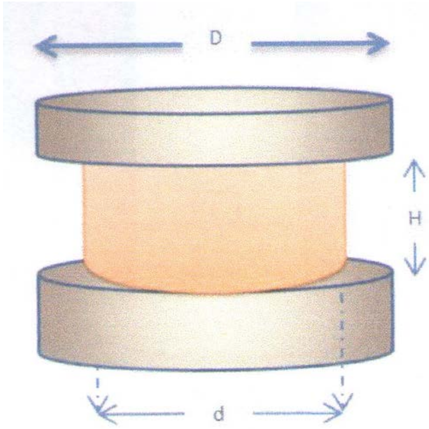

Fig. 9: Detector coil scheme

Chart 2: Mallison configuration characteristics

Configuration	**Mallison**
Number of coils	4
Number of spins per coil	2000
Total resistance	246.6Ω

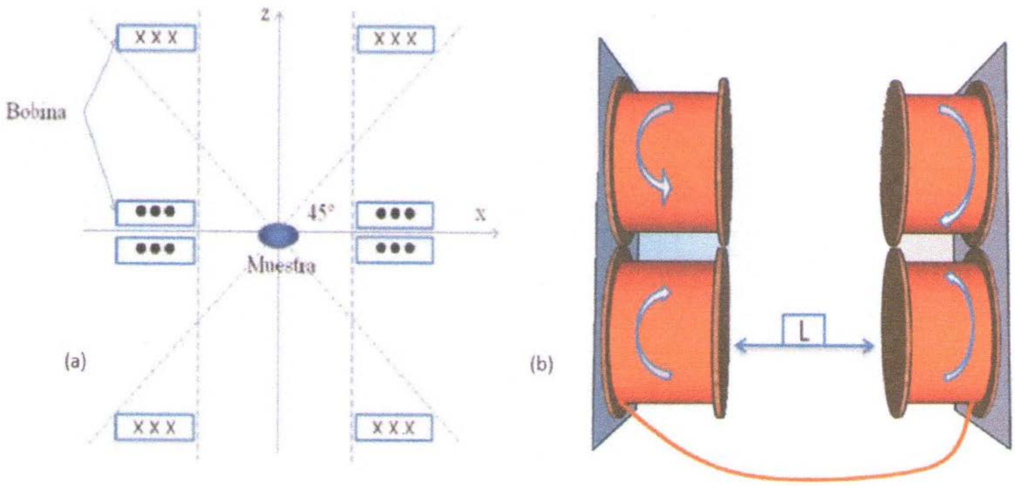

Fig. 10: a) Optimum configuration of Mellison (Figure obtained from the thesis of Luis Aviles) [1]b) Mallison configuration used (arrows indicate the direction of winding)

3.1.5. Sample

Different types of samples were used, such as a pure Nickel disk, and sample sand and Rare Earths, with an approximate size within the range of 150 to 150 microns, the mass used of each of them is approximately 204.6 mg.

3.1.6. LOCK-IN Amplifier

The Lock-I amplifier is an instrument used to detect and measure small AC signals and the presence of potentially high levels of noise. The principle of operation of the Lock-In consists of the modulation of the signal by some reference frequency wr, to then detect and amplify the signal. Mainly, a lock-in consists of electronic circuits that first multiply two signals and separate the result into two components and then send them through a low-pass filter. The following diagram clearly shows us how a Lock-In acts.

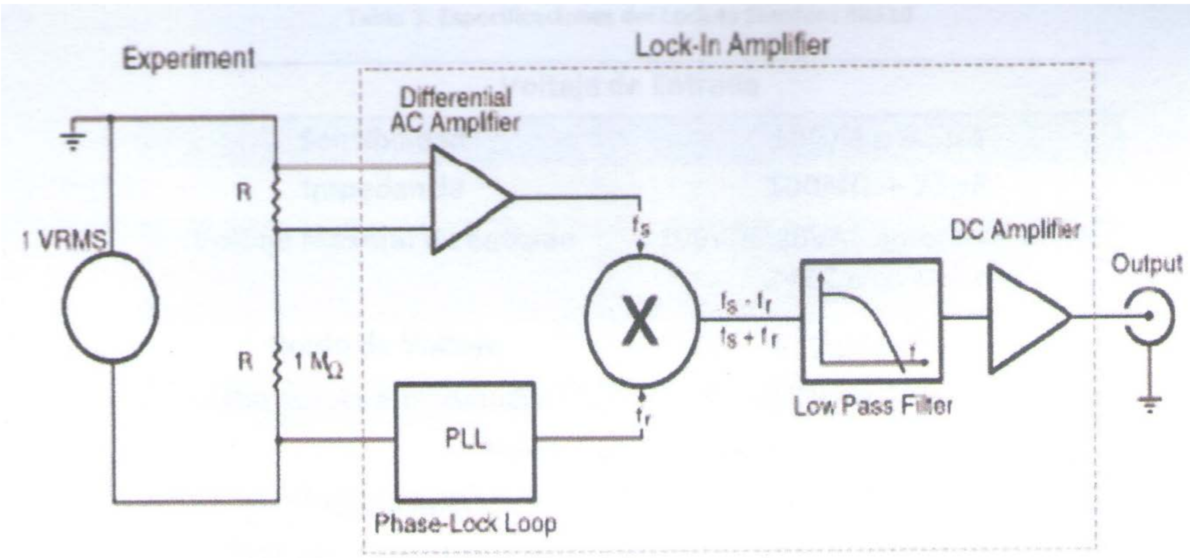

Fig. 11: Block Diagram of the Lock-In Operation

The AC reference signal is used to tell the Lock-In the exact frequency of the signal of interest at the input. The Lock-In Phase Locked Loop (PLL) circuit will follow this frequency without any user-made adjustment.

The output of the PLL provides a signal,

$$\cos(w_r + \phi)$$

The signal at the input is amplified by a high gain AC coupled amplifier. The output of this amplifier is multiplied by the output of the PLL in a Phase Sensitive Detector (PSD). This multiplication changes each frequency component of the input signal w_s by the reference frequency w_r, then the output of the PSD gives us the following,

$$V_{PSD} = \cos(w_r + \phi)\cos(w_s t),$$
$$V_{PSD} = \frac{1}{2}\cos[(w_r + w_s)t + \phi] + \frac{1}{2}\cos[(w_r - w_s)t + \phi]$$

It can be seen that the sum frequency component is attenuated by the low pass filter and the difference frequency components will pass through the narrow bandwidth filter and reach the DC amplifier. Since the typical low pass filter has time constants of up to 100 seconds, the Lock-In can reject noises whose frequency is up to 0.0025 Hz away from the reference frequency. This feature makes Lock-In an extremely useful tool in noise rejection and detection of known frequency signals, and is the reason why Lock-In is the most important tool within the VSM.

The Lock-In amplifier used in the present work is Model SR510 built by Stanford Research Systems, the following table shows the important specifications of the Lock-In used. [8]

Chart 3: Stanford SR510 Lock-In Specifications

INPUT VOLTAGE

Sensibility	$100fA\ a\ 0.5\mu A$
Impedance	$100M\Omega + 25pF$
Maximum input voltage	$100VDC, 10VAC\ damage\ threshold,$ $2VAC\ peak\ to\ peak$
Voltage noise	$7nV/\sqrt{Hz}$
Frequency response	$0.5Hz\ to\ 100kHz$

REFERENCE VOLTAGE

Impedance	$1M\Omega$
Frequency response	$0.5Hz\ to\ 100kHz$
Maximum input voltage	$1Vrms$

3.1.7. Teslameter – SANWA Digital multimeter Model Cd 772

To measure the external magnetic field a Magnetic Field Transducer - TMAGv2 is used. Which uses a Hall sensor which is connected to a digital multimeter that gives us analog values of voltage that is proportional to the measured magnetic field value. The transducer allows measuring magnetic fields up to 2T = 20000G.

3.1.8. (∆) – HALL Sensor

As current flows through the Hall sensor and approaches a magnetic field flowing in a vertical direction to the sensor, then the sensor creates a protruding voltage proportional to the product of the magnetic field strength and current. If the current value is known, then the magnetic field strength can be calculated. If the magnetic field is created by means of current flowing through a coil or a conductor, then the value of the current in the conductor or coil can be measured.

3.1.9. Function Generator

A function generator made by Cast model GF-8026 is used, which fulfills two important functions, the first is as a power supply of the transducer, providing a voltage and a frequency, the second is the TTL output synchronized with the signal Which feeds the transducer is used as a reference signal in the Lock-In which only amplifies the signals to its input, having the same frequency as its reference.

3.1.10. KEPCO bipolar operational power supply

As source of the electromagnet a KEPCO Model BOP 50-8M source is used, which provides us values of currents in the range of -8 to 8 A and voltage values from -50 to 50V.

3.1.11. Electromagnet

The external magnetic field is generated by an electromagnet manufactured by CENCO Instruments, powered by a KEPCO source, and configured in current mode. The schematic of the electromagnet is shown below.

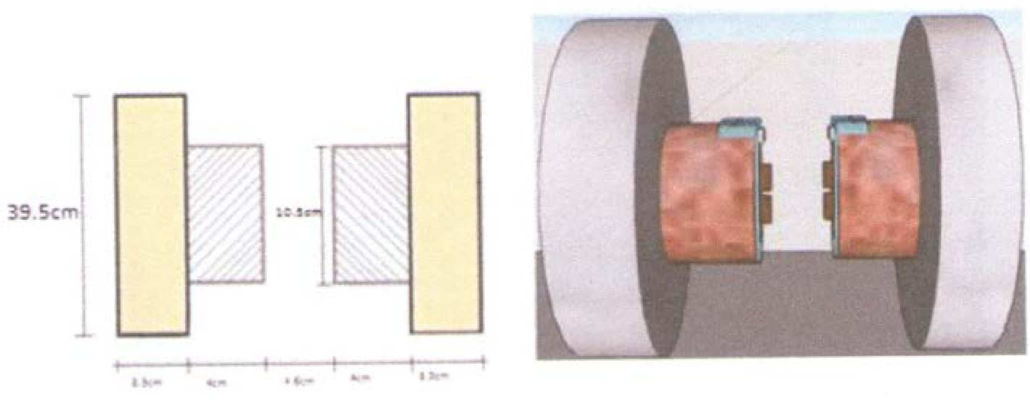

Fig. 12: CENCO Instruments Electromagnetic Scheme

CHAPTER 4: Resultados Experimentales

In this chapter, we will present data obtained in an analogous way, such as the mass of each sample to be measured, as well as obtaining data of the voltage value at the output of the Lock-In and the value of the magnetic field in (Oe) provided By the digital multimeter (doing the voltage conversion to Oe).

Results of sand and rare earth measurements will be presented and compared with a characterization of a sample of nickel taken as a reference.

4.1. Configuration of Detector Coils

The Mallison configuration is used for the measurement of all samples in the present work, the dimensions of this configuration are shown in Table 2. The coils increase the insensitivity to the geometry, position and size of the sample. The external field applied by the electromagnet is in the direction of the "X" axis as in Fig. 12.

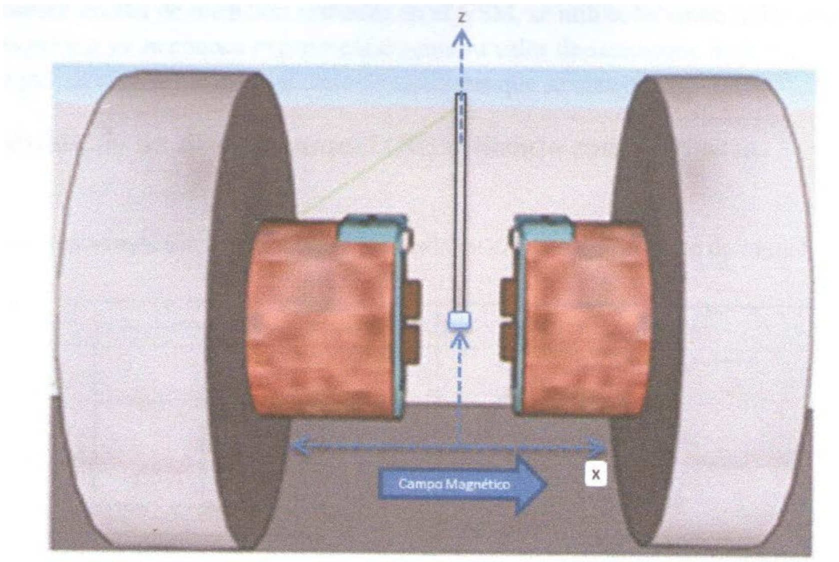

Fig. 13: Mallison configuration diagram and direction of the external magnetic field

As explained above in Chapter 4, the sample is placed on the end of a rod which is also subjected to mechanical oscillation. This suggests that the oscillation of the rod could generate signals because it is subjected to the external magnetic field, thus inducing signal in the coils. As the mass of the rod is much larger, it should not present a significant magnetization, because it would be a source of considerable error in the measurements. Taking into account this condition, a measurement of the Al rod is made, in order to observe its behavior under the influence of the external magnetic field to which it is subjected. The results are shown in the following figure. Fig. 13.

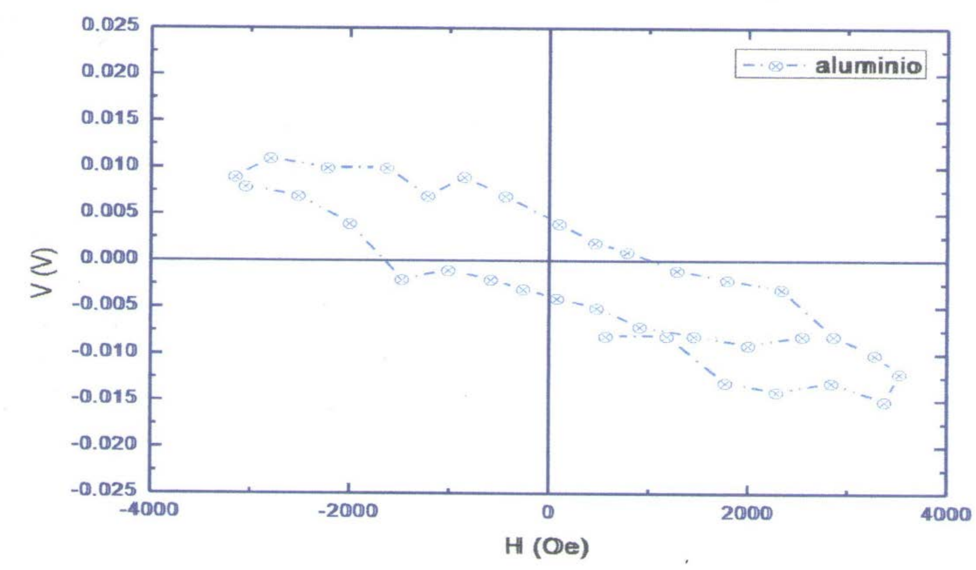

Fig. 14: Hysteresis curve of the aluminum rod

As the first measurement test performed in the VSM, Ni is used as a reference for all measurements because its saturation value is already known experimentally. A nickel disk was taken as the sample, which shows the cycle of hysteresis that was obtained.

4.2. Measurement of a nickel (Ni) disc used as sample

The following is a measurement made on the VSM of a nickel disk (Ni)

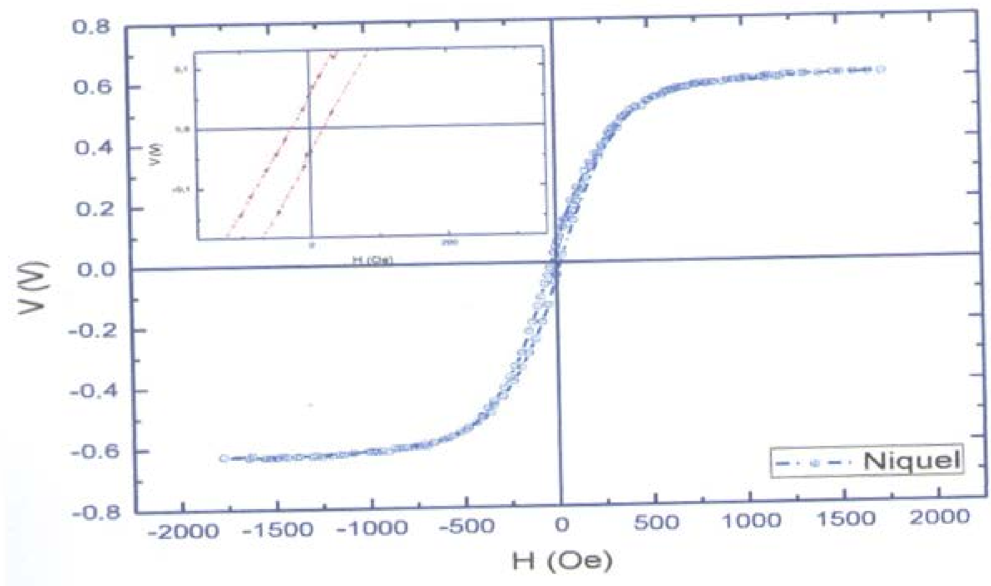

Fig. 15: Cycle of hysteresis of a nickel disk in the configuration of Mallison

By theory we know that $\mu_r = \frac{dB}{dH}$, by means of numerical calculation we can calculate the derivative and obtain the respective graph of Fig. 15, where we appreciate how the magnetic permeability of the material varies depending on the applied field. The nickel permeability curve is presented below.

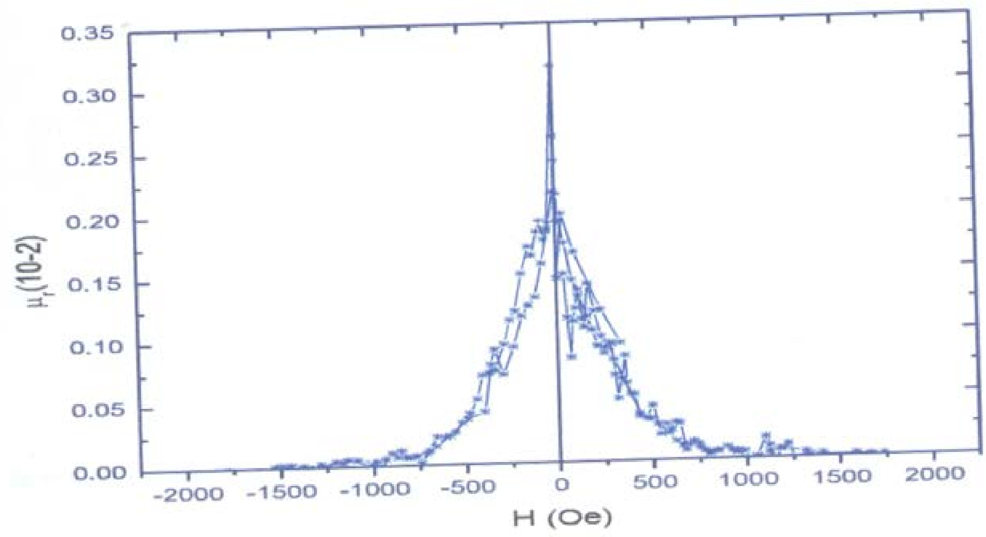

Fig. 16: Permeability of Ni depending on the field applied.

A measurement made on the VSM of a sand sample, magnetically separated with a Frantz Isodynamic magnetic separation equipment, is presented below. The magnetic field is proportional to the current value, ie at higher field strength, so that for very low current, separations of materials with a very large magnetic susceptibility value will be achieved. For the purpose of this work sand was taken as sample, which was subjected to a separation for a current value of I = 0.1A and the mass with which it was characterized is approximately 204mg.

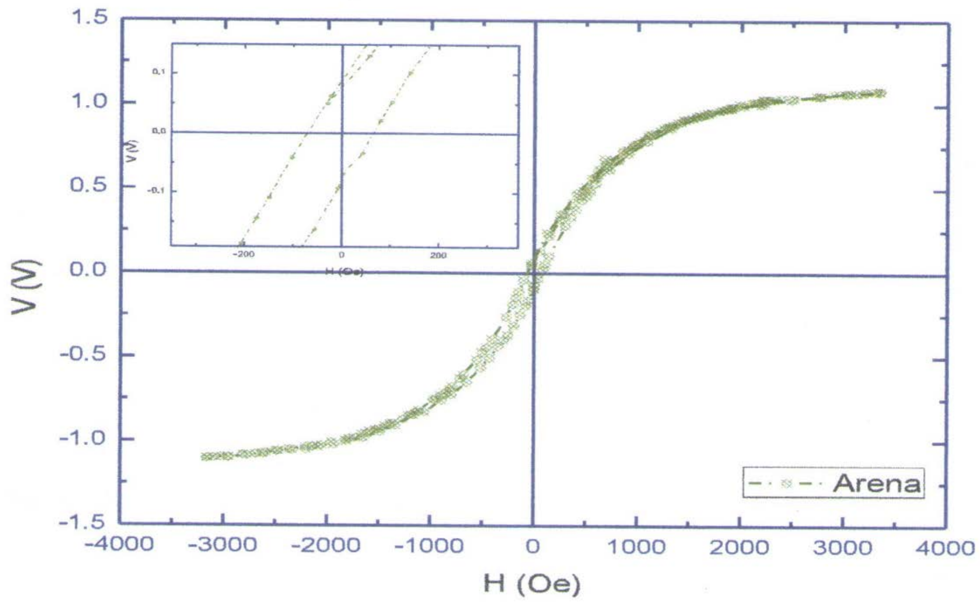

Fig. 17: Cycle of sand hysteresis magnetically separated at a current of 0.1 A in the Mallison configuration

Below is the curve of permeability of the sand.

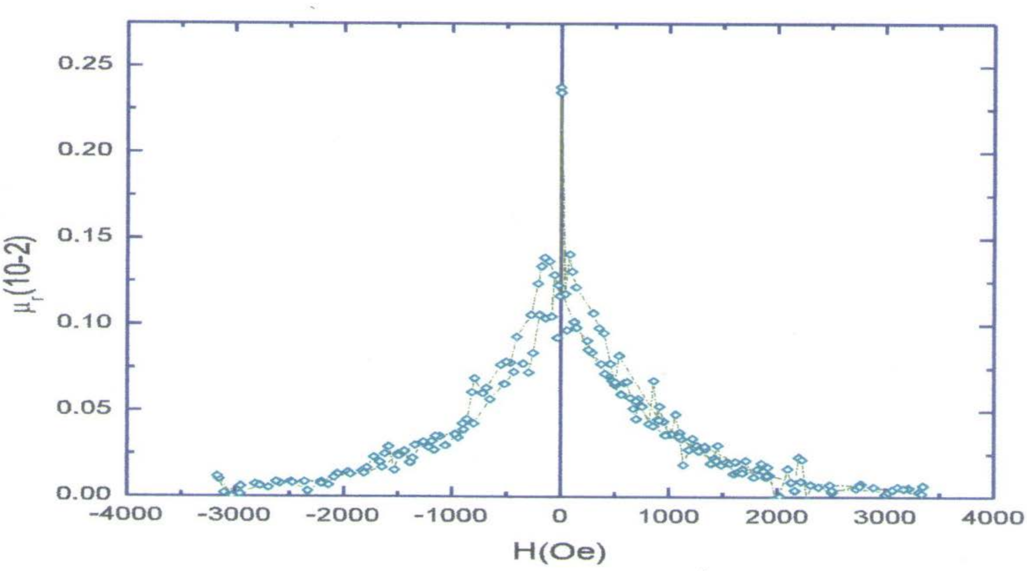

Fig. 18: Permeability of sand magnetically separated to a current of 0.1A depending on the field applied.

A measurement made on the VSM of a sand sample, magnetically separated with a Frantz Isodynamic magnetic separation equipment, is presented, the separation was carried out at a current of i = 0.2A and the mass with which it was characterized is approximately 240mg.

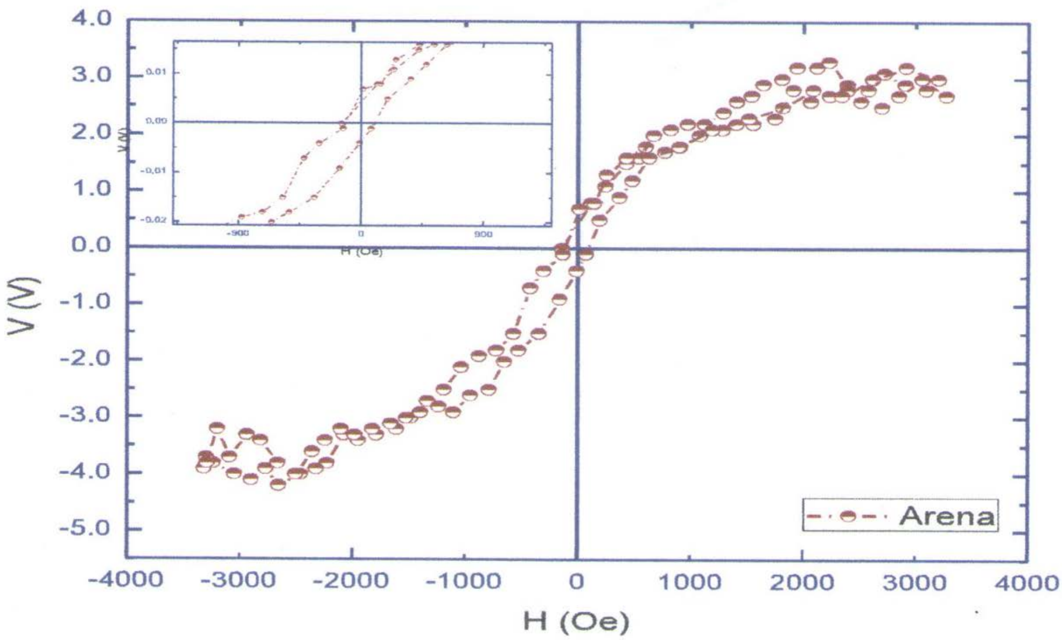

Fig. 19: Sand hysteresis cycle magnetically separated at a current of 0.2 A in the Mallison configuration.

Below is the permeability curve:

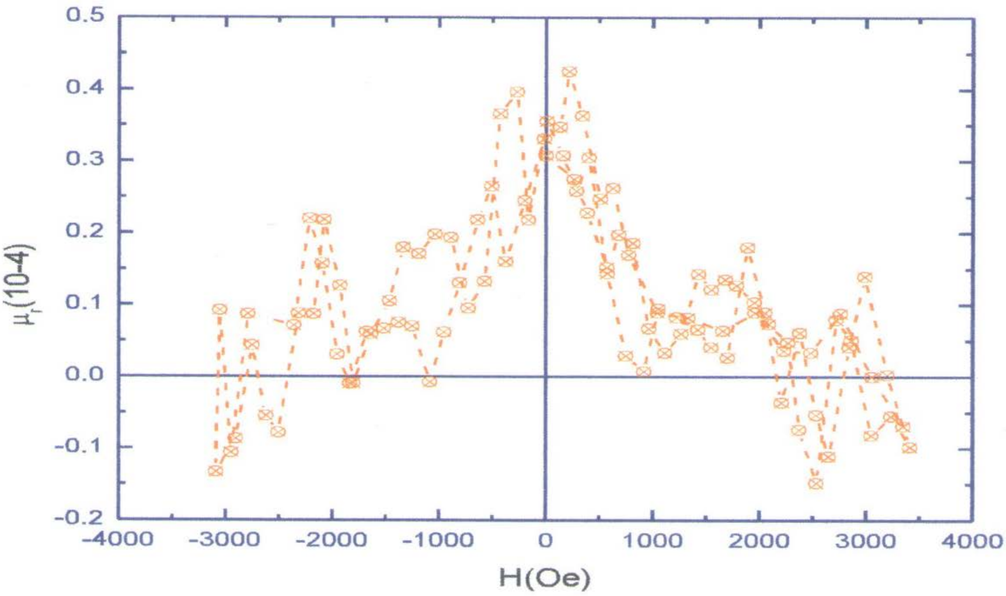

Fig. 20: Permeability of sand magnetically separated at a current of 0.2 A depending on the field applied.

Now we present the hysteresis curves of the sand sample, but magnetically separated to different currents, where we can see that both have different slopes, which is identified, different permeability, one greater than the other.

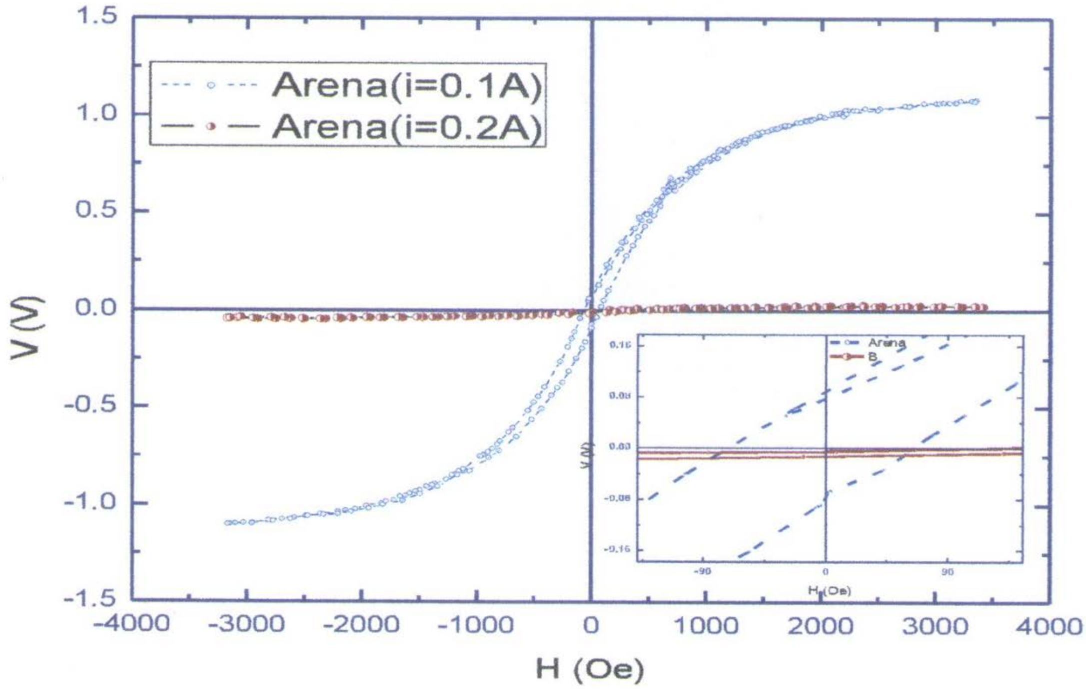

Fig. 21: Sand hysteresis cycles for different current values in the Mallison configuration.

A measurement made on the VSM of a Rare Earth sample (No. 53), magnetically separated with a Frantz Isodynamic magnetic separation equipment, is presented below. The separation was performed at a current of i = 0.1A and a mass of about = 204mg.

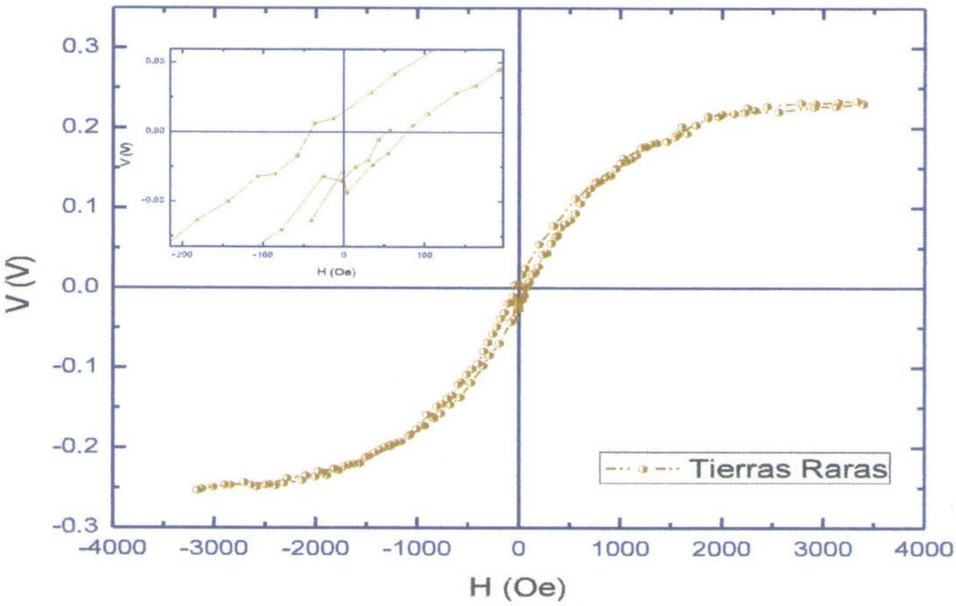

Fig. 22: Cycle of hysteresis of a Rare Earth Sample magnetically separated to a current of 0.1A in the configuration of Mallison

Below is the permeability curve:

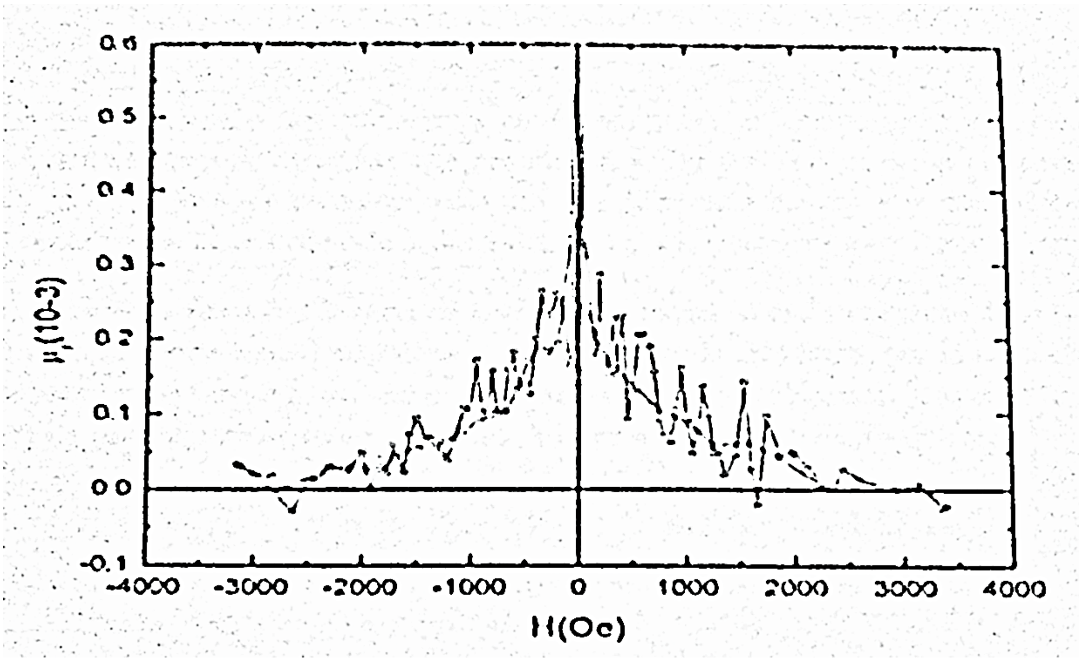

Fig. 23: Permeability of Rare Earth magnetically separated to a current of 0.1A depending on the applied field.

Now we present the hysteresis curves of all the samples that we use in this work, each magnetically separated to different currents, where we appreciate the different slope of each curve, which means the different permeability that they present.

It should be noted that all measurements were made with a reference frequency of approximately 20 Hz, that same frequency is where the sample oscillates between the coils of the Mallison configuration.

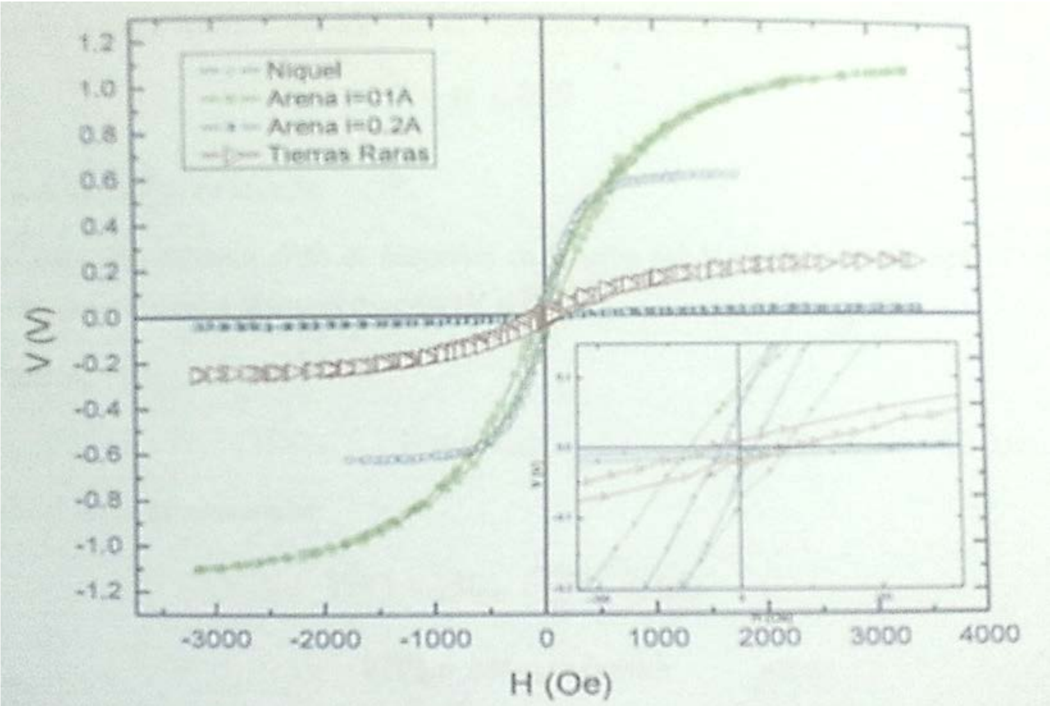

Fig. 24: Cycles of hysteresis of all the samples used in this work in the configuration of Mallison.

CHAPTER 5: Discussions

In this chapter we will focus on the behavior of magnetic moment in a system of particles, which will allow to describe qualitatively the magnetic behavior of a set of particles. Each one with different geometry and magnetic moment, taking into account that in a particle could exist two or more different magnetic moments.

With reference to the curve of hysteresis of the nickel, which has as theoretical and experimental data its magnetic permeability of saturation, therefore we will now concentrate on the sample of sand whose composition probably contains diamagnetic, paramagnetic, ferromagnetic, anti-ferromagnetic materials and ferrimagnetics.

5.1. Estimation of the magnetic moment of a system of particles

Assuming as an ideal case, having as samples to materials with grains (SD or mono domain) and grains (MD or multidomain), being a set of agglomerated particles that do not interact between them and that are separated between them, such is the case that (Magnetite SD), Imperfect Anti-ferromagnetics (Hematite) and other compounds ϖ (where ϖ are diamagnetic, paramagnetic, ferromagnetic materials).

Which the analysis for this case would be the following:

Let be a volume V and let $\vec{m}_1$ be the magnetic moment of each particle. The total magnetic moment within V is the vector sum of all $\vec{m}_1$, then we define magnetization as,

$$\vec{M} = \frac{\sum_i \vec{m}_1}{V_i}$$

Is measured in emu/cm^3 or in emu/g.

In our case we have a cycle of hysteresis as a function of V vs H, so here the voltage is directly proportional to the magnetization ($V \,\alpha\, \vec{M}$).

Where we have,

$$V = \varrho M_{Sat} \dots\dots\dots (1)$$

Calculating the conversion factor,

$$V(V) = \varrho M_{Sat} \left(\frac{emu}{g}\right) . m(g),$$

$$V(V) = \varrho M_{Sat} m(emu),$$

$$\varrho = \frac{V}{mM_{Sat}} \left(\frac{V}{M_{Sat}}\right), \dots\dots\dots (2)$$

Where:

V = Voltage (in V)

M = Magnetization $\left(\frac{emu}{g}\right)$

ϱ = Constant of proportionality

m = Mass

For our case, we use a nickel disk as a pattern, we have the voltage value which saturates, its saturation magnetization and its mass, with these values we obtain the value of constant proportionality (ϱ), and we will use it for all the other samples.

Following with our analysis, in the curve of hysteresis presented in illustration 10, we have both the saturation voltage, then having as data these values we do the following,

$$\vec{M}_{Sat}^{Total} = \vec{M}_{Sat}^{Fe_3O_4} + \vec{M}_{Sat}^{Fe_2O_3} + \vec{M}_{Sat}^{\varpi}. \tag{3}$$

Then from equation (2),

$$\frac{V_{Sat}}{m_{Total}\varrho} = \frac{V_{Sat}^{Fe_3O_4}}{m^{Fe_3O_4}\varrho} + \frac{V_{Sat}^{Fe_2O_3}}{m^{Fe_2O_3}\varrho} + \frac{V_{Sat}^{\varpi}}{m^{\varpi}\varrho}. \tag{4}$$

From this equation, we have theoretical and experimental values of the saturation voltage of both MAGNETITE and HEMATITE, t the mass percentage that is present in the sample, as well as the proportionality constant (ϱ),

Multiplying the equation (4) by the m_{total}, we have,

$$V_{Sat} = \frac{V_{Sat}^{Fe_3O_4}}{\%m^{Fe_3O_4}} + \frac{V_{Sat}^{Fe_2O_3}}{\%m^{Fe_2O_3}} + \frac{V_{Sat}^{\varpi}}{\%m^{\varpi}}$$

$$V_{Sat} - \left(\frac{V_{Sat}^{Fe_3O_4}}{\%m^{Fe_3O_4}} + \frac{V_{Sat}^{Fe_2O_3}}{\%m^{Fe_2O_3}}\right) = \frac{V_{Sat}^{\varpi}}{\%m^{\varpi}}. \tag{5}$$

With this equation we come to see how is the contribution of each particle to the signal formation obtained in the form of the curve of hysteresis.

From Fig. 16 and 18 we can analyze the following, the obtaining of these curves was obtained to the magnetic separation realized by a magnetic separator Frantz Isodynamic, the procedure that followed was the following one:

- A quantity of sample is passed and a determined value of current, with which we obtain in the separator a magnetic field whose varies with the current value that is provided to the separator.
- For our first hysteresis curve, a value of 0.1A is given to the separator which gives a relatively low magnetic field value, then the sample is passed and the procedure is repeated several times, in order to obtain a sample With magnetic and nonmagnetic part, concentrating now on the magnetic part, this sample has a certain magnetic susceptibility, corresponding to a fixed value of current, and the nonmagnetic sample that was obtained is passed through, the

separator, but this To a higher current value for our case was 0.2A, the same procedure is repeated, as the previous case, so we obtain a different value of magnetic susceptibility, this due to the properties of the materials that are in each One of the separations performed, and this is verified in Fig. 20 where we can see how the hysteresis curve is at different current values.

As we know that in our sample both Magnetite and Hematite are present, assuming that in Fig. 16, this sample has Magnetite, this because its magnetic susceptibility is relatively high, and Fig. 18 is the Hematite that has A relatively low susceptibility.

Below is a table of susceptibilities.

Chart 4: Susceptibility of minerals

Minerals	Composition	Magnetic Susceptibility
Magnetite SD		< 1500
Magnetite MD	Fe_3O_4	< 3000
Magnetite PSD		< 5000
Hematite	αFe_2O_3	$2 - 50$

5.2. Importance of size

The size of the particles in this work is 150 to 250 microns, where the magnetic behavior of the small particles, of a single domain (SD or monodomain), is very different from that of the larger particles or multidomain (MD), even if The composition and the quantity of the ferromagnetic materials is the same. In such a way that the magnetization of the SD materials exposed to an external field H parallel to the direction of easier magnetization is only acquired until the field H is very high. This happens subtly when the material reaches its maximum possible magnetization (magnetization of saturation "Ms"), this being also its magnetization remaining; The same is repeated if we then apply a field H in the opposite direction (Fig. 24a). In contrast, the MD materials show immediate changes even in low H-fields, these changes continue to increase as H increases until reaching its Ms, when the field is removed the material retains a "Mr" remnant magnetization, just as in the previous case, The same behavior is repeated if we then apply a field in the opposite direction (Fig. 24b). Some materials with MD grains exhibit similar behavior to SD due to crystalline imperfections that prevent the interaction that normally exists between the different domains of a particle. This behavior is termed as a simple pseudo domain (PSD or pseudomonodomain) and appears to be common in grains no larger than 3 domains. [4]

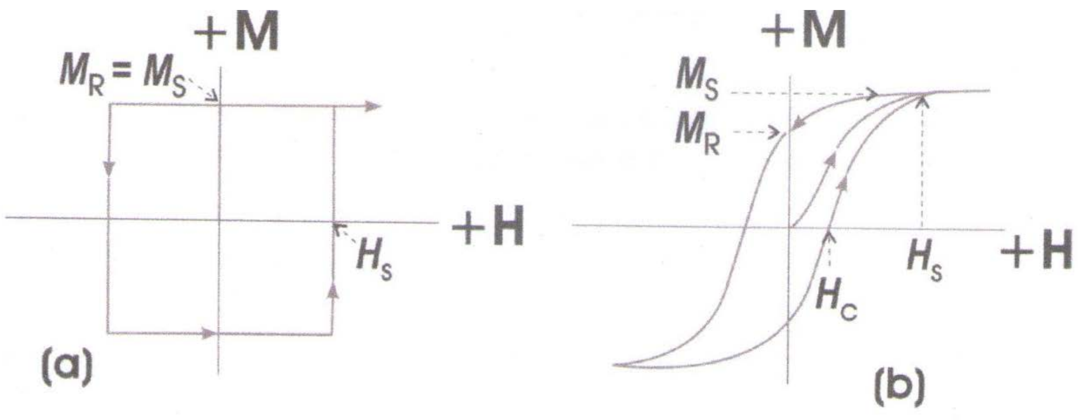

Fig. 25: Behavior of SD (a) and MD (b) materials described by hysteresis cycles

CHAPTER 6: Prospects for future work

6.1. VSM Improvements

The coils used will be optimized, increasing the number of turns to approximately 10'000. With a uniform way of winding the coils, because an ideal way to make the coils requires that the copper wires be coiled as evenly as possible, in order to have a more inductive signal to the coil output.

Elaborate a filter with the objective of eliminating noise coming from electromagnetic signals of the environment, thermal noise and small momentary instabilities that the magnetic field can present. Also, to elaborate an amplifier with the function of increasing the signal induced by the sample increasing the sensitivity of the VSM, giving the sufficient gain to the signal of reference so that it can be used by the amplifier Lock-In. Both the filter and the amplifier will be placed at the output of the detector coils.

The Electromechanical Transducer will be replaced by a system of two audio speakers, with a through axis, this will allow us to passively compensate the vibration, that is, you will have a signal with smaller spurious components. An audio amplifier will be assembled in charge of supplying the current necessary for proper operation of the double speaker.

6.2. VSM Automation

In this work, the tapping was performed manually, so the automation of the VSM requires a means of communication that allows to connect all the equipment to the computer, and of a softwater that controls the devices and that acquires, operates and saves the data for Present them graphically. To communicate the equipment to the computer, a GPIB (General Purpose Interface Bus) controller card will be used and software will be developed.

6.3. Characterization of rare earth sample

With the already improved and automated system, we will proceed to characterize Rare Earth samples, and magnetic susceptibility graphs will be obtained based on the current at which the sample was obtained.

CHAPTER 7: CONCLUSIONS

In the present work we tried to achieve a double objective: on the one hand the magnetic characterization through the measurement of the cycles of hysteresis of samples as minerals. On the other hand, to use the technique of Vibrating Sample Magnetometer (VSM), with the purpose of certifying the process of magnetic separation in minerals.

A VSM was implemented, in order to characterize magnetic samples in the form of powders or grains.

A nickel disk is taken as a reference, because its magnetic properties are already known, so that we can analyze the magnetic compaction of the other samples.

It was possible to observe the different curves of hysteresis of each sample, where the different magnetic susceptibilities are appreciated, as well as the magnetic permeability of each material present in these samples.

It is of great importance to analyze the size of the particle, mainly for the domain that can present, either SD or MD.

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