2.2.4.1.- Vibrating Sample Magnetometry

2.2.4.1.1.- Origins and fundamentals of the technique

The vibrating sample magnetometer (VSM) was invented independently by Van Oosterhout and Foner in 1956 [46,47].

The principle of this magnetometer is to measure the electromotive force induced by a ferromagnetic sample when it is vibrating at a constant frequency, under the presence of a static and uniform magnetic field. According to Faraday's law, the electromotive force induced in a coil, V_{fem}, is proportional to the temporal variation of the magnetic flux passing through it:

$$V_{fem} = -\frac{d\Phi}{dt} \qquad (2.6)$$

where Φ is the magnetic flux and t is time [48,49].

If we have a solenoid consisting of N turns of constant cross section A, the magnetic induction inside the coil, B, can be written as follows:

$$B = \frac{\Phi}{A} N \qquad (2.7)$$

One therefore obtains:

$$V_{fem} = -NA\frac{dB}{dt} \qquad (2.8)$$

Since the sample is vibrating, the moment can be considered as $m(t) = m_0 \sin(ft)$, where m_0 the moment amplitude and f is the frequency. Since $B = \mu_0 H + M$, $M = m/V$ and H is constant, dB/dt becomes $dB/dt = (\mu_0 f/V)\cos(ft)$.

Thus, the amplitude of the induced voltage is proportional to the amplitude of the magnetic moment of the sample:

$$V_{fem} \propto f\, m_0 G(z) \qquad (2.9)$$

where $G(z)$ is a function that defines the dependence of V_{fem} with respect to the sample position in the holder relative to the coils and, hence, it depends on the design of the coils.

The VSM measures the difference of magnetic induction between one region of space with the sample and another without the sample, thus allowing calculation of the magnetic moment, m, generally with a sensitivity in the order of 5×10^{-6} emu.

2.2.4.1.2.- Experimental Method and working conditions

The measurements have been carried out in an Oxford Instruments 1.2 VSM, at the *Laboratori de Propietats Magnètiques i tèrmiques* of the *Universitat Autònoma de Barcelona* (see figure 2.5).

Magnetic hysteresis loops can be performed up to 11 kOe, at different temperatures between room temperature and 850 K. The magnetometer can work either in isothermal or continuous heating regime. It is also possible to measure the temporal or angular dependence of the magnetization. The magnetic field is generated by an electromagnet, with poles of 177 mm in diameter. The gap between the two poles is of approximately 40 mm.

Figure 2.5: Photograph of the vibrating sample magnetometer used in the present study

The sample is located at the end of a rigid holder which is oscillated in the vertical direction, perpendicular to the magnetic field. The oscillation amplitude ranges from 1 to 2 mm and the frequency is 67 Hz.

The oscillations induce an AC signal on the sense coils, which, as has been shown, is proportional to the magnetic moment in the sample. The VSM used in this work has, in fact, four detection coils, oriented perpendicular to the magnetic field. This configuration allows better sensitivity and larger region of homogeneity. Shown in figure 2.6 is a schematic diagram of the VSM.

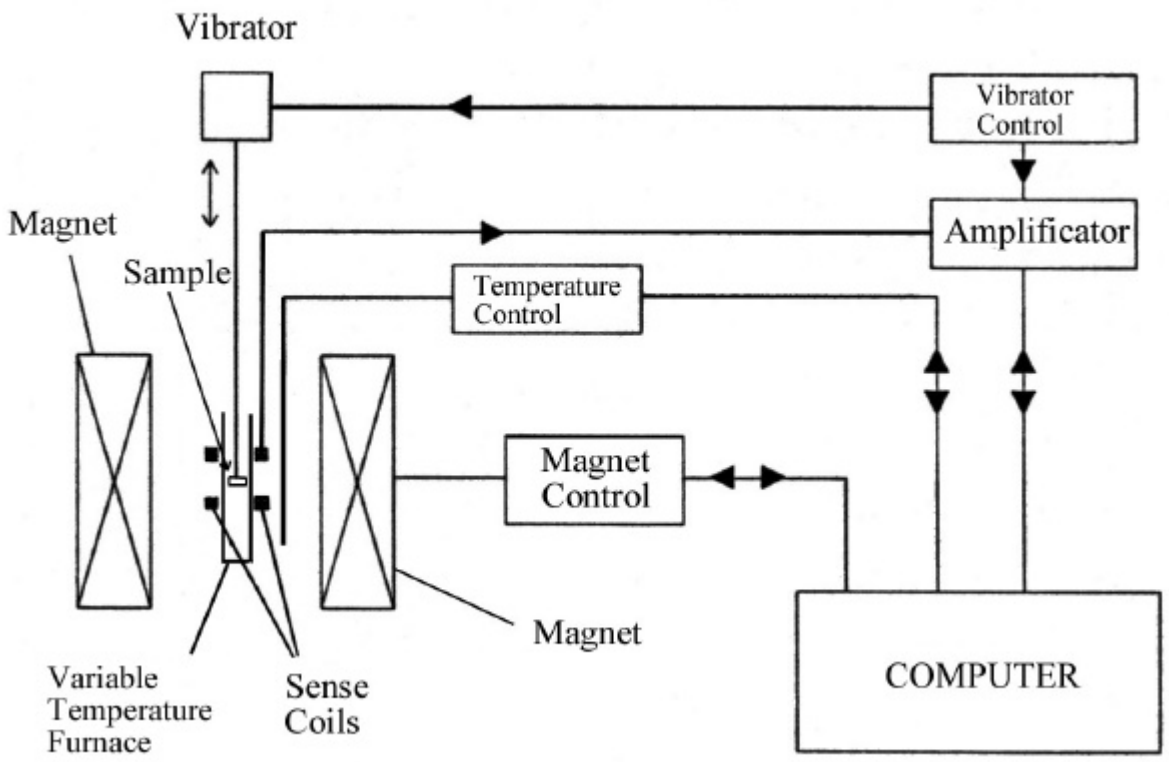

Figure 2.6: Schematic diagram of the different components of the VSM

The powders are weighted, pressed into a small quartz container and finally mounted at the end of a rigid ceramic rod. This allows measurements at high temperatures. To avoid the movement of the powders inside the tube they are compacted using quartz wool. To get optimum signal the samples are previously centered in angle and height, applying a field of 2 kOe. Subsequently they are demagnetized using a sequence of alternating decreasing fields.

The heat treatments were carried out under vacuum (10^{-3} mbar). Some technical specifications and working conditions are the following:

- Maximum Field: 11 kOe
- Magnetic field error: 2 Oe.
- Magnetic field inhomogeneity: < 1%.
- Temperature range: 300 – 850 K.
- Temperature sensitivity: 0.1 K.
- Vibration amplitude: 1.5 mm.
- Vibration frequency: 67 Hz.
- Sample weight: ≈ 40 mg.

The VSM was calibrated in magnetic moment and sample temperature. The magnetic moment calibration was carried out using a Ni standard (sphere) with known saturation magnetization (M_S = 54.4 emu/g). The temperature calibration was performed using ferromagnetic alumel and Ni standards, with Curie temperatures of 438 and 627 K, respectively.

2.2.4.2.- Extraction Magnetometer

2.2.4.2.1.- Origins and fundamentals of the technique

The physical principles of this magnetometer are similar to the vibrating sample magnetometer, i.e. it is also based on Faraday's law. This technique has been used for a long time, i.e. simple designs were already used in the late eighteen hundreds. In general, when a magnetic sample placed at the center of a coil is removed to far from the coil a voltage V_{fem} is induced in the coil, which is the temporal derivative of the magnetic flux produced by the sample (see equation 2.6) [49].

We can schematize the experimental procedure in a very simple way as shown in figure 2.7. When the solenoid is producing a magnetic field H_a and the sample is located at point M (i.e. inside the reading coil) the total flux through the coil is:

$$\Phi_1 = BA = (H_a + 4\pi M)A \qquad (2.10)$$

where A is the search coil (or sample) area (for simplicity, the demagnetizing field created by the sample is neglected). If the specimen is moved to M', then the final flux through the coil will be:

$$\Phi_2 = H_a A \qquad (2.11)$$

The detected flux change will therefore be:

$$\Phi_2 - \Phi_1 = (-4\pi)MA \qquad (2.12)$$

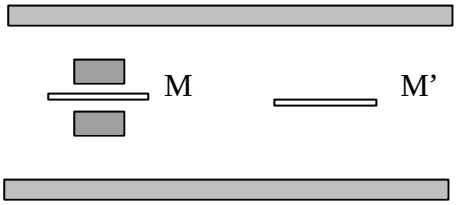

Figure 2.7: Schematic diagram of extraction method with solenoid

2.2.4.2.2.- Experimental Method and working conditions

The extraction magnetometer used in this work is located in the Grenoble High Magnetic Field Laboratory (GHMFL). The magnetometer allows measurements between 1.4 and 400 K in high DC magnetic field, e.g. 230 kOe, created by Bitter magnets [50]. The magnets are made from perforated copper "pancake" segments which constitute a large helical coil, cooled by a continuous flow of water at high pressure. This magnet consists of two concentric coils, each of them using an electrical powder of 5 MW. The use of such large magnetic fields was needed in order to ensure complete saturation of $SmCo_5$ powders.

The *M* measurement rod is a simple thin stainless steel tube, supporting a plastic holder. A general cross-section of the magnetometer in the Bitter magnet is shown in figure 2.8. The main characteristic is a pneumatic jack which moves the sample between the two sets of measuring coils.

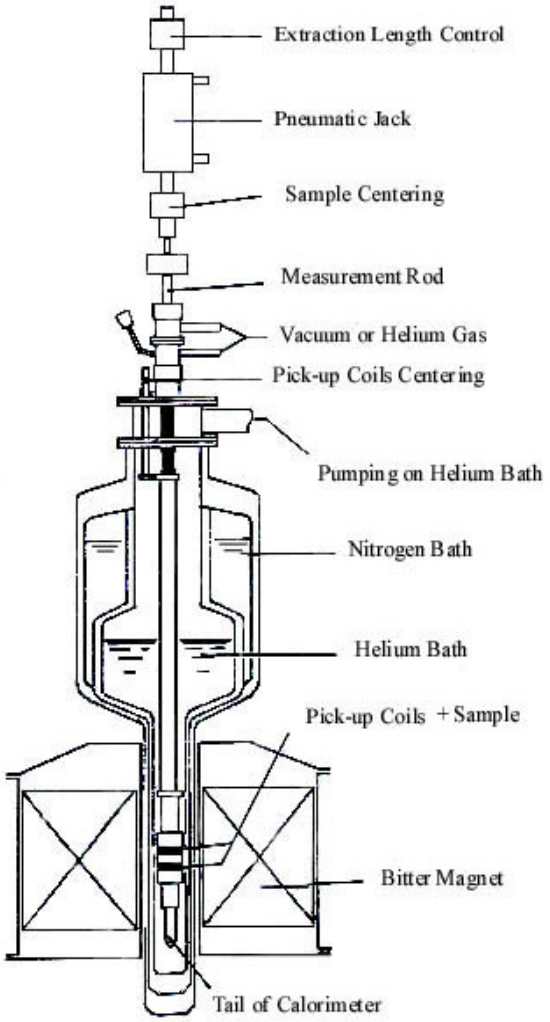

Figure 2.8: General cross-section of the extracting magnetometer

The sensitivity lies around 10^{-3} emu with a relative accuracy between 0.01 and 0.001 %. Standard isotherm *M(H)* or iso-field studies can be performed. In order to avoid movement of the powders during magnetic measurements, they were tightly pressed and put into small plastic containers. The average weight of the samples measured was similar to that of VSM measurements (i.e. 40 mg). In this experimental setup the magnetic sample is placed in a constant field between previously compensated pick-up coils connected in series-opposition, as shown in figure 2.9. The sample is then moved from one set of coils to the other. The

electromotrive force induced when the sample goes out of one set of coils and enters the other is integrated and the signal averaged to obtain the magnetization.

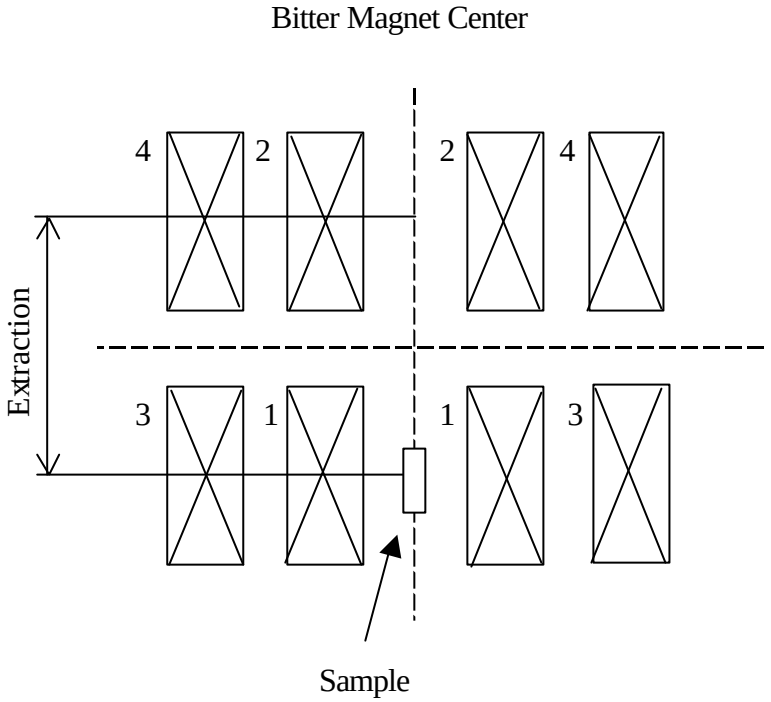

Figure 2.9: The pick-up coils and sample configuration.

Hysteresis loops were carried out in $SmCo_5$ powders milled alone or with NiO or CoO. The loops were carried out at room temperature, although some as-milled powders (of $SmCo_5$ milled alone and with CoO) were also field cooled (H = 50 kOe) and some loops were performed at 100 and 30 K. Some technical specifications and working conditions are the following:

- Maximum Field applied in the hysteresis loops: 230 kOe.
- Magnetic field error: 10 Oe.
- Temperature sensitivity: 0.5 K.
- Sample weight: ≈ 40 mg.

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