Long-pulse magnetic field facility at Zaragoza

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Abstract. The long-pulse magnetic field facility of the Laboratorio de Magnetismo - Instituto de Ciencia de Materiales de Aragón (Universidad de Zaragoza-CSIC) produces magnetic fields up to 31 T, with a pulse duration of 2.2 s. Experimental set-ups for measurements of magnetization, magnetostriction and magnetoresistance are available. The temperature can be controlled between 1.4 and 335 K, being the inner bore of the He cryostat of 22.5 mm. Magnetization is measured using the mutual induction technique, the magnetostriction is determined with the strain-gage and the capacitive cantilever methods, and the magnetoresistance is measured by means of the ac lock-in technique in the 4-probes geometry. An overview of the facility will be presented and the presently available experimental techniques will be discussed.

1. Introduction
The long-pulse field facility of the Instituto de Ciencia de Materiales de Aragón (ICMA) is based in the standard technique of discharge of the energy stored in a capacitor bank in a copper coil. The magnet and power supply were designed and built by the Laboratoire National des Champs Magnétiques Pulsés (LNCMP) at Toulouse, France. The capacitor bank can storage a maximum energy of 1.2 MJ at 7.1 kV, being the total capacity 45 mF. The maximum field is reached in 150 ms. The use of a crowbar circuit gives a quasi-exponential decay of the magnetic field, the time constant is approximately 415 ms. The total pulse duration is 2.2 s.

The conventional coil with uniform current distribution is wound with a copper wire and reinforced with a cylinder of titanium. Top and bottom plates are made of glassfiber composite. The homogeneity of the magnetic field is $10^{-4}$ within a volume of 1 cm$^3$. The magnet is placed in a cryostat and cooled directly with liquid nitrogen. After a pulse of 30 T, the magnet has to cool down during 1.5 h. To reach 31 T, the liquid nitrogen is pumped down to triple point of nitrogen, i.e. a temperature of 63 K.

Different experiments can be performed between 1.4 and 325 K. Above 77 k the nitrogen cooling liquid at the magnet is used to cool the sample. Below 77 K a ‘He bath cryostat, built at the LNCMP, is used. The temperature is measured using a diode positioned at the insert, close to the sample. A temperature controller regulates the temperature within 0.1 K. The inner diameter of the cryostat is 22.5 mm, large enough for allowing a wide range of experiments.

2. Magnetization
The magnetization is measured using the mutual inductance technique. The pick-up coil consists of two cylindrical coils (half-length: 3.62, 4.62 mm; diameter: 2.82, 7.5 mm; number of windings: 4192, 972; wire diameter: 10.40 μm). An additional coil is wound to generate a magnetic field compensation...
signal; which, after adequate attenuation, is subtracted from the signal of the pick-up coil. The coil support is made with machinable glass-ceramic material, which gives an excellent stability and reproducibility of the required compensation signal. The size of the samples is limited to a diameter of 2 mm, and a maximum height of 2 mm. The high sensitivity of the integrator and the pick-up coils makes it normally unnecessary to use bigger samples.

The electronics used to integrate the signal of the pick-up coil, and to subtract the compensation has been carefully designed. The integrators consist of a low-noise high-speed operational amplifier and an auto-nulling circuit to eliminate to a large extent the drift of the integrator due to offsets voltages and bias current. For a more detailed description see reference 1. The final sensitivity of the magnetization measurement is better than $10^{-3}$ emu.

Samples holders are made of PEEK. This material has a minimum magnetization and is resistant against chemical agents. Poly- and mono-crystalline samples are glued with GE varnish and can be oriented within a few degrees. A special sample holder is available for measurements on powder using the free powder method.

Different magnetization measurements have performed using the above describe experimental setup. For instance in different single crystals of $Y_2Fe_17$ and $Y_2Fe_5B$ intermetallic compounds [2] and $Nd_{2x}Sr_{1+2x}MnO_3$ layered manganites [3,4]. In figure 1 we show, as an example, the magnetization measurements obtained in the some $Nd_{2-2x}Sr_{1+2x}MnO_3$, single crystals.

![Figure 1](image.png)

Figure 1 Magnetization isotherms, obtained from some single crystals of the layered Nd-Sr manganites, along the c-direction and the ab-plane at 4.2 K. The anomalies observed correspond to metamagnetic transitions.

3. Magnetostriction
Magnetostriction is the change of length that occurs in a magnetic compound when a magnetic field is applied. In the long-pulse field facility of the ICMA the strain gauge technique is used for determining magnetostriction. The strain gauge is glued to the sample and the relative variation in length ($\Delta l/l$) is obtained by measuring the variation in resistance of the strain gauge and using the relation $\Delta R/R = g \Delta l/l$, where $g$ is the gauge factor. The strain gauges used are model SK-05-031DE-350 (Micro Measurements Division, Measurements Group Inc. Raleigh, North Carolina, USA), which have relatively low magnetoresistance, a room temperature resistance of 350 $\Omega$ and a gauge factor of around 2. The size of the active area of the strain gauge is 1 x 4 mm and can be placed in relatively small samples (see Fig. 2).

The strain gauges are glued with M-bond 600 (Micro Measurements Division, Measurements Group Inc. Raleigh, North Carolina, USA) and baked for 24 h at 100 °C. Samples and wires are glued with GE varnish. Wires are carefully twisted to avoid the pick-up of the magnetic flux, and completely glued to avoid movement due to the Lorentz forces.
A quadratic relation is found between the magnetoresistance of the strain gauge and the magnetic field. The magnetoresistance increases progressively with decreasing temperatures. To compensate for this and other possible spurious effects a “passive” strain gauge is glued on quartz and an “active” strain gauge is glued on the sample. Both strain gauges are carefully selected from the same batch to minimize this error.

The two strain gauges in the sample holder are placed in two opposites arms of an Anderson impedance bridge configuration. The other two arms are formed two high precision resistors. The bridge is balanced for resistive and capacitive or reactive imbalance (due to the impedance of the wires) using the additional variable resistors and capacitance in the bridge. The excitation and detection is performed with a Tektronix 3C66 bridge using a fixed frequency of 25 kHz and a time response of 70 μs. Using this configuration it is possible to measure \( \Delta l/l \approx 5 \times 10^{-6} \).

Figure 2.- Photography of a strain gauge glued in a quartz sample, showing its dimensions in mm.

Figure 3. Volume magnetostriction isotherms of the ErSi$_4$ compounds at some selected temperatures. The metamagnetic-like anomalies correspond to field-induced magneto-structural transitions.

Different magnetostriction measurements have been performed in different samples. A summary of different results is presented in reference 5. As an example the low-temperature volume magnetostriction measured in a poly-crystalline sample of the ErSi$_4$ intermetallic compound is presented. The metamagnetic-like anomalies correspond to a structural phase transition induced by the magnetic field between two crystallographic phases with different volume [6].

4. Magnetoresistance

Resistance measurements are performed by means of the ac lock-in technique in the 4-probes geometry. Samples are diced in bar-shaped samples with a diamond saw, with typical dimensions of 6×2×2 mm$^3$. Electrical contacts between the sample and the wires are done with a two component silver paste epoxy (EPO-TEK H20E/1OZ). The lock-in is a 7265 DSP model from EG&G Instruments, operated by a GPIB board from National Instruments. As shown in Figure 4, the voltage drop at the shunt resistance ($V_s$) of the coil is measured in channel A of the lock-in. The reference signal of the lock-in ($V_r$) is connected to a home-made alternative voltage to alternative current converter, which offers high output impedance. The current can flow either through the sample or through the 1 kΩ shunt resistance of the AV/AC converter. By adjusting the gain of the AV/AC converter and the peak-to-peak amplitude of the reference signal we can select the exciting current for the measurement ($i_{AV}$), and measuring the voltage drop at the AV/AC shunt we can determine $i_{AC}$ with high accuracy (1μA<$i_{AC}$<10 mA). When $i_{AC}$ flows through the sample, the voltage drop between the
probe contacts in the sample, \( v_S \), is measured in channel B. Then, the sample resistance is calculated as \( v_S/i_{AC} \). The lock-in input resistivity in FET mode is 10 M\( \Omega \). In consequence, samples with resistance above 500 k\( \Omega \) can hardly be measured in our set-up. An AC amplifier with huge input resistance between the sample and channel B would be desirable in order to overcome this difficulty. Before the measurement, we perform a phase shift adjust between \( v_{AC} \) and \( v_S \), so that the imaginary part of \( v_S \) is nearly zero. The magnetic field pulse triggers the lock-in memory (we can choose an arbitrary pre-triggering time), in whose buffer 2000 \( v_S \) versus \( V_h \) points can be stored. Since the pulse lasts \( \sim 2 \) s, such buffer allows for a sampling rate of 1 kHz. The operating AC frequency can be varied in the range of 3 Hz (DC measurement) up to 33 MHz, usually a frequency of 3.3 kHz is used. Figure 5 displays an example of measurements of high-field magnetoresistance (HFMR) of the half-metallic double perovskite \( \text{BaSrFeReO}_6 \). The agreement with DC measurements performed at the 12 T continuous field set-up is excellent. The observed HFMR is ascribed to a second order tunnelling processes across insulating grain boundaries.

Figure 4 Sketch of the high-field magnetoresistance set-up.

Figure 5 High-field magnetoresistance of the \( \text{BaSrFeReO}_6 \) compound. Experimental results obtained using continuous field are displayed for comparison.

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